

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 16, 1928.

I.—GENERAL; PLANT; MACHINERY.

Chemical industries and Merseyside. A. HOLT (J.S.C.I., 1927, 46, 439—444 T).—The paper gives a brief *résumé* of the history and development of chemical industries in South West Lancashire and on the banks of the Mersey from early times till about the commencement of the present century.

Scattering of light by graded particles in suspension. T. M. LOWRY and M. C. MARSH (J. Oil and Colour Chem. Assoc., 1928, 11, 3—12).—The light-transmission of suspensions in medicinal paraffin of various sized particles of barytes (graded by elutriation) is recorded. Optical density is proportional to surface-concentration of the powder, but increases as the diameter is reduced. The probable existence of a maximum degree of covering power on continued subdivision is indicated. S. S. WOOLF.

Measurement of particle size in ground powders. J. V. RAMSDEN (J. Oil and Colour Chem. Assoc., 1928, 11, 16—17).—A new projecting microscope is described and illustrated. A very dilute aqueous suspension of the material under test is poured into a transparent dish, which is placed on the stage of an inverted microscope. The image of the particles is projected on to a calibrated screen or photographic plate. All motions of the instrument are on the Kelvin slide system. S. S. WOOLF.

Production of fibrous precipitates. VON WEIMARN.—See V. **Measurement of plasticity.** TALWALKAR and PARMELEE.—See VIII. **Recording Soxhlet extractor.** RANKOW.—See XII.

PATENTS.

Furnace. T. A. MARSH, ASSR. to GREEN ENGINEERING Co. (U.S.P. 1,656,643, 17.1.28. Appl., 29.3.23).—The grate travels between the side walls of the furnace, and pipes of uniform diameter conveying cooling fluid extend the full length of one side of the grate. The forward ends of these pipes are continued past the ignition zone of the fuel bed on the grate and are bent outwards from the grate in advance of the zone to prevent their cooling the zone. The remaining portions of the pipes extend to the rear of the grate mainly in direct contact with the fuel bed. H. HOLMES.

Kiln. H. G. LYKKEN (U.S.P. 1,656,642, 17.1.28. Appl., 15.4.25).—The kiln comprises a floor supporting the goods to be burned, side walls, and a roof. Combustion gases are supplied to the goods under pressure and escape through adjustable openings provided in the side walls above the floor. H. HOLMES.

Dryer. W. C. CARPENTER (U.S.P. 1,655,724, 10.1.28. Appl., 3.12.25).—Material to be dried is delivered into

one end of a rotary drum and discharged from the other end. Ducts for hot air extend along the interior wall of the drum, and their discharge ends are bent back to direct the air through the drum between the ducts.

H. HOLMES.

Centrifugal machine. W. C. LAUGHLIN, ASSR. to LAUGHLIN FILTER CORP. (U.S.P. 1,655,426, 10.1.28. Appl., 23.2.26).—A distributor with a helical flange is mounted within a screen carried by a rotary drum. Blades mounted loosely on the periphery of the flange are maintained in continuous contact with the screen surface solely by centrifugal action. H. HOLMES.

Adjustable plough for centrifugal grinding mills. W. A. GIBSON, ASSR. to BRADLEY PULVERISER Co. (U.S.P. 1,655,499, 10.1.28. Appl., 29.12.24).—A centrifugally-operated grinding member carrying a plough co-operates with an annular die, and means are provided for adjusting the inclination of the plough to the plane of the pan about an axis radial to its direction of movement. H. HOLMES.

Crusher. W. E. KEINE, ASSR. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,656,004, 10.1.28. Appl., 17.5.26).—A crushing head co-operating with a concave surface is gyrated by an eccentric rotatable on a fixed vertical support and driven by a motor mounted co-axially above the support. H. HOLMES.

Mixing machine. W. F. DEHUFF (U.S.P. 1,656,665, 17.1.28. Appl., 23.3.27).—A beater shaft is supported for movement around a driving shaft parallel to it. The former carries an internal gear, engaged by a gear on the driving shaft, and also a second gear engaging a stationary gear. H. HOLMES.

Facilitating the removal of a substance solidified or condensed on cooling drums. APPAREILS ET EVAPORATEURS KESTNER (B.P. 275,952, 19.7.27. Fr., 10.8.26).—A layer of finely-divided dry powder is applied to the drum and forms a non-adherent parting layer so that the substance is easily scraped or broken away from the drum. One means of applying the powder to the bare part of the drum between the knife and the feed bath comprises a long narrow jet as long as and close to the drum which is supplied by one fan with the air-borne dust. Another fan produces a suction through a pair of hoods closely surrounding the supply jet, through which any excess powder is withdrawn, collected in a cyclone, and re-used. B. M. VENABLES.

Mixing and emulsifying apparatus. A. W. EMPSON (B.P. 282,466, 26.6.26).—The apparatus comprises a centrifugal rotor with small labyrinthine passages through which the mixture passes. The outlets from the rotor are still smaller than the passages, so that

the passages are maintained full of mixture and the size of the outlets can be varied by changing grub screws or other means. A collecting casing is provided for excess feed as well as for emulsion, and in the latter are vanes for arresting the motion of the emulsion and aerating it. The amount of aeration is controlled by varying the amount of air allowed to be drawn into the emulsion casing from the excess feed casing.

B. M. VENABLES.

Concentration of liquids. A. HUIILLARD (F.P. 621,358, 7.1.26).—A liquid is heated to a temperature approaching that at which gas bubbles are formed, and the formation of these is limited to the surface of the liquid by maintaining the pressure at that exerted during the heating.

L. A. COLES.

Apparatus for expressing liquid constituents from materials of various kinds. F. KRUPP GRUSONWERK A.-G. (B.P. 273,689, 21.6.27. Ger., 29.6.26. Addn. to B.P. 248,372; B., 1927, 176).—The pressing finger and removing scraper of the device described in the original patent are interconnected so that when the latter moves downwards under increasing pressure of the material, the former moves in the same direction but to a (usually) much smaller extent. The actual travel, the travel ratio, and the spring load are all adjustable.

B. M. VENABLES.

Rotary water-screening plant. F. W. BRACKETT & Co., LTD., and F. W. BRACKETT (B.P. 282,569, 1.2.27).—A rotary cylindrical screen, *e.g.*, for condensing water, is rotated by the filtered water leaving the screen, acting on vanes round the periphery.

B. M. VENABLES.

Separators and filters for liquids. J. H. DINE and C. H. BILLINGTON (B.P. 282,542, 15.11.26).—A number of frames for filter bags are held in place simply by wedging action between an inclined surface on one wall of the tank and the apertured outlet channel along the other side of the tank.

B. M. VENABLES.

Apparatus for controlling chemical reactions. H. S. HATFIELD, and UNITED WATER SOFTENERS, LTD. (B.P. 282,168, 18.9.26).—An automatic machine is provided to analyse a liquid and then to control reactions in the bulk of the liquid in accordance with the analysis. A measured portion of the liquid is taken, and to it is added a reagent in portions at a time until a critical point is reached or over-run. The critical point may be indicated by change in electrical conductivity (or other physical properties) or, as in water testing, by the formation of froth. The number of portions of test reagent will control the addition of the bulk reagent.

B. M. VENABLES.

Means for effecting the chemical analysis of liquids and controlling chemical operations. H. S. HATFIELD (B.P. 282,170, 20.9.26).—A sample of liquid is subjected to a chemical reaction (*e.g.*, boiling to remove temporary hardness of water) and compared with an immediately following sample of the raw liquid by putting the two samples in the arms of a Wheatstone bridge. If the reaction is effected by electrolysis, the same electric current may be used in the bridge and to control, by electro-magnets, the measuring device by which the reaction is caused to regulate the addition of bulk reagent.

B. M. VENABLES.

Liquid and gas contact apparatus. W. J. MCGURTY, ASSR. to BARTLETT HAYWARD Co. (U.S.P. 1,656,591, 17.1.28. Appl., 19.6.25).—A laterally-curved chamber in the upper part of a casing communicates at one end with the interior of the casing through a passage extending centrally thereof, and is connected at its other end to a discharge port. Spaced vertical plates are mounted in the chamber with spaced vertical wires between them.

H. HOLMES.

Treatment of gases or vapours with liquids or liquefiable mediums. E. M. SALERNI (B.P. 282,493, 21.9.26).—The liquid is picked up by a rotating spiral scroll, similar to a Frenier pump, except that there may be several concentric spirals. The gas is forced through the spirals from the axis outwards, *i.e.*, counter-current to the liquid. The pick-up openings at the ends of the spiral may be adjusted to vary the rate of treatment.

B. M. VENABLES.

[Air] classifiers. A. H. STEBBINS (U.S.P. 1,655,575-7, 10.1.28. Appl., [A] 24.11.26, [B] 15.12.26, and [C] 24.12.26).—Modifications of an air separator are claimed in which the material slides down a number of conical or V-shaped baffles, the spaces between which form settling chambers, the whole being enclosed by an outer shell. Air jets blow upwardly in (A), outwardly in (B) from a central supply pipe, or across from wall to wall in (C), and propel the material into the settling chambers, whence the fine material only is carried out by the air currents, aided if necessary by an outlet fan also.

B. M. VENABLES.

Chemical heat storage. F. W. STERLING and H. L. CROOKER, ASSRS. to B. H. DYER Co. (U.S.P. 1,656,366, 17.1.28. Appl., 22.8.23. Renewed 8.4.27).—An unstable solution which evolves heat on crystallisation is used, which consists of sodium acetate, calcium chloride, and water, the water being 46% of the total.

H. ROYAL-DAWSON.

Refining and purifying of liquids. P. T. SHARPLES and L. D. JONES, ASSRS. to SHARPLES SPECIALTY Co. (U.S.P. 1,657,941, 31.1.28. Appl., 2.4.24).—See B.P. 231,877; B., 1926, 1006.

[Continuous] charging of furnaces with solid material. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 283,238, 4.8.26).

[Grooved] building block or brick for use in furnaces and other structures. H. S. WOOD, T. WILLIAMS, and SOUTH DURHAM STEEL & IRON Co., LTD. (B.P. 283,307, 30.10.26).

Device for mixing liquids in variable proportions. H. KAUFERTZ (B.P. 282,533, 24.9.26).

Continuous kilns (B.P. 283,009).—See VIII. **Concentration of materials** (U.S.P. 1,656,042).—See X. **Determination of gas in water** (B.P. 282,487).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Nitrogenous matter in coal. C. W. SHACKLOCK and J. DRAKELEY (J.S.C.I., 1927, 46, 478-481 T).—The quantity of nitrogen extracted by means of dilute sulphuric acid from peat, lignite, sub-bituminous, bituminous, and anthracite coals decreases in the order

given, and is not influenced, under the conditions of the experiment, by particle size provided the coal is first passed through a 30-mesh sieve. Nitrogen in the form of amide compounds, monoamino- and diamino-acid has been distinguished and determined. The monoamino-acids appear to be the most persistent compounds, and in the case of coals account for over 70% of the extracted nitrogen. The diamino-acids exist in coals in amounts which are too small to determine by the methods employed in this investigation. The amide compounds are present in all the fuels, and account for 15–20% of the nitrogen extracted from the coals.

Present-day knowledge of the humus and bituminous constituents of coals. H. TROPSCH (Brennstoff-Chem., 1927, 8, 369–374).—A review of recent work shows that much is known regarding the humus substances in "humus coal" (clarain), but much less of the composition of the bitumens. The humus constituents of peat and the younger lignites, of the older lignites, and of coal are composed of free humic acids, humic acid anhydrides, and alkali-insoluble humins, respectively, all closely related. Oxidation, with air under pressure and in presence of sodium carbonate, of lignin, lignites, and coals gives rise to similar products (*e.g.*, mellitic and benzenepentacarboxylic acids), indicating that the humus substances contain the benzene nucleus, whilst the oxidation of cellulose yields products such as fumaric and succinic acids. A similar conclusion regarding the humus substances may be drawn from the nature of their hydrogenation products, and the phenolic character of humic acids is suggested by their yielding nitrophenols with nitric acid. Conversely, humic acids can be synthesised from the higher phenols and their derivatives. For similar reasons the lignin molecule appears to be to a large extent aromatic and phenolic. Lignin can, moreover, be converted into humic acids, with accompanying loss of methoxyl, by both chemical and biological means. The bituminous portion of lignites contains resins and waxes, the latter being partly composed of esters (*e.g.*, ceryl and myricyl) of normal fatty acids (*e.g.*, the C₂₅, C₂₇, C₂₉, and C₃₁ acids). The bitumen of coal can be separated into oily and solid compounds, the former comprising a mixture of hydrocarbons, whilst the latter contain oxygenated substances.

W. T. K. BRAUNHOLTZ.

Method of testing the strength of blast-furnace coke. W. WOLF (Stahl u. Eisen, 1928, 48, 33–38).—An apparatus for testing the strength and suitability of a coke for blast-furnace use comprises a vertical, rectangular vessel, two opposite sides at the lower end of which are provided with hinged doors which may be kept open at any desired angle by means of weights which are so adjusted that they allow the doors to open further when the pressure of the coke exceeds 2.5 kg./cm.² The vessel is charged with $\frac{1}{3}$ m.³ of the coke to be tested and the doors are opened at an angle of 45°; pressure of 1–2.5 kg./cm.² (according as the coke is hard or soft) is applied by means of a plunger pressing on the top of the coke column, and the material passing through the doors is screened on a sieve with 30-mm. holes. The percentage of undersize is called the "pressure abrasion loss," and the values so obtained are a measure of the strength of the coke in the blast furnace. This

procedure affords a more satisfactory method of classifying coals than does the method involving agitation of the coke in a rotating drum.

A. R. POWELL.

Chemistry of Japanese plants. IX. Studies on the "Tundra" (peat) of Southern Karafuto. S. KOMATSU and O. HIKI (Sexagint [Osaka Celebration], Kyoto, 1927, 229–240).—An examination of peat from different depths shows that as the depth and age increase, the resin, humic acid, and lignin contents rise whilst the sugar and cellulose contents fall. It is concluded that cellulose and other carbohydrates, as well as lignin, play an important part in the formation of coal. Active charcoal, prepared from the peat by washing, drying, carbonising at 500–600°, treating with 25% caustic soda solution, washing, and heating at 700–800° for 1 hr., compared favourably with "norit" in adsorptive power.

C. D. LANGFORD.

Production and composition of decolorising carbons. P. HÖNIG (Chem.-Ztg., 1928, 52, 7–8, 34–35).—The methods of production of the four main classes of absorbent carbons are: (A) carbonisation of raw materials derived from plants, *e.g.*, Bragg's charcoal, Carbox, Suchar, etc.; (B) the action of activating or inflammable gases at a high temperature on carbonised materials, *e.g.*, Norit, Eponit, Supranorit; (C) carbonisation of raw materials mixed with carbonate of calcium or magnesium, *e.g.*, Darco; (D) dehydration of raw materials such as carbohydrates with zinc chloride, *e.g.*, Carboraffin. The products usually contain varying amounts of moisture, ash, and hydrogen, with traces of sulphur and nitrogen. Those of class (B) are richest in carbon, Supranorit containing 98.2%, whilst their absorptive power varies inversely as the hydrogen content. In Darco the lime and magnesia are firmly combined and cannot be removed by washing with acid. Carboraffin may be regarded as a dehydrated hydrocarbon in colloidal dispersion, which is practically insoluble and owes its absorptive power to its highly unsaturated character.

F. R. ENNOS.

Application of distillation under partial pressure of substances of high mol. wt. by means of highly superheated steam in order to effect the "demolisation" of highly "comolised" substances. H. H. WISLICENUS (Z. angew. Chem., 1927, 40, 1500–1506).—[With H. LIANG.] Distillation with highly superheated steam may be carried out in the Schrader aluminium retort (B., 1921, 500 A), or in a specially constructed apparatus, in which superheated steam passes round and then through a central cylindrical retort, which may be placed horizontally or vertically, containing the substance. Highly superheated steam may cause a purely physical dispersion ("demolisation") of highly aggregated ("comolised") molecular systems, which cannot be distilled even under reduced pressure. In other cases it causes definite thermochemical decomposition or simple hydrolysis. When rosin is subjected to distillation with steam at 240–250°, the distillate (or sublimate), obtained in 86–96% yield, comprises pure, crystalline abietic acid, whilst a small quantity (about 0.5%) of carbon dioxide is simultaneously evolved. After recrystallisation from alcohol the acid melts at 154–162° (with selected crystals, 159–164°),

whilst on keeping, even in an evacuated desiccator, the crystals run together to a pale yellow resin. The mol. wt. of abietic acid obtained from commercial rosin, and from freshly collected fir resin, whilst giving the same ultimate analysis, was 308 and 517—518, respectively. The great readiness of abietic acid to "comolise" is evidenced by its separation in large white aggregates from the resin obtained from the living parts of fir trees. The distillation of dammar resin with superheated steam requires a higher temperature (340—350°) than rosin, as well as more steam and a longer distillation time. The distillate does not crystallise, but melts at about 50—80°.

[With E. STELZER.] With superheated steam amber yields non-crystallising oils, of which the viscosity and depth of colour increase the higher the temperature of the superheated steam used (up to 350°).

W. T. K. BRAUNHOLTZ.

Destructive distillation of wood and cellulose under pressure. K. FROLICH, H. B. SPALDING, and T. S. BACON (Ind. Eng. Chem., 1928, 20, 36—40).—The yield of methyl alcohol obtained by the destructive distillation of birch wood is increased by 65—80% over that obtained at atmospheric pressure when the operation is conducted under autogenous pressure at 280—370°. A further increase in temperature causes a considerable decrease in the yield owing to decomposition of the products initially formed. When distillation is carried out under a pressure of 200 atm. in a current of hydrogen, still higher yields (up to 3·11% on the weight of dry wood) are obtained. These increased yields of alcohol are not derived from the cellulose molecule, since only traces of methyl alcohol are formed when cellulose is distilled under the same conditions. Catalysts, in so far as these have been examined, usually reduce the yield of methyl alcohol at atmospheric pressure, and have but a slight effect at higher pressures, though in one case, with a copper-zinc-aluminium catalyst at a hydrogen pressure of 200 atm., 4·77% of methyl alcohol was obtained, possibly due to the fact that the catalyst was added as acetate. With nickel acetate, however, distillation under 200 atm. hydrogen pressure to a final temperature of 500° results in the conversion of more than 90% of the wood into volatile products, including phenols and saturated and unsaturated hydrocarbons. Similar products result when cellulose is substituted for wood; in this case over 97% of the cellulose is converted into volatile products, and even when hydrogen is replaced by nitrogen a conversion of 84% is realised.

D. J. NORMAN.

Hydrocarbon synthesis from carbon monoxide and hydrogen. O. C. ELVINS (J.S.C.I., 1927, 46, 473—478 T).—The interaction of carbon monoxide and hydrogen was investigated at atmospheric pressure and in presence of catalysts at temperatures of the order of 280°. A catalyst prepared from a mixture of 60 pts. of cobalt, 30 pts. of copper oxide, and 45 pts. of manganese oxide was used in conjunction with varying gas mixtures. Contrary to the original statements of Fischer and Tropsch that only aliphatic hydrocarbons are formed by this reaction, definite amounts of water-soluble oxygenated compounds and olefines were found among

the reaction products. A detailed account of the experimental work is given and the preparation of the catalyst from precipitated hydroxides, by ignition of nitrates, and from fused oxides, is described. The size of the granule was also investigated from the point of view of the inherent fouling effect of the waxy products of reaction.

Composition of cracked distillates. J. S. S. BRAME and T. G. HUNTER (J. Inst. Petrol. Tech., 1927, 13, 794—816).—Experiments have been made to distinguish between cracked spirit and straight-run petrol, quantitative information as to the constituents of which is very scanty owing to the difficulty of separation even into unsaturated, aromatic, naphthene, and paraffin hydrocarbons. Fractional extraction was first investigated, with methyl alcohol, sulphur dioxide in acetone, nitrobenzene, aniline, and dimethylaniline, the changes which occurred being traced by determination of the aniline dissolution point, but in no case was a satisfactory separation achieved. The dissolution points of a number of pure hydrocarbons with aniline and with nitrobenzene are also recorded. Attempts to separate unsaturated hydrocarbons, at first from mixtures of known composition, by means of mercury derivatives, proved unsatisfactory because the course of the reaction was uncertain and not quantitative; the solid products were also much contaminated with unchanged oil, and their reconversion into free hydrocarbons was difficult. Successful separation was finally accomplished by bromination followed by fractionation, and the hydrocarbons were recovered by the action of copper-zinc or aluminium-mercury couples. In dealing with the analysis of paraffins and naphthenes, mixtures of hexane and cyclohexane were used. It was found that the addition of a third liquid, followed by careful fractionation, made possible separation to a far greater degree than could be achieved by distillation of the binary mixture; the best results were obtained by the use of aniline. The foregoing methods were applied to a cracked spirit from Russian kerosene, boiling below 120°; this was divided into 11 fractions which were examined in detail, and many individual hydrocarbons were isolated and identified. It is concluded that both paraffins and isoparaffins are present accompanied by nearly equal quantities of naphthenes, and that olefines constitute the bulk of the lower-boiling unsaturated hydrocarbons, but diolefines are also present and cyclic olefines occur in the higher-boiling fractions; only small amounts of aromatic substances are present. R. H. GRIFFITH.

Determination of nitrogen in petroleum and bitumens. E. J. POTH, W. D. ARMSTRONG, C. C. COBURN, and J. R. BAILEY (Ind. Eng. Chem., 1928, 20, 83—85).—Determination of nitrogen in petroleum by the Kjeldahl method has been generally regarded as inaccurate, but the following procedure has been found reliable. The oil is digested with a large excess of sulphuric acid in the presence of potassium sulphate, mercuric oxide, and copper sulphate, and the resulting mixture is treated in the usual way, with precautions necessitated by the large amount of acid employed. Results obtained in this way with numerous oils are compared with those given by the combustion method,

and are considered to be more reliable; they also suggest that figures previously published for the nitrogen content of Californian petroleum are too high. R. H. GRIFFITH.

Effect of sulphur in gasoline on wrist-pin corrosion in automobiles. S. H. DIGGS (Ind. Eng. Chem., 1928, 20, 16—17).—Corrosion of automobile engines due to sulphur in the fuel is particularly noticeable in wrist-pins, but only occurs in cold weather. Tests were made, on an engine fitted with new pistons, rings, and wrist-pins, with three samples of gasoline containing 0.040, 0.151, and 0.458% S, respectively; with the first there was no perceptible corrosion, but with the second it was definitely present, and with the third very serious. Free sulphuric acid was detected in the crank-case, but no other acid was present. All forms of sulphur in the fuel are oxidised to sulphur dioxide, some of which will enter the crank-case and in the presence of water and air will form sulphuric acid, but in warm weather or in engines running continuously insufficient water will condense to make this possible. R. H. GRIFFITH.

Sulphur in gasoline from the automobile point of view. H. C. MOUGEY (Ind. Eng. Chem., 1928, 20, 18—21).—If the permissible sulphur content of gasoline could be raised from 0.1 to 0.3% great reductions of refining costs would result. Attempts have been made to render this possible by crank-case ventilation and control of water-jacket temperatures so that condensation of water is avoided; a large number of tests carried out on a variety of cars show that corrosion can be prevented in this way, but no recommendation is made to allow higher sulphur contents until automobile equipment of this type becomes general. R. H. GRIFFITH.

Preparation of fuel oil by dry distillation of the calcium soap of soya bean oil. IV. Comparison with the magnesium soap. M. SATO. **V. Hydrogenation of the distilled oil.** M. SATO and H. MATSUMOTO (J. Soc. Chem. Ind. Japan, 1927, 30, 242—245, 245—252).—IV. The use of the dry distillation products of the magnesium and calcium soaps of soya bean oil in the preparation of fuel oil has been investigated. The best result was obtained by heating either of the soaps at temperatures below 450° for several hours, followed by distillation above 500°. The time required for completing the distillation of the magnesium soap was less than that for the calcium soap at the same distillation temperature; similarly, the sp. gr. of the distillate of the magnesium soap was somewhat greater than that of the calcium soap. **V.** The light oil (100—175°) and middle oil (175—300°) were hydrogenated in liquid or vapour phase, using nickel reduced at about 370° as catalyst, and the products examined. The hydrogenation of the light oil began at about 60° and was rapid at 80°. In vapour phase it was readily reduced to saturated hydrocarbons at 190°, forming a colourless oil having no disagreeable odour. The middle oil could be hydrogenated in liquid phase at 140° with 2% of nickel, its iodine value being decreased to about 60; similar results were obtained in vapour phase at 320°. Heptane, octane, and nonane were isolated from the hydrogenated light oil. The existence of aromatic hydrocarbons, such as benzene, toluene, and xylene, could not be confirmed. From the hydrogenated middle oil were isolated those constituents which had the

b.p. and ultimate composition analogous to those of $C_{11}H_{24}$, $C_{13}H_{28}$, and $C_{14}H_{30}$, but the sp. gr. of each fraction was greater than that of the corresponding hydrocarbon; it is inferred that some hydrocarbons of the naphthene series accompany the paraffin hydrocarbons, which are the main constituents of the oil.

Y. TOMODA.

Preparation of fuel oil by the dry distillation of the calcium soap of soya bean oil. VI, VII. Reaction mechanism of thermal decomposition of the calcium and magnesium salts of some higher fatty acids. M. SATO, also M. SATO and C. ITO (J. Soc. Chem. Ind. Japan, 1927, 30, 252—260, 261—267).—VI. Calcium and magnesium stearates were heated at 450—480° in a partial vacuum (5—7 mm.). By the isolation of stearone from the distillates obtained, the formation of the ketone by the thermal decomposition of the stearates is confirmed. By similar experiments the formation of oleone from the corresponding oleates was confirmed. The decomposition temperatures of these salts were measured, the results being expressed as curves relating acetone-soluble matter (%) and temperatures. **VII.** Rates of thermal decomposition of calcium and magnesium stearates and oleates at 400° have been measured, and the results are expressed by graphs. Analysis of the gases formed by the decomposition of stearone and oleone at different temperatures showed that the rate of formation of carbon monoxide from the ketones was very small at 300—350°, and increased gradually with temperature. Thus, the carbon monoxide formed during 1 hr. from stearone and oleone heated at 500—550° and 550—600°, respectively, was 48.10% and 66.96% of the theoretical quantity. Saturated and unsaturated hydrocarbons and small quantities of hydrogen and carbon dioxide were also formed by the thermal decomposition of the ketones.

Y. NAGAI.

Constituents of petroleum from Maki, Echigo Province. S. IIMORI and U. KIKUCHI (J. Chem. Soc. Japan, 1927, 48, 520—526).—Petroleum from Maki was unsatisfactorily purified by means of concentrated sulphuric acid, from which a fraction (about 15% of the original oil) distilling at 125—155° was obtained. By bromination and nitration of the fraction, tetrabromoxylene, m.p. 250°, and trinitroxylene, m.p. 174°, were obtained. The bromination and chlorination of the same fraction from more satisfactorily purified oil gave tribromo- ψ -cumene, m.p. 234—237°, and trichloro- ψ -cumene, m.p. 215—216°. The presence of xylene (or a mixture of *m*- and *p*-isomerides) and hexahydro- ψ -cumene in the petroleum was therefore confirmed. The m.p. of tribromo- ψ -cumene was higher than that (222—224°) prepared from Merck's product. K. KASHIMA.

Micro-structure of New Zealand lignites. W. P. EVANS (Fuel, 1928, 7, 75—83).—See B., 1928, 3.

Potassium ferrocyanide. CHEMNITIUS.—See VII. **Catalytic decomposition of soya bean oil.** HAGA.—See XII.

PATENTS.

Treatment of carbonaceous materials. R. LESSING (B.P. 282,874, 27.9.26).—Coal or similar material is cleaned by gravity separation in a liquid of suitable

density, *e.g.*, an alkaline solution of calcium chloride. The coal is introduced below the surface of the separating liquid, which is agitated at the point of introduction. The "float" and "sink" are continuously removed separately, and are washed by upward or downward displacement of the bath liquor with a continuous unbroken body of washing liquid. It is advantageous to remove the greater part of the dust in the coal before submitting it to the process. In a modification of the process the washing may be incomplete and a predetermined quantity of the solute may be left in admixture with the coal to act as a catalyst during subsequent carbonisation, hydrogenation, etc. A. B. MANNING.

Manufacture of fuel briquettes. E. W. BOWEN (B.P. 282,890, 30.9.26).—The fine dust is first removed from the fuel to be briquetted, the remaining granular portion is mixed with the requisite quantity of binder, and then a definite proportion of the dust is re-introduced into the mixture, which is finally moulded and baked. Suitable proportions to use when briquetting anthracite duff are 86% of granular material, 9% of binder (waste sulphite liquor), and 5% of dust. The dust may be separated from the granular material in the drying chamber by an upwardly moving centrifugal draught. Addition of the binder and subsequently of the dust is carried out during the passage of the material through screw conveyors, in which the mass is effectively mixed. A. B. MANNING.

Manufacture of highly active carbon blocks or granules. CHEM. WERKE CARBON GES.M.B.H. (B.P. 266,674, 29.11.26. Ger., 24.2.26).—Finely-divided carbon (1–10 μ in size) is mixed with an organic binding material, *e.g.*, 60–70 pts. of wood charcoal to 30–40 pts. of tar, containing a soluble inorganic substance such as caustic soda lye as a catalyst. The mixture is moulded under pressure (300 atm.), dried, carbonised, and activated in the usual way with gases or vapours. To accelerate the drying and oxidation process, oxides, resinates, or linoleates of manganese, lead, cerium, or cobalt may be added. The inorganic substances are subsequently extracted from the finished product. A. C. MONKHOUSE.

Manufacture of a highly active alkaline coke. N. V. NEDERLANDSCHE MIJNBOW EN HANDEL MAATSCHAPPIJ (B.P. 265,993, 11.2.27. Ger., 12.2.26).—Coal, peat, asphalt, or wood, etc., mixed with alkali or barium carbonates or hydroxides, together with a binder if necessary, and a catalyst, *e.g.*, an iron or nickel compound, if desired, is carbonised to produce a granular active coke suitable for the fixation of nitrogen in the form of cyanide. The finely-divided material is fed continuously on to the external surface of a drum heated internally and rotating inside a gas-tight chamber. Projecting ledges on the drum prevent the material from falling, until it is discharged at the bottom of the chamber, when, if required, it may pass to a second carbonising chamber at a higher temperature. The vapours from the chambers form on condensation a valuable acid-free tar. A. B. MANNING.

Fuel. W. H. ROGERS (B.P. 280,349, 25.10.26).—Road sweepings or waste combustible materials are mixed with an inflammable liquid, *e.g.*, $\frac{1}{2}$ pint of petroleum to

1 cwt. of sweepings, and the mixture is then moistened with water. Clay or similar binder may be added and the material compressed into briquettes. [Stat. ref. to B.P. 230,252, 158,671, 105,340, and 27,334 of 1910.] A. C. MONKHOUSE.

Retort furnaces for the distillation of coal and similar carbonaceous substances. LOW TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P. 281,348, 7.6.26).—The setting of vertical retorts has between each series in line a common combustion chamber in which transverse walls of chequer brickwork are built. These form chambers into which the heating gases from the combustion chamber pass into contact with the retort walls. A. C. MONKHOUSE.

Distillation or coking of coal. A. GAERTNER (B.P. 281,110, 29.12.26).—Finely-divided coal is distilled by being injected with combustion gases into a furnace consisting of a horizontal drum or vertical chamber containing a bell-shaped deflector with a suitable system of baffles or electric dust separator. The distillation gases are burnt in a chamber surrounding the distillation chamber, and the cooler for the gases is used to preheat the powdered fuel. Catalysts may be used in the distilling chamber or injected in the form of finely-divided metal. A. C. MONKHOUSE.

Distillation of fuel. E. ROSER (B.P. 283,420, 29.6.27).—Fuel is carbonised in chambers forming the periphery of a rotary drum, which is heated both internally and externally. The drum is rotated in stages, and during the stationary periods a plunger, operating at a suitable position in the setting, compresses the material in the chamber opposite to it. A compact coke is thereby produced. A. B. MANNING.

Distillation of solid fuel in small pieces. E. HUREZ and P. P. VIRELY (F.P. 620,307, 18.8.26).—The fuel, broken up into small pieces of 10–120 mm. diam., slides over an inclined hearth. L. A. COLES.

Treatment of coal gas etc. and production of ammonium sulphate. C. COOPER, F. B. HOLMES, and W. C. HOLMES & Co., LTD. (B.P. 281,474, 30.11.26).—The cooled gases obtained from the distillation or gasification of fuels are washed in the presence of carbon dioxide with a mixture of ammonia liquor and calcium sulphate. The liquor used is either the condensed liquor or is obtained from it by distillation and, if necessary, is freed from pyridine, tar acids, and cyanogen compounds. The gases after treatment pass through sulphuric acid, which is subsequently used in the treatment of the mixture of ammonia liquor and calcium sulphate after conversion into calcium carbonate and ammonium sulphate. A. C. MONKHOUSE.

Manufacture of gas. H. NIELSEN and B. LAING (B.P. 281,105, 25.8.26. Addn. to B.P. 262,834; B., 1927, 273).—Coke produced by the carbonisation of fuels by internal heating up to 1200° is used for the manufacture of gas as described in the prior patent. A. C. MONKHOUSE.

Washing, purifying, and cooling of producer gases. E. WARNANT (B.P. 282,851, 2.9.26).—A gas scrubber suitable for use on automobiles driven by producer gas comprises a vertical column of small cross-sectional area and considerable length, filled with coke or

metal turnings, down which the gas passes together with water in a fine state of subdivision, and a subsequent separator also filled with coke or turnings and containing water through which the gas is caused to bubble. The separator surrounds the base of the scrubber, the overflow being level with the top of the former. The water is circulated continuously through the scrubber and through a heat interchanger or radiator.

A. B. MANNING.

Means for rendering harmless the exhaust gases of internal-combustion engines. D. A. MACKINNON (B.P. 281,113, 4.1.27).—The exhaust gases from each engine cylinder pass through a separate chamber lined with copper and containing copper screens; a whirling motion is given to the gases by means of deflectors, and air is admitted by pump or fan. A conversion of the carbon monoxide in the gases into carbon dioxide takes place.

A. C. MONKHOUSE.

Heating and cracking of oils. T. E. ROBERTSON. From POWER SPECIALTY Co. (B.P. 283,032, 20.4.27).—Oil is heated to a cracking temperature by passage through tubes heated by the hot gases from a furnace, the rate of passage of oil being rapid enough to avoid a large portion of the oil-cracking effect which would result if the oil were heated to the same temperature by less rapid passage through less highly-heated tubes. The gases leave the oil-heating tube zone at a temperature a little below the maximum temperature at which they may safely be brought into contact with a tubular steam superheater (*i.e.*, gases at about 816° are utilised to superheat steam to about 482°), and the degree of superheating is controlled by the regulated injection of water into the steam. The final residual heat of the flue gases is used to preheat the oil to be treated. C. O. HARVEY.

Refining of petroleum. SHARPLES SPECIALTY Co., Assees. of L. D. JONES (B.P. 259,553, 24.9.26. U.S., 7.10.25).—Petroleum residues remaining after the removal by distillation of gas, naphtha, and burning oils are freed from "non-pressable wax" prior to refining for the removal of impurities (other than hard asphalt), as these impurities assist in the separation of the wax; *e.g.*, the residuum is diluted with naphtha, chilled, and centrifuged. Residua containing excessive quantities of impurities (*e.g.*, hard asphalt) other than those easily removed by clay treatment, cause trouble owing to inefficient removal of wax etc., and such oils are given a preliminary mild acid treatment. Residua containing more asphalt than can be economically removed by an acid treatment can be brought to a condition satisfactory for dewaxing by distilling off all the lubricating content without cracking; the beneficial impurities pass over with the last fractions of the oil, whereas the asphalt remains in the still. The oil should be steam-distilled up to a temperature of about 371°. Oils devoid of the necessary beneficial impurities may be blended with suitable oils prior to dewaxing. The dewaxed oil may be purified by freeing from naphtha, clay treatment, fractionation, etc.

C. O. HARVEY.

Purification of hydrocarbons and the like by liquid sulphur dioxide. ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 268,726, 6.11.26. Ger., 30.3.26).—A preliminary treatment with 10–25% of the total sulphurous

acid is made in order to remove water from the material. This extract is isolated, and the sulphurous acid recovered. The fraction from which the water is removed is treated with the remainder of the sulphurous acid as in the Edeleanu process.

A. C. MONKHOUSE.

Distillation of hydrocarbons. W. M. DUNCAN (U.S.P. 1,654,797, 3.1.28. Appl., 14.7.24).—The hydrocarbons are distilled and cracked under pressures greater than 50 lb./sq. in., and the resulting vapours are passed through a chamber containing a solid hydrocarbon material, whereby the volatile constituents of the latter are removed. The container for the solid material is duplicated so that renewal of the spent material in one container may be carried out while that in the other container is undergoing treatment.

C. O. HARVEY.

Apparatus for treating hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,655,600, 10.1.28. Appl., 28.6.20. Renewed 12.3.26).—A horizontal cylindrical still is provided with a centrally-disposed heating member from which heating gases pass, and around which an annular still section is rotatably mounted. The hydrocarbon oil to be distilled is contained between the rotatable portion and the outer wall of the still.

C. O. HARVEY.

Treatment of hydrocarbons. A. SCHWARZ, Assr. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,655,741, 10.1.28. Appl., 8.5.23).—Predetermined proportions of saturated hydrocarbons of low b.p. and unsaturated hydrocarbons of high b.p. are vaporised and the vapours mixed intimately. After condensation, the uncondensed hydrocarbons are compressed and returned to the vapours that are being mixed. C. O. HARVEY.

Treatment [cracking] of hydrocarbon oils. E. R. WOLCOTT, Assr. to TEXAS Co. (U.S.P. 1,654,771, 3.1.28. Appl., 1.10.24).—Hydrocarbon oils are cracked by introducing them into a chamber along with a combustible mixture, firing the mixture and thereby subjecting the oil to a cracking temperature under a high pressure, and finally releasing the pressure to effect distillation of the products. C. O. HARVEY.

Cracking of hydrocarbon oils and manufacture of cyanides. J. C. CLANCY (U.S.P. 1,651,114, 29.11.27. Appl., 28.12.25).—In the process of U.S.P. 1,567,241 (B., 1926, 486) the carbonaceous material is replaced by hydrocarbon oils.

T. S. WHEELER.

Coking of hydrocarbon oils. R. E. WILSON and H. V. ATWELL, Assrs. to STANDARD OIL Co. (U.S.P. 1,654,200—1, 27.12.27. Appl., [A] 26.11.24, [B] 21.1.27).—The oil is heated to temperatures above 482° on the surfaces of the nickeliferous metal walls of a chamber. Volatile products are withdrawn and scraping means are provided for removing the coke deposit.

C. O. HARVEY.

Reconditioning of crank-case oil. L. L. ODOM, Assr. to PURITY CHEMICAL Co. (U.S.P. 1,655,660, 10.1.28. Appl., 27.9.26).—Crank-case mineral lubricating oil is reconditioned by distilling off the low-boiling diluents and cooling while filtering the residual oil, agitating with sulphuric acid for about 30 min. and washing with water, agitating the washed oil at

a temperature of about 200° with sodium hydroxide solution for about 30 min. and again washing with water, heating the oil and evaporating off any intermingled water, and, lastly, agitating with diatomaceous earth and filtering. C. O. HARVEY.

Desulphurising petroleum and products derived therefrom. A. MAILHE (F.P. 611,890, 16.6.25).—The oil is heated with reducing agents, *e.g.*, with stannous or ferrous salts, or with tin or iron, or their alloys, in the presence of hydrochloric acid, in vessels constructed of material resistant to the action of acid vapours and hydrogen sulphide, and provided with condensing apparatus at the top. L. A. COLES.

Refining of oils. E. A. RUDIGIER, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,656,710, 17.1.28. Appl., 12.3.25).—An apparatus for dehydrating and cracking oils consists of a cracking coil whence the heated oil passes to a conversion chamber and thence to a vapour separator and heat interchanger. The oil to be treated passes through the heat interchanger to a dehydrating chamber and thence to the cracking coil.

C. O. HARVEY.

Counter-flow [oil-refining] still. F. E. GILMORE (U.S.P. 1,655,998, 10.1.28. Appl., 23.4.25).—Sets of horizontal heating pipes are spaced vertically within a shell of rectangular cross-section with baffling material between them. Hot gases are supplied to the lowest set and pass thence through the successive sets above it. Bubbler trays are arranged in the upper part of the shell with baffling material above them. An inlet for hot oil is provided between the trays, and above this an inlet, controlled thermostatically, for cold oil. An outlet for vapour is provided near the top and a float-controlled oil outlet near the bottom of the shell. H. HOLMES.

Purification of benzol, petrol, etc. K. COX and P. J. McDERMOTT, ASSRS. to REFINERS, LTD. (U.S.P. 1,658,285, 7.2.28. Appl., 25.2.26. U.K., 12.1.26).—See B.P. 269,242; B., 1927, 436.

Machines for breaking coal or other friable material. B. NORTON (B.P. 283,835, 9.11.27).

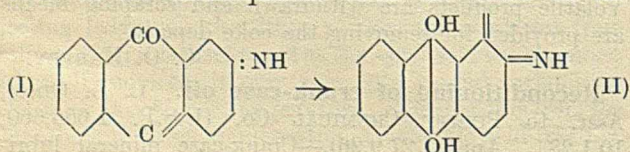
[Valve device for] supplying pulverulent fuels to internal-combustion engines. I. G. FARBENIND. A.-G. (B.P. 266,381, 21.2.27. Ger., 20.2.26).

Gas purifiers. H. H. HOLLIS (B.P. 283,283, 8.10.26).

Activated carbon (B.P. 283,267).—See VII.

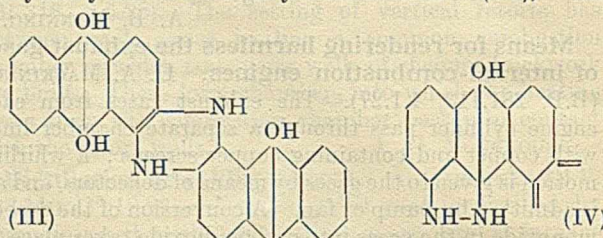
III.—ORGANIC INTERMEDIATES.

Alkaline fusion of anthraquinone derivatives. E. SCHWENK (Chem.-Ztg., 1928, 52, 45—46, 62).—Mechanisms for a number of potassium hydroxide fusion reactions in the anthraquinone series are based on the

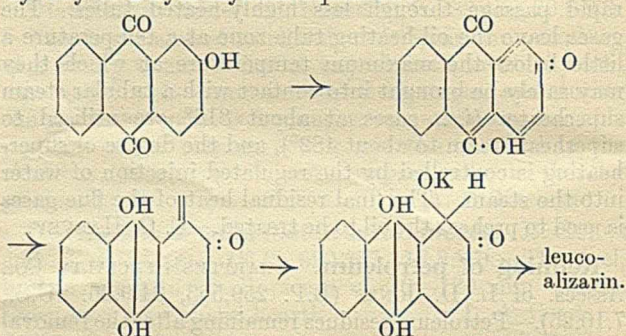


suggestion that one of the rings assumes a *p*-quinonoid configuration. The production of indanthrone, *e.g.*, is regarded as a simple condensation of 2 mols. of quinonoid 2-aminoanthraquinone (I, II). Below 200°

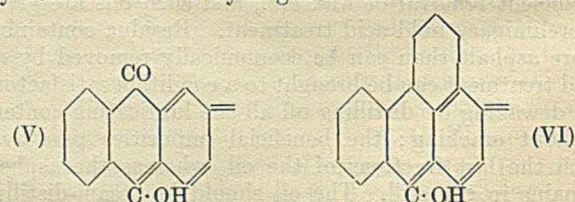
condensation is slow and oxidation to leuco-alizarin by way of 2-amino-1-hydroxydihydroanthraquinone is the main reaction. At higher temperatures tetrahydroindanthrone, accompanied above 300° by dihydroflavanthone, is formed. Since tetrahydroindanthrone undergoes oxidation-reduction with production of equal amounts of dihydroindanthrone and the useless trihydroxy-1 : 2 : 2' : 1'-dihydroanthrazine (III), the



yield of the former can never exceed 50% of the 2-aminoanthraquinone used. The conversion of pyrazolanthrone into Pyrazolanthrone Yellow may be figured similarly as the union of two *o*-quinonoid molecules (IV) to give first the tetrahydro-compound; and the yield of leucovat dye cannot exceed 50%. Alizarin formation from 2-chloro-, 2-bromo-, or 2-sulpho-anthraquinone proceeds by way of 2-hydroxyanthraquinone :



The low yield in absence of an oxidant is due to attack of leuco-alizarin by the hot alkali. A similar addition of K·OH to the *p*-quinonoid form (V) of anthraquinone accounts for the production of 2-hydroxyanthraquinone and leuco-alizarin by alkaline fusion of anthraquinone. Dibenzanthrone formation is figured as union of two *p*-quinonoid benzanthrone molecules (VI) followed by removal of two hydrogen atoms from the resulting



dihydrodibenzanthronyl, and a similar explanation is applied to the alkaline fusion of benzanthronequinoline, naphthalimide, etc. C. HOLLINS.

Activity of a nickel catalyst. E. J. LUSH (J.S.C.I., 1927, 46, 454—456 T).—In the hydrogenation of naphthalene with a nickel catalyst, it was shown that in the vapour phase only tetralin could be formed, whereas in the liquid phase decalin was produced. The total hydrogen absorbed was independent of the relative pro-

portion of tetralin and decalin made. Evidence was produced to show that in the hydrogenation of acetone, vegetable oils, and naphthalene, a given weight of activated nickel turnings used under standard conditions caused the same absorption of hydrogen per hour.

“Demolisation.” WISLICENUS.—See II.

PATENTS.

Laboratory apparatus for the production of absolute alcohol. ÉTABL. POULENC FRÈRES (F.P. 621,388, 12.1.26).—Several types of interchangeable glass dehydrating columns connected in series are attached to a central vessel, the whole apparatus being immersed in a water-bath.
L. A. COLES.

Manufacture of formaldehyde. SOC. CHIM. DE LA GRANDE-PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 272,155, 7.9.26. Fr., 3.6.26).—Formaldehyde is formed by the interaction of carbon monoxide and water (liquid or vapour), thus: $2\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{CH}_2\text{O}$, in the presence of magnesium, zinc, cadmium, manganese, tin, lead, antimony, or bismuth, or their oxides or salts, alone or mixed, at 200–600°, preferably under pressure. The following metals and their oxides must be absent: chromium, molybdenum, tungsten, uranium, vanadium, niobium, copper, silver, gold, the iron group, and the alkali metals. The walls of the reaction vessel are made of inert metals (e.g., aluminium) or their alloys amongst themselves or with metals detrimental to the reaction (e.g., tinned copper). Thus, by using an aluminium tube, heated at 220°, containing manganese carbonate reduced with dry carbon monoxide at 150°, a dilute solution of formaldehyde was obtained, whereas with an aluminium-lined tube, at 340°, containing a mixture of magnesium oxide and antimony trioxide, a 12% solution was formed.
B. FULLMAN.

Method of condensation [of acetone]. N. W. THOMSON, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,654,103, 27.12.27. Appl., 19.3.25).—Diacetone alcohol is obtained by leaving a suspension of potassium hydroxide in a mixture of acetone and benzene at room or lower temperature for 5–12 hrs.
T. S. WHEELER.

Manufacture of dinitrotoluene. E. H. RODD and R. W. EVERATT, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,657,723, 31.1.28. Appl., 8.12.26. U.K., 12.5.26).—See B.P. 263,018; B., 1926, 101.

Preparation of new derivatives of naphthoquinone. A. WAHL and R. LANTZ, Assrs. to SOC. ANON. DES MAT. COL. ET PROD. CHIM. DE ST. DENIS (U.S.P. 1,657,809, 31.1.28. Appl., 12.7.24).—See B.P. 206,150; B., 1925, 200.

Production of α -aminoanthraquinones. J. THOMAS and A. H. DAVIES, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,657,420, 24.1.28. Appl., 20.7.21. U.K., 23.7.20).—See B.P. 173,006; B., 1922, 170 A.

IV.—DYESTUFFS.

Dyestuffs fast to ironing. III. R. HALLER, J. HACKL, and M. FRANKFURT (Kolloid-Z., 1928, 44, 83–84; cf. Haller, B., 1927, 840).—Solutions of dyes fast to ironing contain two or more disperse phases of different colours, which are in equilibrium with each other at a given

temperature. This was investigated by passing an electric current through the solution of the dye between two electrodes of white, semi-conducting material, such as filter paper. When Congorubin was treated in this way, after dialysis, using a difference of potential of 600 volts, the anode became blue, whilst the cathode remained white. With rising temperature, the anode became violet, later tending to scarlet. Addition of electrolytes such as sodium acetate, sodium chloride, ammonium chloride, and magnesium sulphate effected a reversal in the charge of one of the disperse phases, for, on electrolysis, the anode then became blue and the cathode red. Similar results were observed with Diamine Blue 3R, Bordeaux Extra, Benazurin G and 5G, Benzo Brown RC, Brilliant Benzo Violet 2R, and Brilliant Benzo Fast Violet 2RL.
E. S. HEDGES.

Alkaline fusion of anthroquinone derivatives. SCHWENK.—See III. **Dyeing with sandal wood extract.** SOEP.—See XIX. **Light-sensitivity of dyes.** STEIGMANN.—See XXI.

PATENTS.

Manufacture of azo dyes [for wool]. I. G. FARBENIND A.-G. (B.P. 276,353, 19.8.27. Ger., 19.8.26).—2 : 5 : 6-Trichloro-*m*-toluidine-4-sulphonic acid (prepared by trichlorinating *p*-toluenesulphonic acid, nitrating, and reducing the product) is diazotised and coupled with a sulphonylpyrazolone, e.g., 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone, to give acid dyes fast to light.
C. HOLLINS.

Manufacture of new azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,151, 14.9.26).—Monoazo dyes, having as end-component a dihydroxynaphthalenesulphonic acid, are esterified with an arylsulphonyl halide in presence of an acid-binding agent. E.g., the dye *o*-anisidine \rightarrow 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid, or aniline \rightarrow 1 : 6-dihydroxynaphthalene-3-sulphonic acid, is treated with *p*-toluenesulphonyl chloride. Good light- and fulling-fastness on wool is claimed.
C. HOLLINS.

Manufacture of [azo] dyes [pigments and ice-colours]. FABR. DE PROD. CHIM. ROHNER SOC. ANON. PRATTELN (B.P. 275,147, 6.12.26. Fr., 2.8.26).—Arylamides of 2 : 3-hydroxynaphthoic acid are coupled in substance or on the fibre with the tetrazo derivative of a diaminoazo compound obtained by coupling a diazotised mono-oxalyl derivative of benzidine, tolidine, dianisidine, etc. with a middle component and removing the oxalyl group by hydrolysis. Black pigments and dyeings result. An example is 4'-aminodiphenyl-4-oxamic acid \rightarrow α -naphthylamine, hydrolysed, tetrazotised, and coupled with 2 : 3-hydroxynaphthoic α -naphthylamide.
C. HOLLINS.

Manufacture of monoazo dyes capable of being chromed. I. G. FARBENIND. A.-G. (B.P. 263,816, 23.12.26. Ger., 23.12.25).—Chrome red dyes fast to fulling, potting, and light are obtained by coupling diazotised 5-nitro- or 4-chloro-5-nitro-*o*-aminophenol with 1-(2-hydroxy-5-sulpho-3-carboxyphenyl)-3-methyl-5-pyrazolone or the corresponding 3-carboxylic acid or ester.
C. HOLLINS.

Monoazo dyes. K. H. SAUNDERS and H. GOODWIN, Asss. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,657,724, 31.1.28. Appl., 14.4.27. U.K., 17.4.26).—See B.P. 266,561; B., 1927, 325.

Manufacture of azo dyes. A. L. LASKA and A. ZITSCHER, Asss. to I. G. FARBENIND. A.-G. (U.S.P. 1,657,803, 31.1.28. Appl., 10.9.25. Ger., 20.9.24).—See G.P. 430,579; B., 1927, 213.

Inks (B.P. 279,968).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of chlorine and hypochlorous acid on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J.S.C.I., 1928, 47, 4—8 T).—The authors have shown (cf. B., 1926, 480) that the action of hypochlorous acid on wool is of a different nature from that of chlorine. Further investigation indicates that when chlorine reacts with wool soluble and insoluble chloramines are formed, but none are produced by hypochlorous acid, although chlorine enters into combination with the wool proteins. Wool chlorinated with chlorine retains its power of condensing with semicarbazide, the increase in nitrogen content being the same as in the case of the untreated wool. On the other hand, after treatment with hypochlorous acid wool does not condense with semicarbazide. The condensation in the former case is not due to any reaction between the semicarbazide and the chloroamine group. Wool treated with chlorine in presence of sulphuric acid condenses with more semicarbazide than it does when chlorinated in presence of hydrochloric acid. Experiments were made with organic compounds containing CO and CO·NH groups. It is suggested that hypochlorous acid combines directly with the carbonyl groups of the protein molecule in one of the following ways. Direct combination might form ·NH·C(OH)(OCl) groups which if unstable would either give ·N·COCl + H₂O, or the reaction may be ·CH₂·CO + HOCl = ·CH(OCl)·CH(OH).

Action of acids on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J.S.C.I., 1928, 47, 45—49 T).—The rate at which dilute acids are taken up by wool was studied, the period of immersion being 24 hrs. With hydrochloric acid there was a regular increase in the amount of acid removed from the solution as the concentration increased from 0.01M to 1.0M. In the case of sulphuric acid, however, the maximum amount was taken up at 0.075M, and at 0.5M and 1.0M there actually appeared to be more acid present at the end of 24 hrs. than at the beginning of the experiment; similar results were obtained with acetic acid. These abnormal results are believed to be due to hydrogen sulphide or diffusible nitrogen compounds from the wool interfering with the titration, since normal results are obtained when the amount of sulphuric acid remaining is determined gravimetrically. It is suggested that the first stage of the action of acids on wool consists of sorption. More dilute solutions of sulphuric acid remove more nitrogen from wool than concentrated solutions. Probably, at high concentrations the surface film of sorbed acid becomes too viscous to allow diffusion of the products of reaction. Experiments on the effect of agitation on the rate of sorption seem to indicate that

the surface film is of considerable thickness. The effect of p_H on the action of acids on wool is considered and a scheme of equilibria is suggested. Experiments are described showing that hydrochloric acid prevents the condensation of semicarbazide with wool, but no such effect is observed in the case of sulphuric acid. Finally, some effects of acids on the physical properties of woollen yarns are described.

Degumming of silk. I. Action of alkali. II. Effect of formaldehyde in alkaline media. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1927, 30, 568—573, 573—579).—I. The raw silk was treated with potassium hydroxide solutions of various strengths, and the amounts of sericin dissolved and alkali consumed were determined. It was observed that there are three stages in the action of alkali on sericin, viz., glutinisation (that is, swelling and the primary albuminate formation); dispersion; and secondary albuminate formation. The secondary albuminate thus formed stabilises the dispersed sericin particles and prevents them from being again adsorbed on the silk fibre. II. When formaldehyde was added to the degumming bath, or when the raw silk was treated with formaldehyde before degumming, the amount of sericin dissolved was less, and that of the consumed alkali more, than was the case when no formaldehyde was used. This is explained by assuming that formaldehyde lowers the dispersibility of the sericin and increases the reactivity of the latter towards alkali due to neutralisation of its amino-group. Y. TOMODA.

Constituents of the fibres of Japanese hemp-palm and sponge-gourd. S. MASUDA (J. Cellulose Inst., Tokyo, 1927, 3, 321—323).—The fibres, which are very resistant to decay in water, were purified mechanically, cut up, and analysed. The results (calculated on the dry material) for hemp-palm (*Trachycarpus excelsus*, Wendl), and sponge-gourd (*Luffa cylindrica*, L., Roem), respectively, are: water, 11.34% (12.65%); benzene extract 0.77% (0.44%); alcohol extract 1.39% (2.9%); cellulose (on the extracted, dry substance) 42.6% (57.65%); reducible sugar, after hydrolysis of the isolated cellulose, 92.3% (99.7%) of the theoretical yield; lignin 37.5% (14%) with 0.54% (0.18%) of ash; and pentosan 16.8% (18.6%). Mucic acid was not isolated and only a trace of mannose-hydrazone was obtained from hemp-palm fibres, whilst those from sponge-gourd contained mannan 0.4%, galactan 1.8%. In both products, after hydrolysis with 3% sulphuric acid, xylose was identified.

B. P. RIDGE.

Rate of absorption of sodium hydroxide in the decomposition of straw at ordinary temperature. Y. KAUKO (Annal. Acad. Sci. Fennicæ [Komppa Jubilee Vol.], 1927, A29, No. 7. 30 pp.).—The rate of neutralisation of sodium hydroxide solution (6—12%) by straw at ordinary temperature is determined by measurements of conductivity. The velocity of reaction during the first stage is mainly controlled by the rate of diffusion into the tissue of the sodium hydroxide, which is neutralised in part by the silicic acid. This stage is followed by the dissolution of the lignins and the carbohydrates and the breaking up of the structure of the straw. G. A. C. GOUGH.

Pulping flax straw. III. Hydrolysis and delignification with sodium hydroxide and with a mixture of sodium hydroxide and sodium sulphide. M. W. BRAY and C. PETERSON (*Pulp and Paper Mag.*, 1928, 26, 37—41).—The results of a series of experimental cooks on flax straw at 155° using caustic soda alone and mixtures of caustic soda with sodium sulphide indicate that caustic soda-sodium sulphide liquors are more efficient than straight caustic soda liquors of the same available alkalinity (calculated as NaOH on the assumption that $\text{Na}_2\text{S} \rightarrow \text{NaOH} + \text{NaHS}$ by hydrolysis), in that the liquors containing sulphide remove lignin at a more rapid rate, and to a greater extent, whilst causing less destruction of the cellulose itself. For example, digestion for 7 hrs. with 15% of available alkali (13.4% of caustic soda and 3.3% of sodium sulphide) gives a pulp of approximately the same lignin content as is given by a straight caustic soda digestion for 10 hrs. with 20% of caustic soda. Further, chemical examination shows that the cellulose prepared with the sodium sulphide liquors is less degraded than that prepared with caustic soda alone. In comparison with sodium sulphite liquors, both caustic soda and caustic soda-sodium sulphide liquors are more rapid in their action, but cause a greater loss of cellulose for a given amount of lignin removed. D. J. NORMAN.

Sulphite pulp from spruce. D. E. CABLE (*Pulp and Paper Mag.*, 1927, 25, 1215—1222, 1299—1305, 1331—1332, 1405—1408, 1422—1424).—Previous work dealing with the different phases of the sulphite process is reviewed and results are given showing the effects of various alterations in the cooking conditions in the experimental digestion of spruce wood. For spruce wood, though not necessarily for other woods, the pulp shows substantially the same degree of purity irrespective of the base (calcium, magnesium, ammonium, sodium) present in the cooking liquor. The injection of sulphur dioxide at the top of the digester during the first hour of the cook facilitates penetration of the chips by the liquor and reduces screenings (except in the case of sodium bisulphite liquors), with the result that increased yields of pulp of reduced bleach consumption are obtained. The effect of gradually lowering the digester pressure during the last few hours of the cook from the maximum (75 lb./sq. in.) to about 55—58 lb./sq. in., the liquor being maintained at about 148°, is to increase the yield of pulp, more particularly with straight calcium bisulphite liquors, by 1.2—2.7% above that obtained when the pressure is held at 75 lb. throughout the cook. This increased yield is, however, offset to some extent by an increased bleach consumption. D. J. NORMAN.

Sulphite-liquor lactones. B. HOLMBERG (*Annal. Acad. Sci. Fennicae [Komppa Jubilee Vol.]*, 1927, A29, No. 6. 16 pp.; cf. A., 1923, i, 1067; 1921, i, 772, 849, 850).—Sublimation of sulphite-liquor lactone, $[\alpha]_D - 55.2^\circ$ in acetone, at 225—260°/0.05 mm. gives rise to an isomeride, m.p. 206—209°, $[\alpha]_D + 30.7^\circ$, which is converted into β -dimethyl sulphite-liquor lactone by dimethyl sulphate. α -Dimethyl sulphite-liquor lactone is similarly converted into a mixture of almost equal parts of α - and β -dimethyl sulphite-liquor lactones. Treatment of either α - or β -dimethyl sulphite-liquor lactone with alcoholic sodium ethoxide leads to the formation

of a mixture of the two substances. The β -compound is changed to a dimorphous variety, m.p. 155.5—156.5°, by heating at 170°. α -Dimethyl sulphite-liquor lactone is oxidised by sodium hypobromite to oxalic acid; a neutral, optically inactive substance, $\text{C}_{20}\text{H}_{20}\text{O}_5$, m.p. 102—104°, containing 4 methoxy-groups; a dibasic acid, $\text{C}_{22}\text{H}_{24}\text{O}_8$, m.p. 193—194°, $[\alpha]_D + 36.9^\circ$ in acetone, containing 4 methoxy-groups (possibly tetramethyltruxillic or tetramethyltruxinic acid); and an optically inactive, monobasic acid, $\text{C}_{20}\text{H}_{20}\text{O}_8$, m.p. 224—225°.

G. A. C. GOUGH.

Pentosans in the sulphite[*-*cellulose] cooking process. L. SHIERJEBOW (*Zellstoff u. Papier*, 1928, 8, 87—88).—On heating with the cooking acid above 80°, pentosans form a compound with calcium bisulphite in which one pentosan molecule, consisting of four pentose residues, is united to one bisulphite molecule; increasing the temperature to 136° causes a further addition of bisulphite with the formation of a more stable compound in which the bisulphite molecule is united to two pentose residues. The fundamental unit of the pentosans ($\text{C}_5\text{H}_8\text{O}_4$)_n may therefore be regarded as ($\text{C}_5\text{H}_8\text{O}_4$)₄, further hydrolysis causing a splitting of this into two parts in which $n = 2$. Above 137° rapid decomposition of this bisulphite-pentosan compound takes place with destruction of the pentoses, oxidation of the sulphur dioxide to trioxide, and the subsequent formation of calcium sulphate. If too little lime is present free sulphuric acid is formed. Sulphurous acid without bisulphite acts more quickly than bisulphite, but the reactions of the two are sharply distinguished from one another. If magnesium oxide is present instead of lime, the nature of the reaction between the pentosans and bisulphite is unaltered and a sulphur-magnesium compound remains at the end of the cooking. The formation of calcium sulphate is limited by the decomposition of the pentosan-bisulphite compound formed at the beginning of the reaction. The loose combination of bisulphite with the organic constituents of wood, which is destroyed by the action of ammonia with separation of calcium sulphite, or by heating with mineral acids with separation of sulphur dioxide, appears as a result of the addition of bisulphite to lignin or hexoses. The pentosans do not form such a compound.

B. P. RIDGE.

Preparation of cellulose nitrate. E. BERL and E. BERKENFELD (*Z. angew. Chem.*, 1928, 41, 130—132; cf. Berl and Klaye, B., 1907, 1255).—The nitrogen content and viscosity in acetone solution of a sample of cellulose nitrate is dependent rather on the composition of the waste nitrating acid than on that of the original acid mixture used, since an equilibrium is set up between cellulose nitrate and acid. Curves showing the nitrogen content and viscosity corresponding to varying waste acid compositions are given for both linters and wood cellulose, and the results are much more regular than those obtained when these properties are correlated with the composition of the starting acids. Increase of the sulphuric acid content above 60% causes a sharp diminution in the viscosity of the product, and the viscosity of nitrated linters is greater than that of nitrated wood cellulose with the same waste acid composition.

W. J. POWELL.

Effect of temperature on the rate of decomposition of nitrocellulose. R. W. RYAN and E. A. LANTZ (Ind. Eng. Chem., 1928, 20, 40—42).—The stability of nitrocelluloses may be measured by determining the rate of evolution of oxides of nitrogen at different temperatures and plotting the logarithm of the rate of evolution of nitric oxides, calculated as nitrogen, against the temperature. The resulting graphs are straight lines, and, when extended below the lowest temperature at which a test was made, give useful indications of the stability of nitrocelluloses at lower temperatures, whilst at the same time accentuating any differences between various samples. 2—5 g. of the nitrocellulose (previously air-dried and finally dried *in vacuo* over phosphorus pentoxide) are heated in a special decomposition tube provided with a spiral preheating coil for the air current, the whole being accommodated in a suitable vapour bath. Air, free from acid gases and moisture, is drawn through the sample at the rate of 4—5 litres/hr. and the oxides of nitrogen are absorbed in a cooled, neutral 5% solution of potassium iodide, the liberated iodine being titrated electrometrically. The time required to establish a constant rate of evolution of nitric oxides varies from 1—2 hrs. at 130—140° to 16—20 hrs. at 99—100°; titrations are therefore made at intervals of 30 min. at 137—140°, 1 hr. at 116—118°, and 3·5—4 hrs. at 99—100°.

D. J. NORMAN.

Viscosity of nitrocellulose. H. DABISCH (Farben-Ztg., 1928, 33, 1105—1106).—The statement by Merz (cf. B., 1927, 810) that the Engler viscosimeter gives "scientifically exact" measurements of the viscosity of nitrocellulose is criticised on the grounds that all nitrocellulose solutions are more or less plastic, but no distinction between plasticity and true viscosity is recorded in Engler determinations. An ideal viscosimeter will work at sufficiently high pressure to render plasticity effects negligible. From consideration of the influence of various solvents and diluents on the viscosity of nitrocellulose, a mixture of 45 pts. of absolute ethyl alcohol and 55 pts. of pure butyl acetate is recommended as the most desirable solvent. The concentration of the solution is adjusted for each type of nitrocellulose used so that normal lacquer consistency is obtained.

S. S. WOOLF.

Production of fibrous precipitates of substances and the structure of the fibres, particularly cellulose fibres. P. P. VON WEIMARN (Kolloid-Z., 1928, 44, 163—166).—General methods are described by which any insoluble substance can be precipitated in a fibrous form. In a double decomposition between two soluble salts, where one of the products is insoluble in the medium, by employing highly-concentrated solutions of the reactants the precipitate can be shown under the microscope to consist of thread-like structures. The threads are composed of a gelatinous sheath of the insoluble substance, containing a solution of one of the reactants. The further fate of the fibrous structure depends on the relative osmotic pressures of the solutions inside and outside the membrane. The structure of natural cellulose threads is very complicated, the fibres consisting of three concentric tubes enclosing layers of other substances. The outer sheath consists

of a hard skin of waxy material, the middle tube consists mainly of cellulose, and the inner tube of plasma. Between the first and second tubes is a layer of "hide"-cellulose, and between the second and third a layer of "plasma"-cellulose. The middle tube is in itself a complicated system of concentric fine tubes, the walls of which consist of ultramicroscopic fibres of cellulose. The chemical properties of the first and second tubes of wood cellulose are not identical with those of cotton cellulose, a marked difference being observable in the resistance towards the dispersing effect of concentrated solutions of very soluble salts. The high resistance of the outer sheath of cotton cellulose gives rise to an irregular swelling, which gives a characteristic appearance under the microscope.

E. S. HEDGES.

Change of plasticity of viscose with ripening. K. ATSUKI, T. TAKAGI, and T. OHTA (J. Cellulose Inst., Tokyo, 1927, 3, 317—320).—Measurements of the rate of flow of a viscose containing 7% of cellulose and 7·78% of sodium hydroxide, carried out in a modified Ostwald viscosimeter at 25° under pressures of from 3 to 25 cm. of mercury, after ripening the material at 25° for various times from 53 to 240 hrs., show that viscose has a slight but measurable plasticity, since the flow rate—pressure curve at a lower pressure is convex to the pressure axis, giving a yield value. As ripening proceeds, the yield value becomes smaller, reaches a minimum at 121 hrs., and again increases. It is supposed that the disperse phase, at first a continuous system, becomes less continuous by dehydration with the ripening, but that after maximum dehydration, when the yield value is a minimum, it begins to coagulate, forming a firm continuous system giving a yield value. The disperse phase, before and after minimum plasticity is reached, is more or less continuous, but differs in rigidity. In the former case it is easily deformable through being highly hydrated, whilst in the latter it is rigid on account of the formation of micelles of highly hydrated particles.

B. P. RIDGE.

Measurement of hydrogen-ion concentration in the control of pulp and paper manufacture. K. W. FRANKE and J. J. WILLAMAN (Ind. Eng. Chem., 1928, 20, 87—95).—From a review of methods of measuring the hydrogen-ion concentration it is concluded that the hydrogen electrode could be used for control purposes in the preparation of sulphite liquors providing that sulphitation is not carried beyond neutrality; below p_H 7 the readings are unreliable. The quinhydrone electrode is, in general, unsuited for the control of pulp and paper manufacturing operations. The most satisfactory results are obtained with the antimony electrode using ordinary pure stick antimony of radially crystalline structure. Both oxygen and carbon dioxide affect the potential of an antimony electrode, but, if partially closed electrode vessels are used and stirring is accomplished without unnecessary agitation, the results are trustworthy when the readings are taken within 2 min. Further, since all solutions contain oxygen, a tarnished electrode, though somewhat slower in response than a bright electrode, is more dependable. The antimony electrode correctly indicates the progress of the reaction in the preparation of neutral sulphite liquor from

alkaline leach liquors, and, being unaffected by sulphur compounds, is also suitable for determining the p_H value of the spent cooking liquor. For alkaline solutions, its upper limit of usefulness is about p_H 12. Other operations which could with advantage be controlled by the antimony electrode are engine sizing and water purification. The following equation is given for converting antimony electrode potentials into p_H values: $E = 0.050 + 0.054p_H$, at 25° (cf. Kolthoff and Hartong, B., 1925, 154).

D. J. NORMAN.

Economy of the sulphite-cellulose cooking process. A. FROBERG (Zellstoff u. Papier, 1928, 8, 83—87).—See B., 1928, 120.

Action of nitrous acid on wool. WATERMAN and GROOT. Mercerisation. LOTTERMOSER and RADESTOCK. —See VI.

PATENTS.

Treatment of hemp and other vegetable fibres.

L. LAPIERRE (B.P. 279,946, 5.8.26).—The fibres in the form of a mat are placed on rotating screens in an autoclave, in which, after complete degumming, they are treated at 120° for about 2 hrs. with a solution consisting of potassium nitrate 10 kg., ammoniacal potassium sulphide 5 kg., sodium chloride 0.5 kg., sodium borate 0.5 kg., magnesium sulphide 1 kg., ammoniacal copper oxide 0.25 kg., and water 600 litres. The boiled fibre is washed, treated with 2% hydrochloric acid, and again washed until free from acid. "Oxaline oil" (1%) is then applied and the fibre passed through a 1% solution of potassium nitrate. The fibres thus acquire a silky appearance.

D. J. NORMAN.

Preparation of a wool-like material from jute, *Corchorus capsularis*, etc. P. GELLI (F.P. 613,973, 2.4.26).—The fibres are freed from impurities, opened up, and treated with caustic soda solution (d 1.263) at 25—30°. The mixture gradually becomes hotter of its own accord, and, when steam begins to appear, the fibres are removed from the bath, washed, and bleached.

D. J. NORMAN.

Washing or cleaning of wool. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 251,669, 3.5.26. Fr., 4.5.25).—The process differs from that of B.P. 273,755 (B., 1928, 11) in that a substantially high but not necessarily constant concentration of suint liquor is maintained in one or more bowls of a washing set of the periodical-stoppage type, whilst a minimum quantity of fresh liquid (water or weak suint liquor), not substantially exceeding 1 litre per kg. of wool treated, is introduced into the installation. Methods are described for diminishing the losses of suint liquor and for purifying it for re-use.

D. J. NORMAN.

Treatment of fibrous filling material for use in the manufacture of plastic masses. K. RÖMMLER (G.P. 445,535, 31.3.25).—Cotton waste etc. is scoured with a degreasing agent, such as hot sodium silicate solution, after which it is dried, cut up into small pieces, washed to remove residual sodium silicate, and again dried.

L. A. COLES.

Preparation of artificial silk. T. IWASAKI (F.P. 621,543, 15.9.26).—Artificial silk of improved internal structure, *i.e.*, with the particles arranged substantially

in the form of parallel chains, is obtained by converting viscose or similar colloidal silk-forming solution into a linear sol prior to spinning. This is effected by passing the solution through the field of a high-frequency electric current in a vacuum or in an atmosphere of an inert gas such as hydrogen. The solution is then ripened and stretch-spun.

D. J. NORMAN.

Production of high α -cellulose fibre. G. A. RICHTER, ASST. to BROWN Co. (U.S.P. 1,654,603, 3.1.28. Appl., 12.5.25. Renewed 3.6.27).—Sulphite pulp made with a liquor in which soda is the base is washed and digested with an alkaline liquor containing sodium compounds. The resulting high α -cellulose pulp is washed and a portion of these washings used for the alkaline digestion of further quantities of sulphite pulp, whilst the remainder is mixed with the washings from the sulphite process. This mixed wash-liquor is then treated for the recovery of sodium compounds.

D. J. NORMAN.

Manufacture of fine viscose filaments. VEREIN. GLANZSTOFF-FABR., A.-G. (G.P. 444,113, 21.5.19).—By using spinning baths containing considerably more acid than is customary in conjunction with viscose solutions of a suitable degree of ripeness, filaments of any desired denier may be spun through apertures of the usual size, *e.g.*, 0.1 mm., by adjusting the concentration of acid according to the particular denier required.

D. J. NORMAN.

Hydrolysis of cellulose whereby high yields are obtained of products suitable for pure or symbiotic butyric fermentation. SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (F.P. 616,424, 20.5.26).—Cellulosic material, *e.g.*, wood, is partially hydrolysed by the action of sulphuric acid or sodium acid sulphate under such conditions of concentration and temperature that the products of hydrolysis are not destroyed. Hydrolysis is then completed by the action of hydrofluoric acid under pressure, the hydrofluoric acid being obtained by the addition of the calculated quantity of, *e.g.*, calcium fluoride. The resulting liquor is neutralised with chalk and the precipitated calcium fluoride recovered.

D. J. NORMAN.

Apparatus for the dehydration of viscose products. S. A. NEIDICH (U.S.P. 1,654,553, 3.1.28. Appl., 1.5.26).—Wet viscose products are conveyed through a tubular conduit heated at the middle and cooled at the ends. A volatile liquid dehydrating agent is introduced into the conduit at the heated part and is recovered by condensation at the ends.

D. J. NORMAN.

Conversion of cellulose derivatives into a finely powdered form. LONARIT-GES. (G.P. 445,308, 7.7.23).—Plastic material containing, *e.g.*, cellulose derivatives, camphor substitutes, and fillers, heated to render it sufficiently fluid, is forced through fine jets, and as it issues from these the solvent evaporates rapidly, causing the material to swell into porous threads which, after drying, are readily reducible to a fine powder.

L. A. COLES.

Manufacture of sheets, pellicules, or films of cellulose esters and ethers. H. J. HANDS, and SPICERS, LTD. (B.P. 281,803, 24.9.26).—A means is described for producing films etc. exhibiting a progressive variation

in constitution, or in mechanical or physical properties between their opposite faces. The compositions are applied in succession to a support, from which they are finally stripped, in substantially immediate succession, a second composition being applied before any material portion of the solvent has evaporated from the composition first applied.

W. CLARK.

Production of coloured material from cellulose esters. SOC. CHIM. DES USINES DU RHÔNE (F.P. 619,764, 3.8.26).—The colouring matter is formed in a solution of the cellulose ester in a suitable solvent, which is subsequently removed by evaporation; *e.g.*, a blue product is obtained by adding potassium ferrocyanide solution to a well-stirred mixture of cellulose acetate dissolved in acetone with ferric chloride solution, whilst a yellow product is obtained by using cadmium sulphate and sodium sulphide.

L. A. COLES.

Colloidising agent for nitrocellulose. T. L. DAVIS (U.S.P. 1,654,114, 27.12.27. Appl., 25.4.22).—The use of *s*-dialkylcarbamides is claimed.

T. S. WHEELER.

Recovery of by-products from sulphite-cellulose waste-liquor. L. PETIT-DEVAUCELE (F.P. 619,861, 14.12.25).—The liquor is treated with alkali to convert the bisulphite into sulphite and dried to a moisture content of about 10%; 2–10% of gas tar is then added and the mixture completely dried to give a pitch-like product suitable for, *e.g.*, roads. Alternatively, part of the sulphur may be recovered by treating the liquor with hydrogen sulphide, after which air is passed in to convert calcium sulphite into sulphate whereby resins are separated and recovered. Resins may also be separated by treating the heated liquor with crude salt. The resins thus obtained are insolubilised by fusion and polymerisation, optionally after the addition of small quantities of natural resins, or they may be purified by the electrolytic hydrogenation of their solutions in soda.

D. J. NORMAN.

Treatment of the black liquor obtained in the manufacture of wood fibre by the soda process. E. HÄGGLUND (B.P. 273,267, 31.5.27. Ger., 23.6.26. Addn. to B.P. 258,035; B., 1927, 165).—The sodium acetate recovered by crystallisation, when the liquor discharged from the high-pressure tank is treated with a caustic alkali and concentrated, as described in the main patent, is contaminated with other soda salts. In order to avoid this, the liquor from the high-pressure tank is evaporated to dryness, without the addition of caustic alkali, and the sodium acetate recovered by fractional lixiviation of the dry residue, either before or after carbonisation at, *e.g.*, 300° to decompose other organic compounds such as formates, propionates, and butyrates.

D. J. NORMAN.

Separation of copper sludge from the coagulating liquors used in the manufacture of artificial silk. HÖLKENSEIDE G.M.B.H. (G.P. 443,089, 29.6.25).—The liquor is allowed to settle in large vessels with porous walls provided at different levels with valves for withdrawing the clarified liquor and near the bottom with a larger outlet for the sludge. As the clear liquor is removed, partly by decantation and partly by percolation through the porous walls, the sludge residue contracts and detaches itself from the sides of the vessel of its own

accord. Provision is made for mechanically removing the solidified sludge.

D. J. NORMAN.

Interrelated pulp refining and viscose process. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,653,124, 20.12.27. Appl., 29.10.25).—The pulp is purified with caustic soda solution, washed, formed into sheets, and converted into alkali-cellulose. The excess of soda removed from the alkali-cellulose by pressure is used for the preliminary treatment of further quantities of pulp.

D. J. NORMAN.

Production of waterproof paper or paperboard. E. MELLERSH-JACKSON. From BENNETT, INC. (B.P. 280,083, 28.2.27).—A paper furnish comprising waste wax papers alone or in admixture with other paper-making material is beaten at a sufficiently high temperature (about 77°) to soften or melt waxes or any other waterproofing agents, *e.g.*, rosin, which it is desired to incorporate therewith. 5–20% of rosin is then added and dispersed throughout the furnish, after which sodium silicate (*d* 1.41), in the proportion of 3–4 pts. per 1 pt. of rosin and preferably heated at 99°, is incorporated. When dispersion of the wax and rosin is complete the furnish is cooled to 49°, treated with alum until a faint acid reaction is obtained, and run on a paper machine. The resulting sheets are dried and heated to cause the wax and rosin particles to coalesce.

D. J. NORMAN.

Manufacture of a textile material from vegetable raw material. J. C. M. VAN DIGGELEN, Assr. to N.V. OCTROOI MAATSCHAPPIJ "VĒDĒ" (U.S.P. 1,653,138, 20.12.27. Appl., 11.10.26. Holl., 29.4.24).—See B.P. 274,331; B., 1927, 774.

Manufacture of cellulose solution. Manufacture of viscose. L. LILIENFELD (U.S.P. 1,658,606—7, 7.2.28. Appl., 18.6.23. Austr., 15.3.23. Renewed [B] 23.11.27).—See B.P. 212,864—5; B., 1925, 37, 126.

Manufacture of artificial silk. F. W. SCHUBERT, Assr. to BRYSSILKA, LTD. (U.S.P. 1,657,697, 31.1.28. Appl., 21.6.26. U.K., 2.7.26).—See B.P. 258,371; B., 1926, 1009.

Manufacture of hollow artificial silk threads. ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (F.P. 621,181, 8.9.26. Czechoslov., 17.9.25).—See B.P. 258,582; B., 1927, 774.

Method and apparatus for obtaining fibrous material from stalks. E. GMLINDER (B.P. 283,370, 17.3.27).

Photographic supports (B.P. 282,980).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Action of nitrous acid on wool fibre and reactions of the product with azo components. H. I. WATERMAN and J. GROOT (Chem. Weekblad, 1928, 25, 18—22).—The production of colouring matter by treatment of wool fibre with nitrous acid and coupling appears to be due to adsorption rather than to true diazotation. There is no diminution of the nitrogen content of the fibre when it is treated with nitrous acid and boiled; the loss of nitrogen, which must therefore come from nitrous acid held up in some way in the fibre, is about 0.2% on boiling, but the treated fibre will take up considerably more H-acid than is equivalent to this.

The power of forming colouring matter with the coupling substances, also, is not lost even after the fibre has been treated with nitrous acid and then boiled several times. The treated fibre loses the power to become luminous under the influence of ultra-violet rays. S. I. LEVY.

Addition of pyridine to dye-vats. M. FREIBERGER (Z. angew. Chem., 1928, 41, 133; cf. Binz and Prange; B., 1928, 83).—The author refers to his previous publication (B. 1921, 175 A), not cited by Binz and Prange, in which the improvement in the rate of reduction of indigo obtained by the addition of pyridine and similar bases to the vat is described. W. J. POWELL.

Mercerisation of commercial cellulose and cellulose viscose. A. LOTTERMOSER and H. RADESTOCK (Z. angew. Chem., 1927, 40, 1506—1511).—The loss in weight and degree of swelling of various bleached and unbleached celluloses of different degrees of fineness, when mercerised with lithium, sodium, or potassium hydroxides, are determined and plotted graphically. With sodium hydroxide of increasing concentration the loss in weight rises, in every case, to a maximum at a concentration of 3.2 mols./litre, but the value of this mercerisation loss is least for the cellulose which has been most carefully treated in preparation (*e.g.*, unbleached and coarsely ground). The loss increases with the time of steeping up to 12—16 hrs., then remains constant for about 8 hrs., and finally falls to about its initial value after 20—24 hrs. The loss at 100° is not appreciably greater than twice that at 20°. A specially designed volumometer, containing mercury, for determining the degree of swelling of cellulose on mercerisation is described. Of the three alkalis examined, lithium hydroxide gives the greatest degree of swelling, and potassium hydroxide the least, but the curves drawn with molecular concentration of alkali as abscissæ and degree of swelling as ordinates are different in the three cases. With lithium hydroxide the curve has a maximum at a concentration of 2.8 mols./litre, with a horizontal portion on each side; with sodium hydroxide the maximum is at 3.15 mols./litre, and is followed by two horizontal portions; and with potassium hydroxide the maximum is at 3.8 mols./litre, and is followed by one long horizontal portion. The curve connecting time of steeping and degree of swelling is identical in appearance with the loss in weight—time curve. The curves showing the quantity of alkali taken up by cellulose from solutions of different strengths are not characteristic adsorption curves, and it is probable that true additive compounds are formed. This view is supported by the observation that these latter curves have horizontal portions at exactly the same molecular concentrations of alkali as are found in the curves for the degree of swelling. W. T. K. BRAUNHOLTZ.

Dyestuffs fast to ironing. HALLER and others.—See IV.

PATENTS.

Bleaching of vegetable fibres. R. VIDAL (F.P. 617,271, 22.10.25).—The moist material is treated, with or without previous digestion, first with gaseous chlorine in the absence of air, and then with a hypochlorite solution. D. J. NORMAN.

Dyeing, printing, or stencilling of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and W. B. MILLER (B.P. 283,253, 30.9.26).—Acetate silk is dyed (*etc.*) with nitro-derivatives of compounds containing two or more aromatic residues, two of which are united by a single linking not consisting of nitrogen alone, *i.e.*, excluding diphenylamines, azo dyes, and hydrazo compounds. Examples are 3:3'-dinitrobenzidine, 2-nitro-, 2:4-nitro-, and 4-chloro-2-nitro-*N*-benzylanilines, 3-nitro-4-aminodiphenyl ether, 3:3'-dinitro-4:4'-diaminodiphenylmethane, 3:3'-dinitro-4:4'-tetramethyldiaminobenzophenone. Various shades of yellow are obtained. C. HOLLINS.

Application of azo dyes [to regenerated cellulose silks]. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 283,319, 10.12.26).—Level blue shades on viscose are obtained by the use of disazo dyes from *p*-nitroaniline-*o*-sulphonic acid as first component, any suitable middle component, and as end component 2-amino-5-naphthol-7-sulphonic acid or its *N*-alkyl, aryl, or acyl derivatives. C. HOLLINS.

Fixation of dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 283,281, 7.10.26).—Acid or basic (triarylmethane) dyes are dyed on cotton, acetate silk, *etc.* previously mordanted with phosphotungstomolybdic acid, phosphotungstic acid, phosphomolybdic acid, or other derivatives of tungstic or molybdic acid. The mordant is itself fixed on the fibre by means of a preliminary mordanting with a fixing agent other than tannin (*e.g.*, benzidine, phenol, tetramethyldiaminobenzhydroisulphonic acid, dibenzylmetanilic acid, starch, sucrose, dextrose, lactose, cyclohexanol, *etc.*), and/or subsequent reduction with bisulphite, hyposulphite, formaldehyde, *etc.* C. HOLLINS.

Production of colour effects on fabrics. F. EDLINGER (F.P. 619,007, 16.7.26. Austr., 20.4.26).—Fabrics, particularly cotton fabrics, are moistened and passed between hot rollers engraved with the required design. On subsequent dyeing, those parts of the fabric which have been pressed dye to a lighter shade than those that have not been so treated.

D. J. NORMAN.

Increasing the wetting capacity of solutions. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H., G. BASEL, and F. KAUFLE (G.P. 444,966, 20.11.25).—An aliphatic amine such as butylamine, dibutylamine, diisobutylamine, or isoamylamine is used as a wetting agent, *e.g.*, in dyeing with azo dyes. D. J. NORMAN.

Bleaching with hypochlorites. H. WENZL, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,657,140, 24.1.28. Appl., 23.6.26. Ger., 27.7.25).—See B.P. 260,190; B., 1926, 1011.

Dyeing of artificial silk. C. S. BEDFORD (U.S.P. 1,657,656, 31.1.28. Appl., 31.8.26. U.K., 21.9.25).—See B.P. 263,222; B., 1927, 186.

Dyeing of cellulose acetate products in black shades. A. J. HALL, ASSR. to SILVER SPRINGS BLEACHING & DYEING Co., LTD. (U.S.P. 1,657,255, 24.1.28. Appl., 10.6.26. U.K., 10.7.25).—See B.P. 258,699; B., 1926, 976.

Azo dyes (B.P. 275,147).—See IV.

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

Mechanical salt-cake furnaces with indirect heat. The Mannheim furnace. F. BÖHM (Chem.-Ztg., 1928, 52, 101—102).—The Mannheim furnace was introduced about 30 years ago to work with the then abundant nitre-cake. This product was later replaced by sulphuric acid. It effected great economy in fuel and labour over hand furnaces. The nitre-cake was crushed and mixed with rock salt and delivered to the kiln by a screw conveyor. The kiln was fired by producer gas, and a charge required 5—6 hrs. to work, the usual output being 7 tons of salt-cake per 24 hrs., with a fuel consumption of 11—12%. Corrosion of the iron muffle was minimised by the use of dry nitre-cake and by the temperature being maintained at 850° or less. The quality of the iron is important. Parts of the stirring mechanism which were subject to severe corrosion were protected by ceramic or silicon plates. The life of the muffle was an output of 4500—5000 tons, but repairs were needed after an output of 2000 tons. The temperature necessary for calcination of the salt-cake approaches that at which corrosion by hydrochloric acid becomes severe. For working with sulphuric acid a higher temperature is required, and a ceramic muffle replaces iron. Salt and sulphuric acid are introduced separately in various ways, as external mixing gives troublesome incrustations in the feed pipe. The fuel consumption here is 18%. With both types of furnaces the waste heat is used for preheating the secondary air.

C. IRWIN.

Manufacture of potassium ferrocyanide. F. CHEMNITZ (Chem.-Ztg., 1928, 52, 90—91).—Spent oxide from gas purification is ground, extracted with carbon disulphide to remove sulphur, and then washed with water to free it from ammonium compounds. The residue is then treated on a stirring vessel with sodium carbonate, calcium hydroxide, and water in quantities required by its content of "blue." The whole is stirred with air agitation for 3 hrs., tested, and, if decomposition is complete, put through a washing filter press. The filtrate passes into iron tanks. Here, if the calcium content is high it is reduced by treatment with mother-liquor from ferrocyanide crystallisation, and hydrogen sulphide, if present, is removed by blowing. It is then treated in wooden tanks with ferrous chloride, which is preferable to the sulphate. The precipitate of $\text{Na}_2\text{Fe}[\text{Fe}(\text{CN})_6]$ is filter-pressed, washed, and treated with lime and potassium carbonate in a stirring vessel with a heating coil, the quantities again being determined by test. The filtrate from pressing is evaporated in lead-lined pans and crystallised.

C. IRWIN.

Preparation of finely-divided zinc oxide for ointments. E. DÖRZBACH (Pharm. Ztg., 1928, 73, 93—94).—A small low-tension arc is struck between pure zinc electrodes, and the flame is spread by means of an oscillating magnetic field placed at right angles. A mixture of equal volumes of nitrogen and oxygen, compressed to 1000 mm. of mercury, is passed through the arc. The zinc from the disintegrated electrodes is burnt completely to extremely finely-divided zinc oxide, which is carried away by the gas stream and

absorbed directly in molten vaseline, through which the gases are eventually bubbled. The whole apparatus must be highly polished to prevent deposition of zinc oxide. Photomicrographs showing the extremely finely-divided and uniform nature of the product compared with zinc oxide treated in a mill are given.

S. COFFEY.

Determination of iodide in mixtures of halides. H. BAINES (J.S.C.I., 1927, 46, 381—382 r).—The following method is shown to be capable of detecting 0.00005 mg. of potassium bromide. To the solution to be tested a few drops of an acid fluorescein solution are added, followed by dilute chlorine water, when the presence of bromide is indicated by a pink coloration. An alternative single-solution method is described which can be made roughly quantitative by colorimetric comparison. The same method can be applied to the detection of iodides, but chlorides, when freed from tenaciously held traces of bromide by recrystallisation in a rapid stream of chlorine, neither respond to the test nor interfere with the detection of bromide.

Bromine analysis. KUBIERSCHKY (Z. angew. Chem., 1927, 40, 1511—1513).—The determination of bromine by partial precipitation with silver nitrate, for which the equation is found to be $d \text{ Br}/\text{Br} = 43 \times d \text{ Cl}/\text{Cl}$, is not accurate when much chlorine is present (e.g., in debrominated liquors or rock salt). Distillation with permanganate or chromate and sulphuric acid and absorbing the bromine with zinc dust is inaccurate, since the bromine is neither completely liberated nor completely absorbed. If manganese dioxide (4 pts.) is added to the permanganate (1 pt.) the bromine is completely liberated and may be completely absorbed in a mixture of sodium hydroxide and hydrogen peroxide. The quantity of absorbed bromine is found by neutralising the alkaline solution with hydrochloric acid, evaporating to dryness, and weighing the mixed sodium bromide and chloride. To determine chlorine in bromine the sample is added to a concentrated solution of a known quantity of sodium bromide, the liberated bromine is boiled off, the solution evaporated to dryness, and the residue weighed. Small quantities (up to 2%) of chlorine in bromides are determined by adding to the latter potassium permanganate and 4*N*-sulphuric acid, boiling off the bromine, filtering, neutralising the filtrate, and determining its chlorine content by titration. It is found that, whilst the bromine is removed in this way, all the chlorine is retained.

W. T. K. BRAUNHOLTZ.

Fibrous precipitates. VON WEIMARN.—See V. Compounds of ammonia and carbon dioxide. PAUL and others.—See XIX. Washing of silver bromide. LÜPPO-CRAMER.—See XXI. Ferric salts and sludge activation. MOHLMAN and PALMER. See XXIII.

PATENTS.

Production of synthetic ammonia. K. MÜLLER (B.P. 271,482, 18.5.27. Ger., 19.5.26).—Iron carbide is used as a catalyst at a temperature below that at which it decomposes into iron and carbon, its resistance to heat being increased by the addition of amorphous or colloidal substances (e.g., compounds of the elements of group III of the periodic classification, excepting boron, gallium,

indium, and thallium) or sulphur compounds, and the carbide is converted into a more active form by the addition of 3—5% of cyanogen compounds of the alkali or alkaline-earth metals.

W. G. CAREY.

Purification of gases to be used in the production of ammonia. NORSK HYDRO-ELEKTRISK KVAELSTOF-AKTIESELSKAB (B.P. 266,689, 2.2.27. Nor., 25.2.26).—The mixture of hydrogen and nitrogen is subjected to the action of inorganic compounds dissolved or suspended in liquid anhydrous ammonia, *e.g.*, hygroscopic substances, sodium cyanide, ammonium and calcium nitrates, etc.

H. ROYAL-DAWSON.

Manufacture of ammonium chloride. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,654,125, 27.12.27. Appl., 15.1.25).—Hydrogen chloride and aqueous ammonia are added simultaneously to saturated ammonium chloride solution, the heat of reaction being employed to evaporate the added liquid.

T. S. WHEELER.

Production of anhydrous metal chlorides. I. G. FARBENIND. A.-G. (B.P. 275,945, 23.6.27. Ger., 13.8.26. Addn. to B.P. 259,498; B., 1926, 979).—Metallic oxides or substances containing them (other than magnesia) are mixed with carbonaceous material, *e.g.*, peat, sawdust, etc., and shaped into balls, rods, etc., after which they are dried and coked at below red heat. The porous material so formed, preheated at 100—200°, is then introduced into a furnace and exposed to a current of chlorine.

H. ROYAL-DAWSON.

Preparation of table salt. F. W. HUBER (U.S.P. 1,645,238, 11.10.27. Appl., 20.2.23).—Saturated sodium chloride solution is treated at ordinary temperature with an equal volume of concentrated calcium or magnesium chloride solution, and the precipitated sodium chloride is separated and washed with brine. The moist crystals are then mixed with anhydrous sodium carbonate (2—6%) to form a surface layer of calcium carbonate, after which they can be dried without caking.

T. S. WHEELER.

Purification of alkali cyanide solutions containing sulphur. I. G. FARBENIND. A.-G., Asses. of K. HERRDEGEN (G.P. 444,967, 5.12.25).—Sulphur compounds are precipitated by the addition of bismuth hydroxide or salts.

L. A. COLES.

Manufacture of [partially dehydrated] disodium [hydrogen] phosphate. H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,648,656, 8.11.27. Appl., 14.1.27).—Concentrated phosphoric acid is mixed with sodium carbonate.

T. S. WHEELER.

Production of alkali hydrides. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, H. FREUDENBERG, and H. KLOEPFER (B.P. 283,089, 28.9.27. Addn. to B.P. 276,313; B., 1928, 90).—The finely-divided alkali metal is introduced into the reaction space-with or without diluents.

H. ROYAL-DAWSON.

[Simultaneous] production of chromates [and alumina]. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 273,666, 30.6.27).—Sodium chromate and alumina are produced simultaneously by heating at 900—1000°, in an oxidising atmosphere, a mixture of chrome ore and bauxite with sufficient sodium carbonate to form chrom-

ate, aluminate, and ferrate. The product is lixiviated and a solution having d 1.26 (measured hot) is maintained at 100°, or over if in closed vessels, to separate silicic acid as sodium aluminium silicate. The alumina is precipitated by stirring and introducing carbon dioxide and is separated, after which the sodium chromate solution is evaporated to separate the sodium carbonate and is crystallised or converted into dichromate.

W. G. CAREY.

Treatment of leucite and like minerals. P. SPENCE & SONS, LTD., and T. J. I. CRAIG (B.P. 283,087, 29.6.26).—Minerals containing aluminium and potassium oxides are treated with nitric acid, a neutral or basic solution being obtained by removal of free nitric acid and some of the combined acid by heat or by neutralising with alumina or potash, after which iron is eliminated as basic ferric potassium sulphate or by digesting with manganese dioxide, tin oxide, or antimony oxide. The potassium nitrate is separated by crystallising under regulated conditions of temperature and sp. gr., and is washed with a saturated solution of potassium nitrate. Nitric acid is added to the mother-liquor and the aluminium nitrate separated. The mixed nitrates may be cooled below the temperature at which mixed potassium nitrate and aluminium nitrate crystals are formed, and re-warmed to dissolve the aluminium nitrate crystals.

W. G. CAREY.

Recovery of alumina and phosphates from aluminium phosphates. L. MELLERSH-JACKSON. From RHENANIA KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 283,072, 24.6.27).—Aluminium phosphates are mixed with oxides, carbonates, sulphates, or chlorides of alkaline-earth metals and with alkali salts in the proportion of 1 mol. of P_2O_5 to 2 mols. of alkaline-earth metal oxide and 1 mol. of alkali oxide, and 1 mol. of alumina to 1 mol. of alkali oxide, and are heated at 900° or over, the soluble alkali aluminate being extracted with water and the alumina worked up, whilst the phosphate residue is used as a fertiliser. If sulphates or chlorides are used the process is carried out in the presence of steam produced from fuels rich in hydrogen, *e.g.*, water-gas, oil, etc.

W. G. CAREY.

Preparation of an artificial base-exchange substance. E. M. PARTRIDGE and E. W. SCARRITT, Assrs. to PAIGE & JONES CHEMICAL CO. INC. (U.S.P. 1,656,604, 17.1.28. Appl., 8.12.25).—A wet mixture of sodium silicate and trisodium phosphate is combined with a separately prepared wet mixture of aluminium sulphate and magnesium sulphate to precipitate magnesium phosphate and to form a gel which is dried at a relatively low temperature.

W. G. CAREY.

Manufacture of beryllium and aluminium oxides. C. F. BRUSH, JUN., Assr. to BRUSH LABORATORIES CO. (U.S.P. 1,656,660, 17.1.28. Appl., 14.12.26).—Beryllium minerals containing aluminium are treated so as to convert the beryllium and aluminium into sulphates, which are freed from impurities and heated, producing a mixture of their oxides.

H. ROYAL-DAWSON.

Production of sulphur dioxide and magnesium compounds from magnesium sulphate. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Asses. of F. ROTHE and H. BRENEK (G.P. 445,305, 5.6.25).—Mixtures of

magnesium sulphate with silica or material containing it (1 mol. of SiO_2 per 2 mols. of MgO), or with alumina or ferric oxide, alone or in admixture with one another or with siliceous material, are calcined in the presence of steam, which may be added as such to the flame or formed during combustion by using fuel containing a high proportion of hydrogen.

L. A. COLES.

Production of liquid sulphur dioxide from the products of combustion of sulphur or sulphurous pyrites. P. JODECK, Assr. to ALLGEM. GES. F. CHEM. IND. M.B.H. (U.S.P. 1,649,701, 15.11.27. Appl., 12.7.26. Ger., 13.11.24).—The gaseous products are extracted with those hydrocarbons present in naphtha which are soluble in liquid sulphur dioxide and have b.p. 180—300°, and the sulphur dioxide solution obtained is heated to volatilise the gas.

T. S. WHEELER.

Fixation of atmospheric nitrogen. J. BRIGHTMORE (B.P. 283,232, 6.7.26).—Preheated air and superheated steam at a pressure of 4—5 atm. are subjected in a closed chamber to a direct or alternating electric current (30 amp. at 80 volts) in the presence of dry, finely-powdered, hydrated lime and calspar, a catalyst of copper or zinc plates also being present. After treatment for about 3 hrs. the materials are transferred to another chamber and subjected alternately to currents of hot and cold air, the hot air being at 480°, and also to electric currents of increased strength. The nitrogenous content may be supplemented by passing an air-blast through an electric-arc flame, then through a jet of water, on to the material.

W. G. CAREY.

Activation of carbon and production of carbon disulphide. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 283,267, 5.10.26).—Carbon of suitable grain size is treated at a high temperature (800—1000°) with sulphur vapour or with a compound yielding sulphur vapour by decomposition until the required activation is obtained. Carbon disulphide is simultaneously formed.

H. ROYAL-DAWSON.

Preparation of nitrates and a dry mixture of chlorine and nitrosyl chloride. W. DOMENIK (U.S.P. 1,658,519, 7.2.28. Appl., 13.3.26. Poland, 11.3.25).—See F.P. 611,652; B., 1927, 330.

Production of alkali-metal xanthates. R. B. CROWELL and G. F. BRECKENRIDGE, Assrs. to AMER. SOLVENTS & CHEMICAL CORP. OF CALIFORNIA (Re-issue 16,866, 31.1.28, of U.S.P. 1,559,504, 27.10.25).—See B., 1926, 13.

Chemical heat storage (U.S.P. 1,656,366).—See I. Highly active carbon blocks (B.P. 266,674).—See II. Ammonium sulphate (B.P. 281,474).—See II. Recovery of ammonia (B.P. 282,927).—See XVI. Aluminium salts (G.P. 444,734).—See XX.

VIII.—GLASS; CERAMICS.

Properties of soda-baryta-silica glasses and a comparison with the corresponding soda-lime and soda-lead oxide-silica glasses. S. ENGLISH and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 425—432).—In the series of glasses of the molecular type $6\text{SiO}_2 \cdot x\text{BaO} \cdot (2-x)\text{Na}_2\text{O}$ it was difficult to free the glass from seed when the barium oxide content was as

high as 19.38%, but no difficulty in working occurred until the baryta content was 24.25%. It was possible to incorporate up to 41% of the oxide before a melting temperature of 1400° was exceeded. Annealing temperatures varied practically continuously from 485° for the glass SiO_2 72.86%, BaO 3.03%, Na_2O 23.30%, to 510° for the glass SiO_2 62.67%, BaO 24.25%, Na_2O 11.72%. Linear coefficients of thermal expansion decreased from 1102×10^{-8} for the first member to 884×10^{-8} for the last, whilst density increased continuously from 2.4795 for the first member to 2.8841 for the glass SiO_2 59.97%, BaO 26.15%, Na_2O 11.20%. Curves showing the relationship between the above properties and molecular composition of baryta, lime, and lead oxide glasses are given.

A. COUSEN.

Opal glass. Crystal growth and impact brittleness. J. F. HYSLOP (J. Soc. Glass Tech., 1927, 11, 362—369).—In a fluoride opal glass, high viscosity coincides with globular, low viscosity with crystalline, structure of the fluoride particles. When the glass is heated at temperatures below the critical point of Gehlhoff and Thomas (B., 1927, 877), the particle size, for equal times of heating, is inversely proportional to the viscosity. Impact brittleness is chiefly due to the tendency to form sharp angular crystals in the glass, either of silica from the matrix or, with glasses of low viscosity, of the fluoride. One case of brittleness due to the production of heterogeneous glass has been detected.

A. COUSEN.

Detection of selenium in decolorised bottle glasses. E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1927, 11, 386—392).—The qualitative codeine test for selenium (cf. Schmidt, B., 1914, 748) was developed for use with decolorised bottle glasses. Of substances likely to be present in such glasses only manganese interfered with the test, but even when this element was present the test could be carried out by first precipitating the manganese (after dissolving the glass) by means of bromine.

A. COUSEN.

Homogeneity of glass melted in pots on the commercial scale. (Mrs.) G. A. GREEN and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 407—415).—Glasses from a number of pots from different factories, made without mechanical stirring, and including alkali-lead oxide-silica, alkali-lime-silica, and potash-baryta-lime-boric oxide-silica glasses were proved to be very homogeneous by an analysis of samples from the top, middle, and bottom of the pot in each case.

A. COUSEN.

Manufacture and use of glasshouse pots in Great Britain. W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 415—424).—The tabulated results of a survey of the methods of pot manufacture of 14 firms are given, the details of which include particulars of size and method of making, preparation of clay, and the drying, arching, and record of the life of the pots.

A. COUSEN.

Stourbridge fireclays and the manufacture of glasshouse pots. M. H. EDWARDS (J. Soc. Glass Tech., 1927, 11, 400—405).—An account of the mining and subsequent treatment of Stourbridge clay employed for the manufacture of glasshouse pots.

A. COUSEN.

Measurement of plasticity [of clays]. T. W. TALWALKAR and C. W. PARMELEE (J. Amer. Ceram. Soc., 1927, 10, 670—685).—Methods of measuring plasticity are examined, and objection is raised to the significance given by Bingham and Green (B., 1919, 495 A) to the term plasticity, a more limited conception of this quality of clays being preferred. The stress-strain relations in shear of carefully prepared specimens of clay have been determined by means of a torsion machine. It is shown that for the clays examined the relation of stress to strain in shear is the same as that in compression, and that between certain limits the strain is proportional to the stress; beyond this limiting value, which is not the same as the limit of elasticity, the rate of deformation becomes comparatively great. Addition of water to any clay or of graphite to a non-plastic clay increases the rate of deformation beyond the proportional limit. It is suggested that the plasticity index for any clay-water mixture may be expressed as the ratio of total deformation at the point of failure to the average stress beyond the proportional limit. A. T. GREEN.

Quantitative microscopic analysis of commercial felspar. H. INSLEY (J. Amer. Ceram. Soc., 1927, 10, 651—669).—A method for the rapid determination of the constituents of ground commercial felspar is based on the separation of the mineral constituents according to their indices of refraction and the determination of their volume percentages. This method, which gives close agreement with the data calculated from chemical analyses, involves the use of a liquid of index of refraction 1.540 for distinguishing quartz from felspar, and a liquid of index 1.528 for distinguishing potash felspar from plagioclase. Another method in which a partially fused sample is examined microscopically for the quartz content is described. The results of microscopical analysis of nineteen samples of felspar are tabulated. Although chemical analysis is the more accurate, the microscopical methods are valuable in works control, when rapid determinations are necessary. A. T. GREEN.

Refractory material for vertical retorts. STEINHOFF (Gas World, 1928, 88, 72—73).—See B., 1927, 937.

PATENTS.

Continuous kilns for burning [ceramic products] and like purposes. A. E. OSMAN (B.P. 283,009, 19.2.27).—The kiln chambers have upcast shafts at each end, through which the combustion gases from furnaces on either side thereof pass into the upper part of each chamber. The gases from a preceding chamber also pass into one of the shafts, mixing with the combustion gases therein. The hot gases pass down through the chamber into flues running below the centre of the floor, or through perforations in the floor at each side into flues running along each side of the chamber. A suitable arrangement of flues permits the passage of gases or heated air from a chamber being cooled down to another being heated up. A. B. MANNING.

Extraction of clay and other argillaceous raw materials with acids. I. G. FARBENIND. A.-G. (B.P. 278,370, 29.9.27. Ger., 29.9.26).—The acid used for the extraction percolates through a large quantity (100 tons) of the raw material to be treated, and main-

tains a certain liquid level above the charge, the passage of acid used being so regulated by its inflow and outflow that the heat of reaction causes a temperature of at least 105° to be attained in the downwardly travelling zone of maximum reaction in the charge.

H. ROYAL-DAWSON.

Cementing of porcelain. Cementing of porcelain and metal. Cementing of porcelain, and porcelain and iron. STUDIEN-GES. F. WIRTS. U. IND. M.B.H. (B.P. 260,592, and Addn. B.P. 260,593 and 274,408, [A—C] 26.10.26. Ger., [A, B] 27.10.25, [C] 17.7.26).—In (A) a rubber mass containing vulcanising accelerators is used. The whole of the rubber is combined with sulphur and contains filling or other materials to increase its tensile strength. (B) A similar cement is used, but the metal parts are first coated with hard rubber. (C) Sulphur is replaced by selenium or its compounds. A suitable cement for all the purposes enumerated consists of 90% of pure Para rubber, 2% of litharge, 2% of selenium oxide, 3—4% of vulcanising accelerators, e.g., piperidine, and 2—3% of zinc oxide or finely-ground soot. W. G. CAREY.

Manufacture of refractories. M. J. RENTSCHLER, Assr. to W. R. and A. N. JEAVONS (U.S.P. 1,653,123, 20.12.27. Appl., 13.12.20).—A mixture of clay, carbon, firebrick, and water is formed, dried, and coated with a composition containing fireclay, calcium carbonate, sand, sodium silicate, felspar, and water, so that on firing a glaze is formed which protects the carbon body in an oxidising atmosphere. T. S. WHEELER.

Production of a carbon-bonded refractory. E. L. HAUMAN, Assr. to ELECTRO REFRACTORIES CORP. (U.S.P. 1,653,850, 27.12.27. Appl., 28.4.26).—A crucible is formed of graphite, silicon carbide, a salt, and coal tar, and is lined with a mixture of bonding clay, magnesite, and coal tar. The outer portion is then impregnated with borax solution so that when the crucible is heated at 900° in an oxidising atmosphere the carbon is eliminated only from the lining, which is thus rendered resistant to the action of molten steel. T. S. WHEELER.

Manufacture of an abrasive article. D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,655,396, 3.1.28. Appl., 14.4.26).—An intimate mixture is made of the abrasive grains, rubber, and sulphur, together with not more than 30% of a synthetic resin, e.g., bakelite. The mixture is then shaped and vulcanised. F. R. ENNOS.

Production of [cellular] heat-insulating material. A. U. WESTFELT (B.P. 283,055, 28.5.27).—In the production of firebricks, water-glass is mixed with potassium chlorate and, if desired, a powdered silicate, and heated. H. ROYAL-DAWSON.

IX.—BUILDING MATERIALS.

Retarding the setting time of cement by the addition of small quantities of lead monoxide. B. GARRE (Zement, 1927, 16, 469; Chem. Zentr., 1927, II, 726).—The setting time is retarded considerably by the addition of 0.001% of lead monoxide, further additions retarding it still more until with about 1% hardening is incomplete. L. A. COLES.

Suggested standard method of slaking in testing building limes. A. D. COWPER and J. F. WILLIAMS (J.S.C.I., 1928, 47, 31—35 r).—A standard isothermal method for slaking building limes for the purposes of formal tests is proposed, using a large excess of water and rapid stirring, the quicklime being crushed to $\frac{1}{2}$ in. size. Tests at 50° and at 100° are made, and the product of higher volume-yield is chosen for further tests; or a temperature designated by the manufacturer may be used. Results are given for a number of British limes, with analyses, plasticity figures, and comparative results for dry and uncontrolled slaking.

Wood preservation. VI. L. P. CURTIN and W. THORDARSON (Ind. Eng. Chem., 1928, 20, 28—30); cf. B., 1927, 750, 909, 938; 1928, 93).—Further species of wood-rotting fungi have been grown in nutrient media containing sodium alizarinsulphonate and methyl-orange. In all cases sufficient acid was formed to affect the former indicator, and in some cases, notably with *Merulius lachrymans*, a fungus which causes large losses of timber by "dry rot," the culture became acid to methyl-orange. Tables are given showing the "killing points" (minimum concentration that completely inhibits the growth of a test fungus, viz., *Fomes annosus*) of a number of inorganic preservatives, also the toxicity to various fungi of powdered zinc meta-arsenite ("killing point" 0.10) at concentrations up to 0.3%. From the practical point of view zinc meta-arsenite is the most promising of the preservatives studied; it does not increase the electrical conductivity of wood, has no corrosive action on iron, and may be used in standard pressure-treating equipment. Moreover, field tests have shown that wood containing zinc meta-arsenite suffers little loss of preservative during prolonged exposure to the weather.

D. J. NORMAN.

PATENTS.

Manufacture of Portland cement in a rotary kiln. G. MARTIN (B.P. 282,868, 27.9.26).—The output of a rotary kiln is increased by varying the rate of travel of the gases through the zones into which a kiln is naturally divided, the speeds being in some zones substantially greater, in some less, and in some the same as those prevailing in practice. Thus the gases are caused to traverse internal baffles, so that the average linear speed is greater than 22 ft./sec. in the drying zone, and greater than 30 ft./sec. in the clinkering and decarbonating zones.

W. G. CAREY.

Calcining of cement or lime. E. MEIER (G.P. 445,554, 7.9.24).—The finely-divided material falls by gravity down a shaft kiln which is bellied at the combustion zone and at a zone below it from which the waste gases are withdrawn. The combustion gases enter the kiln, and the waste gases leave it in a direction at right angles to that of the falling particles, the waste gases being free from dust.

L. A. COLES.

Grinding of cement and similar materials. C. PONTOPPIDAN (B.P. 283,091, 8.10.27).—In order to absorb the heat produced by grinding and to maintain the temperature below that at which the cement becomes inclined to adhere to the grinding surfaces, a liquid which will evaporate and so abstract heat is added during the final stages through the discharge end of the grinding

machine by means of a pipe directed axially and terminating in an atomiser. The liquid may be supplied through holes in the casing by scoop or blade wheels, and material such as calcium chloride, calcium sulphate, or tannin may be suspended or dissolved in the liquid.

W. G. CAREY.

Preparation of emulsions or suspensions [for roads etc.]. G. BAUME, P. CHAMBIGE, and D. BOUTIER (B.P. 255,074, 7.7.26. Fr., 9.7.25).—The emulsion is prepared by adding to the basic mixture consisting of bitumens, tars, pitch, tar oil, etc., a stabiliser, e.g., stearine pitch, vegetable or mineral oils, a colloid protector, e.g., gelatin, glues, gums, casein, or soaps, and to impart elastic properties tartrates, esters of glycerin or aromatic alcohols. The emulsifying agent is added with a small proportion of water to the basic mixture, and finally the rest of the water is added. These emulsions have a narrow critical temperature range of 5—10°.

A. C. MONKHOUSE.

[Press table for] the manufacture of bricks. SCHEIDHAUER & GIESSING A.-G., and H. ACKERMANN (B.P. 282,711, 23.9.27. Ger., 31.12.26. Addn. to B.P. 267,948).

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

Behaviour of steel under prolonged stress at high temperatures. A. POMP (Chem. Fabr., 1928, 53—54).—Recent work on the "creeping" of steel at high temperatures is reviewed and the necessity of determining the "permanent strength" of steels which are to be used for high-pressure work at elevated temperatures is emphasised. The "permanent strength" of a steel is defined as the highest load which the steel will support at any given temperature without increasing in length after 3—6 hrs. by more than 0.001%/hr. For plain carbon steels this value remains fairly constant up to 300°, then falls very rapidly with rise in temperature; the permanent strength and the rate at which it decreases with rise of temperature increase with the carbon content of the steel up to the eutectoid point, provided that the pearlite constituent is in the lamellar form. Steels in which the pearlite is in a granular form have a much lower permanent strength.

A. R. POWELL.

Hardening by compression and the brittleness of steel. Existence of a limit of brittleness. P. DE-JEAN (Rev. Mét., 1927, 24, 415—417).—When steel is subjected to compression, so long as the load does not exceed a certain critical value, the resilience remains constant irrespective of any previous compression below the critical value to which the metal has been subjected. When the load exceeds this value the resilience falls sharply and the metal exhibits a slight quiver indicating that it has passed from the resilient to the brittle state. The load required to produce the brittle state in a mild steel is greatest after annealing at 900°, quenching in cold water, annealing at 700°, and cooling slowly; forging reduces the critical load, and prolonged annealing at 950° followed by slow cooling in air reduces it to about half that of the metal annealed at 900°.

A. R. POWELL.

Cast iron resistant to acids and alkalis. H. G. HAASE (Stahl u. Eisen, 1927, 47, 2112—2117).—The most satisfactory resistance to attack by mineral acids is shown by cast iron of the following composition: 3.3—3.5% C, 1.3—1.4% Si, about 0.75% Mn, 0.3—0.5% Ni, not more than 0.2—0.3% P, and not more than 0.07% S. Increase of sulphur is particularly deleterious, more manganese increases the rate of corrosion relatively slightly, with silicon maxima occur in the corrosion curve at 2.5 and 7% Si, and with phosphorus and nickel little difference is noticeable in the rate of corrosion with double the above amounts. The cast iron most resistant to alkali contains 3.3—3.5% C, 1.2—1.4% Si, 0.3—0.4% Mn, not more than 0.3% P, not more than 0.08% S, and 0.3—0.5% Ni. Increase of phosphorus reduces the resistance to attack by alkali hydroxides, as also does increase of silicon to a maximum of 7% Si. Metal with the casting skin intact is more resistant than metal from which this skin has been removed.

A. R. POWELL.

[Determination of] phosphorus and silicon in ferrophosphorus. L. E. PITZER (Chemist-Analyst, 1926, No. 47, 8).—The sample (0.25 g.) is heated with 60% perchloric acid (35 c.c.), cooled, made slightly ammoniacal, treated with a slight excess of concentrated nitric acid, and diluted to 75 c.c. The phosphorus is determined with molybdate. For the determination of silicon, treatment with perchloric acid is advised.

CHEMICAL ABSTRACTS.

Analysis of chrome iron ore. E. DITTLER (Z. angew. Chem., 1928, 41, 132—133).—Fusion of this ore with sodium carbonate is never complete, and the analysis is best carried out in the following manner. The finely-ground mineral is fused with ten times its weight of sodium peroxide in a silver crucible, the melt dissolved in water, acidified with hydrochloric acid, and the precipitated silver and silver chloride removed. The filtrate, to which a few c.c. of hydrogen peroxide have been added to reduce the chromium present, is evaporated to dryness and the silicic acid removed by evaporation with hydrofluoric acid; the residue is decomposed with sodium carbonate and a little potassium nitrate, and, after treatment with hydrogen peroxide, the combined oxides of aluminium, iron, and chromium are precipitated with ammonia solution. Provided that excess of hydrogen peroxide is removed before precipitation, all the chromium is obtained as oxide. The three oxides are treated with sodium peroxide, when the iron remains as insoluble residue and the chromate is determined volumetrically in the filtrate after conversion of the manganese present into manganese dioxide, whilst the aluminium content is obtained by difference. In the filtrate from the three oxides, calcium, magnesium, and manganese are determined by the ordinary methods.

W. J. POWELL.

Concentration of ore from the Read-Rosebery mines of Electrolytic Zinc Co. of Australasia, Ltd. E. H. FRASER (Proc. Austral. Inst. Min. Met., 1927, [66], 179—186).—The ore, consisting of blende, galena, and pyrites in a schistose gangue, is ground in a 0.5% solution of sodium carbonate in rolls and Hardinge or tube mills so that 90% passes 200-mesh. The mill discharge

passes to classifiers and the overflow to thickeners, thence to the lead flotation plant. The pulp ratio in this plant is kept at 1:1, the solution containing 0.35% Na_2CO_3 ; the lead sulphide is floated with 0.3 lb./ton of coal-tar distillate in a series of nine sub-aeration cells, and the concentrate cleaned in a series of eight similar cells. The final dried product contains 46% Pb, 19% Zn, 48.5 oz./ton of silver, and 6 dwt./ton of gold. The tailings from the lead plant pass to the zinc plant, the pulp density having been reduced to 1:3 and the alkalinity to 0.05—0.1% Na_2CO_3 ; zinc sulphide is floated with 0.75 lb./ton of coal-tar distillate and 1.5 lb./ton of copper sulphate, the pulp being maintained at 50° by steam injection. The zinc concentrate contains 53% Zn, 6% Pb, 7.4 oz./ton of silver, and 3 dwt./ton of gold. The recovery of zinc is 86% and of lead 60%, whilst 92% of the silver and 70% of the gold are recovered in both concentrates; the total soda consumption is 11 lb./ton of ore.

A. R. POWELL.

Drying and roasting of zinc [sulphide] concentrate at Zeehan Works of Electrolytic Zinc Co. of Australasia, Ltd. E. H. FRASER (Proc. Austral. Inst. Min. Met., 1927, [66], 173—177).—The fine flotation concentrate obtained as described in the preceding abstract is fed to a dryer 45 ft. long and 14 ft. wide, fired with wood fuel and mechanically rumbled. The discharge from the furnace contains 2—3% of moisture, and is fed directly by means of conveyors and elevators to four 7-hearth Herreshoff furnaces, each hearth of which is rumbled mechanically with a cast chromium-steel (27% Cr) rabble provided with eight teeth and rotated 52 times/hr. The furnace is fed with preheated air in such a way that the third hearth is kept at 850°, whilst the seventh is at 600°, no fuel being used. The discharge from the furnace, after cooling somewhat, is damped down and sent to the dissolving plant for the production of electrolytic zinc; it contains 58% Zn, 5.4% Pb, 2.6% S as sulphate, and 4.8% S as sulphides. About 20% of the lead input to the furnace is recovered in fume, and the flue gases contain 2—2.5% SO_2 .

A. R. POWELL.

Theory of the age-hardening process in structural and montegal [aluminium alloys]. W. GUERTLER (Z. Metallk., 1927, 19, 488—491).—The constituent in structural, which renders the alloy amenable to age-hardening, is the compound MgZn_2 , which is not decomposed by aluminium but forms with it a eutectic, m.p. 480°, and a limited series of solid solutions containing up to 28% MgZn_2 at 480° and 4—5% at 20°. For practical purposes the alloy should contain 5—12% MgZn_2 , and the magnesium and zinc content should be so arranged that neither is in excess of the amount required to form this compound. In order to obtain a highly-dispersed separation of the compound during ageing, a temperature above the ordinary is required, the exact temperature depending on the amount of MgZn_2 present. Correct adjustment of composition and heat treatment gives an alloy having a higher tensile strength and ductility than duralumin. Montegal contains calcium silicide as the hardening constituent, and after heat treatment has a tensile strength almost equal to that of duralumin but is

much more ductile, can be rolled into very fine foil, and has a high bending strength and Erichsen value.

A. R. POWELL.

Tensile strength and electrical conductivity of recrystallised aluminium wire, and its practical application in rolling the metal. H. BOHNER (*Z. Metallk.*, 1928, 20, 8—13).—The tensile strength of aluminium wire falls rapidly and the electrical conductivity increases appreciably after annealing for 1 min. at 300° or above. Between 300° and 400° the tensile strength decreases slowly and the conductivity increases slightly over a period of 5 min.; at higher temperatures there is practically no further change after the first minute. With aluminium containing 98—99% Al prolonged annealing at 575° has little effect on the strength or conductivity, whereas with 99.4—99.7% Al the tensile strength decreases with time of annealing at 575° owing to the development of a very coarsely crystalline structure. When soft 98—98.5% Al is annealed at 500° the tensile strength and conductivity remain constant for 45 min., after which the former suddenly increases slightly and the latter decreases. In all the specimens tested the tensile strength increased with the temperature of recrystallisation provided that coarse crystals did not develop; the electrical conductivity showed a corresponding decrease. Under the same conditions of annealing the tensile strength is lower and the conductivity higher the higher the purity of the metal. For the preparation of aluminium wire for electrical conductors the most satisfactory annealing temperature is 350°. The optimum conditions for casting and rolling the metal are briefly outlined.

A. R. POWELL.

Experimental determination of the curve of critical dispersion of the alloy "lantal." K. L. MEISSNER (*Z. Metallk.*, 1928, 20, 16—18).—Specimens of lantal (4% Cu, 2% Si, and 94% Al) were annealed at 510—515° for 20—30 min., quenched in water, and aged for prolonged periods at temperatures between 125° and 220°, the hardness and bending strength being determined periodically during the ageing. At 125—160° the hardness increases from 90 to a more or less constant value of 130 in 12—1 days according to the temperature, at 175° maximum hardness is reached in 12 hrs., and further ageing results in a steady softening of the metal; at 200° the maximum hardness is only 110 and is reached in 5 hrs., and at 220° it is 100, reached in 3 hrs. The bending strength falls slowly to a minimum in 12 hrs. at 175°, then increases again almost to its original value after 24 hrs. The curve of critical dispersion is obtained by plotting the ageing temperature against the time required to reach maximum hardness; it is a hyperbolic curve which falls steeply between 220° and 180°, and very slowly between 150° and 130°. The results obtained in the investigation indicate that the most satisfactory ageing temperature for lantal is 160°, 30 hrs. being required for maximum hardness. Rapid coagulation of the copper constituent occurs above the critical ageing temperature, but at lower temperatures prolonged annealing has little deleterious effect on the properties of the metal.

A. R. POWELL.

Aluminium bronze. J. BOULDOIRES (*Rev. Mét.*,

1927, 24, 357—376, 463—473).—Sand-cast aluminium bronzes containing 80—95% Cu were annealed at 850° for 5 hrs., cooled to 20° during 15 hrs., and heating and cooling curves obtained for the following properties: electrical resistance, thermo-electric power, density, hardness, and expansion. In addition, the alloys were examined by thermal and micrographic analysis. In the case of alloys consisting entirely of α -solid solution, an anomaly was observed in the heating curves at 290—310°, but not in the cooling curves. Alloys consisting of α + eutectoid showed a transformation at 510—520° both on heating and cooling, and those consisting of γ + eutectoid one at 515° on heating and at 493° on cooling; both types of alloys in addition showed the eutectoid transformations at 565—570° and at 690—780°. The change of direction of the various curves for the low-temperature transformations is more marked the higher the aluminium content of the alloys and the more slowly the metal has been cooled after annealing. After rapid cooling or quenching many of these transformations are suppressed, and no deviations occur in subsequent heating and cooling curves. The thermal effect at 300° is not accompanied by any change in the structure or physical properties of the alloy, but that at 520° is accompanied by changes in the density, electrical resistance, and other physical properties, although no structural change can be detected. Quenching from above the eutectoid point (560°) decreases the resistance and hardness of most aluminium bronzes.

A. R. POWELL.

Determination of lead in manganese bronze. F. B. DIANA (*Chemist-Analyst*, 1927, No. 47, 6—7).—The alloy is treated with ferric nitrate solution, then with nitric acid, and, after boiling to remove excess of acid and diluting, the lead is determined electrolytically as dioxide.

CHEMICAL ABSTRACTS.

Analysis of lead-base bearing metal. B. PAXTON (*Chemist-Analyst*, 1926, No. 47, 3).—The sample (1 g.) is decomposed with sulphuric acid (15 c.c.) and potassium sulphate (2 g.), and the cool liquid is diluted with water (100 c.c.) and boiled for 12 min. with hydrochloric acid (10 c.c., concentrated). After rapid cooling, water (200 c.c.) is added, and the antimony titrated with 0.1N-permanganate solution. Lead sulphate is collected from the cold titrated solution, washed, dissolved in ammonium acetate solution, and titrated with molybdate. To the filtrate from the lead sulphate are added concentrated hydrochloric acid (25 c.c.), iron nails, and sufficient of a solution (made by dissolving 1 g. of antimony in 20 c.c. of concentrated sulphuric acid, diluting to 100 c.c. with dilute hydrochloric acid, and adding enough hydrochloric acid to dissolve oxychloride) to dissolve up to 0.15 g. of antimony that may have been precipitated by the iron. The mixture is boiled for 25 min., cooled in carbon dioxide, filtered, and the tin titrated with 0.1N-iodine solution.

CHEMICAL ABSTRACTS.

Intercrystalline brittleness of lead. O. HAEHNEL (*Z. Metallk.*, 1927, 19, 492—496).—Lead cable and water-pipes become rapidly brittle when subjected to vibration owing to loosening of the intercrystalline bond. The rate at which this occurs with pure lead increases with

the rate of vibration and with the temperature. Exposure to temperatures between 40° and 100° results in a slow development of brittleness due to crystal growth; higher temperatures rapidly cause a breakdown of the adhesion between the crystals. Compression, tension, or torsion alone does not cause brittleness, but in conjunction with vibration stresses they increase the rate of breakdown. The resistance to intercrystalline fracture is increased 7–10 times by the addition of 3% Sn or 1% Sb to the lead. A. R. POWELL.

Analysis of acid-resisting bronze, antimonial lead, and babbitt metal. I. R. F. SMITH (Pulp and Paper Mag., 1928, 26, 105–106).—The bronze (1 g.) is dissolved in 30 c.c. of 1:1 nitric acid, the solution evaporated nearly to dryness, diluted to 100 c.c. with hot water, and filtered, and the precipitate of meta-stannic acid is ignited and weighed. The filtrate is neutralised with ammonia solution, treated with 3 c.c. of nitric acid, diluted to 200 c.c., and electrolysed for 1½–2 hrs. with 4–6 volts and 3–5 amp. using a rotating, cylindrical, platinum gauze cathode inside a similar but larger anode. Copper is deposited on the cathode and lead peroxide on the anode. Iron, manganese, and aluminium are removed from the spent electrolyte by addition of ammonia solution, ammonium chloride, and bromine, and the zinc in the filtrate is precipitated as zinc ammonium phosphate and subsequently weighed as the pyrophosphate. An alternative method involving precipitation of the lead as sulphate and of the copper as sulphide by means of thiosulphate, followed by volumetric determination by the iodide method, is described. A. R. POWELL.

Detection of baser admixtures in gold plates. G. TAMMANN and O. LOEBICH (Z. anorg. Chem., 1927, 168, 255–263).—The presence of 0.1% of iron, tin, or antimony in a gold plate can be detected by observing the colour of the surface film produced by heating the plate at 600°. At this concentration the detection of aluminium and tellurium is, however, somewhat uncertain, but by observation of the poisonous action of the plate on a culture of *Bacterium gossypii* or *B. Brassicae* in contact with it, 0.1% of antimony or tellurium will be revealed. If, however, the impurity forms mixed crystals with gold, the bacterial method is, in general, useless. Iron, tin, and antimony cause the appearance of spots when the gold is subjected to the action of dry iodine vapour, but spots are also frequently due to foreign bodies embedded in the surface in such processes as rolling and polishing. The precipitation of radium fluoride from acid solution by a gold sheet is not very satisfactory as a means of detecting foreign metals, as gold itself adsorbs the fluoride to a slight extent, and, further, many metals oxidise before precipitation can occur. R. CUTHILL.

PATENTS.

Heating of smelting furnaces. C. BRACKELSBERG (B.P. 283,381, 13.4.27. Ger., 9.3.27).—Combustion of pulverised fuel is caused to take place directly in a rotary smelting furnace, without a preliminary combustion chamber. A relatively high pressure (400 mm.) of air is used to effect rapid combustion and to carry ash through and out of the furnace, the air at high

pressure being preferably supplied as an envelope to the air-fuel mixture as it leaves the burner nozzle at normal pressure. C. A. KING.

Apparatus for the concentration of ores and other minerals and materials. J. A. CHAMBERS (U.S.P. 1,656,042, 10.1.28. Appl., 9.11.25. S. Africa, 9.9.25).—A pan for wet concentration is provided with means for introducing the ores, an outlet for the lighter constituents and the liquid, and means for withdrawing the concentrates from its lower end. The pan is carried by a plate supported by balls upon a base plate, and is moved in a circular path by mechanism engaging its centre directly. Rotation of the pan about its own axis is prevented by an arm pivoted at a distance and having a sliding connexion with one side of the pan. H. HOLMES.

Ore-grinding machine. C. E. PAYNE (U.S.P. 1,656,756, 17.1.28. Appl., 5.5.27).—A rotary cone is mounted within a stepped casing forming a series of superposed grinding chambers of increasing diameter from top to bottom. The cone carries annular flanges, one projecting into each chamber, and wear blocks are fixed to radial ribs projecting upwards from the flanges. H. HOLMES.

Apparatus for the recovery of minerals from ore. W. C. TRACY (U.S.P. 1,657,097, 24.1.28. Appl., 3.8.25).—On the bottom of a tank is mounted a flat horizontal coil provided with a series of oppositely diverging perforations, and around the inner periphery of the tank is a perforated helical coil for emitting air currents transversely across the tank. Means are provided for supplying air under pressure to the coils. M. E. NOTTAGE.

Smelting of iron in foundry shaft furnaces. F. W. CORSALLI (B.P. 254,727, 30.6.26. Ger., 30.6.25).—For the production of cast iron containing less than 3% C, the air-blast is increased beyond the pressure necessary to produce grey iron, and refining materials, e.g., silicon, are protected in a sheath until the smelting zone is reached. It is preferred to charge only metal into the main shaft, and fuel into subsidiary side shafts communicating at the bottom with the main shaft. C. A. KING.

Manufacture of malleable iron castings. H. A. SCHWARTZ, AssT. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,656,535, 17.1.28. Appl., 23.3.26).—Molten iron is introduced into an acid-lined electric furnace, and, after casting, the metal is subjected to a shortened annealing process. A desulphurising agent is added at a stage prior to casting to nullify the effects of sulphur. H. ROYAL-DAWSON.

Manufacture of iron and steel or iron alloys and steel alloys. F. KRUPP A.-G. (B.P. 268,716, 2.9.26. Ger., 3.4.26).—During manufacture, iron or one of its alloys is deoxidised by means of suitable reagents to an oxygen content of less than 0.005%, which is determined by the absence of any substantial decrease of "tenacity of the notched bar" on subsequent ageing of the metal. Up to 0.5% of nickel, cobalt, chromium, tungsten, vanadium, or molybdenum may be added. C. A. KING.

Pickling of chromium-iron alloys. I. SCHULTE, Assr. to ALLEGHENY STEEL Co. (U.S.P. 1,657,228, 24.1.28. Appl., 2.6.26).—The scale is activated in a solution containing hydrofluoric acid and the salt of a metal positive to iron and chromium, and is then dissolved in a solution containing nitric acid.

M. E. NOTTAGE.

Corrosion-resistant [copper-manganese] alloys. M. G. CORSON, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,657,957, 31.1.28. Appl., 13.4.26).—Alloys resistant to attack by inorganic acids are made by alloying copper with 35–55% of manganese.

F. G. CROSSE.

Flux applied in soldering aluminium and its alloys. O. SPENGLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,482, 20.12.27. Appl., 24.12.23. Ger., 23.6.22).—A mixture of sodium fluoride with lithium, calcium, and zinc chlorides is claimed. T. S. WHEELER.

Manufacture of [dumet] bimetallic wire. B. F. STEEL, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,653,378, 20.12.27. Appl., 1.7.22).—The process of manufacture of the wire is modified in that it is heated rapidly during the first annealing to expand any gas trapped between the core and sheath and thus render visible a defect of this kind.

T. S. WHEELER.

Sintered hard-metal alloys and articles made thereof. F. KRUPP A.-G. (B.P. 279,376, 18.3.27. Ger., 25.10.26).—Material for the production of tools etc. consists of a mixture of the carbides of a hard metal, e.g., tungsten, and of at least one softer metal, e.g., iron, the grains being of such size (less than 1×10^{-4} mm.) that the fracture of the tool reveals no pores under a magnification of 50 diameters. Such comminuted raw material is produced by separately grinding tungsten and iron in a ball mill, carburising the tungsten, and regrinding for at least 50 hrs., afterwards mixing, pressing, and sintering the components. C. A. KING.

Treatment of titanium ores. I. G. FARBENIND. A.-G. (B.P. 267,547, 11.3.27. Ger., 11.3.26).—Finely-ground ilmenite is heated at 180° under pressure with 20–40% sulphuric acid containing sufficient titanous sulphate to reduce the ferric iron in the ilmenite to the ferrous condition. Titanium dioxide is obtained from the resulting solution by hydrolysis without further dilution. The titanous sulphate solution is made by reducing a strongly acid solution containing quadrivalent titanium with the product obtained by heating ilmenite with coal or reducing gases at high temperatures.

A. R. POWELL.

Alloy having a high electrical resistance. Y. KAMISHIMA (B.P. 283,354, 9.2.27).—A non-oxidisable alloy with good magnetic properties contains 1–5% Ni, 7–20% Cr, and 1–6% Si, the remainder being iron and impurities, but free from manganese and having a very low carbon content.

F. G. CROSSE.

Alloy. E. KIAM, Assr. to M. G. COLLINS (U.S.P. 1,653,630, 27.12.27. Appl., 25.2.22. Renewed 19.5.27).—The alloy consists of 30–65% Ni, 15–40% W, 15–30% Cr, 1–8% Mn, and 5–0.5% Mg.

F. G. CROSSE.

Composition of matter [alloy]. T. DE LA CONCHA, Assr. to P. A. HUGHES (U.S.P. 1,653,088, 20.12.27.

Appl., 27.10.26).—The alloy is composed of 30% Pb, 50% Sn, 12% Zn, 5% Al, and 3% of German silver.

F. G. CROSSE.

Production of rare metals and their alloys. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,648,954, 15.11.27. Appl., 29.9.21).—An alloy of a rare metal, e.g., thorium, uranium, or cerium, with aluminium is more stable in air than the pure metal, and can readily be prepared by the thermit process or by treating with aluminium a solution of the double chloride of the rare metal and ammonium, e.g., ammonium thorium chloride, in fused potassium and sodium chlorides. The metal is recovered from the alloy by volatilisation of the aluminium.

T. S. WHEELER.

Production by electrolysis of thin superposed nickel sheets and separation of them one from another. E. BREUNING and O. SCHNEIDER (B.P. 275,221, 22.7.27. Ger., 27.7.26).—A layer of nickel is deposited electrolytically and its surface polarised (e.g., by immersion of the rinsed film in concentrated nitric acid or in a solution of chromic acid, or electrically by means of a 5% sodium hydroxide bath), the operations being repeated for each superposed layer; the layers are then separated by means of weak acid.

H. ROYAL-DAWSON.

Electrodeposition of metals [plating on aluminium and its alloys]. H. WEBB and S. O'BRIEN & PARTNERS, LTD. (B.P. 283,288, 9.10.26).—The metal (aluminium) is cleaned by immersion in sodium hydroxide solution, pickled in concentrated nitric acid, and plated with copper from a copper nitrate bath; alternatively it may be pickled in 5–10% sulphuric acid and plated directly with copper, nickel, or zinc from a slightly acid sulphate solution. In either case the plated article is dried in warm sawdust and embedded in silver sand heated at 180° for a period which varies with the thickness of the metal. After heat-treatment the articles may be plated again with the same or a different metal, e.g., copper-plated aluminium may be coated with silver or gold by plating in the usual cyanide baths.

A. R. POWELL.

Treatment of ores. D. C. HARE, Assr. to INTERNAT. REDUCTION Co. (U.S.P. 1,657,622, 31.1.28. Appl., 26.5.24).—See B.P. 234,524; B., 1925, 676.

Apparatus for treatment of steel ingots. J. V. ELLIS (U.S.P. 1,657,350, 24.1.28. Appl., 12.4.26. U.K., 18.3.26).—See B.P. 268,123; B., 1928, 416.

High-percentage magnesium alloys. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (B.P. 254,741, 2.7.26. Ger., 3.7.25).—See F.P. 618,425; B., 1927, 881.

[Aluminium-silicon] alloys. A. G. C. GWYER and H. W. L. PHILLIPS, Assr. to BRIT. ALUMINIUM Co., LTD. (U.S.P. 1,657,389, 24.1.28. Appl., 22.1.24. U.K., 22.1.23).—See B.P. 219,346; B., 1924, 793.

Magnetic alloy and its application in the manufacture of telegraphic and telephonic cables. W. S. SMITH and H. J. GARNETT (U.S.P. 1,656,959, 24.1.28. Appl., 8.1.26. U.K., 20.11.25).—See B.P. 266,066; B., 1927, 303.

Cementing of porcelain and metal (B.P. 260,592—3 and 274,408).—See VIII.

Rubber coatings on metal (B.P. 283,049).—See XIV.

XI.—ELECTROTECHNICS.

Zinc oxide for ointments. DÖRZBACH.—See VII.

Gelatin. OKA.—See XV.

PATENTS.

[Recording] photometric apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. A. LONG (B.P. 275,597, 22.7.27. U.S., 9.8.26).—A recording photometer comprises a reflector which can be rotated about a lighting unit so as to reflect light upon a photoelectric cell, an oscillograph and a synchronised photographic recorder rotating across the beam of light reflected by the oscillograph mirror. J. S. G. THOMAS.

Electric heating of travelling material. AJAX ELECTROTHERMIC CORP., Assees. of E. F. NORTHRUP (B.P. 279,779, 20.4.27. U.S., 28.10.26).—Alternating current is passed through the travelling material from parallel-connected contacts, substantially equal inductances being arranged in series with the individual contacts in order to distribute the current uniformly. J. S. G. THOMAS.

Electric accumulators. G. FROMONT (B.P. 268,828, 2.4.27. Fr., 2.4.26. Addn. to B.P. 250,985; B., 1927, 914).—The electrolyte consists of a solution of sulphuric acid and sodium or potassium sulphate.

H. ROYAL-DAWSON.

Electrolytic rectifier. LE R. P. BENSING, Assr. to G. P. KOELLIKER (U.S.P. 1,645,085, 11.10.27. Appl., 31.8.25).—An electrolytic rectifier comprises a lead anode, a magnesium or silicon cathode, and an aqueous electrolyte containing 20—25% of potassium fluoride and 1—2% of sodium hydroxide. T. S. WHEELER.

Electrolytic [rectifying] cell. E. ROMANELLI, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,648,963, 15.11.27. Appl., 30.7.26).—The cell comprises a lead anode, a filming cathode of zirconium, and an electrolyte of sulphuric acid, *d* 1.25. T. S. WHEELER.

Impregnation of porous electrodes for primary and secondary cells. K. OETTL (G.P. 444,181, 18.9.25).—High-conductivity electrodes are produced by sucking the impregnating medium into the electrodes by evacuating the air. J. S. G. THOMAS.

Carbon electrode. COMP. LORRAINE DE CHARBONS, LAMPES, ET APPAIRAILLAGES ELECTRIQUES (Addn. No. 31,586, 27.10.25, to F.P. 594,697; B., 1926, 284).—Catalysts used in accordance with the prior patent are employed either in acid or acid electrolytes, but not in both. For use in alkaline electrolytes, salts of cerium and manganese, borates, resinates, oleates, etc. may be employed as catalysts, whilst in acid electrolytes, compounds of copper, iron, etc. may be similarly used.

J. S. G. THOMAS.

Manufacture of preformed positive electrode. R. C. BENNER and J. H. FINK, Assrs. to PREST-O-LITE STORAGE BATTERY CORP. (U.S.P. 1,654,778, 3.1.28. Appl., 10.7.23).—Finely-divided, electrolytically-deposited lead is oxidised electrolytically until 50—90% is converted

into the peroxide, and the product is applied to a support. J. S. G. THOMAS.

Manufacture of preformed negative electrodes. R. C. BENNER, A. J. BRIGGS, and J. H. FINK, Assrs. to PREST-O-LITE STORAGE BATTERY CORP. (U.S.P. 1,654,779, 3.1.28. Appl., 10.7.23).—Finely-divided lead is prepared and applied to a support under conditions substantially preventing oxidation. J. S. G. THOMAS.

Manufacture of electron-emitting bodies [cathodes]. WESTINGHOUSE LAMP Co., Assees. of W. C. SPROESSER (B.P. 281,687, 1.12.27. U.S., 1.12.26).—A fabricated metal core, *e.g.*, of tungsten wire, is impregnated with activating metal, *e.g.*, thorium, by embedding in the latter and heating in a vacuum or inert atmosphere. J. S. G. THOMAS.

Resistance element. R. F. BUCHHOLZ, Assr. to ATWATER KENT MANUF. Co. (U.S.P. 1,656,901, 24.1.28. Appl., 24.6.24).—Lampblack is distributed through a mixture of litharge and glycerin in such proportion that the specific resistance of the product is 0.1—2 megohms/c.c. H. ROYAL-DAWSON.

Apparatus for the electrometric determination of hydrogen-ion concentration. M. TRÉNEL (U.S.P. 1,675,421, 24.1.28. Appl., 19.11.24. Ger., 6.12.23).—See G.P. 399,410; B., 1926, 208.

Manufacture of transparent substances from non-metallic and non-transparent materials [oxides]. F. SKAUPY, H. HOFFMANN, and H. SCHMIDT, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,658,659, 7.2.28. Appl., 5.10.22. Ger., 23.2.23).—See B.P. 211,824; B., 1924, 598.

Resistance material. T. A. F. HOLMGREN (U.S.P. 1,658,334, 7.2.28. Appl., 14.1.25. Swed., 22.1.24).—See B.P. 228,142; B., 1926, 98.

Employment of self-baking electrodes. J. WESTLY, Assr. to NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. OF NORWAY (U.S.P. 1,657,948, 31.1.28. Appl., 5.1.25. Norw., 17.1.24).—See B.P. 227,822; B., 1925, 813.

Metallic [mercury] vapour lamp. R. L. M. BELLEAUD and J. BARROLLIER (U.S.P. 1,656,898, 24.1.28. Appl., 30.1.24. Fr., 14.2.23).—See B.P. 211,455; B., 1924, 860.

Controlling chemical reactions (B.P. 282,168 and 282,170).—See I. **Resistant alloy** (B.P. 283,354). **Nickel sheets** (B.P. 275,221). **Electrodeposition of metals** (B.P. 283,288).—See X. **Insulating material** (U.S.P. 1,656,737).—See XIV.

XII.—FATS; OILS; WAXES.

Acid soaps. M. HARTMANN and H. KÄGI (Z. angew. Chem., 1928, 41, 127—130; cf. A., 1925, i, 1045; B., 1925, 378).—The higher fatty acid *N*-carboxylic esters of asymmetrically-substituted ethylenediamines, *e.g.*, diethylaminoethyloleamide ("sapamine"), yield readily soluble neutral salts with most inorganic and organic acids possessing the properties of soaps. The bases are readily obtained by heating the fatty acid or its chloride with diethylethylenediamine, and may be distilled in a high vacuum. The salts are viscous liquids in the anhydrous condition, and their aqueous or acid solutions

form a lather even at great dilution, the limit being at 0.5 pt. per million. Addition of alkali causes the base to be precipitated and destroys the lathering power, whilst the salt is precipitated in a colloidal hydrated form from concentrated solution on addition of sodium chloride. Unlike alkali soaps, these salts are not precipitated by metallic salts in dilute solution, since, when precipitation would normally occur, the insoluble metallic salt remains in colloidal solution; thus, on adding silver nitrate to the hydrochloride of the base colloidal silver chloride, which is not coagulated by nitric acid, is formed. Colloidal solutions of gold and other metals may be readily obtained by this method. The aqueous solutions of the salts have remarkable oil-emulsifying properties, the surface tension at the oil-aqueous solution interface being too small to measure by known methods. This property makes the solutions of value in the textile and dyeing industries, as in their presence, even in minute quantity, vegetable and animal fibres are very readily wetted. In certain cases where sensitivity to alkali is an objection, the base is converted into a quaternary ammonium base, stable to alkali, by heating with methyl chloride, dimethyl sulphate, or a similar substance. W. J. POWELL.

Lathering power of mixed sodium soap. Y. KAWAKAMI (J. Soc. Chem. Ind. Japan, 1927, 30, 216—221).—The froth value of the soap solution is measured as quickly as possible after vigorous shaking (usually after 1 min.), and the lathering power is classified into three grades, according as the froth value is above 80, between 80 and 50, and below 50. If the specific volume of froth, *i.e.*, (froth volume/froth value) \times 100, is less than 5, after being kept for 5 min., the lathering power is regarded as moderate, even when the froth value is above 80. Thus, the lathering power of the three-component system sodium palmitate-sodium oleate-sodium laurate has been measured, the results being shown in the form of three-dimensional graphs. From the results obtained it is possible to anticipate the lathering power at 20° and 40° of tallow, coconut oil, and olive oil soaps, and the limit of good lathering composition of mixed soap consisting of tallow and coconut oils. Y. NAGAI.

Apparatus for the automatic recording of numbers of extractions in the quantitative analysis of fats and the like with the Soxhlet apparatus. G. RANKOW (Z. angew. Chem., 1927, 40, 1559—1560).—In order to diminish the amount of supervision required in the use of the Soxhlet extractor, a device has been arranged to record the number of extractions actually performed. It consists of a glass or metal cylindrical float in the extraction portion of the Soxhlet suspended from a balance beam, on the other side of which is a metal rod. Each time the extractor fills up an electrical contact is made which operates the recording apparatus and rings a bell. As for any seed etc. a definite number of extractions are necessary for completeness, the process can by this means be somewhat shortened without loss of accuracy. C. IRWIN.

Apparatus for the determination of fatty materials. S. UNO (J. Soc. Chem. Ind. Japan, 1927,

30, 428—429).—A modification of Soxhlet's extraction apparatus. Y. NAGAI.

Recent advances in the hydrogenation of oils. E. R. BOLTON (J.S.C.I., 1927, 46, 444—446 T).—The older methods employing powder catalysts are compared with a new continuous process using a fixed catalyst, which is regenerated repeatedly without loss. The advantages of the continuous hydrogenation process—such as the avoidance of the necessity for filtration of hydrogenated oil—are emphasised, and data given showing the ease of control of the degree of hydrogenation. The composition of oils hydrogenated by different processes is discussed, and it is shown how composition can be altered at will by varying the methods of operating the continuous process.

Selective hydrogenation. K. A. WILLIAMS (J.S.C.I., 1927, 46, 446—448 T).—A method is described by which the extent of the selective effect in the hydrogenation of oils may be measured. The method was applied to the hydrogenation of a number of oils under varying conditions. It was shown that the degree to which the reaction is selective is inversely proportional to the iodine value of the oil treated, and, for a particular oil, directly proportional to the temperature of reaction. With fatty acids the selective effect is directly proportional to the iodine value of the original material.

M.p. of hydrogenated oils. K. A. WILLIAMS (J.S.C.I., 1927, 46, 448—449 T).—The results are given of measurements of the melting points of cottonseed oil hydrogenated, under varying conditions, to different extents. A relation was deduced between the melting point of the hardened product, the temperature of hydrogenation, and the corresponding extent of selection in the reaction.

Hydrogenation of fatty acids and of mixtures of fatty acids with neutral oils. R. G. PELLY (J.S.C.I., 1927, 46, 449—454 T).—In a whale oil containing 12% of free fatty acids the free fatty acids were more fully hydrogenated than the neutral oil. Hydrogenation of neutral oils and fatty acids and of known mixtures with activated nickel turnings or with nickel carbonate-kieselguhr catalyst, followed by examination of the separated neutral oil and fatty acids, showed that (1) the rate of hydrogenation of fatty acids alone is less than that of neutral oil alone; (2) when mixtures of neutral oil and fatty acids are hydrogenated, the rate of hydrogenation of the acid is increased—sometimes beyond the rate of neutral oil alone—while the rate of hydrogenation of neutral oil is greatly retarded; (3) the hydrogenation of neutral glycerides is retarded even by saturated fatty acids. It seems evident that the carboxyl group of the fatty acid molecule is the factor of chief importance.

Acid values of fats and oils. Method of determining the barium values of fats and oils. W. L. DAVIES (J.S.C.I., 1928, 47, 24—26 T).—In titrating the free fatty acids in fats and oils, it is suggested that either alkaline-earth bases should be used as titrating liquids or that excess of neutral alkaline-earth salt solution should be added before titrating with caustic soda or potash, both methods being carried out in alcoholic

solution. For coloured fats in ethereal solution, the end-points can be observed in an aqueous layer underneath containing excess alkaline-earth salt. The insoluble barium value can be determined by ascertaining the amount of neutral barium chloride solution necessary to overcome totally hydrolysis of the saponified fat in dilute aqueous solution, the addition of successive volumes of standard barium chloride generating acidity which can be accurately titrated with alkali. The points obtained mostly lie on two straight lines, the equivalent of the barium chloride value of the point of intersection being the insoluble barium value. This has been determined for a number of fats, and inferences from such data are drawn. The conditions of titrating free alkali when determining the saponification number of a fat are investigated.

Test for refined olive oils in virgin olive oils.

A. BAUD and COURTOIS (Ann. Falsif., 1927, 20, 574—577).—When examined by ultra-violet light filtered through a Wood screen, refined olive oils show a characteristic tint and fluorescence which serves to differentiate them from virgin oils. With transmitted light virgin oils show a yellow-brown colour without fluorescence; refined oils have a blue tint. In very thin films the yellowish colour of the former is not apparent, but the latter show an intensified blue fluorescence. By reflected light the blue coloration is masked by a milky fluorescence.

J. R. NICHOLLS.

Catalytic decomposition of soya bean oil by active carbon. S. HAGA (J. Soc. Chem. Ind. Japan, 1927, 30, 618—624).—Soya bean oil was decomposed by heating with Merck's animal charcoal as catalyst. The cracked oil produced was yellow, had d 0.812, acid value 11.5, n_D 1.4551, and was composed of gasoline, 14.7—23% (containing 71.7% of saturated hydrocarbons, 3.7% of aromatic hydrocarbons, and 24.6% of unsaturated, non-benzenoid hydrocarbons); kerosene, 34.7—38% (containing 72% of paraffin hydrocarbons, 5% of aromatic hydrocarbons, and 22% of unsaturated, non-benzenoid hydrocarbons); and neutral oil, 38—46.6%.

Y. TOMODA.

Examination in Wood's light of some varieties of cod-liver oils. H. MARCELET (Compt. rend., 1928, 186, 226—228).—Twenty-four samples of cod-liver oil, representing varieties used in pharmacy and industry, were examined under Wood's light (3340—3906 Å.) and were found to give a fluorescence which is not destroyed by heating at 150—200°, and which varies from very pale yellow to brown or golden yellow according to the quality of the oil. On account of the difficulty of obtaining the special quartz crucibles most suitable for use as containing vessels, the author places a drop of oil on a sheet of non-fluorescent glass or paper, and examines the streak from the falling drop when the sheet is placed vertically 10 cm. in front of a cardboard screen coated with a non-fluorescent animal charcoal. The drops themselves may appear chestnut-brown in such cases.

J. GRANT.

Lower glycerides of palmitic acid. W. BRASH (J.S.C.I., 1927, 46, 481—482 T).—The lower glycerides of palmitic acid were prepared by heating lead palmitate with the corresponding glycerol chlorohydrin. It was

found impossible to prepare the β -monochlorohydrin in a satisfactorily pure condition. The melting points of the glycerides obtained are: α -monopalmitin 68°, $\alpha\gamma$ -dipalmitin 61.5°, $\alpha\beta$ -dipalmitin 68.5°.

Fatty acids of cohune nut fat. T. P. HILDITCH and N. L. VIDYARTHI (J.S.C.I., 1928, 47, 35—37 T).—Cohune nuts from British Honduras consisted of outer fibrous layer 18.1%, nut (shell) 70.0%, and (kernel) 11.9%; the fat content of the kernel varied from 48—55% and formed a soft, faintly cream-coloured fat (acid value 0.9, sap. value 251.0, iodine value 9.8, unsap. 0.47%; mixed fatty acids: sap. value 268.0, iodine value 10.4, titer 21.2°). After refining and deodorisation the fat was comparable with, but not quite equal to, a high-class coconut fat; there appears to be no reason why cohune nut fat should not be employed for the same purposes as the latter fat; if adequate attention is paid to the collection and transport of mature fruit under hygienic conditions. The mixed fatty acids from the fat were converted into methyl esters and fractionated, and from the results it was estimated that the following acids were present: caprylic 7.5%, capric 6.5%, lauric 46.5%, myristic 16%, palmitic 9.5%, stearic 3%, oleic 10%, and linoleic 1%. The fat is thus very closely similar to coconut fat in its general composition.

Fatty acids of shark- and ray-liver oils. IV. **Fatty acids of Kokonohoshi-Ginzame liver oil.** Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind. Japan, 1927, 30, 116—122).—The authors have examined the fatty acids of the liver oil from Kokonohoshi-Ginzame (*Chimaera barbouri*, Garman), the sample being the same as that used previously (cf. Toyama, B., 1924, 431, 719). This oil, like Yamato-torpedo liver oil, is remarkable for its high content of unsaponifiable matter (32.99% of the oil), which consists essentially of alcohols of the selachyl alcohol group. The fatty acids contain more than about 10% of saturated acids. The unsaturated acids consist for the most part of the acids of the oleic series; the acids more unsaturated than those of the oleic series are present in extremely small quantities only. The saturated acids consist preponderantly of palmitic acid with small quantities of stearic, arachidic, and behenic acids, and an acid $C_{24}H_{48}O_2$. The acids of the oleic series consist of zoomaric and oleic acids, an acid $C_{20}H_{38}O_2$, and cetoleic and selacholeic acids. Amongst the acids more unsaturated than those of the oleic series, the acids $C_{20}H_{32}O_2$, $C_{22}H_{34}O_2$, and $C_{22}H_{36}O_2$ seem to form the main constituents.

K. KASHIMA.

Increase in the free fatty acid content in herring oils during preservation, and influence of various substances on the increase. S. UENO and N. KUZEI (J. Soc. Chem. Ind. Japan, 1927, 30, 275—279).—The acid value of 33 samples of different composition was measured from time to time during the course of 367 days. Oil heated at about 100° showed an increase of acid value almost equal to that of the untreated oil, from which it is concluded that the increase of fatty acid observed during storage of the oils is chiefly due to the decomposition of glyceride by oxygen in air and not to bacteria or enzymes. Oils that had been heated with oleates of sodium, potassium, lithium, calcium, copper,

mercury, or zinc showed greater increase in acid value than the original oil. The degree of acceleration of these salts depends on the total amount of added salts. Boric acid, arsenious acid, and animal charcoal had no accelerating action.

N. KAMEYAMA.

Acetyl value of Japanese fish oils in the hardened oil industry. S. UENO and K. YASUHARA (J. Soc. Chem. Ind. Japan, 1927, 30, 388—389).—The commercial crude oils give variable results and cannot be taken as characteristic of the marine animal oils. The authors suggest that, in determining the quality of fish oils for the hardened oil industry, the acetyl value should be excluded.

Y. NAGAI.

Quality of fish oils from the point of view of the hardened oil industry of Japan. II. Sardine oil. S. UENO and K. YASUHARA (J. Soc. Chem. Ind. Japan, 1927, 30, 348—350).—The characteristics of 75 samples of commercial sardine oils of Japan have been determined. The oils had d 0.927—0.933, n_D 1.479—1.481, saponif. value 187—197, iodine value (Hübl) 160—190, acetyl value 18—30, Hehner value 95—96, and Reichert-Meissl value less than 1.0. Acid value was irregular, but generally lower than that of the herring oil. It is considered that commercial sardine oils often contain some herring oils.

Y. NAGAI.

Composition of the body oil from sperm whale. I. Fatty acids. II. Unsaponifiable matter. Y. TOYAMA (J. Soc. Chem. Ind. Japan, 1927, 30, 519—527, 527—532).—I. The oil prepared exclusively from the body blubber of sperm whale (*Physeter macrocephalus*, L.) deposits at ordinary temperatures large amounts of a crystalline solid having d_4^{20} 0.8806, d_4^{30} 0.8733, n_D^{20} 1.4620, acid value 1.24, saponif. value 131.6, iodine value 82.4, unsaponifiable matter 36.40%, and fatty acids 64.13%. The fatty acids are liquid at ordinary temperature, and have d_4^{20} 0.8918, d_4^{30} 0.8847, n_D^{20} 1.4602, n_D^{30} 1.4564, neutralisation value 199.2, saponif. value 201.8, iodine value 87.4, and ether-insoluble bromides 5.55%. The fatty acids consist of about 10% of saturated and 90% of unsaturated acids chiefly of the oleic series, together with a small proportion of acids still more unsaturated. Myristic, palmitic, stearic, and a small amount of arachidic acids form the saturated acids, palmitic acid being preponderant. Lower saturated acids with less than 14 carbon atoms, probably containing lauric acid together with lower members, are also present in small amount. The acids of the oleic series contain zoomaric and oleic acids, an acid $C_{20}H_{38}O_2$, and cetoleic acid. An acid $C_{14}H_{26}O_2$ is also present in small amount. Lower members of the oleic series with less than 14 carbon atoms are absent or present only in minute amount. The acids more unsaturated than those of the oleic series contain in addition to C_{22} acids also C_{20} and C_{18} acids. The presence of clupanodonic acid, $C_{22}H_{34}O_2$, is confirmed. No evidence is obtained for the presence of physetoleic acid. II. The unsaponifiable matter forms at 20° a crystalline solid, having d_4^{20} 0.8508, d_4^{30} 0.8413, n_D^{20} 1.4500, saponif. value of acetylated product 186.6, iodine value 72.2, and cholesterol content (by digitonin method) 0.44%. It consists chiefly of oleyl alcohol (octadecenol), cetyl alcohol, and octadecanol, of which oleyl alcohol preponderates. It

contains also small amounts of alcohols more unsaturated than oleyl alcohol, among which highly unsaturated alcohols giving ether-insoluble bromides are found. Besides these alcohols, cholesterol is present. Hexadecenol is absent or present in minute amount. Tetradecanol was not detected. Oleyl acetate, b.p. 217—220°/15 mm., yields, on oxidation with potassium permanganate in acetic acid solution, nonoic acid and acetoxynonoic acid; hence the constitution of oleyl alcohol is probably $Me \cdot [CH_2]_7 \cdot CH : CH \cdot [CH_2]_7 \cdot CH_2 \cdot OH$, which is identical with that from liver oil of *Chlamydomonas anguineus*, Garman (J. Soc. Chem. Ind. Japan, 1923, 26, 37; 1924, 27, 468).

K. KASHIMA.

Morning-glory (Asagao) oil. I. Properties of the oil. K. MATSUMOTO (J. Soc. Chem. Ind. Japan, 1927, 30, 205—207).—The black and white seeds of "Asagao" (*Pharbitis Nil.*, Chois.) contain 11.85% and 12.13% of the oil, respectively, which is yellow and has a slightly unpleasant smell. The black seed oil has d_{15}^{20} 0.9255, n_D^{20} 1.4722, acid value 5.3, saponif. value 191.9, iodine value (Hübl) 94.8, Reichert-Meissl value 0.59, and unsaponifiable matter 2.25%. Those of the white-seed oil differ very slightly from those of the black-seed oil. Both oils solidify at about 0°. By the elaidin test, they become turbid after 2 hrs. and yield solid precipitates; after 24—33 hrs. they become buttery masses. The mixed fatty acid (free from unsaponifiable matter) of the oil is a white solid mass yielding no ether-insoluble compounds by bromination, and has m.p. 33—34.5°, iodine value (Hübl) 104.9, neutralisation value 201.4, mean mol. wt. 278.5. Morning-glory oil is a non-drying oil.

Y. NAGAI.

Fuel oil from soya bean oil. SATO with others.—See II. **Activity of a nickel catalyst.** LUSH.—See III. **Coconut oil in butter.** ATKINSON and AZADIAN.—See XIX.

PATENTS.

Preparation of fats of a plastic and lithe consistency. K. ERSLEV (U.S.P. 1,657,380, 24.1.28. Appl., 27.8.26. Holl., 9.5.24).—See B.P. 269,384; B., 1927, 531.

Hydrolysis, and particularly splitting, of oils and fats. P. J. FRYER, ASS. TO CATALPO, LTD. (U.S.P. 1,657,440, 24.1.28. Appl., 9.4.21. U.K., 24.4.20).—See B.P. 166,971; B., 1921, 665 A.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Standardisation of turpentine, "terpenoids," and turpentine substitutes, and a method of estimating their technical qualities. F. HEBLER (Farben-Ztg., 1928, 33, 1106—1108).—Turpentine substitutes other than those (e.g., "terpenoids") that resemble turpentine chemically should at least have similar properties to turpentine from the technological point of view. This is not always the case, but the usual chemical and physical tests do not reveal the differences. The effect on the viscosity and stability of a wax emulsion when small quantities of the solvents under examination are added affords valuable information in this connexion.

S. S. WOOLF.

Facet effects on pigments. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1928, Circ.

No. 321, 97—117).—It was found that the oil absorptions of pigments are appreciably reduced by coating the pigmentary particles with aluminium stearate or other metallic soaps. The laboratory method of achieving this result was to wet the pigment thoroughly with a 2% solution of aluminium stearate in petroleum spirit, that was subsequently evaporated off. Other attempts to "stearate" pigments and reduce the time of grinding of paints gave somewhat anomalous results; e.g., a flat wall paint made by wetting lithopone with aluminium stearate solution and incorporating oil and drier is much heavier bodied than the corresponding paint made on the same formula but omitting the stearate. "Stearated" paints prepared by the laboratory method, however, show less tendency to settle or to "liver" with acid vehicles. Further discussion is given on the possible use of water as a thinner for wall paints, artificial ageing tests on paint (in bulk), and the effects of various vehicles in causing the granulation of paints containing basic pigments. S. S. WOOLF.

Solvents and plasticisers for cellulose esters. A. NOLL (Farben-Ztg., 1928, 33, 1166—1170).—Details of the composition, nature, and various constants of a large number of modern solvents and plasticisers suitable for use in cellulose lacquers are given. S. S. WOOLF.

Brushing lacquers and their future. H. A. GARDNER (Amer. Paint & Varnish Manufs. Assoc., Jan., 1928, Circ. No. 322, 119—129).—The possibilities of the adoption of brushing lacquers for general painting purposes, as distinct from minor decoration, are discussed, it being shown that the use of a primer composed of red lead, zinc dust, aluminium powder, and linseed oil will overcome the difficulties inherent in the use of nitro-cellulose lacquers on wood. The opinions of 12 authorities on these and other related questions are quoted. S. S. WOOLF.

Scattering of light by particles. LOWRY and MARSH.—See I. "Demolisation." WISLICENUS.—See II. Zinc oxide. DÖRZBACH.—See VII.

PATENTS.

Manufacture of lithopone. J. E. BOOGE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,195, 24.1.28. Appl., 6.4.22. Renewed 26.10.23).—Crude lithopone containing less than 0.1% of combined chlorine is concurrently calcined and agitated, air being excluded. H. ROYAL-DAWSON.

Manufacture of inks. BRIT. DYESTUFFS CORP., LTD., C. HOLLINS, and E. CHAPMAN (B.P. 279,968, 19.8.26).—By the addition of 0.1% of isopropylated, sulphonated mineral oil (B.P. 274,611; B., 1927, 841), sulphonated mineral oil (B.P. 279,990; B., 1928, 50), sulphonated formaldehyde-naphthalene or formaldehyde-phenol condensation products, or alkyl-naphthalenesulphonic acids, ink is made to flow more freely. C. HOLLINS.

Treatment of fouled enamel-removing solutions. W. H. ALLEN (U.S.P. 1,656,154, 17.1.28. Appl., 18.12.25).—Chlorine is passed into alkaline solutions which have become fouled through use in removing enamel, varnish, etc., and the supernatant liquid is drawn off and cauterised with lime. H. ROYAL-DAWSON.

Utilisation of scrap from products made from blood, casein, and glue hardened with formaldehyde. PFENNIG-SCHUMACHERWERKE G.M.B.H. (G.P. 444,218, 28.4.26).—The material is rendered plastic by treatment with alkaline hydrogen peroxide solution, which oxidises the formaldehyde to formic acid. L. A. COLES.

Manufacture of condensation products obtained from urea and solid polymerides of formaldehyde. F. E. K. STEPPES, Assr. to H. TRAUN & SÖHNE (U.S.P. 1,658,359, 7.2.28. Appl., 19.8.26. Ger., 4.12.24).—See B.P. 271,264; B., 1927, 564.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

The two coagulations of [rubber] latex. R. AUDUBERT and G. LEJEUNE (Rev. gén. Colloid., 1927, 5, 715—722).—The existence of two zones of flocculation for *Hevea* or *Landolphia* latex with an intervening zone of acidity in which complete coagulation occurs is confirmed using preserved latex. Microscopic investigation of the migration of the particles in an electric field indicates that the electric charge remains negative; with the addition of gelatin or casein, however, the charge of the globules shows a change of sign in the second stable zone. The existence and extent of the intermediate stable zone are also dependent on the concentration of rubber in the latex. D. F. TWISS.

Nature of vulcanisation. III. H. P. STEVENS (J.S.C.I., 1928, 47, 37—43 T).—By vulcanising a sufficiently dilute rubber sol with a limited amount of sulphur chloride so that gelling is delayed and mixing while fluid with a raw rubber sol a mixed vulcanised and raw rubber can be obtained with properties substantially the same as those of a rubber vulcanised to the same extent by sulphur chloride in one operation. Similarly, a series of rubber specimens obtained from mixtures of raw and vulcanised rubber latex behave in the same manner as rubber specimens prepared from vulcanised latex direct. Comparisons are based on swelling in solvents and tensile properties. Load-stretch curves are given. A series of rubber specimens was prepared by incorporating cellulose in a finely divided state by mixing latex and viscose. Increased proportions of cellulose result in decreased swelling in solvents, but the physical effect, although progressive, is not parallel with the vulcanising effect of sulphur. Similar experiments with glue are also recorded. It is concluded that combination of sulphur with rubber is essential to vulcanisation, the physical effect of which is brought about by the deposition of caoutchouc (or polyprene) sulphide in a dispersed state in the rubber. The physical properties of a vulcanised rubber will depend on the degree of dispersion and the shape of the particles, which in their turn are modified by the conditions of vulcanisation.

PATENTS.

Vulcanisation of rubber. ROESSLER & HASSLACHER CHEMICAL Co. (B.P. 265,169, 7.1.27. U.S., 27.1.26).—Compounds of the type $(RO \cdot CS)_2S_x$, where x is greater than 2, are claimed as vulcanisation accelerators. They are made by the action of sulphur chlorides on potassium alkylxanthates, the dichloride giving a trisulphide and the monochloride a tetrasulphide. The preparation

of *thioncarbothoxyl tetrasulphide* ($R = Et, x = 4$), *thioncarbo-n-butoxyl tetrasulphide* and *trisulphide*, all yellow oils, is described. For use as accelerators the thioncarboalkoxyl polysulphides may be mixed with an amine, e.g., dibenzylamine, aniline, ethylaniline, whereby crystalline substances are obtained; or the oils themselves may be used.

C. HOLLINS.

Vulcanisation of rubber. SOC. ITAL. PIRELLI (B.P. 266,732, 23.2.27. Italy, 24.2.26).—Vulcanisation is accelerated by the salts of carboxylic acids, e.g., of the homologous series of fatty acids (including phenylacetic acid), with alkali metals other than sodium or lithium.

D. F. TWISS.

Vulcanisation of rubber. H. GÜNZLER and W. ZIESER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,502, 20.12.27. Appl., 12.4.27. Ger., 21.5.26).—A mixture of zinc oxide and formamide is claimed as an accelerator.

T. S. WHEELER.

Vulcanisation of rubber. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,892, 30.9.26).—Formamide is used as an accelerator of vulcanisation.

D. F. TWISS.

Reclaiming of rubber. G. J. MILLER (U.S.P. 1,657,171, 24.1.28. Appl., 22.12.26).—Old rubber scrap containing cotton is comminuted and heated with a suitable acid solution for the removal of cotton and soluble fillers. After being washed, the rubber is then heated in kerosene oil at 120–150° until the maximum amount of impurities has been removed without dissolution of the rubber. The rubber is then separated, washed with a volatile liquid for the removal of kerosene, and dried.

D. F. TWISS.

Preventing the loss of colour of dyed, unvulcanised rubber. CONTINENTAL-CAOUTCHOUC U. GUTTA-PERCHA Co. (G.P. 445,534, 21.3.25).—The rubber is dyed with solutions of lipoid-soluble dyes or dye bases in mixtures containing acetone, rubber-swelling agents, and suitable quantities of high-boiling fatty acids or their esters, and, if necessary, paraffins in addition.

L. A. COLES.

Production of acid- and alkali-resistant coatings of rubber upon metal objects, with the employment of an intermediate layer of rubber. F. AHRENS (B.P. 283,049, 19.5.27. Ger., 16.4.27).—The metal surface to be covered is first coated with one or more layers of a rubber solution, to which addition of an acid substance, or substances capable of liberating acids, has been made. A hard or soft rubber sheet is then applied and the whole is then vulcanised. The reaction product of an unsaturated oil and sulphur chloride, especially if prepared under increased pressure, is advantageous as the acid-generating substance.

D. F. TWISS.

Production of insulating material [from vulcanised rubber]. A. R. KEMP, Assr. to BELL TELEPHONE LABORATORIES, INC. (U.S.P. 1,656,737, 17.1.28. Appl., 28.2.27).—An insulating material is produced by subjecting a series of mixtures containing rubber and sulphur in different proportions to a temperature above that necessary for vulcanisation and mixing them.

D. F. TWISS.

Treatment of water solutions of rubber. E.

HOPKINSON and W. A. GIBBONS, Assrs. to REVERE RUBBER Co. (Re-issues 16,873–4, 7.2.28, of U.S.P. 1,542,388, 16.6.25).—See B., 1925, 643.

XV.—LEATHER; GLUE.

Evaluation of raw stock [skins]. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1928, 23, 1–3).—Samples of raw stock were cut from different parts and placed in an air-tight container. They were cut into pieces 0.5–1 in. square, with as little loss of adhering dirt, salt, and hair as possible, 100 g. placed in a large jar and shaken for 30 min. with 850 c.c. of water at 21°, then the cover was removed, replaced with a 40-mesh screen to retain the hair, and the wash water decanted. The washing was repeated 9 times over a period of 5 hrs., after which the hide pieces and hair were squeezed as dry as possible and dried to constant weight. Results on a number of skins showed salt 2.25–21.0%, material washed out including sand, dirt, salt, etc. 25.9–69.75%, and actual dry hide (including hair) 32.65–74.1%. Samples from different parts of the skin showed but very small differences in analysis.

D. WOODROFFE.

Deterioration of bookbinding leather. F. P. VEITCH, R. W. FREY, and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1928, 23, 9–12).—Samples of ten different leather bindings from books kept in a relatively pure atmosphere and not exposed to artificial light or in a room heated by coal fires were analysed. Six samples contained no free mineral acid even after 100–400 years. The highest acidity was 0.53%, in a leather containing pyrogallol and pyrocatechol tannins. The other leathers were tanned with pyrogallol tannins mainly. The good condition of the bindings is attributed to the absence of appreciable acidity, the predominance of a pyrogallol tannage, and storage in a non-polluted atmosphere.

D. WOODROFFE.

Chemical nature of vegetable tanning. C. SCHIAPARELLI and G. BUSSINO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 343–361).—The results of a series of tanning experiments with extracts containing 2% of tannin and using pieces of hide instead of hide powder show that the process of tanning is largely dependent on the hydrogen-ion concentration of the bath. The proportion of water-soluble constituents increases, and the resistance of the leather to hot water diminishes as the acidity of the tanning liquid increases. When pure tannin is used in 2% concentration and the p_H value of the bath is above 2.5, 100 pts. of dry protein in ox hide unites at the ordinary temperature irreversibly with about 60 pts. of tannin (cf. B., 1926, 716).

T. H. POPE.

Chemistry of combined tanning. C. SCHIAPARELLI and L. CAREGGIO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 362–367).—Experiments with hides support Wood's theory on combined chrome-tannin tanning (B., 1908, 384, 1164), one part (basic) of the protein molecule absorbing about 60% of tannin, which represents the proportion of tannin irreversibly absorbed, on the average, by 100 pts. of dry protein, whereas another part (acid) of the same molecule absorbs irreversibly 6.18 pts. of Cr_2O_3 , which approximates to the proportion (6.76 pts.) required to form the chromium dicolla-

genate obtained by tanning in a bath of basicity 33 (1 mol. of sodium carbonate per 1 mol. of chromium alum). This combined tanning should be the ideal tanning, since by it all the reactivities of the protein molecule are saturated (cf. Gustavson, B., 1927, 534).

T. H. POPE.

Utilisation of poplar bark as tanning material.

G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 387—402).—Analysis of the barks of various species of poplar gave the following results (%) for *P. nigra*, *P. alba*, and *P. canadensis*, respectively:—Tanning materials, 5.02, 5.26, 4.34; soluble non-tannins, 3.12, 4.40, 4.16; insoluble substances, 81.30, 78.37, 80.55; water, 10.56, 11.97, 10.95. The wood of *P. nigra* contains too low a proportion of tannins to be used for tanning. Extraction of the bark of *P. nigra* with water at about 95° and concentration of the liquid under reduced pressure gave a dry extract which contained 42.00% of tanning materials, 36.09% of soluble non-tannins, 9.83% of insoluble matter, and 12.08% of water, and dissolved completely in hot water, the solution depositing phlobaphens on cooling. For the decolorisation and solubilisation of this extract, the use of blood, oxalic, lactic, or boric acid, or sodium bisulphite proved unsatisfactory, but treatment with a mixture of sodium bisulphite and aluminium sulphate in definite proportions gave good, and that with sulphite-cellulose still better, results. Leather tanned with the crude poplar bark extract, untreated with clarifying or solubilising agents, has a bright chestnut-yellow colour, quite different from the almost dirty colours obtained with other highly coloured tannin extracts. The extract prepared with the help of sulphite cellulose gives a paler yellow, but not a bright, leather, whilst a brown colour is obtained with the extract solubilised with the synthetic coritan. The leathers are of good quality, low ash content, high strength (3.4—3.7 kg./mm.²), and regular elongation (26—27%), and have a soft feel.

T. H. POPE.

Retarding action of tannins in the atmospheric oxidation of the alkali bisulphites.

A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 403—415).—Although alkali bisulphites, even in concentrated solution, are readily oxidised in the air, such oxidation occurs only slowly in presence of tanning materials. In general, the tannin does not combine with or retain the free dissolved sulphur dioxide, which, after vigorous and prolonged aeration of the tanning bath, remains in solution in slight amount, only a small proportion undergoing oxidation during the aeration. The formation of additive compounds of tannin and sulphurous acid is thus excluded, but it seems probable that tannin and an alkali bisulphite unite to give an unstable compound which exists only in solution and is analogous to the bisulphite-aldehyde (or -ketone) compounds. No such additive compounds are formed by tannin and normal sulphites.

T. H. POPE.

Permeability of hide and leather. M. BERGMANN (Collegium, 1927, 572—579).—The water-penetration apparatus consists of a Wolff's bottle containing mercury. The middle aperture in the bottle is fitted with a pressure regulator and overflow tube for the surplus water. Water is forced into one of the side inlets of the

bottle and passes out of the other into a tube, arranged above a clamp containing the leather. A mercury manometer is fitted to this tube. The water is forced through the leather or hide and is collected in a measuring cylinder below. The apparatus for measuring the permeability to gas is similar except that the gas forced through the leather is collected over water. Samples of dried, lightly salted, and dry-salted hide, respectively, were soaked for 24 hrs. and tested for water permeability. The results are expressed as the volume of water which was forced through per hour. Tests were made on similar samples which had been kept for 8 weeks, then soaked, limed, and the swelling reduced with a 2% solution of ammonium chloride. The permeability to water increased by 1.9, 3.0, and 12.7 times, respectively, by liming and deliming dried, lightly salted, and dry-salted hide, respectively. Leather from both flesh and grain sides is equally permeable to gases, but that from the flesh side is much more permeable to water. A piece of leather was split giving a grain split 1.5—2.0 mm. thick and a flesh split 3—3.5 mm. thick. The flesh split was less permeable to water (318 c.c./hr.) from the grain side than the flesh side, and less permeable than the grain split from the grain side (3254 c.c./hr.) even allowing for the difference in thickness. The permeability is affected by retannage. D. WOODROFFE.

Softening of hides. V. CASABURI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 368—373).—A short account is given of a series of experiments on the softening of hide in soft water, water of hardness 50° (French), 0.08% sodium hydroxide solution, 0.12% sodium hydrogen sulphide solution, 0.02N-sodium citrate solution, 0.128% potassium thiocyanate solution, 0.128% sodium arsenite solution, and 0.11% sodium chloride solution. The full results are to be published later. T. H. POPE.

Pickling of hides. E. SIMONCINI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 383—386).—Experiments on the pickling of hides in baths containing up to 2% of sulphuric acid on the weight of hide and a proportionate amount of salt, with a maximum of 25% of the weight of hide, show that the acid is fixed in a comparatively short time, and that, for equal volumes of bath, the quantity of acid fixed in a given time is proportional inversely to the ratio between acid and hide, and directly to the initial concentration of the bath. If the concentration of the acid and the duration of the action are kept constant, the percentage of acid fixed is not altered by varying the concentration of salt in the bath. The principal effect of pickling is to be attributed to the acid, which combines with and stabilises the collagen fibres, these contracting just as in presence of other tanning agents. The salt acts indirectly by impeding dissociation of the hide-acid compound. T. H. POPE.

Leather substitute. R. DITMAR (Gummi-Ztg., 1928, 42, 913).—Finely-ground sole leather waste (15 g.) was mixed with 45 g. of rape oil, 1.5 g. of castor oil, 6 g. of mineral oil, and 15 g. of sulphur dichloride. A reaction began which was complete in 4 min. and gave a moist dark product; incorporation of this with crêpe rubber and sulphur and then vulcanising for 2 hrs. in the press at 150° gave a final product which is not as good as crêpe rubber vulcanised with sulphur and best

French rubber substitute. Better products have been obtained by using 50 g. of rape oil, 16.5 g. of sulphur, and 14.7 g. of leather. A product manufactured by "Georg Grandel," Augsburg, has the following composition:—rape oil 77.34%, sulphur 12.66%, and ground leather 10%, or 30 pts. of pure "substitute" with 14% of sulphur and 10 pts. of ground leather meal. It had d 1.046, acetone extract 19.2%, no free sulphur. The acetone extract is hardly distinguishable from that of "substitute" prepared from pure rape oil.

D. WOODROFFE.

Removal of mineral substances from gelatin by electro dialysis. S. OKA (J. Soc. Chem. Ind. Japan, 1927, 30, 396—402).—After applying a P.D. of 50, 100, or 200 volts to the 3% or 10% gelatin solution for from 7 to 15 hrs., the ash contents of the dried substances decreased from 3.50% to 0.05—0.23%. The progress of demineralisation was followed by the measurement of the rise of the electric conductivity of the distilled water put in each electrode compartment. It was found that the velocity of demineralisation was very great at the beginning and rapidly diminished to a very small and almost constant value. The quantity of the cation removed was found to be far greater than that of the anion. Calcium carbonate was found to be the main constituent of the ash before the treatment, but after the treatment ferric oxide predominated. N. KAMEYAMA.

PATENTS.

Manufacture of tanning agents. I. G. FARBEININD. A.-G. (B.P. 266,697, 10.2.27. Ger., 23.2.26).—By using a sulphonating agent, such as chlorosulphonic acid or oleum, which combines with the water liberated, salicylic esters may be sulphonated at 140—190° without appreciable hydrolysis, tanning agents being produced. Examples are phenyl or β -naphthyl salicylate, acetyl-salicylic acid, salicylides obtained from *o*-hydroxycarboxylic acids and thionyl chloride, phosphorylsalicylic acid, and the compounds described in B.P. 218,316 and 252,694 (B., 1925, 181; 1927, 373). C. HOLLINS.

Manufacture of chamois-tanned leather. A. KEMMLER (U.S.P. 1,657,800, 31.1.28. Appl., 28.9.26. Ger., 29.8.24).—See B.P. 266,622; B., 1927, 306.

[Conveyor for] treating hides, skins, and leathers. MASCHINENFABR. TURNER A.-G. (B.P. 277,349, 8.9.27. Ger., 11.9.26).

XVI.—AGRICULTURE.

Influence of superphosphate on the reaction of the soil. C. ANTONIANI (Atti R. Accad. Lincei, 1927, [vi], 6, 319—325).—When applied to neutral, or almost neutral, naked soils free from lime, at the rate of 240—480 lb./acre, superphosphate produces a gradual acidification, but never causes any marked acidity. The fact, noted by other investigators, that superphosphate renders soils alkaline, is doubtless due to the influence of a growing crop or to other factors. T. H. POPE.

Degree of acidity of Norwegian soils. K. O. BJØRLYKKE (Tids. norske landbruk, 1926; Proc. Internat. Soc. Soil Sci., 1927—8, 3, 36).—Examination of a large number of Norwegian soils revealed the following general relationships. Surface soils generally have lower p_H values than subsoils. Acidity is more pronounced in

the older and more completely weathered soils. Soils rich in colloidal matter are usually less acid than sandy soils, and cultivated soils than uncultivated.

A. G. POLLARD.

PATENTS.

Manufacture of mixed phosphatic fertilisers, and recovery of ammonia from industrial gases. H. HOLLINGS, and GAS LIGHT & COKE Co. (B.P. 282,927, 19.10.26).—An industrial gas containing ammonia, e.g., coal gas which has been stripped of its tar, is passed into an absorber charged with superphosphate of lime, which may be mixed with peat, sewage sludge, or other distributing material, until no more ammonia is taken up. The loosely combined excess ammonia is then expelled from the saturated material by passing through it a current of neutral flue gases. A stable mixed fertiliser of constant composition is thereby manufactured, and the gas at the same time is purified from ammonia. It is advantageous to carry out the absorption at about 40° to minimise any condensation of water, naphthalene, or hydrocyanic acid. A convenient cyclic method of carrying out the process, utilising 3 or 4 absorbers, is described.

A. B. MANNING.

XVII.—SUGARS; STARCHES; GUMS.

Volumetric determination of lactose in presence of sucrose. J. H. LANE and L. EYNON (J.S.C.I., 1927, 46, 434—435 T).—In the analysis of sweetened condensed milk the effect of sucrose on the determination of lactose by the authors' method (*ibid.*, 1923, 42, 32 T), can be allowed for by adding certain volume corrections to the amount of sugar solution required by 10 c.c. or 25 c.c. of Fehling's solution. These corrections are given for sucrose/lactose ratios of 3/1 and 6/1, and for any such ratios up to 10/1 the corresponding corrections can be estimated from these data with sufficient accuracy by inspection.

Caramel. H. DRAKE-LAW (J.S.C.I., 1927, 46, 428 T).—Experiments were made on highly coloured caramel with special reference to the dextrose content. The latter is of importance as the fermenting power of caramel and the physiological action on diabetic patients is approximately proportional to the sugar present. A method of analysis based on Fehling's reduction method gives results which are too high, a contention which is established by converting the sugars present into the osazones. The discrepancy in the two methods of testing is in the proportion of 20% to 5%. Highly coloured caramels are therefore suitable for admixture in foods used for diabetic patients and generally as a non-fermenting foodstuff colour.

Adhesiveness of rice, maize, and wheat starches in comparison with potato starch. E. PAROW, A. STIRNUS, and W. EKHard (Z. Spiritusind., 1928, 51, 23—24).—Samples of starches were examined by the Saare test, pastes being prepared by the usual method followed by heating for varying periods on a water-bath. Rice starch prepared by the alkali process and containing a trace of alkali and some protein showed slightly decreasing Saare values with increased periods of heating. Neutralised rice starch yielded values 3—4 times as great and which increased with the heating period.

Rice starch prepared without alkali showed intermediate values and only slight changes with the heating period. The values for maize starch were generally higher with relatively large increases as the heating continued. For wheat starch the values were intermediate between those of maize and rice and varied irregularly with the heating period. Potato starches varied considerably according to their origin, values generally being below those of cereal starches and tending to decrease as the heating period was prolonged. A. G. POLLARD.

PATENTS.

Clarification of cane-sugar juice. J. V. N. DORR, Assr. to DORR Co. (U.S.P. 1,655,049, 3.1.28. Appl., 8.9.24).—After defecation of the rich juice from the sugar cane, the mud so obtained is defecated with thin juice, and the mud from the latter operation is then separated by a filter press. The filtrate and washings are kept separate, the former being returned to the clarifier and the latter passing to the megass for maceration. The cake is flushed from the filter press with water, and the resultant mud is returned to the megass preceding the last mill. F. R. ENNOS.

Treatment [clarification] of cane juice settlings. J. F. BORDEN, Assr. to OLIVER CONTINUOUS FILTER CO. (U.S.P. 1,653,491, 20.12.27. Appl., 22.9.26).—The liquid is treated at 80° with lime to p_H 8.5 and with phosphoric acid to p_H 6—6.9. T. S. WHEELER.

Economical reduction of composite sugar-bearing solutions. H. J. CREIGHTON, Assr. to ATLAS POWDER Co. (U.S.P. 1,653,004, 20.12.27. Appl., 26.3.26).—The mixture of polyhydric alcohols obtained as described in U.S.P. 1,612,361 (B., 1927, 234) is nitrated without separation of mannitol. T. S. WHEELER.

Manufacture of starch. G. M. MOFFETT, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,655,395, 3.1.28. Appl., 3.3.27).—After steeping the maize in water containing sulphur dioxide, it is washed with water from a preceding separating operation, and then disintegrated. The germ, bran, and fibre are removed in the coarse and fine slop separations, and the starch is tumbled to free it from gluten and water. It is then filtered and washed twice with fresh water to remove solubles. Economy in the use of water is effected by using that from the starch tables partly for steeping and partly for the germ and coarse slop separations, and by employing the first and second washings from the starch filters for the fine slop separation after heating, and for flushing the starch from the tables, respectively. F. R. ENNOS.

Maltose product and production of same. H. C. GORE, Assr. to FLEISCHMANN Co. (U.S.P. 1,657,079, 24.1.28, Appl., 30.3.25. Austral., 28.11.24).—See B.P. 249,428; B., 1926, 458.

Apparatus for diffusing sugar beet or similar substances. J. & F. HOWARD, LTD., and G. E. ROWLAND (B.P. 281,813, 7.10.26).

XVIII.—FERMENTATION INDUSTRIES.

Determination of antiseptic power of hops. T. K. WALKER (J. Inst. Brew., 1928, 34, 61—64).—In the chemical method for the determination of the anti-

septic power of hops, the percentages of α - and β -resins in a hop are determined gravimetrically, and the total preservative power is calculated from the formula $\alpha + \beta/3$, which differs slightly from the formula $\alpha + \beta/4.3$ as given by Ford and Tait (cf. B., 1926, 170). Chapman's biological method gives results which agree well with those obtained from the measurement by titration of the amounts of acid formed by *B. Delbrückii* under certain specified conditions in the presence of decoctions of hops. The gradings of hops with respect to their antiseptic powers according to all three methods are in exceedingly close agreement. C. RANKEN.

Relation between time and temperature of storing and their combined influence on fermentation, increases and acid production in varied length of keeping of beer yeast under water at different temperatures. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1928, 45, 31—37, 49—57).—Bottom-fermentation yeasts kept under water at 0° for periods up to 5 days show a slight uniform increase, proportional to the time, of fermentative, reproductive, and acid-forming power, when compared with yeasts similarly stored at 8°. Storage at 20° markedly decreases these characters, the relative amount of such decrease, in the case of fermentation and acid production, increasing with increasing time up to 4 or 5 days. The application of this to yeast conservation and fermentation control is discussed. F. E. DAY.

Examination of grape juice (must) and improvement of wines. D. SCHENK (Pharm. Ztg., 1927, 72, 1409—1411).—Grape juices and wines are improved by adjusting the sugar and acid contents. Sugaring is carried out by adding cane sugar alone or in aqueous solution, care being taken that the alcohol content produced on subsequent fermentation shall not exceed the desired limit. During fermentation, the acidity usually falls owing to the precipitation of tartrates in the lees and the bacterial decomposition of malic acid to lactic acid. The sugar content is determined by multiplying the sp. gr. of the must expressed in Öchsle degrees at 15° by 0.225. The acidity, calculated as tartaric acid, is determined by titration with sodium hydroxide solution using litmus paper as external indicator. If fermentation has commenced, the Öchsle value for the original grape juice must be found by multiplying the alcohol content by 10 and adding that value to the actual Öchsle value. In the improving process the natural deficiency of sugar is made up and any excess acidity corrected by suitable dilution. The number of Öchsle degrees through which the juice must be raised in the process is called the "elevation value." If sugar alone is used, 0.24 kg. is required per degree in the elevation value per 100 litres of juice, but allowance must be made for the volume change on dissolution of the sugar (1 kg. increases the volume by 600 c.c.). If syrup is used 0.22 kg. is required per degree per 100 litres of juice. When the mash is in full fermentation, the latter should be allowed to finish before tests are made. Excess acid may be removed by means of pure precipitated calcium carbonate, but the latter should not be used in quantities greater than 132 g. per 100 litres, equivalent to 2 pts. per thousand of acid, b

otherwise a bad taste will develop. Wine may be clarified by the correct addition of pure potassium ferrocyanide.

S. COFFEY.

Composition of crude fusel oil according to different methods. B. LAMPE (Z. Spiritusind., 1928, 51, 30—31).—The content of amyl alcohol in crude fusel oil is not correctly indicated by the official method in which 100 c.c. of oil are subjected to simple distillation and the volumes of the fractions obtained up to 100°, 120°, and 130° determined. The use of a double rectifier of high dephlegmating power and the collection of the fraction which distils from 127° to 132° is suggested as a means of more accurate evaluation. In a comparison of six oils by the two methods, the oil which gives the greatest distillate of b.p. 127—132°, by the method of double rectification, does not yield by the official method the distillate of 90 c.c. which is fixed as the minimum amount to be obtained up to 130°. Accordingly, what would be considered as an oil of the highest value by the first method would be classed by the official method as of inferior quality. The difference between the results depends upon the composition of the amyl alcohol-ethyl alcohol-water mixture which governs the tendency of the amyl alcohol in simple distillation to pass over among the first fractions. If these are treated with water and sodium chloride two layers form, from the upper of which appreciable quantities of amyl alcohol can be distilled.

C. RANKEN.

PATENTS.

Preparing a preservable yeast preparation. E. I. LEVIN (B.P. 271,883, 25.5.27. Swed., 26.5.26).—Pressed yeast is heated for approximately 3 hrs. at 54—55°, and the product, which is spread in a thin layer, with or without admixture of a sterile dry substance, is treated with sterilised pure oxygen at 30° for from 20 to 40 hrs. Subsequent repeated heating at 50—60° for 1—2 hrs. destroys the remaining yeast fungi, leaving unaffected the yeast which has been converted into ascospores, and which can be successfully stored for long periods.

C. RANKEN.

Acceleration of fermentation processes. E. BREDO-WEUSTENRAAD (F.P. 621,439, 24.7.26. Belg., 1.9.25 and 11.2.26).—The maturing of must and beer is accelerated by bringing it in contact with air in open or closed troughs as it leaves the heating vessels.

L. A. COLES.

Utilisation of alcoholic fermentation in order to form large quantities of glycerin by use of sulphurous acid. E. A. BARBET (B.P. 282,917, 13.10.26).—In place of the customary large dose of reducing reagent such as sodium sulphite, sulphurous acid, to which the yeast must be first accustomed, is added to the fermentation medium in very small amount so that its sterilising action remains without effect upon the fermentation. The yield of glycerin from beetroot juice is improved by concentrating the beetroot vinasses without deglycerination and adding to the beetroot juice, as a preliminary to the fermentation, a portion of this concentrate with its high content of organic potassium salts.

C. RANKEN.

Production of sparkling wines. A. CHAUSSEPIED (F.P. 621,341, 5.1.26).—The fermentation process, the addition of liqueurs or other liquids, the saturation with

gas, and the cooling are all effected in the same vessel, means being provided to heat the liquid during fermentation.

L. A. COLES.

Cellulose for butyric fermentation (F.P. 616,424).—See V.

XIX.—FOODS.

Appraisalment of contaminated milk. M. FOUASSIER (Ann. Falsif., 1927, 20, 577—580).—Possible sources of contamination are reviewed and a laboratory process is described for removal of insoluble foreign matter by filtration. The interpretation of the results of examination of the sediment is discussed.

J. R. NICHOLLS.

Determination of chlorides in milk. D. RAQUET and A. KERLEVEO (Ann. Falsif., 1927, 20, 580—583).—A criticism of the ordinary methods is followed by a description of two new methods which do not possess the usual disadvantages. (i) To an aqueous extract of the residue from the fat determination by the Adams process 5 c.c. of concentrated nitric acid, 10 c.c. of 0.1N-silver nitrate solution, 5 c.c. of 5% potassium permanganate solution, and about 50 c.c. of water are added. After boiling gently for 10 min. and decolorising, 50 c.c. of water are added and the solution is again heated for 10 min. and then cooled. 5 c.c. of nitric acid and 1 c.c. of 0.2N-ferric alum are added, and the solution is titrated with 0.1N-ammonium thiocyanate. (ii) To 20 c.c. of milk diluted with about 100 c.c. of water is added 1 c.c. of 15% potassium ferrocyanide solution. The mixture, after addition of 1 c.c. of 30% zinc acetate solution, is diluted to 200 c.c. and filtered. To 150 c.c. of the filtrate are added 5 c.c. of nitric acid and 10 c.c. of 0.1N-silver nitrate. The mixture is again filtered, and the filtrate (150 c.c.) after addition of 1 c.c. of ferric alum solution, is titrated with ammonium thiocyanate.

J. R. NICHOLLS.

Coconut oil in butter. H. ATKINSON and A. AZADIAN (Ann. Falsif., 1927, 20, 593—602).—The methods which have been suggested for determining coconut oil in butter are reviewed, and a method is proposed which consists in obtaining "the insoluble silver number" of the fatty acids soluble in water. The neutralised solution after determination of the Reichert value is slightly acidified by the addition of 2 drops of 0.1N-sulphuric acid, 0.1N-silver nitrate solution (10 c.c.) is added, and the solution after diluting to 200 c.c. is kept for 15 min. with occasional shaking. The filtered solution (110 c.c.) is titrated by Volhard's method with 0.1N-potassium thiocyanate. The difference between the titration and a blank is due to insoluble silver salts, and, after correcting for dilution, is expressed as a percentage of the Reichert value. Any increase of the insoluble silver number suggests admixture of coconut or palm-kernel oils; the addition of animal or other fats with a negligible Reichert value, whilst diminishing this value for the butter, has no appreciable effect on the insoluble silver number. Values so obtained for numerous samples of butter from both cow's and buffalo's milk, with and without the addition of coconut and palm-kernel oils etc., are tabulated.

J. R. NICHOLLS.

Baking value of flour. BRAUN (Ann. Falsif., 1927, 20, 588—592).—A method is described for analysing a

small sample of wheat so that within 30 hrs. all the factors necessary for appraising the baking value of the flour produced from it can be obtained. The wheat (25 g.) is ground in a coffee mill to pass 100-mesh, the larger particles being re-ground until the residue weighs 7.5 g. The sieved product represents a 70% flour. The wet gluten is extracted from 10 g. in order to ascertain its quality and its hydration ratio. Then 1 g. of the flour is treated with 10 c.c. of water, collected on a filter, and washed, the nitrogen being determined on the insoluble material; the value obtained, which corresponds to the gluten nitrogen, when multiplied by 6.25, gives the dry gluten. The gliadin is dissolved by treating 5 g. of the flour with 50 c.c. of 0.5% solution of potassium bicarbonate in 70% alcohol for 24 hrs. After filtering, the gliadin nitrogen is determined on 20 c.c. of the filtrate. The moisture of the flour is determined on 1 g.

J. R. NICHOLLS.

Compounds of ammonia and carbon dioxide (salts of hartshorn); ammonium bicarbonate as a raiser in baking. T. PAUL [with M. LANDAUER and F. KRÜGER] (*Z. angew. Chem.*, 1927, 40, 1539—1548).—Five solid compounds of ammonia, carbon dioxide, and water (exclusive of urea) are known with ammonia content ranging from 21.55% to 43.64%. Increase of ammonia content in aqueous solution results in a decrease of the partial pressure of the two other components, especially of carbon dioxide. In the vapour phase at 100° the compounds are largely dissociated, but molecules of ammonium bicarbonate exist. Baking tests with the different solid salts in the proportion of 7.5 g. of salt to 500 g. of flour showed that with increasing proportions of ammonia the lightness of the product (vol./wt.) decreased. The proportion of residual ammonia in the loaf or cake increased somewhat with the ammonia content of the salt. It was further found that the use of ammonia solution in place of water (without any raiser) gave a denser product than with water alone, and it was concluded that ammonia has an actual adverse effect on the raising process. Ammonium bicarbonate is therefore the best medium for the purpose. "Salts of hartshorn," prepared by the old method of distillation of animal refuse, were found to contain 28—36% NH_3 according to the conditions of resublimation. The double salt $\text{NH}_4\text{HCO}_3 \cdot \text{NH}_2 \cdot \text{CO}_2\text{NH}_4$ and ammonium carbonate are unstable, and trade samples proved to be of irregular composition, whilst the bicarbonate is much more stable. It is therefore claimed that the designation "salts of hartshorn" (Hirschhornsalz) should not be limited to products of high ammonia content, as no definite line can be drawn, and for its principal application the bicarbonate is the most efficient form.

C. IRWIN.

Chemical composition of certain foods. L. SETTİMİ (*Atti R. Accad. Lincei*, 1927, [vi], 6, 315—318).—Results of analysis are given for a number of samples of the following foods, in common use in Italy: tinned meat of various ages, fresh lean beef, cheese, stockfish, lean bacon, and dried mushrooms (*Boletus edulis*).

T. H. POPE.

Nutritive value of the nitrogenous substances obtained from certain kinds of preserved foods.

Experimental researches on white rats. S. BAGLIONI and L. SETTİMİ (*Atti R. Accad. Lincei*, 1927, [vi], 6, 377—383).—The soluble proteins from certain common Italian foodstuffs (cheese, stockfish, dried mushrooms) and the insoluble proteins from cheese, stockfish, and casein not only maintain the nitrogen balance in the organism of white rats, but also effect a storage of nitrogen and an increase in body weight. The insoluble proteins from mushrooms are not able to do this.

O. J. WALKER.

Detection [in sausage] of dyeing with the colouring matter of sandal wood. L. SOEP (*Analyst*, 1927, 52, 696—699, and *Chem. Weekblad*, 1927, 24, 624—625).—To detect sandal wood dye in sausage, 50 g. of sausage are boiled with absolute alcohol, the mixture is cooled and filtered, and the filtrate examined spectroscopically. Uncoloured sausage extract shows only very little absorption at 0.59 μ , 0.54 μ , and 0.44 μ , but the coloured extract shows three distinct bands. The lead salt of santalic acid is then precipitated by basic lead acetate, and very small quantities give the precipitate a violet tinge. The precipitate is then collected, washed with hot alcohol, suspended in 50 c.c. of *N*-sulphuric acid, and again collected on a filter. The filtrate shows the three typical bands in the spectroscope. The santalic acid is transferred to ether and the ethereal solution evaporated, the residue dissolved in dilute acetic acid, and tested with hydrogen peroxide for the formation of colourless needles in addition to an amorphous substance, and with potassium nitrite for formation of a brown precipitate and yellow solution. Finally, a confirmatory microscopical examination is made. An alcoholic solution of santalic acid dyes woollen fibre orange to pink in presence of bisulphate, and produces a dirty violet colour on fibre mordanted with ferric chloride with no bisulphate.

D. G. HEWER.

PATENTS.

Sterilising, ageing, and bleaching of flour, meal, etc. E. STAUDT (B.P. 263,760, 9.11.26. Switz., 23.12.25).—The flour etc. is treated with the minimum quantity of halogen oxides, e.g., 1—2 g. of chlorine dioxide to 100 kg. of flour, suitably diluted with an inert gas, powdered silica gel, or a solvent such as water or oil.

F. R. ENNOS.

Extraction of pectin from fruit. H. T. LEO (U.S.P. 1,654,131, 27.12.27. Appl., 7.2.27).—Citrus fruit is subjected to pressure to extract the juice, and the rind, after slicing, is treated with strong alcohol in order to render inactive the enzymes present and to inhibit the action of the acid juices on the pectin. The alcohol is then removed, and the pectin is dissolved out from the rind with a hot weakly acid solution and concentrated to a definite gel standard.

F. R. ENNOS.

Manufacture of powdered or granular jellifying manufacture. H. T. LEO (U.S.P. 1,655,398, 3.1.28. Appl., 12.7.21. Renewed 6.5.27).—A solution of pectin in boiling water is prepared and, after the addition, preferably first, of the sugar and then of a fruit acid, e.g., tartaric or citric acid, the product is at once dried by atomisation.

F. R. ENNOS.

Apparatus for treating chocolate. BAKER PERKINS, LTD., W. E. PRESCOTT, J. P. BUNCE, and ANO.

ÉTABL. A. SAVY JEANJEAN & CIE. SOC. ANON. (B.P. 283,716, 17.12.26 and 13.6.27).

Method and apparatus for treating crystallised margarine mass. C. HILDEBRANDT-SØRENSEN (B.P. 267,928, 15.3.27. Denm., 16.3.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of theobromine. E. A. MAUERSBERGER (Chem.-Ztg., 1928, 52, 89—90).—Theobromine is extracted from the waste products of cocoa and chocolate manufacture by either alcoholic or aqueous extraction, the material freed from fat and dried containing 0.8—3.5% of the alkaloid. It is ground, mixed with dry slaked lime, and stirred with water. Combined theobromine is liberated and ammonia and methylamine are evolved. The older method of extraction with 80% alcohol at 60—70° is now uneconomic owing to the excessive loss of solvent involved. The author employs 35% alcohol at 50°, using a filter press in which the cake is washed with water. The washed cake is free from theobromine and contains only 3—8% of alcohol, which is recovered from a dryer. The filtrate is distilled in iron stills until free from alcohol, and then concentrated and neutralised in enamelled pans. Theobromine separates with but little formation of resin. Processes of extraction with hot or cold water involve either concentrating the filtrate until theobromine mixed with albuminous substances separates, or rendering it alkaline and allowing it to settle. A higher filtration pressure is needed, and the wear on filter cloths is greater. Crude theobromine is freed from albumin by converting it into the calcium compound, and from colouring matter by treatment with soda. C. IRWIN.

The DM1 and DM2 indices and the Warsaw Congress. F. DE MYTTENAERE (J. Pharm. Chim., 1928, [viii], 7, 14—25).—The de Myttenaere indices for the toxicities of arsenobenzene preparations were adversely criticised at the Warsaw Congress. Experiments are described showing that the DM index has a precise chemical significance, depending on the amount of arsenobenzene oxidised to arsenobenzene oxide, and that these indices do afford information regarding the composition of commercial arsenobenzene preparations; a case is cited where a sulphoxylate compound of diaminoarsenobenzene contained a considerable quantity of a non-nitrogenous impurity along with some disulphoxylated compound. The DM index very often serves to correct erroneous results obtained for the toxicity of an arsenobenzene preparation by *in vivo* experiments. The latter are not as satisfactory as would be desired. Of two perfectly satisfactory preparations one was toxic to rabbits but non-toxic to mice, whereas with the second the reverse was true.

S. COFFEY.

Determination of impurities in crude camphor.

I. Determination of water and solid matter. S. YAMADA and T. KOSHITAKA (J. Soc. Chem. Ind. Japan, 1927, 30, 356—359).—For the determination of water in crude camphor by distillation with volatile solvents, a device which is a modified form of Normann's apparatus (B., 1925, 512) was used. It is claimed that adhesion of condensed water on the wall of the still-head is com-

pletely avoided by the use of a still-head provided with a built-in reflux condenser having an inverted part at its end. The use of toluene or xylene instead of benzene is also recommended. More accurate results were obtained than by the usual method of centrifugal separation. Y. TOMODA.

Californian lemon and orange oils. L. BONACCORSI (Boll. Uff. R. Staz. Sperim. Ind. Essenze, 1927, 2, 1—3).—A sample of pale yellow Californian lemon oil had d^{15} 0.8580, α^{15} + 63.8°, n^{20} 1.4709, aldehydes (as citral) 2.35%, saponif. value 9.33, fixed residue (water-bath) 2.75% having saponif. value 193.9, solubility in 90% alcohol 1 : 7. A sample of Californian orange oil gave d^{15} 0.8518, α^{15} + 98.5°, n^{20} 1.4693, aldehydes (as citral) 2.62%, saponif. value 4.58, fixed residue (water-bath) 3.46% having saponif. value 160.91, solubility in 90% alcohol (turbid) 1 : 8.

T. H. POPE.

Constituents of Indian essential oils. XXII.

Essential oil from flower heads of *Cymbopogon coloratus*, Stapf. P. P. PILLAY, B. S. RAO, and J. L. SIMONSEN (J.S.C.I., 1928, 47, 52—54 r).—Oil from the flower heads of *C. coloratus*, Stapf., grown in the North Arcot district had d_{20}^{30} 0.9183, n_{20}^{30} 1.4819, $[\alpha]_D^{30}$ —20.7°, acid value 2.3, sap. val. 39.8, sap. val. after acetylation 111.2, aldehydes (sodium bisulphite) nil. The principal constituents found were *l*-camphene (15%), *l*-limonene (7%), camphor (?) (trace), *l*-borneol (8%), geraniol (10%), sesquiterpene oxide (?) (2—3%), sesquiterpenes (35%), sesquiterpene alcohols (8%). The absence of citral is noteworthy (cf. Bull. Imp. Inst., 1912, 10, 670; Proc. Chem. Soc., 1914, 30, 10).

Characters of essences of aromatic plants cultivated in Calabria. L. BONACCORSI (Boll. Uff. R. Staz. Sperim. Ind. Essenze, 1927, 2, 4—5).—The flower-heads (leaves) of *Majorana hortensis* gave 0.48 (0.43)% of pale yellow essential oil having d^{15} 0.8912 (0.8868), α + 7.2° (9°), acid value 0.28 (0.56), saponif. value 13.07 (11.20), saponif. value after acetylation 64.72 (73.77), carbonyl compounds (as citral) 1.67 (2.03)%, solubility in 82.5% alcohol 1 : 2.2 (1 : 1). The essence of *Thymus citriodorus* gave d^{15} 0.9020, α + 3.6°, acid value 4.48, saponif. value 30.01, carbonyl compounds (as citral) 13.50, phenols trace; soluble in 2 vols. of 70% alcohol. The essence of *Salvia trilobica* gave d^{15} 0.9200, α + 10.4°, acid value 1.68, saponif. value 8.66, saponif. value after acetylation 50.38, carbonyl compounds (as citral) 5.84; soluble in 1 vol. of 80% alcohol. Oil of *Pelargonium radula*, Ait (?) cultivated on the plain (hill) gave d^{15} 0.8970 (0.8966), α —9° (—9.2°), n^{20} 1.4639 (1.4643), acid value 13.44 (12.30), saponif. value 62.71 (70), saponif. value after acetylation 277.81 (260.32), esters (as geranyl tiglate) 26.43 (29.50)%, total alcohols (as geraniol) 76.40 (72.12), free alcohols 59.20 (52.88), citronellol 36.70 (33.36), solubility in 70% alcohol 1 : 2.1 (1 : 2.7).

T. H. POPE.

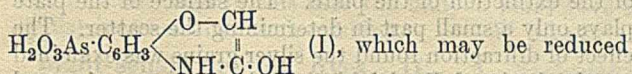
Australian sandal woods and their essences. E. PERROT (Bull. Sci. Pharmacol., 1927, 34, 609—640).—The botanical and histological characteristics of *Santalum spicatum* A. DC. (*Eucarya spicata*) and *Santalum lanceolatum* are described and the chemical and physical

constants of the oils derived from them are compared. From a review of many published values for these constants the following values for the two oils are derived: *S. spicatum*, d_{15}^{20} 0.968—0.972, n 1.498—1.512, α -3° to -9° , alcohols (as santalol) 90—96%, solubility in 70% alcohol, 1 in 3 to 6 vols.; *S. lanceolatum*, d_{15}^{20} 0.968—0.973, n 1.498—1.512, α -30° to -40° , alcohols 90—95%, solubility in 70% alcohol, 1 in 3 to 6 vols. Australian sandalwood oil is produced almost exclusively from *S. spicatum*, and in therapeutic action is not inferior to Mysore oil. E. H. SHARPLES.

Zinc oxide for ointments. DÖRZBACH.—See VII.

PATENTS.

Manufacture of organic compounds of arsenic. G. NEWBERY, and MAY & BAKER, LTD. (B.P. 280,613, 8.7.26).—By the action of suitable reducing agents the arsenic acid derivatives of *o*-nitrophenoxyacetic acids are converted into arsenic acid, arsenoxide, or arseno-derivatives of 3-hydroxybenz-1:4-oxazines. 3-Nitro-4-carboxymethoxyphenylarsinic acid (Christiansen, A., 1922, i, 1203) is reduced by ferrous sulphate and sodium hydroxide to 3-hydroxybenz-1:4-oxazine-6-arsinic acid,



by sodium hyposulphite and magnesium chloride to the 6:6'-arseno compound. The corresponding 8-acetamido derivatives of these are prepared from the 6-acetamido-derivative of (I) obtained by interaction of chloroacetic acid and 2-nitro-6-acetamidophenol-4-arsinic acid (itself prepared by nitration). C. HOLLINS.

Production of aluminium salts of organic acids. H. JUNG (G.P. 444,734, 1.3.25).—Products of therapeutic value as intestinal disinfectants, especially against oxyuriasis, are prepared by treating water-soluble basic aluminium salts of acetic acid or its homologues with aromatic carboxylic acids. *E.g.*, basic aluminium propionate solution, prepared by the action of propionic acid upon a mixture of aluminium sulphate solution and calcium carbonate, is boiled with benzoic acid, when a basic salt with a benzoate:propionate ratio of 1:2 is precipitated, which is collected on a filter, washed, and dried at 100° . L. A. COLES.

Manufacture of a substance producing hypoglycæmia. H. E. DUBIN and H. B. CORBITT, ASSRS. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,653,452, 20.12.27. Appl., 23.8.23).—Cabbage is extracted with alcohol, and the solution formed is treated with charcoal, which is then extracted with glacial acetic acid. The acid is evaporated *in vacuo*, and the residue, dissolved in water, is then treated with dinitrosalicylic acid to precipitate the required product. T. S. WHEELER.

Production of santonin. SOTERIA G.M.B.H., CHEM. PHARM. FABR. (G.P. 444,850, 24.11.22. Addn. to G.P. 346,947; B., 1922, 521 A).—The leaves of indigenous species of *Artemisia*, *e.g.*, *Artemisia monogygna* from the Baltic coast, or *Artemisia maritima*, are used for the production of santonin by the process described in the prior patent. L. A. COLES.

Production of a [stable] colloidal silver-tannin-albumin combination soluble in water. H. COHN and C. STEBERT (U.S.P. 1,653,087, 20.12.27. Appl., 28.12.26. Ger., 14.3.23).—Diacetyltannin dissolved in sodium carbonate solution is mixed with silver-albumin solution, and the liquid is evaporated *in vacuo* to yield a soluble product of therapeutic value. T. S. WHEELER.

Manufacture of derivatives of organic arsenic compounds. J. PFLEGER and A. ALBERT (U.S.P. 1,653,227, 20.12.27. Appl., 10.6.25. Ger., 19.6.23).—See B.P. 235,864; B., 1925, 738.

Manufacture of alkyl derivatives of cyclotri-methylenearypyrazolones. C. MANNICH (U.S.P. 1,657,544, 31.12.28. Appl., 29.11.26. Ger., 31.12.25).—See B.P. 263,773; B., 1927, 869.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photochemical] bleaching-out paradoxes. LÜPPO-CRAMER (Phot. Ind., 1927, 1099—1100).—It has been shown previously that the photochemical bleaching of pre-exposed silver bromide plates which have been treated with a solution of potassium iodide containing a safranin dye, and the desensitisation of silver iodide plates which have not been pre-exposed, decrease instead of increase with increase in concentration of the dye. Similar results have now been found with un-ripened diapositive silver bromide plates, free from all traces of soluble bromide, and without potassium iodide treatment. In certain cases the densities rise to a value higher than that due to the pre-exposure. The effect is probably due to a peculiarity of the emulsion. W. CLARK.

Anomalous destruction of nuclei. LÜPPO-CRAMER (Phot. Ind., 1927, 1286—1287).—Bleaching of the latent image with Phenosafranine first increases with dilution of the solution (from 1:20,000 to 1:80,000) and then begins to decrease. It is considered that the bleaching is due to the combined action of the Phenosafranine acting as oxidiser and the bromine remaining adsorbed on the grains and being set free by pre-exposure. There will be an optimum concentration of the dye at which these two results will show a maximum. The dye has a replacing effect on the bromine adsorbed to the grain and the extent of replacement will depend on the dye concentration. W. CLARK.

Denucleation and desensitisation. LÜPPO-CRAMER (Phot. Korr., 1927, 63, 362—363).—The action of a desensitiser must be on the latent image while it is in the act of being formed and not on the latent image already formed. The actual latent image probably contains silver in a condition too coarse for it to be acted on by the desensitiser. It is possible that the ripening nuclei would be attacked by the desensitisers, and that the action of a desensitiser on a plate treated with chromic acid to remove the ripening nuclei would be less than on a plate not so treated. Experiments show this to be the case. W. CLARK.

Experiments on hypersensitisation. K. JACOBSON (Phot. Ind., 1928, 56—62).—For the hypersensitisation of kinematograph film, a mixture of Pinacyanol

and Pinaflavol is not altogether satisfactory. If pyridine is added the results are better, but spots are liable to form. Best results are obtained with a mixture of Pinachrome and Pinachrome Violet and ammoniacal silver chloride. By appropriate pre-exposure after sensitisation the sensitivity may be still further increased. Hypersensitised film must be exposed and developed within 48 hrs. of sensitisation. W. CLARK.

Ageing phenomena of sensitised material. A. FUNGER (Phot. Ind., 1927, 1203—1204).—A general discussion of the keeping qualities of sensitive material, with special reference to edge fog. Colour sensitive materials do not keep so well as unsensitised emulsions because no bromide is added to them before coating. W. CLARK.

Sensitometry of reversal emulsions. I. LOBEL and J. LEFÈVRE (Bull. Soc. Franç. Phot., 1927, 14, 288—297).—The sensitometry of emulsions intended for the production of direct positives is considered from first principles. The gamma of the positive curve is equal to that of the negative curve, and is symmetrical to it about a line drawn parallel to the exposure axis through the ordinate $D/2$, where D is the maximum density developable with the emulsion. The position of the positive curve depends on the value of D alone. For correct positive reproduction, the emulsion should have a gamma-infinity equal to unity, and the solarisation density (*i.e.*, the density corresponding to an exposure at which solarisation commences, development being to gamma infinity) must be equal to the maximum density obtainable by complete reduction of all the silver halide. If it is desired to render pure whites, the exposure must be such that the value of It is equal to the exposure value at which solarisation commences. The speed of an emulsion for reversal is higher the thinner the emulsion coating. W. CLARK.

Error in sulphide-toned images. K. KIESER (Phot. Ind., 1927, 1257—1258).—In the ordinary process for sulphide toning, using ferricyanide and sulphide, care must be taken that all the ferrocyanide and ferricyanide is washed out of the emulsion before sulphiding. The sulphide will reduce any ferricyanide to ferrocyanide, and this, on keeping, and especially on exposure to light, decomposes with the formation of blue products which give markings on the prints. W. CLARK.

Tentative hypothesis of the latent image. II. A. P. H. TRIVELLI (Phot. J., 1928, 68, 67—72; cf. B., 1928, 107).—A probable elementary photo-electric cell on the surface of the silver halide grains of an emulsion is pictured, based on the conclusions of Wightman and Quirk (B., 1927, 268), and of Clark (*ibid.* 507), that the sensitising specks of high-speed emulsions as well as the latent image probably consist of both silver and silver sulphide. In the photo-electric system, silver is regarded as the cathode, silver sulphide as the anode, and silver bromide as a solid electrolyte. A mechanism of this cell in producing sensitivity is described. The theory is also applied to the explanation of the action of a flash exposure in increasing desensitisation by chromic acid. On the assumption that optical sensitising is due to an increase of photo-conductivity for longer wave-lengths, a mechanism is given of how dyes or silver sulphide could

act as optical sensitisers, based on the assumption that dye specks or finely-divided silver or silver sulphide lying in the field of the photo-electric cell $Ag-AgBr-Ag_2S$, increases its photo-conductivity to long wave-lengths above that of a similar cell without these substances present. W. CLARK.

Photographic density. J. C. VAN SELMS (Dissertation, Utrecht, 1927).—The extinction of a photographic plate is in general independent of the wave-length for ordinary development. With plates strongly exposed and developed for a very short time, the extinction increases with decreasing wave-length. A method is described for measuring in an absolute manner the intensity of light scattered at all angles by a photographic density. Measurements with blue and with red light showed that the intensity of light scattered at different angles is independent of the wave-length. The intensity of scattered light was small at big angles, and very high at small angles, but no relation could be deduced between intensity and angle. A curve is given showing the intensity of the scattered light at different angles as a function of the plate extinction. The intensity of light scattered at a given angle is in certain cases independent of the extinction of the plate. The surface of the plate plays only a small part in determining the scatter. The effect of diffraction round the silver grains was examined on the basis of Rayleigh's law, but the experimental results did not agree with the theoretical. The theory of Mie would probably be more applicable. The intensity of the scattered light could be explained if the variation of the refractive index of the gelatin in the neighbourhood of the silver grain were known. W. CLARK.

Errors produced in slow development with glycine. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1927, 14, 306—308).—The observations of Lobel and Lefèvre (B., 1927, 461), that as far as the final result is concerned, the actions of glycine and metol-quinol are identical, is confirmed. In the use of a dilute solution of glycine as a tank developer, however, troubles occur owing to the formation of a film of oxidised developer at the plate surface. This film is invisible and prevents the developer from wetting the plate at the parts of the sensitive surface where it is formed. The trouble can be partly but not entirely eliminated by wetting the emulsion surface before developing. W. CLARK.

Silver content of photographic layers. F. WEIGERT and F. LUHR (Naturwiss., 1927, 15, 788).—The authors have improved the electrometric titration of silver to such an extent that on titration with 0.0001N-potassium iodide it is possible to determine 0.001 mg. of silver in 20 c.c. of solution. Using this method, the amount of free silver in unexposed photographic plates was measured. In four different commercial plates the amount of silver varied from 1.88×10^{-4} to 2.80×10^{-4} mg./cm.² That this is ripening silver is proved by the fact that the amount of free silver increased with the time of ripening, the increase being greatest with silver chloride, less with silver bromide, and nil with silver iodide. When the plate is treated with persulphate, the amount of free silver decreased and the sensitivity

diminished, in accordance with the observations of Clark (B., 1927, 507). Silver sulphide is not attacked by persulphate under the conditions studied. W. CLARK.

Toning with selenium compounds. J. MILBAUER (Chem. Listy, 1927, 21, 509—515).—A large number of selenium compounds is examined from the point of view of their direct or indirect use in toning photographic images. The procedure recommended is the following: Prints or lantern slides are fully developed, fixed, and washed, and then treated in a bath containing 80 c.c. of a 1% solution of crystalline sodium sulphide and 10 c.c. of a 1% solution of sodium selenite or selenious acid. After 20 min. or less in this bath, intense brown tones are obtained with chlorobromide papers, or brown-violet tones with pure bromide papers. W. CLARK.

Fastness to washing of dyed silver bromide. LÜPPO-CRAMER (Phot. Korrr., 1927, 63, 193).—Sensitisers adsorbed to silver bromide in photographic plates can be removed by washing. Plates sensitised with Pinaflavol, Rhodamine B, or Isoquinoline Red lose their yellow sensitiveness on washing. With erythrosine there was no decrease, but an increase on prolonged washing, although in this case the undyed plate gave a similar increase after such washing. W. CLARK.

Light-sensitivity of dyes. II. A. STEIGMANN (Kolloid-Z., 1928, 44, 173—175).—Theories of the sensitisation of photochemical reactions by colouring matters are discussed. E. S. HEDGES.

PATENTS.

Obtaining photographic images. E. GAY (B.P. 283,274, 6.10.26. Addn. to B.P. 282,894; B., 1928, 173).—The light-sensitive diazo compounds of the prior patent are mixed with *O*-acyl derivatives of the phenols or naphthols used as coupling components in development. The phenols or naphthols are liberated by development with ammonia gas or a dilute alkaline bath, and a coloured positive image is obtained. Examples of suitable *O*-acyl derivatives are phenyl acetate, α -naphthyl benzenesulphonate, 2-acetamido-8-acetoxynaphthalene-6-sulphonic acid (from γ -acid), 1-acetamido-8-acetoxynaphthalene-3:6-disulphonic acid (from *H*-acid), and 1:8-diacetoxynaphthalene-3:6-disulphonic acid (from chromotrope acid); the last-mentioned gives with 2-diazo-1:4-dimethoxybenzene a violet-red image. C. HOLLINS.

Sensitising photographic films or plates. O. LOISA (G.P. 443,677, 25.1.25).—Sensitising in solutions of basic sensitisers is carried out for 10—30 min. with simultaneous cooling of the bath to below 15°, and drying the bathed material at below this temperature. Correct colour-tone rendering can be obtained without the use of a filter. W. CLARK.

Protective layers for photographic gelatin emulsions. P. G. and C. GLASER (F.P. 617,497, 14.6.26).—The emulsion is coated with a varnish consisting of a solution of celluloid and gum lac, to which is added a mixture of glacial acetic acid, ethyl acetate, and amyl acetate. W. CLARK.

Actinism-proof cellulose ester composition. J. H. CLEWELL, JUN., Assr. to E. I. DU PONT DE NEMOURS &

Co. (U.S.P. 1,647,435, 1.11.27. Appl., 7.1.24).—Transparent sheets of pyroxylin are coated on both sides with a colourless, transparent, oil-resin varnish, e.g., a copal-linseed oil mixture, impervious to ultra-violet light.

T. S. WHEELER.

X-Ray photography. F. SIMON (B.P. 276,678, 25.8.27. Ger., 25.8.26).—In *X*-ray photography, the difference in wave-length of the rays penetrating the object to be photographed is registered in visible colour tints by interposing between the object and the sensitive material a screen, the elements of which are formed of different transparent material of different extinction coefficient for *X*-rays, and at the same time rendered distinct by being coloured. A suitable screen is composed of starch grains of three kinds, of which the first are saturated with a lead salt, the second with a copper salt, and the third with an aluminium salt. Each of the kinds of starch is arbitrarily dyed with a different colour. W. CLARK.

Process of photogravure. E. STRAUB, Assr. to J. GRIFFITHS (U.S.P. 1,656,843, 24.1.28. Appl., 26.3.26. Fr., 10.2.26).—A plate is coated with a thin layer of dichromated gum arabic, dried, exposed through the object to be reproduced, rubbed with a solution of glycerin, water, and acetic acid until the image appears, and then washed in a solution of alcohol and acetic acid. A quick-drying varnish is then rubbed in to penetrate the bare parts of the plate, the plate is washed, dried, and inked with printing ink, after which the light-affected layer is removed with hydrochloric acid and the plate is etched. W. CLARK.

Compositions containing derivatives of carbohydrates [used as photographic supports]. H. J. HANDS, and SPICERS, LTD. (B.P. 282,980, 29.12.26).—Sheet material for forming photographic supports consists of compositions containing derivatives of carbohydrates having the empirical formula $(C_6H_{10}O_5)_n$, in particular the ethers and esters of cellulose and similar carbohydrates, in association with a relatively pure cellulose in a finely-divided form, and an opaque white pigment. The composition may be used for preparing compound sheets according to B.P. 281,803 (B., 1928, 187). W. CLARK.

Manufacture of bodies of desensitising action. R. SCHULOFF, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,314, 20.12.27. Appl., 10.12.26. Ger., 11.12.25).—See B.P. 262,816; B., 1928, 69.

Photometer (B.P. 275,597).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Preparation of cellulose nitrate. BERL and BERKENFELD. **Decomposition of nitrocellulose.** RYAN and LANTZ.—See V.

PATENTS.

Initiating composition [for explosives]. E. VON HERZ (G.P. 443,551, 15.6.26).—A mixture of lead thiocyanate and lead azide is used. The mixture, which may be in the form of a mixed salt obtained by simultaneous precipitation, is more sensitive than lead azide alone. S. BINNING.

Composition producing yellow smoke. G. BOCK (G.P. 443,901, 5.2.26).—A mixture of powdered pitch and powdered sodium salts (*e.g.*, borax) is recommended. The volume of smoke may be further increased by addition of powdered glue. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Ferric salts as coagulants for activated sludge prior to filtration. F. W. MOHLMAN and J. R. PALMER (Eng. News-Rec., 1928, 100, 147—150).—Laboratory and large-scale experiments at Chicago indicate that ferric chloride is the most suitable reagent for conditioning activated sludge prior to filtration. The use of a mixture of ferric sulphate and ferric chloride, produced by chlorinating ferrous sulphate, is suggested as a means of overcoming difficulty in obtaining adequate supplies of ferric chloride at a reasonable cost. C. JEPSON.

Dissolved oxygen absorption-time relation of activated sludge effluents. P. GAUNT and W. E. ABBOTT (J.S.C.I., 1928, 47, 14—16 T).—When diluted sewage or polluted water is incubated at 21°, the rate of dissolved oxygen absorption during the carbonaceous fermentation, lasting from 5 to 19 days, is represented by Phelps' equation, although the constant *K* has a more widely varying value than was previously recognised. With reasonably purified effluents, nitrification, commencing usually before the third day, causes rapid absorption of oxygen, and this equation does not then apply. A new equation is suggested to meet these conditions. The rate of acceleration of the absorption due to nitrification is shown, on certain simple assumptions, to be such as might be anticipated from the action of nitrite-producing organisms developing by binary fission. The time elapsing between successive fissions is shown to vary from one to three days. C. JEPSON.

Determination of dissolved gases in water. H. RICHTER (Chem.-Ztg., 1928, 52, 47).—Samples of water from pressure boilers should be cooled before releasing the pressure. Boiler water at 138° and 45 atm. had after releasing the pressure a temperature of 96° and showed no trace of dissolved oxygen. A similar sample, cooled to 30° before expansion, showed 0.2—0.3 mg. of oxygen per litre. C. HOLLINS.

PATENTS.

Purification and sterilisation of contaminated air by means of ozone. A. WOLFF (G.P. 445,502, 30.12.24).—The air mixed with ozone is brought in contact with aqueous solutions, atomised if necessary, containing acids or acid salts and catalytic material, *e.g.*, ferric chloride, until ozone is no longer present. L. A. COLES.

Apparatus for automatically determining the amount of gas existing in water, and the automatic control of chemical operations. H. S. HATFIELD (B.P. 282,487, 20.9.26).—The apparatus refers particularly to the determination of temporary hardness in water. When a measured quantity of hard water is boiled the amount of carbon dioxide evolved by the decomposition of bicarbonates will be large in comparison with that merely dissolved in the water. The evolved gases are separated from the steam in a con-

denser, and then measured before and after passing over an absorbent of carbon dioxide. The measuring bells are connected to the outer wheels of a differential gear, and the inner wheel operates a pointer and/or electrical control arm by which means the hardness of the water may be indicated and/or caused to control the flow of a softening reagent. B. M. VENABLES.

Preventing the formation of incrustation or scale in boilers caused by salts of silicic acid. J. OSTERTAG (U.S.P. 1,657,443, 24.1.28. Appl., 17.6.27. Ger., 26.11.25).—The boiler is "blown off" and make-up water which has been treated so as to provide an excess of sodium carbonate over sodium silicate in the water in the boiler is added. The amount of blow-off and the amount and degree of alkalinity of the make-up water are calculated to maintain the total concentration below a predetermined maximum. M. E. NOTTAGE.

Prevention and removal of incrustation in boilers.

J. BILLWILLER (B.P. 281,361, 25.8.26. Addn. to B.P. 257,915; B., 1928, 72).—Fur and incrustation in boilers may be removed, or their formation prevented, by addition to the boiler or feed water of resin acids which have been slightly oxidised or polymerised by heating them above their m.p.; or resin acids containing "bound" water, obtained, *e.g.*, by hydrolysis of resins with alkali etc. and acidification, may be used. Gums may be added to the acids. B. FULLMAN.

Water-softening system. G. S. APELDORN (U.S.P. 1,646,270, 18.10.27. Appl., 26.6.25).—A zeolite water-softening plant comprising in particular a zeolite container connected at a relatively low point, by piping and valve, with the bottom of a second container of small capacity. The latter, which serves as a regenerating tank, is provided with means at the base for retaining solid reagent, and at the upper portion for supplying a stream of water to the reagent and for automatically cutting off the supply when the level of liquid in the vessel reaches a given height. When regeneration of the base-exchange media is necessary the zeolite container is drained, the required amount of salt is added to the second container, the two are placed in communication, and water is then supplied to the small vessel until the liquid reaches the desired level and the levels of liquid in the two containers are equalised. W. T. LOCKETT.

Purification of water. W. H. GREEN and A. S. BEHRMAN, Assrs. to GEN. ZEOLITE CO. (U.S.P. 1,653,272, 20.12.27. Appl., 9.12.21).—The hardening constituents of water are precipitated by the addition of lime, freshly precipitated sludge, and aluminium sulphate. H. ROYAL-DAWSON.

Purification of hard water by base-exchanging bodies. E. M. E. FRÉCHOU (U.S.P. 1,657,822, 31.1.28. Appl., 24.9.25. Fr., 19.12.24).—See B.P. 244,713; B., 1926, 390.

Apparatus for adding reagents to water. J. F. JONES (B.P. 282,193, 1.11.26).

Analysis of liquids (B.P. 282,168 and 282,170).—See I.

Base-exchange substance (U.S.P. 1,656,604).—See VII.