# BRITISH CHEMICAL ABSTRACTS

# **B.**—APPLIED CHEMISTRY

NOVEMBER 26, 1926.

# I.-GENERAL; PLANT; MACHINERY.

Apparatus for the automatic regulation of furnaces. VERNEY (Compt. rend., 1926, 183, 561-562) .---By means of two insulated copper tubes, mounted on the same axis in the form of a horizontal arch, contact may be made with a strip of malleable copper previously bent in the shape of the time-temperature curve required. The vertical stand which supports the copper strip is moved regularly in the direction of its plane, and the strip thus makes contact with one of the copper tubes. An endless screw which is attached to the torsion head of a galvanometer and is actuated by means of an electric motor, brings back the insulated portion of the arched contact on to the copper strip again. The galvanometer thus indicates the temperature corresponding to the ordinate of the curve, and sets in action a relay which adjusts the temperature of the furnace. J. GRANT.

Recent work on the measurement and industrial importance of particle size. J. PARRISH (J. Oil and Colour Chem. Assoc., 1926, 9, 252–259).—A general survey is given of the literature on particle size, including methods of its measurement and its importance in relation to the paint, rubber, paper, and cement industries. D. F. Twiss.

Value of a direct measurement method for particle size determination. H. GREEN (J. Oil and Colour Chem. Assoc., 1926, 9, 226—234).—A direct or photomicrographic method for the measurement of particle size has the value that it gives a "particle-distribution" curve showing the relative frequency of occurrence of various diameters. Such a method also does not always involve the necessity to assume that the particles are cubical or spherical. The particle distribution curve also enables a calculation as to the particular average diameter needed for special work. With certain clays and the so-called inert powders especial care is required to ensure that the sample examined is truly representative. D. F. Twiss.

Apparatus for the separation of grit and coarse particles from fine powders. G. GALLIE and B. D. PORRITT (J. Oil and Colour Chem. Assoc., 1926, 9, 235 -238).—The apparatus consists of a metal funnel terminating in a removable cup, 25 mm. in diameter, the bottom of which is formed of wire gauze of any desired mesh. A weighed quantity of the powder is introduced into the funnel, and water (filtered through metal gauze) is added until the funnel is approximately half filled. A jet of filtered water issuing from a tube reaching almost to the bottom of the funnel, and arranged so that its distance from the gauze bottom is adjustable, forces the particles through the sieve and disintegrates loose agglomerates. The residue in the cup is finally washed, the cup being then removed and dried at 100° preparatory to weighing. D. F. Twiss.

Apparatus for determination of specific gravity of liquids. A. N. N. TRENITÉ (Chem.

Weekblad, 1926, 23, 465).—The apparatus consists of a glass tube, A B C D E, 12 mm. in diameter, bent through 180° at B, C, and D so as to form two U-tubes, A B C, E D C, connected at C; stop-cocks are fused on at B, C, and D. Each of the four vertical limbs so formed is provided with a millimetre scale. Water is introduced through the limb, A, and the fluid to be tested through the limb, E, thus enclosing air in the inner limbs and the bend, C. The differences in height of the two water levels in A B C and of the two fluid levels in E D C give the specific gravity directly.



# S. I. LEVY. PATENTS.

**Open-fire kiln.** P. A. MEEHAN, Assr. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,596,502, 17.8.26. Appl., 21.8.24).—Door-locking devices for the exit end of a continuous tunnel kiln are described.

#### R. B. CLARKE.

Cylindrical dryer for low-temperature drying. J. BRABAEK (G.P. 431,564, 3.10.22).—Materials for which high temperatures are unsuitable are dried by a dry air blast in a cylinder around the upper surface of which are a large number of nozzles arranged radially, and connected with an air pressure main.

## W. G. CAREY.

Heating, humidifying, and evaporating apparatus. R. J. KRAUSE (E.P. 257,468, 10.12.25).—The apparatus comprises a number of rotating concentric drums supported on a disc-like driver; alternate drums project further from the driver than the others, and the projecting drums have spaces left between them and the driver, so that when material is supplied to the drum nearest the axis it will spread over the interior surfaces of all the drums in succession, being held inside each drum by centrifugal force. Heating means such as electric resistances may be applied to the exterior surfaces of the drums. B. M. VENABLES.

Centrifugal evaporation. M. J. KERMER (U.S.P. 1,598,282, 31.8.26. Appl., 16.1.25).—The stationary housing round a rotating cylinder forms a steam chamber, and is provided with an annular chamber which contains fluid as a seal. S. BINNING.

Centrifugal separators. C. S. GARLAND, J. W. HINCHLEY, and C. S. WATSON (E.P. 255,914, 4.2.25).—

A centrifugal separator designed for the continuous discharge of both separated constituents, which may be a solid and a liquid or heavier and lighter liquids, is constructed with two conical bowls, 5, 6, with their bases together (see Fig. 1). The lower bowl is rotated by a rigid connexion to the shaft and the upper bowl by a resilient connexion combined with a screw thread on the shaft so that when the upper bowl lags behind the lower the common bases of the bowls separate slightly



and allow the collected solid matter (or heavier liquid) to fly out. The lag of the upper bowl is produced by friction between a fixed surface, such as a flanged sleeve, 11, and the accumulated solid matter. The lighter constituent discharges over an internal rim in the usual way. Alternatively the upper bowl, 5, may not rotate except slightly on a fixed screw thread (see Fig. 2); the whole of the upper bowl then forms the frictional surface, and is held in the closed position by fixed external springs, 43, easily accessible for adjustment.

#### B. M. VENABLES.

Centrifugal separating machines. R. A. STURGEON (E.P. 257,422, 1.10.25).—A centrifugal separator is provided with means for removing separated solid matter without stopping the rotation comprising a pair of pistons and a cap vertically fixed but rotating with the hollow vertical shaft of the machine. Embracing the pistons is a cylinder, also rotating, which can move vertically, and is divided into three compartments. The uppermost compartment forms the collecting surface for solids, and is closed when the cylinder is raised against the cap and opened for discharge of solids when lowered. The middle compartment is filled with water to raise the cylinder, the lowest is filled with water to lower it. The water for operation is supplied to the appropriate compartment under no particular pressure, the pressure for operation being obtained by the rotation of the B. M. VENABLES. apparatus.

Treatment of solid materials with liquid reagents. [Preparation of aluminium sulphate from bauxite etc.] H. N. SPICER, and DORR CO. (E.P. 257,643, 28.4.25). —A continuous process of leaching in a series of agitators which are so arranged that the coarser particles are retained sufficiently long so that they do not overflow until they have been reduced to fine particles by the action of the solvent. The strong solution is recovered, and the fine particles are washed by decantation in a series of thickeners. The process is particularly suitable for the preparation of aluminium sulphate solution of high concentration from bauxite and sulphuric acid. The bauxite, acid, and part of the overflow from the second thickener are mixed in the first agitator, the strong aluminium sulphate solution overflows from the first



thickener, and water is added to the last thickener. Instructions are given for calculating the size of the thickeners, viz.: For clear overflow, free falling rate of particles multiplied by area of thickener = volume rate of overflow. For thickest possible underflow, the necessary hindered settling period multiplied by volume rate of underflow = volume of thickener, which divided by the area already calculated gives the necessary height. Since heating increases the efficiency of the thickeners the incoming liquids or pulp may be heated externally to the thickeners, but not in them on account of convection currents. B. M. VENABLES.

Liquefaction and separation of gaseous mixtures. R. C. E. MEWES (E.P. 257,325, 25.5.25).—A mixture of liquefied gases such as air is introduced simultaneously into two rectifying stills, one of which operates under moderate pressure and the other at nearly atmospheric pressure. The low-boiling constituent obtained from a preliminary rectification in the pressure column is used both to heat the still of the atmospheric column and, after reduction of pressure, as washing liquid in the same column, together with a portion of the low-boiling liquid obtained from the bottom of the atmospheric column and that used to heat the still of the pressure column. The cold to make up losses is provided by low-boiling constituent (or air) supplied under high pressure to the still of the pressure column. B. M. VENABLES.

Refrigerating machines for absorption of ammonia or any other substance having the same properties. A. E. ANDERSON, G. MAIURI, and R. F. BOSSINI (E.P. 257,458, 21.11.25).—In an absorption refrigerating apparatus the inert carrier gas is caused to circulate by thermo-siphon effect, the flow side being heated in the boiler by vapour therefrom, or in a separate heater. The return is cooled in the condenser. Heat interchange may be provided between the gas arriving at and leaving the evaporator, also between the liquor to and from the boiler. B. M. VENABLES.

Purification of liquids by adsorption. P. W. PRUTZMAN and A. D. BENNISON, ASSTS. to General PETROLEUM CORP. (U.S.P. 1,598,254-6, 31.8.26. Appl., [A] 17.8.25, [B] 23.3.26, [C] 23.3.26).—Liquids are purified by treatment with the following finely divided substances: (A) natural magnesium silicate, (B) magnesium silicate after treatment with hydrochloric acid, or (c) magnesium silicate after treatment with sulphuric acid. S. BINNING.

Roll or cylinder mills for grinding paint and other substances. A., O., and W. BUHLER (BUHLER BROS) (E.P. 257,727, 18.8.25).-A number of rolls are arranged over each other, but the axes are not in the same vertical plane, and the circumferential distance between the lines of contact of adjacent rolls is less than one-third of the circumference of a roll. The velocity ratio between adjacent rolls lies between 3.2:1 and 2:1. With this arrangement the peripheral speed of the fastest roll may be higher than 750 ft./min., the ordinary limit for paint. Arrangements are described for very fine adjustment of the rolls, and feed guides are provided so that the material is spread over the whole length of the rolls, any excess working over the ends B. M. VENABLES. being caught in other guides.

Pulverising apparatus. R. J. TUGWOOD. From JEFFREY MANUF. Co. (E.P. 257,665, 4.6.25).—A disintegrator is provided with yielding hammer-pieces in which two holes are formed, and a pin passes through each hole connecting the hammer with the driving discs or spider. One hole is circular, the other is a curved slot, so that when the hammer hits an unbreakable obstruction it may turn aside. The hammers may be held in their operative position entirely by centrifugal force, or, in addition, a thin, brittle, and easily renewable piece of metal may be placed across the slotted hole to prevent the movement of the hammer.

#### B. M. VENABLES.

Furnaces. O. Y. IMRAY. From T. E. MURRAY (E.P. 258,760, 23.12.25).

Furnaces. J. Woods (E.P. 258,347, 17.6.25).

Bearings for centrifugal extractors. W. H. SUMBLING (E.P. 246,153, 14.1.26. Conv., 15.1.25).

Safety devices for centrifugal machines. T. BROADBENT & SONS, LTD., and H. BROADBENT (E.P. 258,789, 6.3.26).

Air filters. NATIONAL AIR FILTER Co., Assees. of H. E. BIRKHOLZ (E.P. 249,851, 16.3.26. Conv., 30.3.25).

Devices for separating liquids of different specific gravities. E. W. GREEN (E.P. 258,662, 24.6.25).

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

Application of X-rays to the laboratory jigtesting of coal. C. N. KEMP (Proc. S. Wales Inst. Eng., 1926, 42, 411-437).—A method is explained for testing in the laboratory samples of coal from certain aspects of its ash content. The apparatus consists essentially of a U-tube of square cross-section; near the bottom of one limb is a grid upon which, and nearly filling the limb, the sample of coal under test rests. The U-tube is nearly filled with water, to which rhythmic impulses are imparted by the action of compressed air at the top of the second limb, causing it to surge to and fro through the coal sample. The coal sample is observed by the passage through it of X-rays, by means of which the separation of the adventitious ash may be followed. Exhaustive separation of this matter may be effected. The relative depths of the coal (which shows light) and of the ash (which shows dark) give a key to the proportions of them in the raw coal. The test may be used to show the amount of dirt in the coal, and whether a sample of washed coal can be improved by further washing, or whether washing has been carried too far in that the dirt contains "clean coal." Hence it may serve to control washing processes and to determine the relative values of samples of coal. R. A. A. TAYLOR.

Volatile matter in coke. G. E. Foxwell (Gas World, 1926, 85, Coking Sect., 102-105).-From a survey of the experimental researches of Porter and Ovitz and of Burgess and Wheeler, the advisability of expelling the whole of the volatile matter from coke is discussed. Every 1% of volatile matter left in the coke corresponds to a deficiency of 4 therms in the gas per ton of coal carbonised. The expulsion of 1% of volatile matter requires 1 therm of heating gas and a further hour's heating in the oven. The by-products remaining in high-temperature coke are of no value. It is concluded that for coke-oven plants having no sale for their gas the last traces of gas are not worth the additional time, wear and tear necessary; for gas works, the therms in the gas are of more importance, consonant with the production of good coke. A. C. MONKHOUSE.

Coking capacity of coal. D. J. W. KREULEN (Chem. Weekblad, 1926, 23, 449—453; cf. B., 1925, 160). —Mixtures of the same coal reduced to different particle sizes with varying proportions of sand and of graphite were coked under exactly the same conditions, and the resistances to impact of the resulting coke examined. The resistance is greater the smaller the size of the particles of added indifferent material, and reaches a maximum in all cases with addition of about equal total surfaces of added material. The cokes containing sand show much lower resistances than those containing graphite. The proportion of non-coking coal which may be added to a coking coal to form a good coke may be increased by finer division of the former. S. I. LEVX.

Low temperature carbonisation : the economic side. O. J. PARKER (Proc. S. Wales Inst. Eng., 1926, 42, 403-410).—Attention is directed to the lowtemperature carbonisation of lower-grade non-coking coals. The aim is to provide a smokeless coke for domestic use, together with other remunerative products. Mere increase in the calorific value of the coke over that of the raw fuel does not in itself constitute grounds for treating the raw fuel, especially as in the higher grades the increase goes down in proportion. Combustibility and robustness of the product must be considered. Graduated heating up of the charge is advocated to enhance the yield and value of the tars, and to attain in them a partial fractional distillation. Briquetting is advocated for slacks, smalls, and washery slurries. Costs are quoted for a German (the Delkeskamp) plant meeting these requirements.

# R. A. A. TAYLOR.

Value of coal carbonisation products and its determination in the laboratory. R. GEIPERT (Gas-u. Wasserfach, 1926, 69, 861-862).—The coal sample (10 g.) is dropped, in the form of a briquette, through the vertical limb of a T-piece attached to the top of a silica tube placed vertically in a furnace maintained electrically at 1100°. The volatile carbonisation products pass out through the horizontal limb of the T-piece, through a short tube packed with glass wool to catch the tar, and the gas is collected over water in a graduated vessel. Three determinations should be made and can be completed within 1 hr., and the cal. value and composition of the united gas may be determined in the usual manner. From the value of the therms obtained, and of the coke and tar, the total value of these products per ton of coal is ascertained, and forms a basis for comparing different classes of coals. In determining the value of the coke, corrections are introduced for its ash content and mechanical quality, and account is taken, in the final valuation, of the moisture content of the original coal. W. T. K. BRAUNHOLTZ.

Steaming in continuous vertical retorts. M. BARASH (Industrial Chemist, 1926, 2, 351-355; cf. Fuel Res. Bd., Tech. Paper 8, B., 1923, 1163 A).—The history of the continuous vertical retort is reviewed with special reference to steaming. A series of tests is recorded in which the yield of gas per ton of coal carbonised was increased by additional steaming from 16,970 cu. ft. of calorific value 503 B.Th.U. per cub. ft., to 70,000 cub. ft. of calorific value of 350 B.Th.U. per cub. ft. All tests, including those in which the quality of the gas lay intermediate between these two values, gave results in agreement with theoretical calculations.

# S. PEXTON.

Relationship between the calorific value of a gas and either the oxygen necessary to burn it, or its products of combustion. H. FAHRENHEIM (Gas- u. Wasserfach, 1926, 69, 838-840).-Taking the water formed as steam, the volume of the products of combustion of unit volume of coal gas or coke-oven gas or mixtures of these bears a strikingly constant ratio to the gross calorific values of the respective gases. Owing to the condensation of the water after explosion, however, the volume of the products cannot be measured, but the oxygen used in the explosion bears similarly a ratio which is nearly constant to either the gross or nett calorific value. The amount of oxygen necessary to burn 100 c.c. of gas gives the gross calorific value when multiplied by 50.9, and the nett when multiplied by 45.85, these figures being the mean for a series of gases, and giving a result in kg.-cal. per cub. m. The greatest deviation from the calorific value calculated from the analysis is 1%. Extended to blast-furnace and producer gases not containing unsaturated hydrocarbons, similar relationships hold, the ratio of oxygen to nett calorific value being about 56.5; the deviations from one gas to another are greater, but the maximum error is only 1.2%. To find the gross calorific value, the nett has to be multiplied by a factor particular for each type of gas: blast-furnace gas, 1.03; producer gas, 1.05; blue water gas, 1.09. R. A. A. TAYLOR.

Gas purification. H. BLAKELEY (Gas J., 1926, 175, 478-480).-Purification of gas from hydrogen sulphide is reviewed from the plant manufacturer's point of view. In laying down new plant it is desirable for future developments, to provide two or three proportionately larger iron oxide purifiers than four smaller purifiers. When using purifiers with water lutes it is essential to prevent foul oxide from falling into the lutes and producing acidulated water which results in extensive corrosion of the side plates and covers. Also the valve system should be such that each purifier can be independently controlled and that any sequence of purifiers can be worked. The resistance of a purifying installation can be halved by dividing the oxide in each purifier into two shallow layers, and leading the gas from the intervening space through the two layers in parallel.

S. PEXTON.

Burkheiser process of gas purification. W. BURKHEISER (Gas- u. Wasserfach, 1926, 69, 765-771).-The various stages in the evolution of the process are described. In the latest form the gas, after removal of tar, passes into a rotary scrubber containing a suspension of iron hydroxide and free sulphur where the gas is purified from cyanogen and hydrogen sulphide. The mixture in the purifier is pumped to an enclosed stirrer where it is mixed with ammonium sulphide (concentrated gas liquor) and the sulphur is dissolved. The mixture is then transferred to a closed settling tank where the ammonium polysulphide and thiocyanate are separated. The ammonium polysulphide is decomposed to ammonium sulphide and free sulphur, which latter is burnt to sulphur dioxide, as in the old process, and used for the recovery of the ammonia in the gas as ammonium sulphite. The iron sulphide from the settling tank is pumped to a rotary scrubber where, after admixture with freshly revivified material to remove any polysulphide, it is revivified with air and subsequently pumped back to the purifier. This last stage of development, viz., "wet" purification and "wet" revivification, awaits large-scale experiment. A. C. MONKHOUSE.

Refrigeration of gas for the removal of naphthalene, water and ammonia. F. LENZE and RETTEN-MAIER (Gas- u. Wasserfach, 1926, 69, 689—691).—Foul gas leaving a works condenser at 10—30° was further cooled in a refrigerating apparatus to 0°, where the naphthalene content was reduced from 13—26 to  $1\cdot7$ —  $2\cdot6$  grains per 100 cub. ft., and the ammonia content reduced from 75—175 to 30—45 grains per 100 cub. ft. with no measurable loss in the benzol content or the calorific value of the gas. The condensate from the refrigerator contained  $9\cdot6\%$  of ammonia. The partial pressure of ammonia in equilibrium with ammonium carbonate solutions of varying strengths and also the freezing points of ammonium carbonate solutions were determined. S. PEXTON.

Direct recovery of pure sulphur from gases containing hydrogen sulphide by means of solutions of copper salts. F. FISCHER and P. DILTHEY (Brennstoff-Chem., 1926, 7, 300).—If a gas containing hydrogen sulphide is passed through a solution of cupric chloride Th in brine at a slightly elevated temperature a yellow crust of almost pure sulphur is obtained. The purity of the product increases with increasing concentration of the cupric chloride and with increasing temperature, suitable conditions being a 10% solution at 70—80°. The th cuprous chloride simultaneously formed is converted back into cupric chloride by blowing air through the in solution, oxidation being favoured by increased tempera-

ture and retarded by increased concentration. W. T. K. BRAUNHOLTZ. Reduction and hydrogenation of carbon mon-

oxide. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1926, 7, 299—300).—The formation of a small quantity of "synthol"-like products under certain conditions from water gas at atmospheric pressure, recorded by Elvins and Nash (cf. A., 1926, 917), had been previously observed by the present authors, though the production of hydrocarbons (cf. B., 1926, 475) can be so conducted as to eliminate this reaction, which, whilst proceeding to a considerable extent under pressure, is here only a subsidiary one. The theory that unstable carbides are intermediate in the formation of higher hydrocarbons at atmospheric pressure is reaffirmed.

# W. T. K. BRAUNHOLTZ.

Chemical changes in the "Berginisation" of coal. A. SPILKER and K. ZERBE (Z. angew. Chem., 1926, 39, 1138-1143).-Treatment of naphthalene in an autoclave with hydrogen at 100 atm. (initial) and 470° results in transformation of about one-third into hydronaphthalenes, mostly tetralin. At lower temperatures and pressures less is attacked ; at higher temperatures, hydrobenzenes and other decomposition products are formed. Similar results are obtained in presence of aluminium chloride, the yield of higher-boiling products being somewhat greater. Hydrogenation of anthracene at 450° and an initial pressure of 75-80 atm. yields a completely liquid product of mixed hydroanthracenes. Phenanthrene and acenaphthene under the same conditions yield 30 and 90%, respectively, of hydrogenated liquid mixtures. Fluorene yields 30% of liquid hydrofluorenes when treated at 465°; carbazole remains unattacked under the same conditions. S. I. LEVY.

Conversion of lignin into humic acid and humin in the formation of humus and lignite from conifer remains. W. GROSSKOPF (Brennstoff-Chem., 1926, 7, 293-299).-A comparison of the chemical composition of successive stages in the conversion of green pine needles, through beds of dead needles and dry peat, into lignite. Pentosans were determined by conversion into furfuryl alcohol, and cellulose by chlorination. By extraction with cold, 42% hydrochloric acid lignin and humus remain undissolved, and of these the humus alone remains undissolved on treatment with acetyl bromide. In the series from fresh pine needles to lignite the content of pentosans shows a gradual but steady decrease, and the same appears to apply to hexosans; no appreciable formation of oxycellulose occurs during the production of humus. The content of cellulose also gradually diminishes, the substance being changed into a slack material having the properties of neither oxycellulose, humus, nor coal. Cellulose is absent from lignite. The rapid increase in the content of humus corresponds roughly with the rapid decrease in that of lignin, and indicates the conversion of the latter into the former, the change taking place in two stages, viz., the formation of humic acid reaching its maximum in dry peat, and the subsequent formation of humin. It is considered that humus beds composed of conifer remains constituted, in the Tertiary period, the beginnings of present-day lignites. W. T. K. BRAUNHOLTZ.

Carbonisation of oil shale in rotary retort. E. FLEISCHMANN (Brennstoff-Chem., 1926, 7, 229-232).-A Thyssen rotary retort of 80 tons per day capacity was heated by two rows each of 10 double-heating shafts, fired with a mixture of shale coke and shale. The ash is used in the cement manufacture. The shale to be carbonised passes through the retort in 2 hrs. The gases are scrubbed with heavy oil, which extracts the heavier fractions, and then cooled to 100°, purified with milk of lime, and debenzinised by a Linde refrigerator. The heat value of the shale was 100 cal. and of the shale coke 500 cal. per kg. One ton of shale yielded in practice 36.90 cub. m. of gas and 30.11 kg. of oil. The heat balance of the process and analyses of the products are given. H. MOORE.

Cracking petroleum by the Dubbs process. F. MEVER (Brennstoff-Chem., 1926, 7, 232-235; cf. Dunstan and Pitkethly, B., 1925, 60).—A description of the Dubbs cracking process and comparison of the advantages of different methods of cracking. H. MOORE.

Diesel oil. H. KÜHL (Petroleum, 1926, 22, 977—983). -The water content of Diesel oils should not exceed 1%, the oil should still be liquid at 5°, or, in case of coal tar oil, nothing should separate out at 15°; in general, viscosity should not exceed 3° Engler at 20°. Flash point (Abel-Pensky) should lie between 55° and 150°. The coke residue should not exceed 8, 1, and 3.5% for crude mineral oil, gas oil or kerosene, and coal tar oil, respectively. The ash of crude or residue oils should not exceed 0.03-0.05%; amount insoluble in benzene or xylene should not exceed 0.3%. A hydrogen content of  $11\frac{1}{2}$ %, or for tar oil  $6\frac{1}{2}$ %, is minimum. A small content of sulphur is unimportant, but much is said to attack the exhaust system. Calorific value is a measure of the energy obtainable from the oil. For crude oil, 9800; gas oil, 10,000; residue oils, 9900; kerosene, 9600; coal-tar oil, 8600 kg.-cal. per kg. are minima. l the crude oil, 20; gas oil, 60; residues, 20; tar oil, 75% at least should come over at 350°, whilst kerosene should give at least 50% at 300°. The oil should not contain more than 0.3% of mineral acid (as SO<sub>3</sub>) nor more than 0.05% of chlorine. An oil which fails in some of these respects may still, however, be usefulexperience alone can settle the point. The present tendency is to filter or centrifuge the ash and coking constituents out of mineral residue oils, with what success is still doubtful. The merits of oil from petroleum, lignite, and coal, and low-temperature carbonisation, and the probable duration of the petroleum supply, are discussed. W. N. HOYTE.

Activated charcoal for gasoline recovery. G. MÜLLER (Petroleum, 1926, 22, 1043—1047).—The quantity of steam used on the works scale in regenerating the charcoal is found to be less than that predicted from laboratory experiment and calculation, on account of the "rinsing" effect of the current of steam. The use of the compression method, in which the steam is passed through a heat exchanger, and serves to generate fresh steam under a partial vacuum, economises steam and prevents loss of gasoline. W. N. HOYTE.

Simple viscosimeter with scale [for oils]. R. VON DALLWITZ-WEGNER (Petroleum, 1926, 22, 1048— 1049).—A worm rotating at constant speed in a cylindrical chamber, heated to the desired temperature and containing the oil under test, causes the oil to rise in a vertical glass tube, with scale, to a height depending on the viscosity of the oil. The scale can be divided in any of the usual units. W. N. HOYTE.

#### PATENTS.

[Coal] pulverising apparatus. W. R. WOOD. From INTERNATIONAL COMBUSTION ENGINEERING CORP. (E.P. 257,436, 16.10.25).—Pulverisers for coal are found to work better when the coal is slightly moist, but this moisture might seriously interfere with the subsequent handling of the finely powdered fuel; therefore the circulating carrier air on its passage from the pulveriser to the cyclone collector is relied on to dry the coal. On the return journey from the cyclone the air is cooled and the moisture condensed out of it, and it is then re-heated before re-entering the pulveriser. B. M. VENABLES.

Drying and low-temperature carbonisation of fuels. R. DRAWE (G.P. 432,251, 23.4.25. Addn. to G.P. 425,316).—The flues of the original patent (B., 1926, 573) have internal projections, affording a more efficient heat transfer from the heating gas to the material being heated... The inlets and outlets of the flues are so proportioned that part of the heating gas is passed through the material under treatment. W. T. K. BRAUNHOLTZ.

Coke-ovens. J. STEPHENSON (E.P. 244,772, 14.12.25. Conv., 20.12.24).—A regenerative oven is described (see diverted at the centre of the setting to the top of the conjugate set of flues heating the other half of the oven face, where they pass downwards and meet further supplies of fuel gas. The waste gases pass through the regenerators to the main flue. Periodically the direction of flow of the gases is reversed. The arrangement of flues ensures a very uniform heating. A further feature is the flue 9 (Fig. 1), which connects with the oven at 11; this flue allows coal gas, otherwise wasted, to be collected during the charging of the ovens. S. PEXTON.

Gas-making and carbonising apparatus and process. W. W. ODELL (U.S.P. 1,598,217, 31.8.26. Appl., 17.12.23).—Fuel slowly gravitates through a vertical retort. Part of the fuel is burnt by the admission of air through a number of ports arranged around the retort at different levels. The fuel core, as it passes through the surrounding zone of combustion, is carbonised and the gaseous products are withdrawn through offtakes located within the fuel. S. PEXTON.

Utilising waste heat in the production of watergas. BAMAG-MEGUIN A.-G. (E.P. 253,088, 11.5.26. Conv., 4.6.25).—It has become the general practice to utilise the water-cooling chamber surrounding a watergas generator as a steam-raising boiler. It is difficult to construct such a boiler to withstand high pressures, and also difficult to remove scale from its surface. The present invention uses the cooling chamber for circulating the hot water produced therein, through a closed system in which the heat recovered is transferred to the water being fed to the usual high-pressure boiler, which recovers waste heat from the "blow gases."

S. PEXTON.

Means for detecting the presence of methane or other hydrocarbon gases. J. SEJVL, R. WINKLER, and M. PALKON (E.P. 238,531, 27.7.25. Conv., 15.8.24).— Two spirally coiled filaments, one of tungsten, enclosed in a hermetically-sealed bulb, and the other of some metal which catalyses the combustion of hydrocarbons, and surrounded by a shade through which a current of the gas to be tested is drawn, are connected in series or in



figs.). When the flue at the left of the centre of the base is in connexion with the chimney, air enters the regenerators 26, 27, 29 and 28, and passes via 36 and 35 to the sole of the oven. Fuel gas is admitted to the combustion flues 23, at several points 41, and meets the hot'air from the oven sole. The combustion completed, the products containing excess of secondary air are

parallel with an electric battery. In the centre of each filament is a thermopile, the two being connected in opposition through a galvanometer the null point of which is determined by introducing the apparatus into an atmosphere devoid of hydrocarbons. When the apparatus is brought into the presence of combustible hydrocarbon vapours the active filament increases in temperature and glows more intensely than the standard filament. Also the thermopile circuit is disturbed to an extent dependent upon the proportion of hydrocarbon vapour present. The galvanometer in the thermopile circuit can be calibrated so as to make the instrument quantitative. S. PEXTON.

Recovery of benzol from gases. O. HEITMANN (G.P. 431,923, 3.7.21).—The gas is washed with oil of b.p. 180—220° derived from crude benzol, and the benzol absorbed is driven off in stills operating without free steam. The process is characterised by extreme simplicity. W. T. K. BRAUNHOLTZ.

Distillation of [benzol] wash oil. BERGBAU A.-G. LOTHRINGEN (G.P. 432,378, 1.3.24).—In order to prevent thickening of the wash oil, the benzol is distilled from it by means of steam which is free from oxygen and carbon dioxide. The steam may be generated from the aqueous condensate in an evaporator placed between the still and the condenser for benzol and steam.

#### W. T. K. BRAUNHOLTZ.

Breaking of [petroleum] oil-water emulsions. J. C. WALKER, ASST. to DOHERTY RESEARCH CO. (U.S.P. 1,597,700, 31.8.26. Appl., 5.12.21).—The emulsion is treated with a mixture of formaldehyde and steam.

W. N. HOYTE.

Cracking of [hydrocarbon] oils. H. L. DOHERTY, Assr. to DOHERTY RESEARCH CO. (U.S.P. 1,597,674, 31.8.26. Appl., 22.11.22).—The oil is heated in a pipe still, and the vapours and oil issuing therefrom are passed under pressure and counter-current to one another through a cracking chamber. Pressure on the oil is lowered on entering the evaporator. Residue vapours are condensed by heat-interchange with untreated oil, and residue oil is returned to the cracking chamber. W. N. HOYTE.

Still for cracking [petroleum] oils. D. PYZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,597,821, 31.8.26. Appl., 25.6.24).—The oil passes through heating coils to a reaction chamber, and vapours evolved therefrom pass up a dephlegmator. The runback from the dephlegmator is passed again through the heating coils at a rate which varies with the amount of condensate from the dephlegmator. W. N. HOYTE.

Cracking of [hydrocarbon] oils. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,598,136, 31.8.26. Appl., 12.7.22).—Oil is heated in a large still under pressure to cracking temperature until the deposition of pitch begins, fresh stock is then fed in to keep the pitch in solution, and towards the end of the run pitchladen oil is withdrawn from the still at such a rate that pitch is not deposited. W. N. HOYTE.

Cracking of [hydrocarbon] oils. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,598,368, 31.8.26. Appl., 23.3.21. Renewed 14.1.26).—The oil is passed through heating coils to a large elongated horizontal expansion chamber, which is fitted with several residuum draw-offs connected to a common header. Fluid under pressure is forced through the header to withdraw residuum quickly from the expansion chamber. W. N. Hoyte.

Lubricating compounds. G. WISNER (E.P. 244,086, 1.12.25. Conv., 5.12.24).—The lubricating qualities of mineral oils are improved by adding 0.5% to 5% of a saturated fatty acid of high molecular volume and containing at least 16 carbon atoms, the best results being obtained when the oil is nearly saturated with respect to the acid; the resistance of the film may be further increased by adding a triglyceride. W. N. HOYTE.

Lubricating compounds. G. WISNER (E.P. 247,520, 1.12.25. Conv., 10.2.25. Addn. to E.P. 244,086). Sulphur is added to the mineral oil in quantities varying from 0.5% to 5%, and fatty acids and triglycerides are then added as set forth in E.P. 244,086 (cf. preceding abstract). W. N. HOYTE.

[Acetylene or oxygen] gas generators. C. CHOFFEL and A. P. JACQUELIN (E.P. 243,369, 19.11.25. Conv., 20.11.24).

Gas burners. G. HELPS (E.P. 258,632, 17.4.25).

Liquid fuel burners. H. KNUDSEN (E.P. 258,657, 23.6.25).

# III.—TAR AND TAR PRODUCTS.

Determination of anthracene by the Rütgers method. J. SIELISCH (Z. angew. Chem., 1926, 39, 1248-1249).-The method is based on the oxidation of anthracene to anthraquinone, solution of the crude anthraquinone in sodium bisulphite solutions and filtration from impurities, followed by reoxidation of the anthraquinol, the anthraquinone thus precipitated being finally weighed. The procedure is as follows :- To 1 g. of the sample, dissolved in 45 c.c. of boiling acetic acid, a solution of 15 g. of chromic acid in 10 c.c. of acetic acid and 10 c.c. of water is added at the rate of 1 c.c. per min. until the green solution becomes definitely brown. After boiling for a further hr., the cooled contents are diluted with 400 c.c. of ice-cold water and after 1/2 hr. filtered through a glazed filter. The precipitate is washed with ice-cold water then with (at most) 200 c.c. of 1% hot sodium hydroxide, It is then transferred to a 200 c.c. Erlenmeyer flask, and warmed with 15 c.c. of filtered reducing agent (10% sodium hydroxide containing 10% of sodium bisulphite) for a few minutes at 60-80°. The red solution is filtered into a warmed suction flask through filter paper in a Gooch crucible, which has previously been filled with reducing solution diluted 10 times. The crucible is not allowed to run dry throughout the filtration. The residue is submitted to a second reduction with 5 c.c. of reducing The solution is filtered as before and the warm agent. filtrates are oxidised to complete decolorisation with dustfree air or preferably with 5 c.c. of concentrated hydrogen peroxide. The precipitated anthraquinone is filtered through a porcelain filter dish (7 cm. diam.), washed with hot water till neutral, well drained, and dried at 100°. The determination is complete in  $3-3\frac{1}{2}$  hrs. instead of a day as in the modified Höchst test (B., 1926, 734) and the experimental losses do not exceed 1% (2% in the Höchst). R. BRIGHTMAN.

Principles of the determination of anthracene by the Rütgers method. J. SIELISCH and P. KÖPPEN-KASTROP (Z. angew. Chem., 1926, **39**, 1249–1253).— A detailed analysis of the procedure in the Rütgers method (cf. preceding abstract) in comparison with the Höchst test. R. BRIGHTMAN. Modification of the Höchst method for the determination of anthracene. F. JACOBSOHN (Brennstoff-Chem., 1926, 7, 311-313).—See B., 1926, 734.

Bromometric determination of the cresols. P. W. DANCKWORTT and G. SIEBLER (Arch. Pharm., 1926, 264, 439-447).-The official (German) method of analysing liquor cresoli is unsatisfactory. Good results for water, cresols, neutral oils, and soap are obtained by Arnold and Mentzel's procedure (Apoth.-Ztg., 1903, [16]), whilst the following method is applicable when the cresols are to be more exactly determined (cf. also Ulrich and Kather, B, 1926, 306). All the cresols take up three atomic proportions of bromine, but *m*-cresol is brominated much more rapidly than its isomerides. To prevent loss of bromine, a stoppered conical flask is used; the potassium iodide solution (10 c.c. of a 20% solution) is placed in a small sturdy container (e.g., a small bottle) standing inside the flask; and the stopper of the flask is sealed with a little potassium iodide solution. Into the flask are run successively 30 c.c. of 0.1N-potassium bromate solution, 25 c.c. of the solution to be analysed (e.g., one containing about 1.5 g. of cresol per litre), 30 c.c. of dilute sulphuric acid (1 part to 5 parts of water), and, after careful mixing, 10 c.c. of 25% potassium bromide solution. The flask is quickly stoppered, and the contents are mixed by swirling, care being taken not to upset the potassium iodide solution. After an appropriate time has elapsed ( $\frac{1}{4}$  hr. for *m*-cresol; 24 hours for a mixture; up to 72 hours for o- or p-cresol) the potassium iodide is upset, and the mixture is titrated with thiosulphate. "Lysol" is acidified and steam-distilled, the distillate being analysed as described. In analysing creolin preparations the neutral oils must be removed from the distillate, either by filtration or a suitable extraction process. W. A. SILVESTER.

#### PATENT.

Prepared tar for road-making etc. L. H. SENSICLE (E.P. 257,560, 21.5.25).-Prepared tar suitable for roadmaking can be produced without distillation from a by-product plant by dehydrating a tar or mixture of tars from which part of the condensate rich in naphthalene and phenols has been kept out. In a direct recovery process plant, practically the whole of the output can be converted into prepared tar by collecting separately the condensate from the serpentine coolers and multitubular coolers, removing part of the naphthalene and phenols therefrom, and then mixing the product with the remainder of the tar in the proportions required to produce a tar with the necessary viscosity, distillation range, etc. to comply with the specifications. This prepared tar has the advantage of a low naphthalene and phenol content. If desired the composition can be adjusted by the admixture of tar distillate fractions. B. W. CLARKE.

# IV.—DYESTUFFS AND INTERMEDIATES.

New synthesis of phenylrosinduline. A. WAHL and R. LANTZ (Chim. et Ind., 1926, 16, 355—366).— A résumé of work previously published (cf. A. 1923, i, 209; 1926, 296, 530, 617). J. S. H. DAVIES. Determination of dyes. A. NoLL (Papier-Fabr., 1926, 24, 593—595; cf. *ibid.*, 1924, 22, 361).—Methods are given for the determination of auramines and of the sulphuric esters of *leuco*indigoid dyes in the commercial products such as Auramines O and G and Indigosols O and O4B. For the auramines the dye is hydrolysed by boiling dilute hydrochloric acid to the corresponding benzophenone derivative, which is weighed. The soluble indigo derivatives are converted, by hydrolysis and oxidation with dilute sulphuric acid and sodium nitrite, into the corresponding indigos, which are weighed.

A. DAVIDSON.

## PATENTS.

Anthraquinone-2-glycine-3-carboxylic acid. GES. F. CHEM. IND. IN BASEL (Swiss P. 109,067, 30.11.23. Addn. to 103,217).—2-Chloroanthraquinone-3-carboxylic acid is heated, for example, with glycine (1 mol.), magnesium oxide, water, and copper powder for a long time at 120—130°. The product is acidified, and the precipitate filtered off and purified by dissolving in sodium carbonate solution and reprecipitating. Anthraquinone-2-glycine-3-carboxylic acid is an orange powder, its solution in sulphuric acid being deep orange, in alkalis red, in alkaline sodium hyposulphite wine-red. It may be used as an intermediate for dyes.

A. DAVIDSON.

Manufacture of 2-mercaptoanthracene-3-carboxylic acid and anthracene-2-thioglycol-3-carboxylic acid. Soc. CHEM. IND. IN BASLE (Swiss P. 110,414 and 110,415, 22.7.24. Addns. to 103,647).— 2-Mercaptoanthraquinone-3-carboxylic acid is reduced, *e.g.*, by heating with aqueous ammonia and zinc dust for 4—5 hrs., and the product condensed with chloroacetic acid. 2-Mercaptoanthracene-3-carboxylic acid on warming with chloroacetic acid (1 mol.) and alcoholic potash yields the anthracene-2-thioglycol-3-carboxylic acid described in the chief patent. A. DAVIDSON.

Manufacture of anthraquinone-thioxanthones and -acridones. Soc. CHEM. IND. IN BASLE (Swiss P. 110,749, 111,500, and 111,501, 16.4.24) .- A 1-chloro-4-acylaminoanthraquinone or 1-chloro-4-aminoanthraquinone (1 mol.) is condensed with 5-chloro-1-mercaptobenzene-2-carboxylic acid or 5-chloroaniline-2-carboxylic acid (1 mol.), the N-acyl group in the products eliminated where necessary, and ring closure to thioxanthones or acridones brought about by condensing agents such as chlorosulphonic acid. For example, 1-chloro-4-carbethoxyaminoanthraquinone is heated at 120-130° with 5-chloro-1-mercaptobenzene-2-carboxylic acid, potassium or sodium carbonate, and amyl alcohol, the alcohol distilled off, the residue extracted with dilute sodium hydroxide, and the alkaline solution acidified. The violet-brown flocks of 4-carbethoxyaminoanthraquinonyl-1-mercapto-5'-chlorobenzene-2'-carboxylic acid are hydrolysed by 90% sulphuric acid at 15-20° to the free amino-compound. On adding this to chlorosulphonic acid and keeping the solution at 40° till a sample, diluted with ice water, gives a pure blue precipitate, anthraquinone-4'-chloro-4-amino-2: 1-thioxanthone is formed. It gives a reddish-orange solution in sulphuric acid and a violet vat with alkaline sodium hyposulphite. 4-Aminoanthraquinonyl - 1 - mercapto - 5' - chlorobenzene - 2' - carboxylic

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acid, violet-brown, can also be obtained by condensation of 5-chloro-1-mercaptobenzene-2-carboxylic acid with 4-chloro-1-aminoanthraquinone in presence of aqueous sodium hydroxide and alcohol at 80°. On heating 1-chloro-4-carbethoxyaminoanthraquinone with 5-chloroaniline-2-carboxylic acid, potassium carbonate, a little cuprous chloride and amyl alcohol for a long time under reflux there is formed 4-carbethoxyamino-1:5':2'-chlorocarboxyphenylaminoanthraquinone. This is hydrolysed by 85% sulphuric acid at 15-20° to 4-amino-1:5':2'chlorocarboxyphenylaminoanthraquinone. On heating the latter with chlorosulphonic acid at 15° till the blue solution becomes red, ring-closure takes place with formation of anthraquinone-4'-chloro-4-amino-2:1-acridone, blue, giving a violet vat with alkaline sodium hyposulphite. The products are used for the preparation of dyes. A. DAVIDSON.

Manufacture of 2:3-anthraquinonylindoxyl. Soc. CHEM. IND. IN BASLE (Swiss P. 110,416, 30.11.23. Addn. to 103,648).—Anthraquinone-2-glycine-3-carboxylic acid is heated under reflux for some time with anhydrous sodium acetate and acetic anhydride. The brownishyellow crystals which separate are washed with acetic acid, and then with water. Anthraquinonyl-2:3-indoxyl dissolves in cold concentrated sulphuric acid to an orangered solution from which water precipitates greenishyellow flocks. With alkaline sodium hyposulphite it gives at first a brown, then an olive-green vat. It serves as an intermediate for dyes. A. DAVIDSON.

Manufacture of anthracene-2 : 1-thioindoxyl. Soc. CHEM. IND. IN BASLE (Swiss P. 111,997, 14.1.25).condensation of 2-mercaptoanthracene with chloroacetic acid or by reduction of anthraquinone-2-thioglycollic acid, is treated with condensing agents. The anthracene-2-thioglycollic acid, yellow, m.p. 105°, is converted by phosphorus pentachloride into anthracene-2-thioglycollyl chloride. This is dissolved in chlorobenzene, treated with aluminium chloride, and warmed for several hours until evolution of hydrogen chloride ceases. The reaction mixture is added to ice and hydrochloric acid, the chlorobenzene driven off by steam, and the residue extracted with dilute sodium hydroxide to obtain anthracene-2:1thiondoxyl, yellow, m.p. 172°. It finds application as an intermediate for dyes. A. DAVIDSON.

Anthraquinoneacridine dye. Soc. CHEM. IND. IN BASLE (Swiss P. 111,502, 13.5.24. Addn. to 97,059).— 8-Amino-2: 1-anthraquinoneacridone (less than 3 mols.) is mixed with nitrobenzene, cyanuric chloride (1 mol.) is added, and the mixture warmed gradually to 190—200° and stirred for several hours at this temperature. The condensation product, when washed with nitrobenzene and alcohol, is obtained as a dark violet powder, which dyes cotton from the vat in reddish-violet shades fast to light, washing, and chlorine. A. DAVIDSON.

Aromatic amines (G.P. 421,151).—See XX.

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

Determination of the dry weight of wool. S. G. BARKER and J. J. HEDGES (J. Text. Inst., 1926, 17, T 453-456).—A comparison of the actual moisture content of wool with the commercial regain as determined by an ordinary conditioning oven and by the Bradford Conditioning House has shown that the latter figure is only about 0.5% less than when specially dried air is used in the drying vessel, and is therefore sufficient for practical purposes. The common ovens, however, give results about 1% too low, and frequently they may be found in works near steam-using plant or other places where the air is unduly moist. J. C. WITHERS.

Gel structure of the wool fibre. J. B. SPEAKMAN (J. Text. Inst., 1926, 17, T457-471).-In this and a subsequent paper (ibid., 472), it is shown that the wool fibre has a true yield point and, after extension, remains permanently more extensible under low loads. Such observations are held to necessitate the replacement of the theory that wool has a two-phase gel structure by a four-phase theory. It is argued that the wool fibre consists of two gels arranged, as it were, in parallel. The first may be called the petrified gel, and comprises an elastic cell wall enclosing a fibrillar structure which is not in physical equilibrium with a viscous phase. The second, which fills the interstices of the petrified gel, is gelatinous and capable of reversible solution in, and deposition from, water. Gelation occurs at 0° in water, and at ordinary temperatures in unsaturated air and, under such conditions, the medium possesses the usual two-phase structure of gels. In reality, therefore, the wool fibre consists of four phases, but for convenience these may be reduced to three, viz. (1) an elastic cell wall enclosing (2) a fibrillar structure which is not in physical equilibrium with (3) a viscous, gelatinous medium included in its interstices. The experiments described deal with breaking loads, extensions at break, and breaking stresses, and include measurements of the effect on these factors of diazotisation, formaldehyde, benzoquinone, boiling water, and chlorine water.

J. C. WITHERS.

Extension of wool fibres under constant stress.— J. B. Speakman (J. Text. Inst., 1926, 17, T472—481).

Viscose. E. BERL and J. BITTER (Cellulosechem., 1926, 7, 137-145).—The xanthate reaction is a specific reaction of the alkali alkoxide group, and depends intimately on the alcohol-alkoxide equilibrium; in the case of polyhydric alcohols considerable resistance is experienced. Primary monohydric alcohols, like ethyl alcohol, react easily and completely, glycol forms a xanthate slowly with simultaneous formation of byproducts, glycerol under ordinary conditions does not form a xanthate at all. Methylation alters these relationships so that glycol monomethyl ether and glycerol dimethyl ether react easily, like ethyl alcohol, whilst glycerol monomethyl ether reacts like glycol. Surface phenomena also play a part, so that in the presence of active charcoal even glycerol may form a xanthate. Viscoses can be prepared from cellulose monoalkyl ethers, but the dialkyl ethers cannot be brought into reaction; hence the presence of two free hydroxyl groups the case of cellulose is essential to xanthation. ın The alkali first produces the sodium alkoxide, for which purpose in order to establish a favourable equilibrium it must be concentrated, and then hydrates closed anhydride linkages, thus altering the physical structure

of the cellulose, increasing its internal surface development and setting up the initial stages of dispersion. The absence of stoicheiometrical proportions between the reacting carbon disulphide and cellulose suggests that while xanthate groups are formed as the result of the alkali alkoxide equilibrium the most important feature of the reaction is the breakdown of the cellulose substance into a dispersed condition. Whereas xanthation occurs in the presence of a restricted quantity of water, hydrolysis proceeds in the presence of an excess of water. During the ripening process, the ultramicroscopic, dispersed particles show a progressive slowing down of their Brownian motion with ultimate cessation; this is accompanied by a noted decrease in the number of particles and increase in their size until incipient coagulation is indicated by a slightly coherent structure which finally develops to a honey-comb gel. The hydrolysis of the xanthate in dilute solution is characterised by the formation of free cellulose xanthic acid and caustic soda in equilibrium. The free xanthic acid dissociates into dispersed hydrated cellulose and carbon disulphide which reacts with the sodium hydroxide. The equilibrium is thus upset and the hydrolysis proceeds further, while the accumulation of dispersed free cellulose induces re-aggregation of the particles. J. F. BRIGGS.

Viscose. E. BERL and A. LANGE (Cellulosechem., 1926, 7, 145-147).-Viscose prepared from cotton linters has a higher viscosity than that from wood cellulose, under identical conditions, corresponding with the higher micellar molecular weight of the cotton cellulose. Both types of viscose during the ripening process undergo an initial fall in viscosity which is attributed to the degrading action of the sodium hydroxide. After this initial fall there is a continuous rise in viscosity. If the viscose be precipitated at the different stages of ripening, either by neutral or acid coagulating agents, converted into hydrated cellulose, and if this regenerated cellulose be nitrated under standard conditions, the viscosity of the nitrocellulose solutions in acetone may be taken as a measure of the integrity of the cellulose molecule in the viscose at that stage of ripening. The results showed that the nitrocelluloses from the cotton viscose had consistently higher viscosities than those from wood pulp viscose. The nitrocelluloses from freshly prepared viscose had higher viscosities than those which had ripened for a few hours. On the other hand, the subsequent stage during which the viscosity of the viscose continuously increases during ripening is not correspondingly reflected in the viscosity of the nitrocellulose prepared from the regenerated cellulose. After the initial rapid fall in the viscosity of the nitrocellulose, this remains constant during further ripening of the viscose. J. F. BRIGGS.

Experimental method for investigating thermal properties of cotton fabrics. J. GREGORY (J. Text. Inst., 1926, 17, T 553-566).

Changes in strength and elongation of artificial silk caused by moisture. Y. KAMI (Cellulose Ind., Tokyo, 1926, 2, 25).—The elongation of artificial silk generally increases 10-65% on wetting, the average being 40%, but the elongation of some kinds diminishes. The tensile strength diminishes by 50-70%. These

changes are independent of the duration of moistening, and the original properties of the silk are almost recovered on drying. The cross-section of the dried thread is elliptical or rectangular, but when wetted it becomes almost circular and 2-2.5 times larger in area.

A. GEAKE.

Viscose. V. G. KITA and R. TOMIHISA (Cellulose Ind., Tokyo, 1926, 2, 26–28).—During the ripening of viscose, xanthate is formed by the action of carbon disulphide on alkali cellulose, and sodium carbonate and thiocarbonate are formed from carbon disulphide and free sodium hydroxide. The first reaction is the faster, but the xanthate again decomposes when the concentration of carbon disulphide and sodium hydroxide is reduced by the second reaction. Eventually, all the free alkali is exhausted, and the viscose is coagulated by the accumulation of salts. Rise of temperature favours the formation of carbonate and thiocarbonate because the xanthate decomposes more rapidly. During spontaneous coagulation all free alkali disappears. A. GEAKE.

Viscose. VI. G. KITA, K. AZAMI, and R. TOMIHISA (Cellulose Ind., Tokyo, 1926, 2, 28-30; cf. preceding abstract) .-- Increasing the amount of carbon disulphide from 100% to 150% of the weight of cellulose increases the viscosity, but does not affect the proportion of combined alkali (about 0.65 mol.). Increasing the amount of alkali diminishes the viscosity and increases the proportion combining; the ratio of carbonate to thiocarbonate is also increased. If xanthate is diluted with 5% sodium carbonate solution instead of with water, the combination is stronger, and the viscosity is diminished. Addition of sodium hydroxide, which has been shaken with carbon disulphide until no free alkali remains, results in less combined alkali and a higher viscosity than when the sodium hydroxide and carbon disulphide are added without previous combination. Combination after a definite time of ripening is increased by the addition of sodium hydroxide to a maximum, which is probably determined by the exhaustion of the carbon disulphide; combination is then further increased by the addition of carbon disulphide. A. GEAKE.

Cellulose esters. G. KITA, I. SAKRADA, and T. NAKA-SHIMA (Cellulose Ind., Tokyo, 1926, 2, 30-32).—A cellulose ester containing 0.29 mol. of fatty acid may be further esterified after treatment with sodium hydroxide. After 14 esterifications the proportion of fatty acid reaches a maximum of 2.11 mols. About half of this ester is soluble in benzene, the soluble part containing  $2 \cdot 32$ , and the insoluble  $2 \cdot 00$  mols. of fatty acid. Unchanged cellulose may be extracted with cuprammonium hydroxide solution, or by the viscose process, from esters containing 1 mol. of fatty acid to 2, 3, or 4 CgH1005 groups, and the composition of the undissolved residue tends to approach 1 mol. of fatty acid to one C6H10O5 group. It is concluded that in cuprammonium hydroxide solution cellulose is dispersed into C<sub>6</sub>H<sub>10</sub>O<sub>5</sub> units. Ripened xanthate does not combine with fatty acid chlorides after purification with acetic acid and salt solution, but when precipitated with alcohol a sulphurfree ester may be obtained. A. GEAKE.

waxes from vegetable fibres and textiles prior to bleaching, magnesium oleate is added to the alkaline water to the extent of 10% of the alkali present. R. B. CLARKE.

[Electrolytic] manufacture of cellulose. T. A. HILL, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,595, 29.6.26. Appl., 26.5.20. Renewed 17.6.24). —A cuprammonium solution of cellulose is fed continuously to an electrolytic cell having a platinum or platinum-plated belt as anode and a suitable metallic cathode. The belt travels in the same direction as the solution, and cellulose is deposited on each side of the belt in increasing thickness, which can be regulated by suitable means. The films of cellulose are removed by means of brushes and passed through a tank containing dilute sulphuric acid, and finally washed and wound. R. B. CLARKE.

[Electrolytic] manufacture of cellulose products. E. TAYLOR, ASST. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,603, 29.6.26. Appl., 17.6.24).—In the process of U.S.P. 1,590,595 (cf. preceding abstract) the platinum or platinum-plated belt is replaced by a copper belt. R. B. CLARKE.

Manufacture of artificial filaments [cellulose acetate]. G. B. ELLIS. From Soc. FABR. DE LA SOIE "RHODIASETA" (E.P. 248,696, 27.5.25).—In the process of dry-spinning solutions of cellulose derivatives in volatile solvents, the cross-sections of the filaments are controlled, the flat type giving scintillating effects being avoided and the rounded or star-shaped type being produced, by ensuring at the spinning apertures an input of heat, maintaining at the spinning dies and in the immediate neighbourhood a temperature regulated and varied according to the demand of the other spinning conditions, independently of the temperature of the remainder of the cell. J. F. BRIGGS.

Subjecting wood and similar materials to a preliminary treatment for the manufacture of paper board etc. H. BRÄUNLICH (U.S.P. 1,597,717, 31.8.26. Appl., 29.5.25. Conv., 30.5.24).-The process of steaming wood for the manufacture of paper boards is divided into four separate stages. The material is first subjected to a slow preparatory heating up to a steam pressure of 2-4 atm. according to quality; in the second step this pressure is kept approximately constant for 2-4 hrs., and in the third step it is gradually reduced to atmospheric pressure within 3-8 hrs. by regulating the steam supply. The fourth step consists in admitting water to the boiler, with or without the admixture of chemicals, and saturating the material J. F. BRIGGS. therewith for 4-5 hrs.

Collapsible reels for winding filaments, yarns, threads, silks, etc. BRYSILKA, LTD., and F. W. SCHUBERT (E.P. 258,377, 2.7.25).

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

PATENTS.

Machines for dyeing and similarly treating fabrics. F. RILEY (E.P. 258,736, 28.9.25).

Yarn beams on which yarns are to be wound for fluid treatment. J., T., and J. BRANDWOOD (E.P. 258,309, 15.5.25).

Polysaccharides. XXXV. Enzymic degradation of artificial silk and natural cellulose. (P. KARRER and P. SCHUBERT (Helv. Chim. Acta, 1926, 9, 893—905). —Different varieties of cellulose and artificial silks show widely differing reactivity towards cellulase, previous treatment by alkali greatly aiding reaction. Tables are given showing rate of destruction for many commercial preparations, with diagrams showing the magnified cellulose particles, from which it appears that increase of surface favours enzyme action. Artificial silk which is easily dyed is easily attacked by cellulase. The velocity of decomposition of filter-paper by cellulase was determined, but no velocity constant could be found.

E. W. WIGNALL.

Semi-chemical pulping process. J. D. RUE, S. D. WELLS, F. G. RAWLING, and J. A. STAIDL (Pulp and Paper Mag., 1926, 24, 1163-1167).-A modified digestion process is described which is particularly applicable to hard woods such as aspen, birch, and the gums. High yields (70-85%) are obtained, and the pulp is suitable for medium- and low-grade printing papers and certain classes of board. The process involves impregnation of the chips under pressure, mild digestion with chemicals which are practically neutral, and are capable of maintaining approximate neutrality during the digestion operation, and reduction of the softened chips by mechanical means. A suitable digestion liquor consists of a mixture of sodium sulphite and sodium bicarbonate, conveniently prepared by absorbing sulphur dioxide in soda ash solution; the ratio of sodium bicarbonate to sodium sulphite may vary within wide limits, depending on the character of the wood and the quality of pulp required : the total quantity of chemicals need not exceed 15 lb. per 100 lb. of wood. The chips, after steaming for at least 3 hr. at atmospheric pressure, are impregnated with the cooking liquor under a pressure of 100 lb./sq. in., preferably at  $120-125^{\circ}$ , for  $1-1\frac{1}{2}$  hrs. After removal of the excess of liquor the temperature of the cook is quickly raised to 140-160° and held there for 1-6 hrs. The digested chips are washed and finally converted into pulp by passage through a rod mill. Semi-chemical pulp from aspen and birch may satisfactorily replace over 50% of a standard newsprint furnish, or may be used with ground wood alone; in the latter case, however, the paper, though strong when dry, is relatively weak in the wet state and somewhat difficult to run on the machine. In the manufacture of boards, less care is necessary in the selection, preparation, and cooking of the wood, and dark woods, such as chestnut, elm, etc., may be used. Chestnut chips (after extraction of tannin) have been treated by this process on a commercial scale with satisfactory results. There appears to be no advantage in treating coniferous woods by a semi-chemical process since the resulting pulp lacks D. J. NORMAN. strength and felting qualities.

Micro-organisms concerned in the decomposition of cellulose in the soil. WAKSMAN and SKINNER. —See XVI.

# PATENTS.

Treating textile material. R. A. PHAIR, Assr. to H. KOHNSTAMM & Co., INC. (U.S.P. 1,598,305, 31.8.26. Appl., 16.7.21).—In the process of boiling out fats and 946

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

Neutralisation of ammonium sulphate. A. E. THAU (Gas- u. Wasserfach, 1926, 69, 832-834).—A résumé of Bateman's work (B., 1926, 404).

R. A. A. TAYLOR.

Manufacture of sodium dichromate. L. L. POPOV (J. Chem. Ind. [Russia], 1925, 2, 465-474; Chem. Abstr., 1926, 20, 3063).—Rich chromium ores are more advantageous than poor ores, as regards both speed of oxidation and yield of dichromate; poor ores must be finely pulverised. The amount of lime must be 80% of the weight of ore if the latter contains 30-40%  $Cr_2O_3$ , 90% for 40-50%, or 120-130% for a content greater than 50%. These quantities must be increased if the temperature of operation is above 1100°. The theoretical quantity of sodium carbonate is employed. Rich ores are calcined for  $1\frac{1}{2}$  hrs. at 1100°; poor ores for a longer time at 1200°. A. A. ELDRIDGE.

Synthesis of galena crystals. W. OGAWA, C. NEMOTO, and S. KANEKO (Res. Electrochem. Lab., Japan, 1926, [173], 1—59; Chem. Abstr., 1926, 20, 2768).— Galena crystals, suitable for use as radio detectors, were obtained by fusion of (i) lead sulphide 92.5, silver sulphide 7.5, and sulphur 1.5 parts, or (ii) lead sulphide 99.5, thallous sulphide 0.5, sulphur 1.5 parts.

A. A. ELDRIDGE.

Recovery of sulphur from gases. FISCHER and DILTHEY.—See II.

#### PATENTS.

Apparatus for hydrating lime. H. MISCAMPBELL (E.P. 257,793, 26.11.25).—The lime, after treatment in a pulveriser situated below ground level, is discharged by a vertical elevator into a bin, and is fed by a reciprocating feeder and a spiral conveyor into a horizontal mixer fitted with rotating paddles. Water is added, and the lime and water are discharged into a hydrator pan rotating upon rollers or ball bearings; in the pan are hung stationary ploughs which stir the mixture during the slaking process. The hydrated lime is discharged through the bottom of the rotating hydrator, and is elevated to a series of horizontal cooling conveyors in which the lime is carried in one direction through one conveyor, and in the opposite direction through another, until it is discharged and elevated to a final pulveriser. The dust and fumes evolved during the mixing and hydration are condensed in a dust collector provided with water-spraying nozzles, and are sent back with the water to the mixer. W. G. CAREY.

Production of pure hydroxides of barium or strontium. RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of B. STUER (G.P. 432,114, 9.8.24).—Barium or strontium oxides or hydroxides containing sulphide, which have been obtained from the sulphur compounds by hydrolysis without the use of ammonia, are crystallised from water in presence of ammonia.

#### W. G. CAREY.

Production of sodium sulphate from ferrous sulphate and sodium chloride. VEREIN FÜR CHEM. U. METALL. PROD. (G.P. 432,201, 21.10.24).—After heating, the undecomposed sodium chloride is removed by extraction with water at so low a temperature that the smallest amount of sodium sulphate is dissolved, the residue containing sodium sulphate is then dissolved in water at a suitable temperature and the sodium sulphate is separated. W. G. CAREY.

Simultaneous production of potassium phosphate and phosphoric acid. W. H. Ross, R. M. JONES, and A. L. MEHRING (U.S.P. 1,598,259, 31.8.26. Appl. 4.3.24).—A mixture of phosphate rock, a potash silicate, and a carbonaceous material is heated in a reducing atmosphere at 1300°. The evolved fumes are burnt at the outlet of the furnace and the resulting product is recovered in a Cottrell precipitator.

S. BINNING.

Calcium cyanogen compounds. R. W. POIN-DEXTER, ASST. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,596,119, 17.8.26. Appl., 18.2.26).—Solid cyanides of the alkaline-earth metals are prepared by treating a mixture of the oxide of the metal and a monohydric alcohol, *e.g.*, methyl alcohol, with an excess of hydrocyanic acid. R. B. CLARKE.

Cyanide product [containing combined ammonia]. R. W. POINDEXTER, ASST. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,596,120, 17.8.26. Appl., 13.4.26).—The compound Ca(CN)<sub>2</sub>,2NH<sub>3</sub> is prepared by the interaction of hydrocyanic acid and calcium nitrate tetrahydrate in anhydrous liquid ammonia. The product decomposes in air and is used as an insecticide. R. B. CLARKE.

Production of arsenic trisulphide. I.-G. FARB-ENIND. A.-G., Assees. of U. DREYER (G.P. 432,358, 16.1.25).—In the production of arsenic trisulphide from arsenious oxide and hydrogen sulphide, the arsenious oxide is suspended in water or in a salt solution, which preferably is warmed. W. G. CAREY.

Manufacture of hydrogen. J. H. BEAUMONT (E.P. 257,434, 14.10.25).-Pure precipitated ferric oxide is reduced to iron by producer gas in an upper vessel which connects through its base with a lower vessel so that the iron passes into the latter by gravity without contact with air. The iron is treated with steam in the lower vessel, giving pure hydrogen, and being oxidised to ferrous oxide, which is then elevated by suitable means to the upper vessel and again reduced. The reduction is carried out between 500° and 550°, since to ensure the production of pyrophoric iron the temperature must not exceed 600°, and the re-oxidation takes place between 500° and 650°, the temperature being kept below 700° to minimise the formation of magnetic iron oxide, which requires more carbon monoxide for its reduction. The temperatures are controlled by external heating.

W. G. CAREY.

Treatment of solids with liquids (E.P. 257,643). —See I.

# VIII.—GLASS; CERAMICS.

Effects of thermal treatment on glass as shown by precise viscosimetry. V. H. STOTT, D. TURNER, and H. A. SLOMAN (Proc. Roy. Soc., 1926, A, 112, 499-521; cf. B., 1925, 921).—A method of determining the viscosity of molten glass by measuring the thickness of the film which adheres to a fine iridioplatinum wire withdrawn from it at a known rate is described, the attainable accuracy being  $\pm 3\%$ . By this means numerous experiments have been made with the glass N.P.L. 15. It is found that the viscosity of this glass at temperatures above 1200° is not altered by cooling the glass to the ordinary temperature and reheating, unless the glass is kept too long within a certain temperature range below 1200°. In this region the glass is apparently capable of existing in at least two states of differing viscosity. These facts may have some connexion with the known effect of variations in, *e.g.*, the moisture content, heat treatment, or proportion of cullet on the brittleness and mechanical behaviour during working of glasses of the same chemical composition.

#### R. CUTHILL.

Machine-made table glass. W. SCHMIDT (Keram. Runds., 1926, 34, 521—523; Chem. Zentr., 1926, 97, II, 1567).—Ordinary table glass is not adapted to mechanical manufacture, on account of a tendency to devitrification. A glass containing 11% of calcium oxide and 19% of sodium oxide is suitable for the Foucault process but it is not very stable. Stability is conferred by the addition of alumina, and such a glass is "long," very resistant to tension and pressure, tough, and, at the same time, elastic. Alumina can be introduced in the form of an aluminous sand. Koalin is incorporated into the glass with difficulty, alumina more easily, and felspar most easily of all. L. S. THEOBALD.

Separation of metals from glass melts. L. SPRINGER (Keram. Runds., 1926, 34, 503—504; Chem. Zentr., 1926, 97, II, 1567).—Lead is produced on melting lead-glass under reducing conditions. Melts of copperruby glass and silver-yellow glass yield copper and tin, and silver respectively. In another case copper and iron separated, the latter probably arising from ferrous oxide. A melt of table glass gave copper and iron either in a compact form or as iron tinsel. A piece of cast iron added to the melt absorbed copper, but was still undissolved after 3 days. A change in shape and loss of weight, however, were observed. L. S. THEOBALD.

Vitreous silica and vitreous quartz. W. W. WINSHIP (Trans. Amer. Electrochem. Soc., 1926, 50, 177—188).—Vitreous silica and vitreous quartz exhibit the following properties : indifference to acid reagents, small expansion coefficient, high temperature electrical resistance, low solubility in water, good light transmission over the whole spectrum, low permeability to gases, low dielectric loss, and simple chemical structure. Applications of these products to various chemical, electrical, and mechanical purposes, also to optical apparatus and in the gas industry, are described, all being dependent upon one or more of these properties. F. G. CROSSE.

Temperature diffusivities and thermal conductivities of silica and fireclay refractories. A. T. GREEN (Gas World, 1926, 85, Coking Sect., 112—114).— The diffusivities of silica products at lower (700°) and at higher temperatures (1200°) are, in general, higher than those of fireclay products, although individual products may vary from this generalisation. The lower values of the apparent specific gravity of well-fired silica bricks tend to give lower values for the thermal conductivities when compared with fireclay materials. Well-fired silica bricks of good quality, in some cases, are better conductors of heat than the average fireclay brick at 800°, whilst many silica and fire-bricks have approximately equal conductivities at this temperature. At higher temperatures (1200°) silica bricks are the better conductors, except in the case of those which have been inadequately fired. The rate of increase of diffusivity and conductivity with temperature is generally greater with silica products. The effect of porosity and texture on conductivity is briefly discussed. L. S. THEOBALD.

Effect of industrial usage on the thermal conductivity of a semi-silica material used in a cokeoven wall. A. T. GREEN (Gas World, 1926, 85, Coking Sect., 111—112).—Diffusivity and thermal conductivity data, determined by the method previously described (Trans. Cer. Soc., 1922, 21, 394; cf. B., 1922, 547 A), are reported for semi-silica material which had been in use for 18 years and for a sample of the original, unused material. The used material shows higher values for thermal conductivity over the range 500—1300°, and this is ascribed to alterations in the constitution of the material and in the nature of its porosity. L. S. THEOBALD.

Thermal expansion of some fused oxides used as refractories. G. E. MERRITT (Trans. Amer. Electrochem. Soc., 1926, 50, 283-291).-The thermal expansions of silica, zirconia, thoria, the refractories made of magnesia and alumina, and of a mixture of equimolecular proportions of thoria and zirconia, between the temperatures 25° and 1000°, were measured. The results are exhibited graphically and compared. The introduction of 3.5% SiO<sub>2</sub> + 0.35% TiO<sub>2</sub> to pure zirconia appreciably raises the thermal expansion. From the form of the curves, and other evidence, it is concluded that combination takes place when the equimolecular mixture of thoria and zirconia is fused at about  $3500\pm500^\circ$ , giving a crystalline thorium zirconate. The average coefficients of expansion of these oxides from room temperature to 800° are tabulated.

F. G. CROSSE.

Application of recrystallised silicon carbide. F. A. J. FITZGERALD (Trans. Amer. Electrochem. Soc., 1926, 50, 279–282).—Recrystallised silicon carbide, made by mixing silicon carbide with glue as a binder and heating to about 1800°, has been satisfactorily used as a refractory material in furnaces for firing porcelain.

F. G. CROSSE.

Refractories for melting metals. JORDAN and others.—See X.

Thermal insulation of electric furnaces. HART-MANN and WESTMONT.—See XI.

Refractories for induction furnaces. UNGER. —See XI.

#### PATENTS.

Manufacture of refractory substances from highgrade oxide of zirconium. DEUTSCHE GASGLÜHLICHT-AUERGES. M.B.H. (E.P. 245,101, 16.12.25. Conv., 24.12.24).—Refractory substances capable of resisting temperatures exceeding 2000° are manufactured by mixing up to 5% of the oxide of an alkaline earth, *e.g.*, magnesium oxide, with high-grade zirconium oxide, pressing the wet mixture, and sintering at 1300° preliminary to finishing at a higher temperature. More porous articles can be prepared by dispensing with the preliminary sintering. B. W. CLARKE.

Manufacture of glass, sand, or flint paper, emery cloth, and like abrasives. C. A. KLEIN and R. S. BROWN (E.P. 258,412, 26.8.25).—The base of sheet abrasive material is first waterproofed by passing it through a bath of linseed oil and wax kept at 100—170°. The abrasive is fixed to this base by means of an adhesive, similarly waterproofed with a drying oil and a gum or synthetic resin. The material thus prepared is unaffected by moisture. F. G. CROSSE.

**Producing moulded articles from refuse or its constituents.** A. GROTE (E.P. 257,854, 14.4.26).— Calcium carbonate and silicious materials are added during the fusing of refuse etc. so that the silica-lime ratio is from 2:1 to 4:1, the slag produced still remaining plastic when cooled to 900°. In this condition it can be moulded under pressure to form homogeneous moulded articles which are subsequently subjected to a tempering process, the final product resembling basalt in character. B. W. CLARKE.

Kilns and other heat treatment furnaces. MORGAN CRUCIBLE Co., and C. W. SPEIRS (E.P. 257,472, 18.12.25). —Electrically heated tunnel kilns are provided with trucks for supporting the ware, furnished with tables arranged to rotate as the truck is traversed through the kiln, whereby the articles are subjected to an approximately uniform heat treatment. B. W. CLARKE.

Oil- or gas-fired [pottery] kilns. W. S. MURRAY (E.P. 258,318, 4.6.25).—In kilns used for firing pottery, a heat economy is effected by having the kilns arranged in tandem or series each communicating with the next by a passage at the bottom, and the last leading into a shaft. Burners are so arranged inside each kiln that the products of combustion, after heating the first kiln, pass on to the next, which thus becomes partly warmed. F. G. CROSSE.

Manufacture of sheet glass. E. C. R. MARKS. From E. DANNER (E.P. 258,694, 6.7.25).

# IX.—BUILDING MATERIALS.

Mortar sands. H. BURCHARTZ (Zement, 1926, [8]; Mitt. Materialprüf., 1926, [3], 60—62).—Sand from the Rhine will vary according to its source in particle size and density (bulk weight) with corresponding variations in strength of the mortar produced. It should not be used without preliminary tests being carried out. The density (bulk weight) of a sand is not always a measure of its suitability for use as mortar. Although the addition of fine sand to coarse sand, to increase the density, will also increase the strength of the mortar produced, yet a further increase of density obtained by the addition of stone dust will not bring about a corresponding increase in strength of the mortar, owing to the excessive increase in the surface area of the particles which must be covered by the cement.

# B. W. CLARKE.

Coloration of cement, concrete, and other building materials. C. J. GOODWIN and G. N. WHITE (Chem. Age, 1926, 14, 435).—A solution of a suitable dyestuff or its derivatives is used instead of water for gauging cement or concrete, so that during mixing and setting, oxidation takes place and the original dyestuff is precipitated in the cement. In this way the particles of cement become covered with the particles of dyestuff, and it is possible to obtain suitable shades of colour with the use of 0.1-0.6% of colouring matter reckoned on the dry weight of the concrete. The depth of the tint produced depends only on the ratio of dyestuff to cement, being unaffected by the addition of aggregate. The dyestuff used must be fast to light, moisture, and atmospheric influences, and in the finished state must be completely insoluble in water. B. W. CLARKE.

Analysis of burnt chalk and magnesite after storage in air. A. STETTBACHER (Z. angew. Chem., 1926, 39, 1151—1154).—Drying the stored material in phosphorus pentoxide-sodium desiccators shows that a proportion of the water taken up is not chemically combined. Freshly-ignited pure magnesium oxide after exposure to air for 3—4 days is found to contain  $1\cdot 8-2\cdot 1\%$  of water removed on keeping in the desiccator. Calcium oxide similarly treated and kept for three days lost  $0\cdot 6-0\cdot 7\%$  in weight on keeping in a vacuum desiccator. S. I. LEVY.

Painting of treated wood: M. E. DUNLAP (Ind. Eng. Chem., 1926, 18, 1091).—Paint exposure-tests were carried out on wood panels previously treated with creosote, mixtures of creosote and montan wax, gas oil, pine oil, sugar, borax, zinc chloride, sodium fluoride, mercuric chloride, and two proprietary water-soluble articles, Triolith and Ac-Zol. While the water-soluble preservatives give rise to no apparent detrimental action, discoloration, bleeding, checking, and peeling are observed in the other cases. The strengths of solution and average absorptions of the preservatives by the wood are tabulated. S. S. WOOLF.

# PATENTS.

Continuous furnace for preparing molten aluminous cement, etc. G. M. J. DUMAS (E.P. 257,819, 3.2.26).-A continuous furnace for producing aluminous cement or other products having a high melting point consists of a melting chamber of the reverberatory type provided with a water jacket for cooling the walls at the places most exposed to corrosion, and a feeding channel, preferably a vertical furnace of the cupola or water-jacket type, through which only part of the burnt gases is passed while the material to be treated is delivered. The material in the reverberatory furnace moves down a slope and is heated by one or more flame jets under pressure (preferably burning pulverised coal), the pressure in the furnace being kept above atmospheric by a proper adjustment of the draught and of the flames. Openings are provided which serve as tap holes for the molten cement and for the escape of part of the exhaust gases, being made large enough to allow the passage of the whole of the burnt gases without affecting the rate of combustion if the feeding channel becomes choked through partial fusion of the material to be treated. In normal conditions, some of these openings are kept closed by clay stoppers, which are broken when the feeding channel is choked, and are arranged so that the flames in this event are directed against the partially fused lump of material, thereby freeing the channel and ensuring continuity of operation. B. W. CLARKE.

Apparatus for calcining and clinkering with recovery of by-product heat and by-products. R. D. PIKE (U.S.P. 1,596,509, 17.8.26. Appl., 21.8.24). —The raw mix for making Portland cement clinker, after being calcined in a neutral atmosphere, is passed to an inclined rotary kiln into which sulphur dioxide is passed from sulphur burners. The kiln is heated to 1370—1540° and a free supply of air is maintained. The kiln gases are then treated for the separation of alkali sulphates as described in U.S.P. 1,594,689 (cf. B., 1926, 879). R. B. CLARKE.

Protection of wood from natural discoloration. G. GRAU. (G.P. 432,300, 8.5.25).—The wood is coated with a solution of p-toluenesulphonamide hydrochloride or one of its soluble salts, and containing an alkali carbonate, so that it is protected from decomposition by metal salts. The solution itself causes no discoloration and offers the discolouring fungi an alkaline medium, in which the growth of the fungus *Ceratomella* and of other moulds causing discoloration of wood is very markedly inhibited. For example, the wood, as soon as possible after cutting, is painted with or steeped in a 0.5-1%solution of p-toluenesulphonamide hydrochloride or one of its soluble salts to which is added a 5% solution of sodium carbonate. W. T. K. BRAUNHOLTZ.

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

Passivity and corrosion of iron. L. McCulloch (Trans. Amer. Electrochem. Soc., 1926, 50, 197-205).-Of a number of small particles of electrolytic iron isolated from one another, only a certain proportion will rust in the presence of air, moisture, and sodium chloride. The smaller the particles, the greater the proportion which is "passive," but if the particles are in contact with one another they all rust. Again, in a solution of ammonia and ammonium chloride pieces of soft iron may either dissolve readily or remain unattacked. "Passivity" in this solution is produced by heating until temper colours appear, or often merely by cleaning with coarse emery and exposing to the air. The metal is activated by contact with zinc or active iron in the solution or by cathodic treatment at a sufficiently negative potential. Any of these activating agencies will also cause mill-scale or ferrous oxide to dissolve in this solution, so that the observed passivity of iron is ascribed to a superficial oxide film. The progressive corrosion of iron in the presence of air and natural waters may be explained without assuming the intervention of carbon dioxide. Electrolytic action will produce at anodic areas in the presence of oxygen a coating of ferric hydroxide containing within its pores ferric chloride and hydrochloric acid (derived from sodium chloride), both of which attack the underlying metal chemically. Since sodium hydroxide produced at cathodic areas is readily washed away, excess of iron salts spreads over the surface and electrolytic action is displaced by chemical corrosion by oxygen

with these salts as intermediaries. Experimental evidence is cited in support of this theory.

H. J. T. ELLINGHAM.

Properties of electrolytic iron. G. P. FULLER (Trans. Amer. Electrochem. Soc., 1926, 50, 189-195).--Electrolytic iron manufactured by the Niagara Electrolytic Iron Co., using the Boucher-Bouchayer process, contains, after annealing to remove hydrogen : 99.965% Fe, 0.015% Cu, and about 0.005% each of C, Si, S, and P, and has a scleroscope hardness 14-16, a yield point of about 23,000 lb./sq. in., and a tensile strength of about 40,000 lb./sq. in. The metal can be annealed at a high temperature and quenched at once in cold water without appreciably changing its properties. Tubes about 13 ft. long and 9.5 in. diameter are deposited directly and cold-worked to other diameters and wall thicknesses : for a 2.5 in. diameter tube the walls may be as thin as 0.02 in. The high resistance of electrolytic iron to chemical corrosion is further increased by cold-working. H. J. T. ELLINGHAM.

Corrosion of steel by acids. V. DUFFEK (Korrosion u. Metallschutz, 1926, 2, 152—154; Chem. Zentr., 1926, 2, 1328—1329).—The author worked with 2% solutions of sulphuric, nitric, hydrochloric, and oxalic acids at  $20^{\circ}$ , immersing special steels for 24 hrs. in 30 c.c. of the acid, and using test pieces  $10 \times 10 \times 20$  mm. Highly polished steels are attacked less than rougher samples, but for accurate results the solution must be agitated, and the author concludes that several samples should not be placed in the liquid together because of the influence of solubility. The test pieces should be of the same shape and have the same surface area. W. G. CAREY.

Effect of manganese, silicon, and phosphorus on the pearlite interval. B. KJERRMAN (Trans. Amer. Soc. Steel Treat., 1926, 9, 430-451).-The pearlite interval of a number of steels made by different processes was studied by the electric resistance method. In the absence of manganese, silicon, or other alloying element Acl occurred at a definite temperature or over a very small range. Anomalies were found which were thought to be connected with non-metallic inclusions, as crucible steel showed smaller intervals. Manganese depressed the beginning of the interval and widened the range. Silicon raised the interval and extended it. In a siliconmanganese steel the interval was doubled. Heat treatment tending towards homogeneity of structure reduced the range caused by manganese but did not affect the range caused by silicon. The effect of these two elements is explained on the assumption that at the same temperature manganese and silicon are respectively more and less soluble in austenite than in ferrite. The behaviour of phosphorus was similar to that of silicon.

T. H. BURNHAM.

Dependence of the electrical resistance of nickel steel on composition, temperature, and heat treatment. F. RIBBECK (Z. Physik, 1926, **38**, 772–787, 887–907).—After quoting figures showing the lack of agreement obtained by other workers for nickel steels, the author describes his method of determining the resistance and gives his results for iron as pure as possible,  $10 \cdot 10$  microhms/cm., corrected for impurities to  $9 \cdot 62$ ; a table gives the variation in resistance up to  $1000^\circ$ . For 950

the purest nickel obtainable the resistance was 10.6microhms/cm., with an uncertain correction to be made for impurities; the variation up to 500° is given. Tables are given showing the resistances of steels containing various proportions of nickel and carbon for a range of temperature from about 20° to 300°. To obtain the samples as nearly as possible in a condition of equilibrium they were heated at 800° for an hour and then slowly cooled. Some steels, however, showed an "induced effect" ("Anlasserscheinung"), possessing a lower resistance when cooled from 300° to a given temperature than when heated to that temperature. One steel, with 9.55% nickel and 0.59% carbon, was so hard that it could not be turned on the lathe ; it became rather softer after heating at 500° for an hour followed by slow cooling. Reversible and irreversible steels (cf. Osmond, B., 1899, 278) are discussed. In all cases the resistance increases with rise in temperature (unlike certain nickel-chromium alloys), the temperature coefficient being about 0.075microhm/cm./degree. With increasing nickel content the resistance increases rapidly at first, then slowly from 5% to 20%, then again more rapidly; with 29.9% of nickel the resistance is eight times that of pure iron.

E. B. LUDLAM.

Dilatometric method of heat treatment. O. E. HARDER, R. L. DOWDELL and A. C. FORSYTH (Trans. Amer. Soc. Steel Treat., 1926, 9, 403-419).--- A dilatometric heat-treatment furnace is described in which a fused silica rod rests vertically on the steel to be hardened, and the expansion of the steel operates the movable plunger of a dial indicator. The specimen is heated a few divisions above the arrest point and then quenched. By this device tools and dies were more accurately hardened as regards final size than by other methods. For the investigation of cast iron a dilatometer was constructed in which two alined silica push-rods touch the ends of the specimen placed horizontally in the heating furnace. One rod works against the diaphragm of a mercury reservoir fitted with a capillary tube indicator, the other being connected to a micrometer arrangement. During heating the level of the mercury was maintained constant by adjusting the micrometer, the dilation being read off directly on the micrometer dial.

#### T. H. BURNHAM.

Welding of high-chromium alloys intended to meet extreme conditions. S. M. NORWOOD (Trans. Amer. Electrochem. Soc., 1926, 50, 133-136).-Brittleness caused by welding alloys containing more than 10% of chromium is considered a defect inherent in the alloy rather than due to the method of welding. The addition of about 8% of nickel to high-chromium alloys has been found beneficial since it refines the grain and improves the ductility and shock-resistance of the alloy. To counteract the tendency to diminish the resistance of the alloy to corrosion by sulphur gases, the silicon content is increased to about 2%. It has also been found that if the manganese content is made approximately equal to that of the silicon a flux-covered rod becomes unnecessary, the products of oxidation being self-slagging. C. A. KING.

Voltage in copper refining cells. C. G. FINK and C. A. PHILIPPI (Trans. Amer. Electrochem. Soc., 1926, 50, 125-131).-The fall of potential in copper-refining cells, where two copper electrodes are immersed in a dilute sulphuric acid-copper sulphate bath, depends on the fall of potential through the solution and on polarisation of the anode and cathode. Variation of these factors with changes in temperature, acid and copper concentrations of the bath, has been investigated. Rising temperature or acid concentration lowers the potential fall in the solution and decreases the potential difference due to anodic and cathodic polarisation but, with regard to the latter factor, the effect becomes less marked as the acid concentration rises. Increase in copper sulphate concentration causes increased potential fall in the solution but reduces the polarisation effects to a diminishing extent. There is, thus, an optimum copper concentration for production of the minimum overall voltage. Introduction of gelatin into the electrolyte has no effect on the anode polarisation or solution potential fall, but causes a very marked increase in the cathode polarisation, which becomes constant after the addition of a small amount of the colloid.

L. M. CLARK.

Influence of shrinkage on the mechanical properties of alloys of copper and of aluminium. L. GUILLET (Compt. rend., 1926, 183, 541-544).-The physical properties in the untreated and annealed states, and the degree of shrinkage due to forging at high temperatures of various alloys of copper and of aluminium have been determined. The shock tests are affected most by the degree of shrinkage and show distinct maxima, which are highest in the case of aluminium alloys. A great difference exists between the results obtained when the shock tests are applied in the same direction as and perpendicular to the lamination of the sample, and this difference increases beyond a certain degree of shrinkage. Thence the most favourable "degrees of shrinkage" are found to be  $2 \cdot 0$  and  $2 \cdot 5$  for alloys of copper and of aluminium respectively. J. GRANT,

Properties of addition agents in electrodeposition. G. FUSEYA and K. MURATA (Trans. Amer. Electrochem. Soc., 1926, 50, 87-114).-Determination of iron concentrations and of migration numbers shows that copper and silver form complex cations with metaphosphoric acid, organic hydroxy- and amino-acids. Sugars show little, if any, tendency to form such complexes; when copper or silver salt solutions, to which have been added these complex-forming materials, are electrolysed, some of the added material is deposited with the metal. It is suggested that this explains the diminution of grain-size in such deposits. No change of grain-size was noted with substances which do not form complexes, and which are not deposited with the metal during electrolysis. No formation of colloidal particles was observed on mixing silver nitrate and glycine or tartaric acid solutions, and the production of finegrained deposits cannot, therefore, be ascribed to this cause. L. M. CLARK.

Acid zinc plating baths. M. R. THOMPSON (Trans. Amer. Electrochem. Soc., 1926, 50, 219-243).—The importance of the  $p_{\rm H}$  value of the plating bath is indicated, the best value for acid zinc plating in the body of the solution being from 3.5 to 4.5, range measurements being made with bromophenol-blue. At a lower  $p_{\rm H}$  the cathode efficiency is decreased especially at low current densities, and at a higher  $p_{\rm H}$  the deposits are coarse-grained and spongy. The  $p_{\rm H}$  value of the cathode film probably approaches 6.2, i.e., the precipitation point of zinc hydroxide. To maintain the desired  $p_{\rm H}$  the solution must be buffered with aluminium salts from which the hydroxide is precipitated at or before  $p_{\rm H}$ 4.5, so preventing the  $p_{\rm H}$  of the solution from increasing, at least until nearly all the aluminium is precipitated. The deposits from such solutions are whiter and brighter. It was not found possible to increase materially the throwing power of an acid zinc bath as the cathode polarisation is low. The addition of dextrose, dextrin, glycerin, and  $\beta$ -naphthol causes a slight increase in polarisation and throwing power, giving smoother but darker deposits. The conductivity is increased by using zinc chloride instead of sulphate, and adding a large amount of sodium or ammonium chloride; thus, higher current densities can be used. Zinc deposited at low current densities tends to form isolated crystal groups but at higher values the deposits are continuous. A good plating bath was obtained by using a solution containing zinc chloride 136 g., sodium chloride 234 g., and aluminium chloride 20 g. per litre at a current density of 1.5-5 amp./dm.2 for articles of not too irregular shape or from 10 to 15.amp./dm.<sup>2</sup> in agitated baths.

#### F. G. CROSSE.

Equilibria between lead, oxygen, and sulphur in the roasting [of lead sulphide]. SCHENCK (Metall u. Erz, 1926, 23, 326; Chem. Zentr., 1926, II., 1576—1577; cf. B., 1925, 287, 995).—The vapour pressure relationships of lead sulphide are discussed, and the existence of three basic lead sulphates is demonstrated. All these, as well as lead oxide and lead sulphate, can be converted into metallic lead and sulphur dioxide by heating with lead sulphate, if certain conditions are observed. Under other conditions, basic sulphates or lead oxide and sulphur dioxide are formed. In all, 13 equilibria are possible, of which 8 or 9 are accessible to observation. C. T. GIMINGHAM.

Relatively fine-grained deposits from "unsatisfactory" electrolytes. E. A. VUILLEUMIER (Trans. Amer. Electrochem. Soc., 1926, 50, 83-86).—A dense, fine-grained deposit of lead is obtained by electrolysis of a lead acetate solution on a copper cathode which is covered with a film of glycerin, whereas a loose, rough coating is produced in the absence of the glycerin film. The effect is not so strongly marked when the acetate solution is acidified with nitric in place of acetic acid, nor when a concentrated sugar solution is substituted for the glycerin. A more dense deposit of silver from a nitrate solution is similarly produced by the use of a gold or silver cathode, previously wetted with distilled water or glycerin. L. M. CLARK.

Cadmium : its electrodeposition for rust-proofing purposes. C. M. HOFF (Trans. Amer. Electrochem. Soc., 1926, 50, 269—278).—A comparison of the physical and chemical properties of cadmium with those of zinc indicates that the former should be the better for the rust-proofing of iron; it is less active chemically, forms a protective oxide film, is not amphoteric in character, is on the alkaline-earth side of iron in the electromotive series of metals, and, although softer than zinc, is more ductile. A satisfactory plating bath consists of a mixture of cadmium hydroxide, ammonium sulphate, and "gulac" dissolved in a solution of sodium cyanide and sodium hydroxide, using cadmium anodes. This solution has a specific resistance of 4.9 ohm./c.c. at  $24^{\circ}$ . The anode polarisation potential is 0.24 volt, the cathode 0.44 volt at a cathode current density of 2.7 amp./dm.<sup>2</sup> The best results are obtained with an anode current density of 6.5—7.6 amp./dm.<sup>2</sup> F. G. CROSSE.

Metals to resist corrosion or high temperatures. H. J. FRENCH (Trans. Amer. Electrochem. Soc., 1926, 50, 47-81).-A résumé of the characteristics of metals and alloys used industrially to resist high temperatures or corrosion. The metals copper, aluminium, lead, tin, silver, nickel and iron, and their alloys, are considered. Tests on the steam erosion of metals made in Switzerland, in which a jet of steam was directed upon specimens of different metals at a stated velocity of 3600 to 3900 ft. per sec. for 70 hrs., showed that both low- and mediumcarbon high-chromium steels, e.g., stainless steel and iron, possessed favourable characteristics, but the best result was obtained with high-carbon nickel steel which also contained appreciable proportions of chromium, silicon, and manganese. C. A. KING.

Refractories for melting pure metals : iron, nickel, platinum. L. JORDAN, A. A. PETERSON, and L. H. PHELPS (Trans. Amer. Electrochem. Soc., 1926, 50, 115-124).-Crucibles made from commercial fused oxides, alumina, zirconia, and magnesia, were unsuitable for melting pure iron, the alumina and zirconia because of contamination of the melt by silicon and the magnesia because of sulphur. Strong and dense crucibles can be made from calcined, chemically pure magnesia mixed with 2% of magnesium chloride and about 10% of water, and such crucibles fired to about 1600° enable pure melts to be obtained. Magnesia crucibles bonded with shellac and alcohol are also satisfactory, and materials found suitable for melting pure iron were satisfactory also for nickel. Crucibles made of zirconia freed from carbon. and iron soluble in acid proved suitable for melting pure platinum and platinum-rhodium alloys, providing the melting is done under oxidising conditions. Electrically sintered zirconia is extracted with hydrochloric acid (1:1), roasted, and prepared as a casting slip by grinding with about 4% of kaolin. Crucibles are then cast, dried, and fired carefully to 1700°. Although hard and strong, zirconia crucibles do not withstand sudden temperature changes, and are best used with an outer supporting crucible of alundum or porcelain.

C. A. KING.

Failure of metals by fatigue. H. F. MOORE (Trans. Amer. Soc. Steel Treat., 1926, 9, 539-552).—When slip occurs in a metal crystal due to applied stress, the crystal splits into laminæ in a direction determined by the planes of weakness in the grain. Under repeated stresses the serrated edges of the crystal and the roughened surfaces of the laminæ behave like minute files. The result of slip under alternating stresses is the net balance of two actions, an increase in the resistance to slip, and a gradual filing action tending to start and spread cracks. After many cycles at low stresses, mild steel resists reversed stresses many times better than if initially unstressed. Cold work by tensile stress lowers the resistance to repeated stress, but rolling packs the crystalline grains closer and tends to prevent the filing action due to slip. T. H. BURNHAM.

# PATENTS.

Production of cast iron. MEIER & WEICHELT (E.P. 234,106, 12.5.25. Conv., 13.5.24).-Instead of producing the graphite in cast iron by means of the secondary decomposition of cementite, in the form of laminæ or veins, a finer grade of cast iron is obtained, consisting of mixed crystals and a eutectic mixture of graphite and iron, by taking an ordinary iron from a cupola furnace (Si about 3.5%), and rapidly cooling in an iron mould. The rate of cooling may be reduced in proportion as the carbon content of the iron is lower. The eutectic graphite so produced is distributed in fine particles, the size of which is no larger than the breadth of the finest striations of lamellar pearlite, and is especially useful for the production of high quality grey iron having a tensile strength of 30-40 kg./mm.<sup>2</sup> and bending strength of 50-80 kg./mm.<sup>2</sup>. If, however. a little white iron be also produced, this is easily broken up by short heating at about Acl, when the cementite is very rapidly decomposed into ferrite and temper carbon.

F. G. CROSSE.

Production of copper from spent pyrites. F. CURTIUS & Co. (G.P. 431,387, 31.1.25; see G.P. 427,011). —The burnt ore is leached, and when dry subjected to magnetic separation. The magnetic portion is treated for copper by subjecting to a chloridising roast, or by heating with fresh pyrites, according to G.P. 427,011 (cf. E.P. 230,415, B., 1925, 597), while the non-magnetic fraction is used direct for the production of iron. The additional preliminary leaching is preferable to magnetic separation only. W. G. CAREY.

Depositing copper from solutions. ORKLA GRUBE AKT. (E.P. 248,724, 16.2.26. Conv., 6.3.25).—Copper from non-metallic ore products, such as pyrite cinders subjected to chlorination roasting, is recovered by leaching them and depositing the copper from solution by means of scrap brass or other alloy of copper rich in zinc. F. G. CROSSE.

Imparting various colours to brass articles. F. A. E. STAHLSCHMIDT (E.P. 258,796, 24.3.26).—The articles are immersed in a cold solution of potassium hydroxide, potassium sodium tartrate, and copper sulphate, the time of immersion determining the final colour of the articles. A final treatment with dilute acid produces a golden appearance. F. G. CROSSE.

Lead and silver from lead-zinc sulphide ores. E. LANGGUTH (G.P. 430,919, 1.1.25).—The recovery of lead and silver from sulphide ores by treatment with zinc in a melt of zinc chloride and alkali chloride is modified by the addition of alkaline-earth chlorides, or such alkaline-earth compounds as will be converted into chlorides by the zinc chloride, to the fluxing salts or to the ore, in an amount at least equivalent to the sulphate content of the ores. The high consumption of zinc due to the sulphate content of the ores before subjection to the froth flotation process is thereby reduced.

Lead alloys. C. T. J. VAUTIN and C. V. STEPHENS (E.P. 257,676, 5.6,25).—An alloy of lead containing 0.1-2.0% of cadmium is used for extruded metal work, an alloy of such composition forming no eutectic which segregates on solidification from the molten state. The alloys are stronger and less corrodible than lead itself; their uses include the production of composition piping and sheathing for electric cables. C. A. KING.

**Decomposition of chromium ores.** I.-G. FARB-ENIND. A.-G., Assees. of R. CASPARI (G.P. 431,644, 23.3.24).—The ore, mixed with an alkaline material, is spread in a thin layer, and is exposed without agitation to the action of oxidising gases. W. G. CAREY.

**Preparation of alloys [of beryllium and silver].** BERYLLIUM CORP. OF AMERICA, Assees. of H. S. COOPER (E.P. 257,473, 21.12.25. Conv., 2.12.25).—The introduction of beryllium into an alloy of silver increases the resistance of the alloy to tarnishing in a marked degree. Such alloys may comprise beryllium and silver only or may contain up to 10% of each metal. Alloys of this character are made by electrolysing a beryllium salt using a cathode of molten silver. C. A. KING.

Concentration of ores containing metals of the platinum group. MINERALS SEPARATION, LTD., and J. C. MOULDEN (E.P. 258,648, 22.6.25).—In the frothflotation concentration of an aqueous pulp of ore containing metalliferous matter and gangue in the presence of a protective colloid (e.g., starch, glue, gelatin, etc.), better separation may be secured by adding alkaline xanthate to the ore pulp so that it is present with the colloid. A Norite sulphide ore from South Africa which before treatment assayed 0.21% Ni, 0.11% Cu, and 2.1 dwt. of platinum metals per ton, gave a concentrate containing 73.7 dwt. of platinum metals, 7.68% Ni, and 3.75% Cu. F. G. CROSSE.

Preventing formation of crystals and deposition of incrustations on metal surfaces. ANTISCALE Co., LTD. (E.P. 236,534, 15.6.25. Conv., 3.7.24).—The walls of the containing vessel are subjected to the action of a magnetic field or weak electric current. Thus crystals form as a fine loose powder, and boiler scale as a soft pulp or sludge. F. G. CROSSE.

Tungsten wire (E.P. 258,642).—See XI.

Heat treatment by electricity (E.P. 258,049).—See XI.

# XI.—ELECTROTECHNICS.

**Refractories for induction furnaces.** M. UNGER (Trans. Amer. Electrochem. Soc., 1926, **50**, 41-46).— For induction furnaces of the horizontal ring, iron-core type, operating at 1500° to 1800°, magnesium oxide proved to be the most satisfactory lining, and when properly constructed would have a life of 400 heats with slags containing 20 to 25% of silica. Proper grading of the magnesium oxide is important, and pitch was found to be the best bonding material. To obtain a lining of maximum density a mixture known as "Furnite" (U.S.P. 1,444,527, B., 1923, 310 A) was rammed carefully to form the contour of the melting crucible. Inorganic bonds, *e.g.*, magnesium salts, ball clay, sodium silicate, proved unsatisfactory. C. A. KING.

Thermal insulation of electric furnaces. M. L. HARTMANN and O. B. WESTMONT (Trans. Amer. Electrochem. Soc., 1926, 50, 155-176).-Thermal conductivities of a high-temperature insulating refractory (B., 1925, 761), of fused alumina and fused magnesia refractories, and of fireclay, have been determined and their variations with temperature recorded on a graph, with the corresponding data for carborundum refractories (B., 1925, 549) and silica. The chemical composition, bulk specific gravity, and porosity of these materials are stated, and available data for their mean specific heats have been collected. From these figures and values for heat losses from rough, vertical, plane surfaces (Trinks, "Industrial Furnaces"), calculations are made of the temperature gradients, heat capacities, and heat losses of furnace walls of various thicknesses and inside temperatures, these walls being either of simple refractories or of refractories with an external insulation. The use of such external insulation is important with "super-refractories" such as carborundum, since otherwise heat losses can only be kept low by having very thick walls, thereby increasing the external surface and heat capacity of the walls. A high heat capacity limits flexibility of operation, especially where intermittent working is desired. The above-mentioned insulating refractory can be used up to 1450° and is therefore suitable for backing relatively thin walls of super-refractory. Such composite walls may be designed to give greatly improved energy conservation. Present methods of insulating various types of electric furnace are briefly reviewed.

#### H. J. T. ELLINGHAM.

Products formed during the working of lead accumulators. L. MAZZA (Atti R. Accad. Lincei, 1926, [vi], 4, 215—218).—By means of the Debye-Hull powder method it is shown that the substances formed during the charging and discharging of lead accumula tors include pulverulent and massive lead, lead sulphate, lead peroxide, and mixtures of these substances in various proportions. T. H. POPE.

See also pages 950, Copper-refining cells (FINK and PHILIPPI); Addition agents in electrodeposition (FUSEYA and MURATA); Acid zinc plating baths (THOMPSON). 951, Fine-grained deposits from "unsatisfactory" electrolytes (VUILLEUMIER); Electrodeposition of cadmium (HOFF).

#### PATENTS.

Tungsten wire [for electric lamps]. EDISON SWAN ELECTRIC CO., LTD., and G. A. PERCIVAL (E.P. 258,642, 20.6.25).—The introduction of beryllium silicate in small quantities, not exceeding 0.9%, into tungsten wire, for the purpose of filaments in electric lamps, is found to minimise the tendency of the wire to change its crystalline structure when heated by an electric current. Colloidal solutions of pure silica and beryllium oxide are mixed in such proportions that on fusion the resultant compound is equivalent to the meta- or ortho-silicate.

F. G. CROSSE.

Galvanising bath. W. A. F. PFANHAUSER, and LANGBEIN-PFANHAUSER-WERKE A.-G. (E.P. 259,072, 21.12.25).—In a galvanising bath of the travelling type in which articles to be treated are suspended and subsequently removed from a travelling device at substantially the same point, the electrolyte is continually pumped through a filter into the galvanising chamber in such manner that the bath liquid is agitated and anode slime removed, thus enabling a high current density to be employed to galvanise the articles during a single passage through the bath. J. S. G. THOMAS.

Heat treatment [surface hardening] of materials by electricity. W. J. BROWN, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (E.P. 258,049, 23.7.25).— Material to be treated, *e.g.*, metal, is placed within a solenoid through which is passed an alternating electric current of such frequency that heat is developed chiefly at the surface of the material. The material is quenched while still subjected to the alternating field.

J. S. G. THOMAS,

# XII.—FATS; OILS; WAXES.

Olive oil production in Tunis. M. ROUSSEAU (Bull, Mat. Grasses, 1926, 163-193).-The number of olive trees in Tunis is about 8,500,000, and is increasing; the yield of oil is irregular, with an annual mean of 300,000 quintals, Tunis being fifth in order of productivity after Spain, Italy, Greece, and Portugal. A commission under the Department of Agriculture of Tunis examined in 1923-1925 the various factors affecting the separation of olive oil from the fruit pulp and the yield and quality of the oil. The fruit was broken up in a mill, subjected to preliminary and final pressing in a modern type of mechanically-operated oil press, and the oil decanted from the pulp by some form of separator. The following results are reported :--(1) Elimination of leaves and stalks and washing of the olives are essential to the production of oil of superior quality; the type of plant to carry out these operations is briefly indicated. (2) Influence of duration of storage of olives in ordinary silos is seen in rapid deterioration of the quality of the oil by reason of fermentation set up; olives kept two days gave oil of free fatty acids 0.5%, and kept 18 days gave oil of free fatty acids 3%. (3) Under comparable conditions the results when storing in closed silos were worse than when using ordinary ones. (4) When stored on wooden screens placed one above the other stagewise, the olives yielded better oil than when stored in silos because of freer circulation of air, less internal heating, and therefore less chance of fermentation. (5) The influence of salting on olives kept for 36 days is to hinder the development of moulds on the fruit and high acidity of the oil and equally to hinder elevation of temperature of olives stored in heaps and to diminish the intensity of any fermentation. It is advisable, however, to apply salting only to those olives of poor quality or to those that have commenced to undergo change, since salting imparts an objectionable flavour to the oil. (6) When investigating the duration and fineness of the breaking or crushing of the fruit it was found that the total yield of olive oil increases with duration of the crushing; the yield of oil of first pressings increases with duration of crushing, while the yield of oil at second pressing diminishes. The quality of oil diminishes with increase in the duration of the crushing and this diminution begins to be felt when the duration is beyond 30 to 40 minutes. (7) A comparison of the methods of separating the oil from the accompanying pulp afterpressing shows that the most advantageous is the use of the Hignette centrifuge, the Sharples centrifuge closely following in merit, whilst the automatic washing separator appears least satisfactory. Moreover, the quality of oil when using the last-named method of separation is certainly inferior to that obtained by the other methods, the acidity of first pressings oil being 0.75%, and of second pressings 1.9%, the acidity of the oil in the olives being originally 0.5%. H. M. LANGTON.

Vegetable oils containing glycerides of erucic acid. J. J. SUDBOROUGH, H. E. WATSON, and P. R. AYYAR (J. Indian Inst. Sci., 1926, 9A, 25—70).—Full analytical constants of the oils are given and the solid and liquid acids, separated by Twitchell's lead salt-95% alcohol method (B., 1921, 817 A) have been analysed by analysis of the fractionated methyl esters. The refractive index-iodine curves for the hardened oils have also been studied (cf. B., 1924, 755).

[With N. R. DAMLE.] Expression of the seeds of Brassica campestris var. napus yielded 38% of rape oil having an acetyl value of 31, due to the presence of mono- or di-glycerides. The composition of the acids from the oil was : myristic 1.5%, stearic 1.6%, behenic 0.5%, lignoceric 2.4%, oleic 20.2%, erucic 57.2%, linoleic 14.5%, linolenic 2.1%, and from the hardened oil : myristic 1.5%, stearic 38.5%, behenic 57.6%, lignoceric 2.4%.

[With V. M. MASCARENHAS.] A yield of  $35 \cdot 2\%$  of Indian mustard oil ("rai") was obtained by extraction of seeds of *Brassica juncea*, D.C. The acids from the oil had the composition : myristic 0.5%, stearic nil, behenic  $3\cdot8\%$ , lignoceric  $1\cdot1\%$ , oleic  $32\cdot3\%$ , erucic  $41\cdot5\%$ , linoleic  $18\cdot1\%$ , linolenic  $2\cdot7\%$ , and those from the hardened oil : myristic 0.5%, stearic  $52\cdot1\%$ , behenic  $46\cdot3\%$ , lignoceric  $1\cdot1\%$ .

[With T. J. MIRCHANDANI.] Appreciable differences existed between the extracted and expressed Jamba oils from the seeds of *Eruca sativa*, Lam, the latter closely resembling Indian rape oil and having an acetyl value of 22. The acids from the expressed oil consisted of : stearic 4.2%, behenic 4.5%, lignoceric 1.8%, oleic 28.7%, erucic 46.3%, linoleic 12.4%, linolenic 2.1% and those from the hardened oil : stearic 47.4%, behenic 50.8%, lignoceric 1.8%. A sulphur compound, which inhibited hydrogenation, could only be removed by hot extraction of the oil with 88% alcohol. Maceration of the seeds with water containing sodium fluoride followed by steam distillation gave an average yield of 0.8% of a volatile oil having a penetrating odour and containing sulphur and nitrogen, but differing considerably in properties from the essential oil of rape seed. Attempts to isolate the glucoside were unsuccessful.

[With N. R. DAMLE]. The seeds of *Tropxolum majus*, Linn., on extraction yielded 7.2% of oil having  $d^{15\cdot5}$ 0.9092,  $n_D^{40}$  1.4568, iodine value 77.5, saponif. value 172.6, and unsaponifiable matter 1.1%. The mixed fatty acids had Hehner value 95.1, iodine value 72.9, and mean mol. wt. 312.8. The solid acids ( $45\cdot7\%$ ) had iodine value 72.9, mean mol. wt. 330.0, and the liquid acids ( $54\cdot3\%$ ) had iodine value 72.6 and mean mol. wt. 298.0. Tribrassidin (m.p. 56—57°,  $n_D^{60}$  1.4547) and tribehenin (m.p. 81—81.5°,  $n_D^{85}$  1.4391,  $n_D^{89}$  1.4375) were prepared from trierucin (m.p.  $30 \cdot 5 - 31 \cdot 0^{\circ}$ ,  $n_D^{40}$ 1.4630,  $n_D^{60}$  1.4560,  $n_D^{81}$  1.4475) crystallised from the oil. The m.p. of purified behenic acid prepared by six different methods ranged from 79.3° to 80°. The solidifying point curves of mixtures of methyl stearate and methyl behenate and the solidifying and melting point curves of mixtures of stearic and behenic acids have been determined. E. H. SHARPLES.

Oil of Pongamia glabra. G. D. BEAL and M. C. T. KATTI (J. Amer. Pharm. Assoc., 1925, 14, 1086-1096; Cham. Abstr., 1926, 20, 2895).-The seeds contain 27-33% of oil of a bitter taste and unpleasant odour, saponification value 189.1, iodine value (Hanus) 89.1, Reichert-Meissl value 1.04, unsaponifiable matter 2.4%, acid value as oleic acid 8.36%, soluble acids as butyric acid 1.96%, insoluble acids together with unsaponifiable matter 93.25%, neutralisation value of insoluble acids 196.1, mean mol. wt. of insoluble acids 286.0, iodine value (Hanus) of insoluble acids 92.6. Distillation in steam removes most of the odorous constituents, and the bitter principle (unsaponifiable) is extracted with 76% ethyl alcohol. A substance, m.p. 157.5°, becoming red in sunlight, constituted or contained the bitter principle. A. A. ELDRIDGE.

Effect of  $p_{\rm H}$  on the germicidal action of soaps. A. H. EGGERTH (J. Gen. Physiol., 1926, 10, 147-160).— The action of various fatty acids (butyric, caproic, capric, caprylic, undecoic, lauric, tridecoic, myristic, pentadecoic, palmitic, stearic, oleic, and ricinoleic, and their salts at different hydrogen-ion concentrations on a number of bacteria (*Streptococcus pyogenes*, *B. diphtheriæ*, *Staphylococcus aureus*, *B. typhosus*, and *Vibrio choleræ*) depends both on the acid and the particular bacterium used, but in general the lower acids of the series are more active at an acid reaction, whereas the higher numbers are more active at an alkaline reaction. W. O. KERMACK.

#### PATENT.

Manufacture of soaps. A. H. BAILY and J. N. KIRBY (E.P. 256,362, 6.6.25).—Hard soaps particularly suitable for degreasing fibres and for the degumming of certain vegetable fibres are made by adding an alkali protein solution to the usual mixture of saponified fatty oils. The protein solution, made by dissolving caseinogen or the like in excess of alkali and adding 0.5-1.0% of formalin, is added in quantities as required to give the desired quality of soap. Similarly, the protein solution may be used with the desired combination to give liquid soaps and dry soaps. H. M. LANGTON.

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

Importance of particle properties in paint pigments. C. A. KLEIN (J. Oil and Colour Chem. Assoc., 1926, 9, 192—196).—Both chemical and physical investigations are necessary in connexion with the problems offered by pigments. The variation and irregularity in the particle shape and size are matters of much importance to the paint manufacturer. The presence of oversized particles has an effect disproportionate to their number. Much has still to be learnt as to the effects produced by so-called "inert extenders." Various problems awaiting attention are indicated.

D. F. Twiss.

Influence and elimination of coarse particles [from pigments]. N. HEATON (J. Oil and Colour Chem. Assoc., 1926, 9, 215—218).—The particles of pigments can be conveniently classified into three groups, viz., "coarse"—over 60 microns in diameter, "intermediate"—between 10 and 60 microns, and under 10 microns. In most pigments the intermediate and coarse particles are present only in small percentage. Probably the most difficult pigments to obtain in a satisfactory degree of fineness are those prepared by successive precipitation and calcination. D. F. Twiss.

Use of asbestine (micro-asbestos) in the paint industry. H. ROSENBERG (Farben-Ztg., 1926, 31, 2936).—The properties of European and American asbestine are discussed, the latter being almost pure magnesium silicate, whilst the composition of a typical sample of the former is as follows :—silica  $49 \cdot 81\%$ , alumina  $2 \cdot 19\%$ , iron oxide  $2 \cdot 80\%$ , magnesia  $27 \cdot 96\%$ , lime  $13 \cdot 21\%$ , water  $3 \cdot 91\%$ , and traces of manganous oxide. Asbestine finds application in the paint industry as an inert extender, a base for lake pigments, and a desirable constituent of acid-, alkali-, and fire-resistant paints. S. S. WOOLF.

Wetting power of solvents, and their behaviour on evaporation. H. VOLLMANN (Farben-Ztg., 1926, 31, 2932-2934).-Various solvents used in the paint and varnish industry were dropped from a capillary pipette on to brass and glass plates, and on to duplicate plates that had been washed with the particular solvent and wiped dry. The volume of a drop, extent to which it spread, its appearance and subsequent behaviour in the course of volatilisation are tabulated and discussed. Greater spreading and more rapid evaporation were found on brass than on glass. Preliminary washing of the plates increased the spreading power, by displacement of an adsorbed gas-layer by a layer of the solvent. The wetting power of solvents is dependent to some extent on their chemical structure, paraffins, for instance, showing marked differences from benzene and its S. S. WOOLF. homologues.

Rapid evaluation of baked japan finishes. E. M. HONAN and R. E. WATERMAN (Ind. Eng. Chem., 1926, 18, 1066—1068).—It is proposed to evaluate the service life of a japan film baked on metal by immersing test pieces in an 8.5% solution of phenol in water (which has constant composition at normal room temperature) until at least half the japan has wrinkled or has peeled off. For the same japan, the time of resistance to phenol is found to depend on stoving time and temperature, nature of metal coated, and condition of the latter before coating. The method gives results consistent with those of parallel service tests. S. S. WOOLF.

Condensation of formaldehyde with phenols and urea. H. BARTHÉLEMY (Chim. et Ind., 1926, 16, 367— 372).—A discussion of the mechanism of condensation. By addition of aqueous alcohol to an alcoholic solution of the partially condensed product a mixture of two colloidal suspensions is obtained, which can be separated into positive and negative components by the action of an electric field. These suspensions, which possess the Brownian movement, are regarded as ionic miscelles having the constitutions :  $[(\Sigma_1^n C_6H_4O, CH_2)H]OK, +ve$ , Painting treated wood. DUNLAP.-See IX.

Detection of grit in rubber pigments. MURPHY.— See XIV.

#### PATENTS.

Production of [green] pigment colours. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 257,528, 21.4.26).—The shade of the green pigment colours produced by the process described in E.P. 181,584 (B., 1922, 600 A) is influenced by the addition of basic dyestuffs, preferably of the malachite-green series, the fastness to light and alkalis being unimpaired and sometimes improved. Improvements involving the addition of Turkey red oil, or some other dispersing agent, and an alternative order of precipitation are also claimed. S. S. WOOLF.

Production of permanent pigments. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (E.P. 242,282, 28.10.25. Conv., 31.10.24).—Small quantities of acid adhering to pigments after precipitation (particularly titanium dioxide) are neutralised by zinc oxide or zinc hydroxide in aqueous suspension before the filtering and drying processes. Kneading the moist pigment with zinc oxide is an alternative method of neutralisation.

S. S. WOOLF.

Resinous condensation products from phenols and formaldehyde. Soc. VERRERIES FOLEMBRAY (F.P. 563,777, 27.6.22).—Mixtures of phenols with about an equal volume of 40% formaldehyde solution containing 5—10% of calcium chloride are heated until the plastic products are no longer sticky when cooled. The products when first formed are soluble in alcohol and acetone, and can readily be moulded into shape or rolled into thin sheets, or they can be mixed with rubber to accelerate vulcanisation, but on further heating or on prolonged storage at the ordinary temperature they turn hard and insoluble; they are especially adapted for the manufacture of varnish for wooden floors and ships' hulls.

L. A. Coles.

Resinous condensation products from phenols and formaldehyde. J. BRUHAT (F.P. 575,532, 21.3.23). —Liquid phenol-formaldehyde condensation products are mixed with esters of polyhydric alcohols and organic acids. For example, the product obtained by condensing phenol with hexamethylenetetramine, trioxymethylene, or paraformaldehyde in the absence of a catalyst, is mixed with 10% of glycerin mono-oxalate. The product can be partially hardened by storage at 15° for several hours before it is moulded into shape, and the hardening process completed by heating, e.g., at 80—100°, or it can be moulded immediately and hardened by heating to 60—100° in the air, or solutions of it in alcohol or acetone can be used as varnish.

L. A. Coles.

Resinous condensation products from phenols and formaldehyde. E. ROPP (F.P. 564,575, 12.7.22).— Phosphorus pentachloride, with or without the addition of sodium acetate, phosphorus oxychloride, or acetyl chloride, is used as catalyst in the condensations. For example, a mixture of phenol, b.p. 182-184°, purified by distillation over aluminium filings, with 40% formaldehyde or trioxymethylene free from iron, and a little phosphorus pentachloride or oxychloride, is heated to  $45-50^{\circ}$ , whereupon the temperature rises to  $97^{\circ}$ . The mixture is then heated at the boil for 20 min. After withdrawing the aqueous layer, the product is washed, heated to 100°, concentrated in vacuo at 160-170° until it is of the consistency of honey, and then heated to 180-190° in an autoclave for 2 hours. The product is hard and transparent, and possesses high dielectric properties. Products obtained in a similar manner from the homologues of phenol are usually cloudy. L. A. Coles.

Preparation of resinous bodies derived from acetaldehyde. L. H. BAEKELAND and A. H. GOTTHELF, Assrs. to BAKELITE CORP. (U.S.P. 1,598,546, 31.8.26. Appl., 19.12.19).—An infusible condensation product results from the reaction of a substance containing an active methylene group with a condensation product of a phenol and acetaldehyde. S. S. WOOLF.

Roll or cylinder mills for grinding paint etc. (E.P. 257,727).—See I.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Röntgen effect of stretched gels, especially of caoutchouc. W. OSTWALD (Kolloid-Z., 1926, 40, 58-73) .- Natural caoutchouc has a typically cellular structure; the cells consist of latex drops, of which the outer membranes are composed of highly polymerised hydrocarbons, whilst the gelatinised cell-contents consist of a network of uniform rod-like isoprene chains or polymerides, embedded in a dispersion medium of hydrocarbons of lower molecular weight. In the unstretched condition the distances between the meshes of the net are greater than the X-ray wave-length, but with sufficiently great stretching in one direction, a spindleshaped or cylindrical latex cell may be formed in which (a) the net meshes occupy a uniform three-dimensional arrangement in space; (b) the rod-like micelles forming the net approach one another until they come within the range of the X-ray wave-length; and (c) the net, previously not necessarily rigid, becomes an immovable lattice by the strong tension on the cell membrane. The latex cell thus becomes a crystal lattice body. The theory is confirmed by the quantitative observations of Hauser and Mark (cf. B., 1926, 334, 761).

#### L. L. BIRCUMSHAW.

[X-Ray] interference phenomena in stretched rubber. S. REINER (Gummi-Ztg., 1926, 41, 133—135).— A review of recent investigations and theories as to the structure of rubber, with especial reference to the results of X-ray spectrographic research. D. F. Twiss.

Has caoutchouc been synthesised? J. R. KATZ (Koll.-Chem. Beih., 1926, 23, 344—348).—Synthetic caoutchouc prepared by the polymerisation of isoprene has not the same elasticity as the natural substance. On freezing in liquid air and then smashing by a blow, the cleavage is quite different. X-Ray examination of strongly stretched natural caoutchouc reveals a crystal-

line structure caused by parallel arrangement of the particles. No such structure is revealed by X-ray examination of synthetic caoutchouc. E. S. HEDGES.

Influence of particle size in rubber manufacture. S. S. PICKLES (J. Oil and Colour Chem. Assoc., 1926, 9, 204–207).—The influence of particle size of compounding ingredients on rubber is discussed, with special reference to varieties of litharge, zinc oxide, and carbon produced by different processes. D. F. Twiss.

Particle shape [of powders for incorporation in rubber]. P. SCHIDROWITZ (J. Oil and Colour Chem. Assoc., 1926, 9, 208—210).—A review of the effect of the shape of the particles of a rubber ingredient on the development of "calender grain" in the manufactured article. D. F. Twiss.

Particle size effects in rubbers subjected to repeated stress. T. R. DAWSON (J. Oil and Colour Chem. Assoc., 1926, 9, 211-214).-Mixtures of a parent stock (100 vols.), containing rubber (95 pts. by weight) and sulphur (5 pts.), with various fillers (20 vols.) were submitted to comparative tests. The effect of the powders (barytes, clay, ordinary and "colloidal" zinc oxide, magnesium carbonate, lamp black, and gas black) on the tensile strength and resistance to extension is greater the smaller the particle size; the hysteresis (at 260 stresses per min.) is more marked the smaller the particles, as also is the scleroscope hardness. Volume increase under strain, however, is only approximately related to particle size, the degree of adhesion of the particle to the rubber being also probably a serious D. F. Twiss. factor.

Importance of particle character in a rubber "pigment." D. F. Twiss (J. Oil and Colour Chem. Assoc., 1926, 9, 197—203).—A summary of the various effects produced by different finely-divided compounding ingredients in rubber, and of the particle features associated with these effects. The abrasion resistance of vulcanised rubber containing carbon black is affected in a less degree than the tensile strength by the presence of coarse particles in the carbon black; the "resilient energy" of such a mixture, therefore, does not supply a simple measure of the abrasion resistance.

# D. F. Twiss.

Detection of grit in rubber pigments. E. A. MURPHY (J. Oil and Colour Chem. Assoc., 1926, 9, 219—225).—A modified elutriator permitting the economical use of special media for the avoidance of incomplete dispersion, is described. For most powders a solution containing 0.5% of sodium hydroxide and 0.1% of glue in distilled water is advantageous. The usefulness of an elutriator, however, is limited, and for most purposes the sieving apparatus of Gallie and Porritt (p. 935) offers advantages. D. F. TWISS.

Origin of [X-ray] interferences in the stretching of rubber. E. A. HAUSER (Z. Elektrochem., 1926, 32, 463-467).—See B., 1926, 638.

#### PATENTS.

Concentrating caoutchouc latex etc. K. D. P., Ltd. (E.P., 243,016, 13.11.25. Conv., 14.11.24).— Natural, preserved, or vulcanised latex of rubber, guttapercha, balata, or analogous materials is dehydrated in a horizontal rotating cylindrical chamber by the action of a current of heated air or other gas, which is first led to the bottom of the chamber so as to effect initial contact with the bulk of the latex ; evaporation is aided by rotary discs, concentric with the chamber, which dip in the bulk of the latex and continually expose fresh surface films to the action of the gas. With this method of procedure frothing and skin formation are avoided. D. F. TWISS.

Utilisation of rubber-bearing plants. F. T. LAHEY (U.S.P. 1,597,807, 31.8.26. Appl., 25.4.23).— The material of a rubber-bearing plant is reduced to a plastic mass, dried, and vulcanised. D. F. TWISS.

Process for devulcanising vulcanised rubber. C. F. WILLARD (U.S.P. 1,598,470, 31.8.26. Appl., 4.1.26).—Vulcanised rubber, together with fibrous material, is heated with a solution of an emulsoid colloid, the fibre being concurrently converted into colloidal cellulose; the mixture of devulcanised rubber and cellulose constitutes a product of superior quality and increased yield. D. F. Twiss.

Vulcanisable plastic material. F. T. LAHEY (U.S.P. 1,597,808, 31.8.26. Appl., 18.10.23).—A vulcanisable plastic material is produced by milling ground vulcanised rubber, and concurrently introducing rubber latex and emulsified oils. D. F. Twiss.

Manufacture of rubber with innumerable microscopically small pores. H. BECKMANN (E.P. 257,561, 3.8.26. Conv., 1.9.25; cf. E.P. 240,430, B., 1925, 1000). Latex mixed with sulphur or other vulcanising agent is coagulated by means of a manganese salt, particularly manganese sulphate; the coagulum forms a firm jelly which may be vulcanised in the manner described previously. The coagulant is removed by washing either before or after vulcanisation. D. F. TWISS.

Caoutchouc compositions. A. BIDDLE (E.P. 257,718, 31.7.25).—Latex of rubber, gutta-percha, balata, or similar substances, mixed with tapioca meal, with the addition of casein and filling materials if desired, is of use in the manufacture of insulating materials, adhesives, binding materials, etc. D. F. TWISS.

# XV.—LEATHER; GLUE.

Rôle of colloid mills in utilising tanning materials in the tanning of skins. U. J. THUAU (J. Soc. Leather Trades Chem., 1926, 10, 258-263).-The difference between the results obtained by tanning hides with liquors extracted from the tanning material, e.g., oak bark, quebracho wood, and liquors prepared from extracts of these materials is due to the agglomeration of colloidal tan (cf. B., 1923, 367). The concentration of the weak tan liquors results in the aggregation of the coarser colloidal particles and consequent precipitation when diluted again. When divi divi powder containing 21.9% of insoluble matter was powdered in a Kek (" colloid ") mill (7500 r.p.m.), a 20% colloidal solution of the product contained 1.8% of insoluble matter and 7.1% of tans; after mixing the treated powder with water and passing it through the mill, the insoluble matter was 2.3%, and the tan 8.0%; after keeping the mixture for 8 days its tannin content had fallen to 6.4%. When the divi divi powder was bruised in

a mortar the solution obtained contained only 1.3%of insoluble matter, but the clear liquor obtained after keeping for 8 days contained only 4.0% of tans. Similar tests on mimosa bark and sumac are described. D. WOODROFFE.

Determination of the degree of tannage [of leather] by means of the water-resistance test. Influence of drying on the water-resistance of hide powder. O. GERNGROSS and R. GORGES (Collegium, 1926, 391-397).—The water-resistance of a leather is defined as the percentage of hide substance in a leather which remains undissolved after treatment with boiling water for 7 hrs. The equivalent of 1 g. of dry hide substance is weighed into a flask fitted with a stirrer, and 100 c.c. of boiling water are poured into the flask, which is kept at 100° in a water bath for 7 hrs. The liquor is then strained through linen, 2 c.c. of the filtrate are analysed by the micro-Kjeldahl process, and the percentage of dissolved hide substance is determined. The percentage of undissolved hide substance, i.e., the water-resistance, is found by difference. Drying hide powder at ordinary temperatures increased the water-resistance slightly, but drying at 110° for 24 hrs. increased it from 2 to 41. D. WOODROFFE.

Effect of hydrogen-ion concentration and neutral salts on the intensity of formaldehyde tannage. O. GERNGROSS and R. GORGES (Collegium, 1926, 398-408).—Hide powders of  $p_{\rm H} 3.0$  to 12.0 were tanned for 5 hrs. with 0.95% formaldehyde solutions, and the intensity of the tannage was determined by the water-resistance figure (cf. preceding abstract). There was scarcely any tanning effect at  $p_{\rm H}$  3.0, but it increased gradually to  $p_{\rm H}$  6.3. The isoelectric point had no significance for the formaldehyde tannage. Between  $p_{\rm H}$  6.3 and 7.0 there was a considerable sudden increase in the tanning intensity. Above  $p_{\rm H}$  7.0 the tanning intensity increased slightly to  $p_{\rm H}$  8.0, from which point it was practically constant. A four-fifths saturated sodium chloride solution and N- and 0.1N-solutions of potassium thiocyanate diminished the degree of tannage in alkaline solutions, whilst the sodium chloride had no effect in acid solutions. Experiments with hides and sheepskins confirmed the bad effect of formaldehyde tanning in strongly alkaline solutions. The degree of tannage was high, but the fibres were overswollen, and there was a kind of dead tannage. The tanning of sheepskins with formaldehyde in strong salt solutions failed in acid solutions, but resulted in a very good leather in strongly alkaline solutions ( $p_{\rm H}$  11.2 before, 8.6 after the tannage), and the leather had a water-resistance of 87. An after-treatment of this leather with egg yolk did not alter this figure.

D. WOODROFFE.

Liming with sulphide. V. CASABURI (Boll. Uff. Ind. Pelli, 1926, 4, 236—259).—The theory of the treatment of hides with alkaline sulphide is discussed and the rationale of this treatment when applied in the case of sole leather, kid, glove leather, etc., considered in detail.

Т. Н. Роре.

Measurement of hydrogen-ion concentration in its applications to the tanning industry. G. BAL-DRACCO (Boll. Uff. Ind. Pelli, 1926, 4, 227-235).—A summary of the uses to which a knowledge of the hydrogen-ion concentration is applied in tanning process. The colorimetric method of measurement is recommended.

T. H. POPE.

Adsorption of acids by hide in connexion with swelling phenomena. I. P. N. PAVLOV (Kolloid-Z., 1926, 40, 73-90).-From the change of solution concentration occurring on the adsorption of mineral acids by hide, the apparent adsorption isotherm can be determined, the concentration change being caused by the taking up of both solvent and solute by the hide. Simultaneous observations of the change of solution concentration and of the adsorbent swelling lead to the determination of the true adsorption of the solute. The adsorption coefficient of hydrochloric acid, sulphuric acid, and nitric acid is independent of the bath volume and of the amount of hide; from this it is inferred that the adsorption process consists in the formation of solvates of the acid molecules and ions with the collagen. The apparent adsorption isotherms of the acids form a curve with two branches. The swelling curve of hide in hydrochloric acid shows two minima, corresponding to two isoelectric points. The isotherms of the apparent " dry " adsorption and the swelling curve are comparable, a rise in the adsorption curve corresponding to a rise in the swelling curve, whilst the horizontal, or almost horizontal, branch of the adsorption curve corresponds to a fall in the swelling curve. Calculation of the true adsorption of the acids shows that, in the case of hydrochloric and sulphuric acids, the process follows the usual adsorption isotherm, and may be expressed by the Boedecker equation. With nitric acid, the true adsorption isotherm has not the simple form of those of the other acids, perhaps owing to the higher dissolving power of the acid for the collagen. The acids are adsorbed by hide in the order  $H_2SO_4/2 > HCl > HNO_3$ . L. L. BIRCUMSHAW.

Extraction of chromium from leather. L. MASNER and N. J. BERESTOVOJ (Chem. Listy, 1926, 20, 468—469). —The degree of extraction of chromium from leather by means of Rochelle salt depends on the reaction of the medium. Using sufficient of a 2.5% solution of Rochelle salt to give 2 g. of the salt for every gram of leather, a minimal extraction is found at  $p_{\rm H}$  3.58. In order to avoid hydrolysis of the leather it is recommended that solutions of moderate acidity be used. When extraction is carried out by sodium hydroxide, the hydrolysis is considerable and the amount of chromium extracted is approximately proportional to the amount of leather entering into solution. C. RANKEN.

Treatment and disposal of waste [tannery] waters. J. NOVER (J. Soc. Leather Trades Chem., 1926, 10, 263—265).—The waste liquors from an alum tawing works are alkaline. They contain protein matter, lime, and traces of sulphide. The settling tank has an inclined floor, and is divided into two parts, and the hair, flesh, and a little lime settle out here. The decanted liquor is agitated in a purification tank, divided into three compartments, with 50 g. of ferrous sulphate, 100 g. of sulphuric acid, 10 g. of aluminium sulphate, and 5 g. of sodium hypochlorite per cubic metre, and agitated. The ferrous sulphate forms ferrous sulphide and ferrous

hydroxide, which is oxidised to ferric hydroxide and coagulates the protein. The treated liquor is filtered through coke, and if necessary softened with permutit. The liquor is then neutral, clear, and odourless. The sludge contains 5-6% of nitrogen, and has manurial value. D. WOODROFFE.

Water content of glue and its importance in glue valuation. F. BAUM (Chem.-Ztg., 1926, 50, 649-651, 691-692, 742-745).-The inadequacy of chemical analysis alone in judging the quality of a glue is discussed, and the behaviour of different grades on treatment with water described. Discrepancies in analysis are due partly to the difficulty of obtaining a representative sample, which is obviated by the preparation of a uniform glue jelly from which portions are taken for the various determinations, and also to the extreme slowness with which a solid glue attains the thoroughly airdried condition. It is important to make determinations both of the total water and of that lost on air-drying. The water content of the glue in the completely air-dried condition is related to the quality, and in good varieties is about 11%. The composition of a glue may be expressed thus :- the essential air-dried glue substance with its combined water + the soluble matter lost during soaking + the excess of water removed by air-drying. The last-named constituent may vary considerably, and the marketing of glue on the basis of a standard airdried condition is recommended. F. R. Ennos.

Analysis of lactic acid. VIDAL.-See XX.

#### PATENT.

Tanning. J. R. GEIGY SOC. ANON., Assees. of CHEM. FABR. HALTINGEN, JUCKER & Co. (E.P. 256,628, 2.9.26. Conv., 5.8.25).—Hides or skins are treated with hydrofluosilicic acid or a salt thereof prior to or simultaneously with the tanning operation. This treatment is said to facilitate the tanning process. D. WOODROFFE.

# XVI.—AGRICULTURE.

Origin and nature of soil organic matter or soil "humus." II. Method of determining humus in the soil. S. A. WAKSMAN (Soil Sci., 1926, 22, 221-232; cf. B., 1926, 892).-Preliminary treatment with hydrochloric acid does not increase the amount of "humus" extracted by subsequent treatment with alkaline solutions. Sodium hydroxide solution is preferable to ammonium hydroxide for humus extraction. The method recommended for the determination of humus consists in heating the soil with 2.5% sodium hydroxide for 30 min. at 15 lb. pressure in an autoclave, or allowing it to react for 48 hrs. in the cold, and filtering. Fresh sodium hydroxide is added and filtration and washing are continued till the filtrate is no longer coloured. The combined filtrates are precipitated with excess of warm 10% hydrochloric acid. The precipitate ( $\alpha$ -fraction) after collection and warming is dried at 65-70° for 12-24 hrs. and weighed. Ash and nitrogen are determined in portions of the precipitate. The filtrate from the  $\alpha$ -fraction is treated with 2-3% sodium hydroxide solution, until maximum precipitation occurs, to obtain the  $\beta$ -fraction. G. W. ROBINSON.

Absorption of iron by soils. H. C. DOYNE and C. G. T. MORISON (Soil, Sci., 1926, 22, 163-173).-Data illustrating the absorption of iron from ferric chloride solution are given for a number of different types of soil. Four factors appear to be concerned in the absorption of iron, namely, calcium carbonate, gross amount and activity of the clay present, ferric oxide, and organic matter. Calcium carbonate acts by neutralising the free hydrochloric acid resulting from the hydrolysis of ferric chloride. The iron is then precipitated as ferric hydr-Free hydrochloric acid is also neutralised by oxide. clay and there is a mutual flocculation of ferric hydroxide and clay. Ferric hydroxide gel acts simply by neutralisation. A number of curves are given to show the effect on the absorption of iron by different soils of adding varying amounts of calcium carbonate. In the case of sandy soils there appears to be a maximum absorption of iron which cannot be exceeded until sufficient calcium carbonate has been added for complete precipitation. The form of the curves obtained is discussed.

#### G. W. ROBINSON.

Loss of nitrates from cropped soils. J. HENDRICK (Agric. Progress, 1925, 2, 69—71; Chem. Abstr., 1926, 20, 3056).—Crops appear to take up nitrates practically as fast as they are formed from the nitrogenous fertilisers applied, since no appreciable loss was observed, even in very wet weather. Drainage waters from uncropped soils contained 3—7 times as much nitrogen as those from cropped soils. A. A. ELDRIDGE.

Micro-organisms concerned in the decomposition of cellulose in the soil. S. A. WAKSMAN and C. E. SKINNER (J. Bact., 1926, 12, 57-84; Chem. Abstr., 1926, 20, 3056).—The amount of cellulose decomposed in soil under aerobic conditions depends on the amount of available nitrogen; under anaerobic conditions much less nitrogen is required. In normal soils, organisms causing anaerobic cellulose decomposition are scarce. There is a direct correlation between cellulose decomposition, the development of fungi, and the transformation of soil nitrogen into microbial protoplasm. Nitrogenfixing bacteria probably do not increase the store of soilnitrogen under aerobic conditions, especially in humid soils, when cellulose and straw are added as sources of energy. Nitrogen fixation occurs when starches and lower carbohydrates are also introduced.

#### A. A. ELDRIDGE.

Agricultural chemical investigations. F. MÜNTER (Landw. Jahrb., 1926, 64, 65-127; Chem. Zentr., 1926, 97, II, 1569).-(1) In vegetation experiments, the application of large amounts of nitrogen increased the yield of dry matter and of protein in leguminous plants. This is contrary to the results of field experiments. A sufficient supply of water appeared to be decisive. In a dry period, salts containing nitrogen act unfavourably on young lupins by making the salt concentration in the soil too high. (2) It is advantageous to plough in fertilisers on heavy deep soils. (3) In the presence of ammonium sulphate, the uptake of phosphoric acid by plants is increased by adding potassium salts, but not by adding sodium nitrate. (4) "Cave "phosphate, discovered by Willner, has little manurial value. (5) Addition of lime about six months after manuring with superphosphate increased the yield of sugar beet by 50%; lime given eight months and more after

superphosphate had no effect. (6) Of the newer phosphate fertilisers, Rhenania phosphate showed the highest percentage of  $P_2O_5$  utilised by plants. (7) Sugar beet and barley tolerate a relatively alkaline soil reaction; carrots, potatoes, red clover, flax, and yellow lupins are more sensitive. Sulphur, calcium sulphate, and manganous sulphate decrease alkalinity and increase yield. The existence of two optima for barley was confirmed $p_{\rm H}$  4.8 and  $p_{\rm H}$  7.6. Calcium cyanamide acts best, and sodium nitrate badly, on strongly acid soils. Acid soils are specially unfavourable in dry periods. (8) Seed stimulants are not likely to be useful in Germany since the arable soils are generally sufficiently supplied with salts. (9) Japanese results with Asahi Promoloid are not confirmed. (10) Barley, flax, and clover proved to be the best crops in regard to their influence on a following crop; sugar beet was the worst.

#### C. T. GIMINGHAM.

Manuring of potatoes. LAMBERG (Z. Pflanz. Düng., 1926, B5, 380—382).—From considerations based on Mitscherlich's theory, it is concluded that applications of large amounts of nitrogen would be justified by the increased yields of potatoes, whereas this would not be the case with large applications of potassium salts. The collated results of a large number of experiments confirm this conclusion. C. T. GIMINGHAM.

Effect of nitrogenous manuring of grass land on the proportion of grasses and clovers. W. JESSEN (Z. Pflanz. Düng., 1926, **B5**, 394—396).—With reference to a paper with this title by Maiwald (*ibid.*, **B4**, 531), it is pointed out that the subject was previously investigated by Lemmermann, and his conclusions are quoted (cf. Landw. Vers.-Stat., 1907, 67, 207). C. T. GIMINGHAM.

Manuring straw crops with increasing amounts of sodium nitrate. KUHNERT (Z. Pflanz. Düng., 1926, B5, 386—389).—In plot experiments with a mixture of summer rye and oats, the highest application of sodium nitrate tried gave a paying increase of yield. The experiments have reference to the difficulty of obtaining satisfactory crops of oats in certain parts of Schleswig-Holstein. C. T. GIMINGHAM.

Field experiment on the effect of an acid soil reaction on different [systems of] manuring. E. MÖLLER-ARNOLD (Z. Pflanz. Düng., 1926, B5, 376-379).-A field experiment is reported on the effect of various types of manuring (acid, neutral, or alkaline) on the yield of potatoes on a very acid soil  $(p_{\rm H} 4.0)$ . The greatest increase of yield (83%) over that of the unmanured plot was given by an alkaline mixture of manures (sodium nitrate, basic slag, and potassium sulphate) with chalk in addition, though the acidity of the soil was not greatly reduced. The same mixture without chalk gave only an insignificant increase. An acid mixture (superphosphate, ammonium sulphate, and 40% potash salts) also gave an insignificant increase, whereas the same mixture without superphosphate caused a small decrease in yield. C. T. GIMINGHAM.

Influence of calcium and nitrogen on the protein metabolism of the soya bean plant. J. M. GINSBURG and J. W. SHIVE (Soil Sci., 1926, 22, 175—192).— Soya bean plants grown in limed and unlimed soils, in soil extracts with calcium carbonate, and in complete

culture solutions containing varying concentrations of calcium and nitrogen, were analysed for protein nitrogen, total nitrogen, calcium, and magnesium. There was a definite correlation between calcium in culture solutions and the calcium content of the resultant plants. No definite correlation was observed in the case of nitrogen. Plants showed a higher nitrogen content, however, where calcium carbonate had been added to the medium. Calcium as chloride or nitrate had no influence on the total nitrogen or protein content. High total nitrogen was associated with low hydrogen-ion concentration in the medium. The increased rate of nitrogen absorption in the presence of calcium carbonate had no effect on the protein content. Plants grown with calcium chloride or nitrate required less iron than those grown with calcium carbonate. The plants grown in soil were similar in composition to those grown in culture solutions, but calcium carbonate had a more marked accelerating effect on plant growth in the soil cultures.

# G. W. ROBINSON.

Influence of nutrient supply on earliness of maturity in cabbage. J. E. EDMUND and E. P. LEWIS (Michigan Agric. Sta. Tech. Bull., 1925, [75], 1—10; Chem. Abstr., 1926, 20, 2890).—The greatest response of cabbage plants grown in sand cultures with a modified Pfeiffer's nutrient solution was observed with plants which received applications during the middle growth period; applications during the early growth period caused least response. A surplus of nutrient material, particularly in the middle growth period, increased the length of the internodes. A. A. ELDRIDGE.

Carbon dioxide fertiliser. W. RIEDE (Z. Pflanz. Düng., 1926, **B5**, 383–385; cf. B., 1925, 684).—A "carbon dioxide fertiliser" ( $58 \cdot 5\%$  peat, 18% wood charcoal,  $3 \cdot 5\%$  pyrolusite, and 20% nutrient salts) tested under field conditions gave small increases of yield with potatoes and soya beans, but had no effect on cabbages and other market garden crops.

#### C. T. GIMINGHAM.

Sources of ammonia. J. J. SKINNER and T. S. BUIE (S. Carolina Agric. Exp. Sta. Bull., 1926, [227], 1—32; Chem. Abstr., 1926, 20, 2890).—New fertiliser materials, obtained from nitrogen-fixation processes, gave favourable results with cotton; with maize the results were good, although sodium nitrate and ammonium sulphate are preferable. A. A. ELDRIDGE.

#### PATENTS.

"Seed pickling " materials. HOLZVERKOHLUNGS-IND. A.-G. (Austr. P. 100,210, 13.12.21).-Thiocyanates of various metals or mixtures containing the thiocyanate compounds, if necessary with other disinfecting materials, are claimed for use in seed disinfection. For example : (a) a solution of barium thiocyanate is added to a solution of aluminium sulphate and the precipitate filtered; (b)arsenious oxide and potassium thiocyanate are dissolved in water; (c) 10% ferric chloride solution is mixed with 83% of potassium thiocyanate; (d) silver nitrate is dissolved in concentrated solutions of potassium or ammonium thiocyanate. For use, such solutions are diluted with water so as to contain, e.g., 0.3% Al, or 0.1% As, or 0.14% Fe, or 0.6% Ag. Other examples are various mixtures of solutions of ferric, magnesium, zinc, mercury, lead, silver, chromium, and copper thiocyanates.

Addition of potassium permanganate increases the disinfecting power of the thiocyanates. Addition of phenol, pyridine, or their derivatives is also advantageous. No injury to germination occurs from the use of these solutions. C. T. GIMINGHAM.

Manufacture of cleansing and disinfecting materials, and of fungicides and insecticides. R. VIDAL (F.P. 566,406, 8.8.22; and Addns. 27,591, 30.1.23; 27,779, 14.2.23; 27,784, 19.2.23; 28,059, 10.3.23;28,060, 15.3.23).-Phenols, or tar oils containing phenols, are converted into alkylated phenols by warming with alcohols in the presence of anhydrous zinc chloride and hydrochloric acid gas. The alkylphenols, e.g., n-butyl o-cresol, are dissolved in aqueous solutions of alkali and solutions of the alkali soaps of ricinoleic acid are added ; or the free phenols are mixed with ordinary soap solutions, castor oil, and aromatic hydrocarbons, carbon disulphide, or tetrahydronaphthalene. The stable emulsions, obtained on dilution with water, find uses in the textile dyeing, tanning, and fur industries, and for disinfection of houses, as well as for destruction of pests of all kinds, in the soil and on plants or animals. Treatment of oleic, palmitic, and stearic acids with alkali hydroxides or ammonia in presence of alkylphenols, phenol, resorcinol, or quinol gives products which, without addition of ricinoleates, can dissolve large amounts of free fatty acids, aliphatic, aromatic, and terpene hydrocarbons, carbon tetrachloride or disulphide. In place of alkylphenols, benzyl-phenols or -cresols, or alkali salts of phenol- or cresol-sulphonic acids, or of m-hydroxybenzoic or hydroxytoluic acids can be used. Some of these preparations possess strong lathering properties with sea water or very hard waters. C. T. GIMINGHAM.

**Preparation of fungicidal adsorption compounds.** CHEM. FABR. VON HEYDEN A.-G., Assees. of D. LAMMERING (G.P. 432,399, 9.7.24).—Soluble copper salts are mixed with alkali aluminates, or only a part of the alkaline reagent required to precipitate the copper is replaced by alkali aluminate. For example, a solution of copper sulphate is poured into a solution of sodium aluminate with stirring; the greater part of the water is removed from the precipitate, which, while still moist, is intimately mixed with fine clay and dried with gentle heating. The product is ground and yields a powder, readily distributable and with good adhesive properties. It is used as a spray fluid in water, or for seed disinfection, or as a fungicidal dust. It remains suspended in water for a long time and adheres specially well to foliage.

C. T. GIMINGHAM.

# XVII.—SUGARS; STARCHES; GUMS.

Effect of moisture on the loss of sugar from beets in storage. D. A. PACK (J. Agric. Res., 1926, 32, 1143—1152).—In order to conserve the maximum amount of sugar in beets, storage must be so controlled that the water content of the roots is kept at its normal, at which point it is under the optimum conditions for growth and respiration. A temperature of about  $1.7^{\circ}$  inhibits growth and reduces respiration to a minimum. By thus keeping the tissues alive and as dormant as possible, beets may be kept healthy and at a high sugar content for eighteen months. J. P. OGILVIE.

Dehydration of the sugar beet (De Vecchis process). B. J. OWEN (Intern. Sugar J., 1926, 28, 542-546).-By using thin beet cossettes, by blowing the air through the sliced material, and by using a temperature not exceeding 110°, it was found possible to dehydrate them to  $7\frac{1}{2}$ % of water without the formation of invert sugar or caramel. Syrups of 50° Brix were obtained by lixiviation of the dried cossettes. Following treatment with lime and phosphoric acid, a single filtration only was sufficient, but this depended on the quality of the beets. An estimate made of the consumption of steam in manufacture gives 195 lb. of coal per ton of roots worked for ordinary diffusion, but only 50 lb. for the dehydration process on the same basis. It is calculated that by the dehydration process the cost of manufacture would be £5 14s. 5d. per ton of sugar made for a factory slicing 1000 tons of roots per day. J. P. OGILVIE.

Coloration of beet juices during evaporation. V. STANEK and J. VONDRAK (Z. Zuckerind. Czechoslov., 1926, 51, 1—8, 9—15, 17—22).—An apparatus designed to reproduce in the laboratory the conditions obtaining in the factory in respect of the darkening of juice during evaporation demonstrated, *inter alia*, the following figures : thin-juice, double carbonated, 70—77; thin-juice, carbonated once and sulphited, 19—21; and thin-juice, triple carbonated and unsulphited, 30—34. An increased addition of lime in carbonatation enhanced the resistance to coloration, whilst over-saturation of the first carbonatation gave juices which both before and after evaporation were darker. Regarding the effect of various metals on darkening during evaporation, iron gave the highest figure (124, compared with 102 *in vitro*).

## J. P. OGILVIE.

Danger of inversion with sulphurous acid in sugar manufacture. L. VAN DER HEIDE (Intern. Sugar J., 1926, 28, 558—559).—In the sulphitation of cane juice, owing to the presence of buffers, it is possible to carry the operation to a relatively high content of sulphur dioxide without the occurrence of inversion. A  $p_{\rm H}$  less than 7 can be reached provided sufficient buffer salts are present, otherwise there is considerable danger. As a means of obviating this, the addition of some suitable salt, e.g., calcium acetate, in sufficient amount is advised. J. P. OGILVIE.

Formation of caramel substances in the presence of nitrogenous compounds. B. RIPP (Z. Ver. deut. Zuckerind., 1926, 627-655).-Caramel formed by heating a pure solution of lævulose on the water-bath was shown to have a formula identical with that of caramelan (Gelis), possessing four alcoholic hydroxyl groups in the molecule, and behaving as a carbohydrate, i.e., possessing a cupric reducing power and yielding lævulic acid, hydroxymethylfurfuraldehyde, and dextrose on hydrolysis. When the lævulose solution contains nitrogenous substances, the caramel formed no longer behaves in a general way as a carbohydrate, though it still possesses a reducing power. On heating dextrose and lævulose solutions with amino-acids, the depth of colour of the resulting product is due to the interaction with the nitrogenous substance, though not propor-J. P. OGILVIE. tionately so.

Nature and distribution of the non-sugars affecting the quality of white beet granulated sugars. H. S. PAINE and R. T. BALCH (Intern. Sugar J., 1926, 28, 472-477) .- Particles of impurities large enough to cause considerable turbidity in solutions of white-beet granulated sugars were in some cases found in greatest proportion near the centre of the crystals, indicating that such may act as nuclei. During growth the sugar crystals adsorb continuously colloids of the type which have the property of reducing surface tension and of becoming concentrated at the interface between crystal and liquid, as constituents were found to be distributed through the individual crystals, though present mostly in the external portions, probably owing to the presence of films of mother-liquor on the surface. J. P. OGILVIE.

Determination of sucrose by means of the interferometer. L. HORACEK (Z. Zuckerind. Czechoslov., 1926, 51, 25-30).—Sucrose may be determined by making two refractometric observations, one before and the other after hydrolysis (preferably using invertase), the increase in the reading being determined by means of the Zeiss interferometer. Although rapid, the method has an accuracy which does not exceed that of the double polarisation process. J. P. OGILVIE.

Fermentation of bagasse. Owen and BENNETT.— See XVIII.

Determination of sucrose etc. in "stroop." KRUISHEER.—See XIX.

# XVIII.—FERMENTATION INDUSTRIES.

Brewing in its relations with other fermentation industries. A. FERNBACH (J. Inst. Brew., 1926, 32, 454-462).-The Nathan process for the manufacture of lager beer supplies one of the most striking examples of the strict application of the principles of pure fermentation, and the methods evolved in its development have been successfully extended, with slight modifications, to the manufacture of vinegar, acetone, butyl alcohol, cider, and wine. The "Amylo" process used in distilleries is the first instance of strictly aseptic work on a large scale in a fermentation industry, and many of its working details are based on brewery methods. The correction of the hydrogen-ion concentrations of worts by the addition of lactic acid ferments has been introduced from distillery procedure into brewing as a means of counteracting alkaline brewing waters. The knowledge of fermentation temperatures acquired in lager beer manufacture and the methods to combat its excessive rise have been applied to distilling for the suppression of the loss of alcohol by evaporation. In wine making, the introduction of refrigeration appliances, suggested by brewing experience, has improved the soundness of the final product. C. RANKEN.

Measurement of foam in beer and the factors which influence it. K. GEVS (Woch. Brau., 1926, 43, 439-444).—When using the apparatus devised by Lüers for foam measurements decarbonation of the beer due to handling should be avoided. The hydrogenion concentration and humulon, with its sensitiveness to the hydrogen-ion concentration, are the two governing 962

factors in head-retention of beer. Fermentation at low temperatures results in low hydrogen-ion concentrations, whilst the quantity of yeast present has also an effect. Especially with greater quantities of yeast, there is a tendency, towards the end of the fermentation, for the stage of increasing or constant acidity to be followed by a reversion to a more alkaline period. Increasing hydrogen-ion concentration leads to coagulation of the proteins with promotion of foam formation and head-retention, whereas either a constant or a reverting hydrogen-ion concentration causes peptisation of the ultramicrons of the beer, with the result that little foam is formed and the head-retention is poor. This latter result is also assisted by the fact that, with peptisation, the highly charged colloidal particles have no tendency to go to the surface, and, in addition, owing to the extreme smallness of the particles, there are no nuclei at which the carbon dioxide can be evolved. C. RANKEN.

Influence of the brewing water on the acidity of the wort and beer. W. WINDISCH and P. KOLBACH (Woch. Brau., 1926, 43, 423-428, 444-447).-Comparing the effect of the addition of various salts to the brewing water on the worts obtained from the same malt, the authors find that when calcium sulphate is present in the brewing water, the concentration of the hydrogen ions is raised, the amount of buffer substances diminishes owing to the precipitation of calcium phosphate due to the increasing concentration of the calcium ions, but the titration acidity is affected only to a very small extent. Magnesium and sodium carbonates lower the hydrogen-ion concentration, the titration acidity, and also the amount of buffer substances. The loss of buffer substances originates chiefly from the loss in phosphates brought about by the augmented formation of insoluble secondary calcium phosphate by the increased alkalinity. Calcium carbonate diminishes the hydrogen-ion concentration, but to a less extent than does an equivalent amount of magnesium or sodium carbonate. It lessens the titration acidity, and, as the concentration of the calcium ions is increased as is also the alkalinity, these two factors induce a greater precipitation of phosphate with a correspondingly greater decrease of buffering substances. Similar titration curves to those for the worts are found for the beer, but the differences between those obtained for the various salts are very small. No definite conclusions can be reached from the amounts of acidity formed during fermentation, which are deduced from the difference between the titration acidities of the wort and of the corresponding beer. In the majority of cases, the amount of buffer substances in the beer is less than that in the wort, but in a few cases the converse holds. C. RANKEN.

Influence of temperature on the optimal hydrogen-ion concentration for amylolytic action. H. LÜERS and S. NISHIMURA (Woch. Brau., 1926, 43, 415-416).—The results of the authors are not in concordance with those of Olsen and Fine (B., 1924, 992), who had previously showed that the optimal  $p_{\rm H}$ for the amylolytic action of malts varied from 4.3 to 6.0 for the temperature range of 25-70°. Considering that the experimental conditions of the previous workers were not sufficiently definite and controlled,

they have repeated the investigation, using a solution of soluble starch, which, after being buffered by acetateacetic acid mixtures to give  $p_{\rm H}$  values varying from 3.5 to 6.0, was fermented by an extremely active form of amylase prepared according to the method of Sherman and Schlesinger. For  $15-70^{\circ}$  the optimal  $p_{\rm H}$  was found to lie within the limits  $p_{\rm H} 4.4$  to 4.6, and the zone of optimal action became more restricted with rising temperature. C. RANKEN.

Determination of the colour of wort and beer by colorimeter constructed according to Ostwald's theory. F. MESTAN (Z. angew. chem., 1926, 39, 1336— 1340, and Chem. Listy, 1926, 20, 462—468).—The author discusses the defects of the different instruments for the measurement of colour on the basis of Ostwald's principles (B., 1919, 914 A), and describes an improved form of apparatus which is based on the use of wedges of coloured glass, the rays transmitted through these wedges being compared with those passing through the liquid under examination. C. RANKEN.

Fermentation of bagasse in relation to the yield of industrial alcohol. W. L. OWEN and N. BENNETT (Intern. Sugar J., 1926, 28, 463-469).—Addition of bagasse to molasses worts decreases the efficiency of the fermentation (making allowance for the sugars introduced), this probably being due to an inhibiting effect of gums, non-fermenting sugars, and other substances present. J. P. OGILVIE.

Determination of tartaric acid in wine. DUBOUX.—See XXIII.

#### PATENTS.

Obtaining acetone and other products by fermentation of materials containing carbohydrates. N. MOSKOVITS (Austr. P. 102,927, 6.3.22).—The materials are treated in closed vessels with organisms which generate acetone. The fermentation is carried out in the same vessel in which the steaming of the raw materials and the preparation of the mash are performed. The vessel is connected with a gasholder containing, under pressure, a gas (e.g., air, oxygen, nitrogen, or ammonia) which does not hinder fermentation so that, when the connexion is opened after steaming, the gas enters as the pressure in the fermentation vessel falls on cooling. The entrance of foreign organisms before and during fermentation is thus prevented. In other respects the process follows the usual method for obtaining acetone by fermentation of materials containing carbohydrates, with, e.g., B. macerans. Indifferent substances such as asbestos, filter-paper, beer refuse, potato skins, straw, and nitrogenous organic materials, like compressed yeast, yeast extract, or malt embryos, may be added to the mash, and, in sterilising the mash, acids may be added. A. DAVIDSON.

# XIX.—FOODS.

Refractive index and density of butter fat. A. SCHNECK (Milchw. Zentr., 1926, 55, 113—116, 153—156). —The values of the specific refraction, obtained from the refractive index and density by the Lorentz-Lorenz formula, of a number of butter fats show only slight variations among themselves, and approximate closely to the theoretical values calculated from the percentages of esters and the refractivities of their constituent atoms. CL. XX.-ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Assuming a mean specific refraction, the density of a butter fat may be calculated from its refractive index and vice versa, the results differing from the observed values by no more than one or two units in the third decimal figure. Summer butter has a higher specific refraction than winter butter; also butter from the later stages shows higher values than that from the earlier stages of lactation. F. R. ENNOS.

Determination of flour in sausages. H. S. J. F. SNETHLAGE (Chem. Weekblad, 1926, 23, 465—466).—As the extract with 25% hydrochloric acid recommended for the determination by Dutch law is found to give rapidly diminishing rotations on keeping for a few hours, it is recommended that the extract be diluted to contain 15% of acid, before taking the polarimeter reading. Direct extraction with 15% acid is not satisfactory.

S. I. LEVY.

Determination of sucrose and invert sugar, and of glucose (starch) syrup in "stroop." C. I. KRUISHEER (Chem. Weekblad, 1926, 23, 430-437) .--In order to arrive at a suitable method of analysis for the syrup commonly used as a sugar foodstuff in Holland under the name "stroop," and which consists of a mixture of glucose (starch) syrup and molasses, samples of these component materials from the bestknown Dutch makers of the material were analysed separately, and mixtures in various proportions were then prepared and tested. The method of analysis adopted for the mixtures, which is accurate to within about 2%, consists in warming with dilute acid to cause inversion, making alkaline, adding excess of iodine, removing the excess exactly, and determining the reducing sugars expressed as lævulose by Schoorl's method (cf. Kolthoff, B., 1923, 467A); the figure obtained  $\times 1.9$  gives the sucrose and invert sugar together expressed as sucrose. For determination of the starch syrup the sample is hydrolysed by boiling for 1 hr. with hydrochloric acid, and the reducing sugars are determined as before. Formulæ are given for expressing the composition of "stroop" from these determina-S. I. LEVY. tions.

Composition of the food grains, vegetables, and fruits of Western India. D. L. SAHASRABUDDHE (Dept. Agric. Bombay Presidency Bull., 1925, [124], 1-38; Chem. Abstr., 1926, 20, 3049).

# PATENT.

Making edible alkali caseinate. H. F. ZOLLER (U.S.P. 1,598,334, 31.8.26. Appl., 8.5.22).—Sufficient alkali solution to dissolve the casein is added with stirring to a suspension, in a dilute alkali phosphate solution, of casein which has been precipitated by acid, the use of considerable excess of alkali being avoided.

# L. A. Coles.

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

Production of ether by solid catylysts. S. K. K. JATKAR and H. E. WATSON (J. Indian Inst. Sci., 1926, 9A, 71-109).—A detailed account of results previously published (J.S.C.I., 1926, 45, 168 T).—Measurements of the equilibrium constant made by determining the quantity of alcohol formed when mixtures of ether and water are passed over the alum catalyst indicated that the most probable value is  $7 \cdot 3$  at 500° Abs.

E. H. SHARPLES.

Crystallisation of citric acid from lemon juice. G. AJON (Riv. Ital. Essenze Profumi, 1925, 7, 125—127; Chem. Zentr., 1926, II, 1597—1598).—The juice, which is prevented from coming in contact with iron by the use of porcelain presses and basins, is stored for several days, preferably with the addition of some fermenting juice, when it is concentrated to one-fourth of its volume, and inorganic constituents are precipitated and removed. After further concentration on the water bath, and the addition of a little sulphuric acid, citric acid separates in fine crystals. Large crystals are obtained by subjecting the juice to osmosis after removing the inorganic constituents (cf. B., 1925, 227; 1926, 214).

L. A. Coles.

Analysis of commercial lactic acid. U. J. THUAU and M. VIDAL (J. Soc. Leather Trades Chem., 1926, 10, 257-258).-Sulphuric acid can be determined in lactic acid by the Meunier method, in which the sulphates are precipitated by means of 95% alcohol, the latter evaporated off from the filtrate, and the sulphuric acid determined by means of barium chloride. In the Balland method, the total  $SO_4$  is determined by means of barium chloride, the combined SO<sub>4</sub> by determining the sulphates in the ash, and the difference is due to free sulphuric acid. In both methods the result should be converted into the equivalent of lactic acid and deducted from the percentage of the latter as determined by Thompson's method (cf. B., 1918, 343 A). To determine hydrochloric acid, a weighed amount of the sample is neutralised with alkali, evaporated to dryness, heated carefully without calcining, extracted with hot water, and the total chlorides determined (I). The determination is repeated, omitting the neutralisation (II). The difference (I-II) gives the chlorides due to free hydrochloric acid. A sample of lactic acid analysed by the authors contained 50.70% of lactic acid as determined by the official method, but 2.27% of this was due to free sulphuric acid, so that the real content of lactic acid was only 48.43%. D. WOODROFFE.

Stability of hexylresorcinol in pharmaceutical preparations. W. A. FEIRER and V. LEONARD (J. Pharm. Exp. Ther., 1926, 28, 395-397).—Hexylresorcinol, in solution in olive oil enclosed in soluble gelatin capsules, does not deteriorate on keeping for one year at the ordinary temperature.

H. I. COOMBS.

Terpineol content of turpentine obtained in the production of terpin hydrate. SUSSKIND (Trans. Sci. Chem. Pharm. Inst. Moscow, 1923, [26], 29—30; Chem. Abstr., 1926, 20, 3076).—The mother-liquor from the preparation of terpin hydrate from turpentine and sulphuric acid, when distilled in steam, yields terpineol (8-9%) and pinene (38%). A. A. ELDRIDGE.

Automatic devices for extracting alkaloidal solutions. II. Application to nux vomica and belladonna alkaloids. H. R. WATKINS and S. PALKIN (J. Amer. Pharm. Assoc., 1925, 14, 1099–1104; Chem. Abstr., 1926, 20, 2895).—Automatic extractors are preferable to hand-shaken separators. The quantity of ammonia or ammonium sulphate present is immaterial when chloroform or benzene is used; small losses of atropine were observed when ethyl ether was employed in the presence of ammonia. A. A. ELDRIDGE.

Iodine value of commercial peptones. A. BERTHE-LOT and M. CNADUC (Bull. Soc. Chim. biol., 1926, 8, 936—939).—The iodine value (determined titrimetrically) in acid and in alkaline solution is suggested as a useful method for the comparison of commercial peptones for bacteriological purposes. R. K. CANNAN.

Activity of the commercial extracts of parathyroid glands. M. KOHLER (Biochem. Z., 1926, 175, 27-30).—Extracts prepared by Collip's method (A., 1925, i, 754, 1017) gave often a transient fall followed by a considerable rise, gradually attained, in blood calcium. Of four commercial extracts two had no effect, one caused a slight fall, and the fourth a slight rise in blood calcium. Hæmocrinine, a dry blood preparation containing the hormone, also was inactive. P. W. CLUTTERBUCK.

Volatile oil of Collinsonia anisata. E. R. MILLER and J. K. HUNT (J. Amer. Pharm. Assoc., 1925, 14, 1096— 1098; Chem. Abstr., 1926, 20, 2895).—The oil (yield 0.138%) had  $\alpha_D - 2.34^{\circ}$  to  $-0.4^{\circ}$ ,  $n_D^{19.2}$  1.5185— 1.5225; about 80% is methylchavicol. Salicylic acid, safrole, and an unidentified ketone, terpene, and aldehyde are also present. A. A. ELDRIDGE.

Volatile oil of Myrica asplenifolia, Endl. M. A. BRAUN (J. Amer. Pharm. Assoc., 1926, 15, 336–337; Chem. Abstr., 1926, 20, 2896).—The oil (yield 0.02%) had  $d^{25}0.8945$ ,  $\alpha_{\rm D}$ — $3.75^{\circ}$ . Aldehyde was absent from the lowest fraction (below 100°) but present in the third (150–200°). The 200–220° fraction had an ester content of 28.8%, whilst the total alcohol content was 45.74%. A. A. ELDRIDGE.

#### PATENTS.

Recovery of alcohol, ether, aldehydes, etc. from gases. E. BERL (G.P. 432,357, 3.3.22).—Organic vapours which are present in dilute form in air, hydrogen, or waste fumes are recovered by absorption in relatively small quantities of coal tar phenols or lignite tar phenols, any residual vapour being adsorbed by activated charcoal or colloidal silica. W. G. CAREY.

Manufacture of phosphoric esters of polyhydric alcohols. P. E. GOISSEDET and A. L. HUSSON, ASSTS. to Soc. CHIM. DES USINES DU RHÔNE (U.S.P. 1,598,370, 31.8.26. Appl., 15.9.25. Conv., 27.4.25).—Polyhydric alcohols are treated with phosphoric anhydride in the presence of tertiary bases. The esters are precipitated as their calcium salts. W. N. HOYTE.

Manufacture of primary and secondary aromatic amines. E. MERCK, Assees. of W. KRAUSS (G.P. 432,151, 21.8.24; Addn. to 407,487, B., 1925, 474).— The chief patent and G.P. 417,926 (B., 1926, 173) are varied by treatment of secondary or tertary amines of the general formula Aryl·NRR' (R = H, alkyl, or benzyl; R' = benzyl or substituted benzyl) with hydrogen in presence of metal catalysts. For example, ethylbenzylaniline and alcohol are shaken with hydrogen in presence of palladium black. Exactly 1 mol. of hydrogen is absorbed yielding toluene and ethylaniline. The following amines are decomposed in a similar manner :---methylbenzylaniline into methylaniline and toluene; benzylaniline into aniline and toluene; dibenzylaniline into aniline and toluene; 3:4-methylenedioxybenzylaniline into aniline and 3:4-methylenedioxytoluene; o-hydroxybenzylaniline into o-cresol and aniline. A. DAVIDSON.

Preparation of di- and poly-sulphochlorides of homo- and hetero-cyclic mono- and poly-nuclear aromatic compounds and their substitution products. O. LUSTIG and E. KATSCHER (Austr. P. 101,667, 9.5.23).—The starting material is heated with an excess of chlorosulphonic acid (at least 5 times the weight) for a long time (5 hrs. at least) at the b.p. of the reaction mixture or just below it. Examples are given of the preparation of m-xylenedisulphonyl chloride from xylene, of 1-chlorobenzene-2: 4-disulphonyl chloride from chlorobenzene, and of carbazoletetrasulphonyl chloride from potassium carbazolesulphonate. Yields of 75—80% of crystalline products are obtained. A. DAVIDSON.

Preparation of carboxylic esters of halogenated polyhydric alcohols. S. Rosenzweig and H. Leger-LOTZ (Austr. P. 101,671, 9.11.23).-A mixture of a polyhydric alcohol and a carboxylic acid is treated with dry hydrogen halide at a suitable temperature. For example, dry hydrogen chloride is passed into a mixture of p-acetamidobenzoic acid and glycol at 110-120° for several hours, and the glycolchlorohydrin produced is distilled off. On pouring the residue into an excess of sodium carbonate solution,  $\beta$ -chloroethyl p-aminobenzoate, m.p. 85-86°, crystallises out. Similarly from p-nitrobenzoic acid, glycol, and hydrogen chloride at 100-110° is formed β-chloroethyl p-nitrobenzoate, m.p. 54-55°. By passing dry hydrogen bromide into a mixture of equal parts of anhydrous glycerol and acetic acid at 140–160°, the ester produced being simultaneously distilled off, treated with dilute sodium carbonate, dried with calcium chloride, and distilled in a vacuum, monobromohydrin acetate, CH<sub>2</sub>Br·CH(OH)·CH<sub>2</sub>·O·COMe, b.p. 172-174°/ 100 mm., is obtained. A. DAVIDSON.

Manufacture of an acid crystalline saponin from Primula species. L. KOPLER and S. ROSENZWEIG (Austr. P. 101,486, 21.3.24).-Primula plants are extracted with aqueous alkaline solutions, and the saponin is precipitated from the extracts by acidification and filtered off. The crude saponin is dissolved in hot dilute alcohol, the solution treated with decolorising charcoal, filtered, and after distilling off most of the alcohol, the saponin is precipitated by gradual addition of water and crystallised from suitable solvents. Alternatively, the plants are extracted with hot dilute alcohol and, after distilling off most of the alcohol, the saponin is precipitated by water, filtered off, again dissolved in hot dilute alcohol, and purified as above. The end products or intermediate products of this process are subjected to electrodialysis in alkaline aqueous solution. A. DAVIDSON.

Alkylhydroxyalkyl- and dihydroxyalkyl-arsinic acids. ÉTAB. POULENC FRÈRES, and C. J. OECHSLIN (F.P. 585,970, 15.11.23; cf. E.P. 206,152, B., 1925, 83).— Alkyl or hydroxyalkyl halides are allowed to act on sodium salts of hydroxyalkyl- or alkyl-arsinic acids. For

example, an aqueous solution of sodium methylarsinate is reduced at 45° with sulphur dioxide and neutralised with sodium hydroxide solution ( $d \ 1 \cdot 332$ ). Glycolchlorohydrin is added to the solution at 30°, the mixture stirred 6-8 hrs., acidified with sulphuric acid, evaporated in a vacuum, the residue extracted with hot alcohol, and the alcoholic solution allowed to crystallise or precipitated with acetone or ether. The methylhydroxyethylarsinic acid, OH·CH2·CH2·AsMe·O2H, softening between 80° and 103°, forms water-soluble salts. The same acid is obtained from sodium hydroxyethylarsinate and methyl iodide. By the action of glycolbromohydrin on sodium hydroxyethylarsinate, dihydroxyethylarsinic acid is formed. From sodium hydroxyethylarsinate by treatment with allyl bromide at 60°, allylhydroxyethylarsinic acid is formed. A. DAVIDSON.

Manufacture of mercurated hydroaromatic F. NEUMANN (Austr. P. 100,723, hydrocarbons. 14.11.23).-Mono- or poly-cyclic aromatic compounds, fully or partly hydrogenated, are treated with mercuric salts at about 150-250°. For example, tetrahydronaphthalene is heated at about 200° for  $\frac{1}{2}$  hr. with less than 1 mol. of mercuric acetate; the mercurated tetrahydronaphthalene forms needles of no definite melting point and decomposes slowly on heating. The preparation of analogous compounds from decahydronaphthalene and cyclohexanone is described. The products are soluble in indifferent organic solvents such as carbon disulphide, are strongly toxic, and find application as disinfectants and as a means of combating plant pests.

# A. DAVIDSON.

Preparation of highly purified physiologically active substances from female internal secretive organs. Ges. FÜR CHEM. IND. IN BASEL (Swiss P. 113,835, 5.12.24) .- Fatty acids and other acid impurities may be removed from extracts of organs by treatment with alkalis or alkaline earths, the extracts being dissolved in solvents in which the resultant soaps etc. are insoluble; or the alkalis etc. may be dispersed on an indifferent carrier (carbon, kieselguhr, etc.), the treatment being carried out as before, or by impregnating the loaded carrier with the extract and extracting with a solvent in which the resultant soap etc. is insoluble. The alkalis etc. may be dispersed by, e.g., treating animal charcoal with an aqueous alcoholic solution of sodium hydroxide or sodium carbonate and evaporating. Examples are given of the purification of extracts of placenta, ovary, and corpus luteum. B. FULLMAN.

Preparation of a remedy for diabetes. J. BUR-MANN (Swiss P. 107,092, 23.5.23).—Fresh glands with internal or external secretion, such as pancreas or liver, from healthy animals are freed from tissue and fat, treated with physiological serum and 1% of their weight of hydrochloric acid, and ground in an indifferent gaseous atmosphere. Sterile sand is added, the mass ground in absence of air at 0°, and filter-pressed. The resultant liquid contains all the soluble enzymes (especially the glycolytic ones) as well as the hormones and hydrochlorides of the active bases of the glands. It is dried, and the residue, after removal of fat by treatment with light petroleum, dissolved in water and the solution added to ether, when a fine powder is precipitated. This has a strong diastatic action, and gives good results in the treatment of diabetes. The same product is obtained from yeast of the species *Saccharomyces ellipsoideus glykolyticus*. The yeast is cultivated in a medium containing glycerophosphoric acid. On completion of the fermentation, the yeast is allowed to settle, washed with water faintly acidified with hydrofluoric acid, ground, and then treated as above. B. FULLMAN.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Ripening of photographic silver halide emulsions. W. JENISCH (Z. wiss. Phot., 1926, 24, 248-256). -Lüppo-Cramer's experimental proof of the nucleus theory of sensitivity depends on the observation of the removal of the nuclei with chromic acid and the resultant decrease in sensitivity. Conversely, it follows that the addition of nuclei should result in an increase of sensitivity. The observations of Lüppo-Cramer and others, that colloidal metal sols added to a finished emulsion before coating are without influence on speed and fog, were confirmed. Colloidal gold, present in an acid emulsion during precipitation, gave a marked increase of sensitivity with no fog. Increase in the amount of gold gives a bigger speed increase and, eventually, fog. Ammonia-emulsions behave similarly. The influence of the colloidal metal depends on its dispersity, the sensitising action increasing with increasing dispersity, and the amount of metal sol that can be added without inducing fog increases also. In the case of a fine gold sol prepared by phosphorus reduction it was possible to add a certain amount of sol without producing any effect. Silver and platinum sols give increased sensitivity to about equal extents, but not so markedly as the highly dispersed gold sols. The results establish that part of the sensitivity increase in ripening is due to formation of nuclei in the last part of the ripening process, but the nuclei are not necessarily of silver. W. CLARK.

Silver chloride grain. R. E. LIESEGANG (Phot. Ind., 1926, 867-868).-Sodium chloride spheres, 5 mm. in diameter, were converted into silver chloride spheres by immersing for long periods in concentrated silver nitrate solution, and these were used to imitate the microscopic grain of the photographic plate. If such spheres are exposed and developed in metol-adurol, reduction occurs at the surface with formation of greywhite metallic silver, and the shape is unaltered. Oxidation of the developer is more rapid in the neighbourhood of the grains than at the surface of the solution by the air. A model of a photographic plate was prepared by immersing a silver chloride sphere in a gelatin sol, and setting. When a developer was poured on the jelly surface, it became dark brown when it had diffused to the sphere. If, after development, the gelatin was melted, the sphere remained surrounded by an envelope of tanned gelatin. Sulphide toning may be illustrated by treating the developed sphere with sodium sulphide solution. The solvent action of thiosulphate and ammonia may also be observed; the action begins at some particular point on the sphere, and is not regular. W. CLARK.

Desensitisation. LÜPPO-CRAMER (Phot. Ind., 1926, 948-952).-The paper deals essentially with the statement of Lumière and Seyewetz that phenosafranine can be completely washed out of a plate, with restoration of the original sensitivity, and the conclusion that this indicates that desensitisation is not an oxidation effect, but is due to the formation of a less light-sensitive complex between the desensitiser and the silver bromide. Desensitisation is certainly decreased by washing, but it is not completely destroyed, the extent of the decrease varying with the desensitiser. If the statement of Lumière and Sevewetz were correct it would not disprove the oxidation theory of desensitisation. The results of Hübl (B., 1926, 612) are quoted in support of the W. CLARK. arguments.

#### PATENTS.

Film for photocollographic printing plates. M. DE'SPERATI, ASST. to ARGENTOGRAPHICA, LTD. (U.S.P. 1,598,061, 31.8.26. Appl., 28.11.25).—A film for photocollographic printing plates has a gelatin layer on each face. One of the layers is prepared so as to dissolve at a lower temperature than the gelatin of the other layer. W. CLARK.

# XXII--EXPLOSIVES; MATCHES.

#### PATENTS.

Liquid air explosives. LIQUID OXYGEN EXPLOSIVES, LTD. From LES PETITS-FILS DE F. DE WENDEL ET CIE. (E.P. 257,828, 26.2.26).—A liquid explosive charge contains cooling salts such as sodium chloride or sodium carbonate, and blast-furnace dust as an absorbent material for the liquid air. S. BINNING.

Detonator [priming composition]. E. VON HERZ (E.P. 241,892, 15.10.25. Conv., 23.10.24).—Metal salts of the *iso*nitroamines are used in priming compositions for detonators and percussion caps. The lead salts of methylene di*iso*nitroamine and ethylidene di*iso*nitroamine are specially mentioned. They may be used in detonators in conjunction with lead styphnate. A mixed salt obtained by the simultaneous precipitation of the lead salts of an *iso*nitroamine and of styphnic acid is very effective as a priming composition. S. BINNING.

# XXIII.—SANITATION; WATER PURIFICATION.

Water purification studies by U.S.A. Public Health Service. H. W. STREETER (J. Amer. Water Works Assoc., 1926, 16, 336-341).- A preliminary review of experimental work completed and in progress. The results obtained in a collective survey of a group of 17 municipal filtration plants made in 1923-4 to determine the maximum limits of raw water pollution which are consistent with the production of effluents meeting given standards of bacterial purity have been confirmed by the experimental work in which a small but fully equipped rapid sand filter plant was used. The results indicate that the maximum B. coli index of the raw water consistent with the production of chlorinated filter effluents conforming to the revised Treasury Department standard is approximately 5000-6000 per 100 c.c., and as regards unchlorinated effluents 60-100 B. coli per 100 c.c. Although overall efficiency of bacterial removal, as measured in terms of 37° plate count, is influenced decidedly by turbidity and seasonal changes, the corresponding overall efficiency of removal of organisms of the *B. coli* group appears to be affected only to a minor, if any, extent by either of these factors; and in general, the overall efficiency of bacterial removal, and more especially of *B. coli* removal, is affected to a considerably greater extent by variations in the density of bacteria in the raw water. W. T. LOCKETT.

Sand filtration. M. E. DICE (Chem. Met. Eng., 1926, 33, 529-533) .- Air injected during the softening of water becomes entrained in the water and penetrates into the sand of gravity filters, thus making a porous structure, and allowing the precipitate to work its way into the sand instead of forming a film on the surface. The air can be removed by a vacuum process or by raising the pressure sufficiently to dissolve the air, for example, by increasing the hydrostatic head in the filter tank, but this procedure is not advisable where the removal of bacteria is desired. No penetration of air into the sand bed occurred with a head of  $12 \cdot 3$  ft. or more of water. An empirical formula for the rate of flow of water through sand was checked experimentally and was accurate for clear water, and a formula was evolved for determining the rate of filtration at any time for the particular sediment-bearing water examined. Sand for filtration should have a low " coefficient of uniformity," this being the ratio of the sieve opening which will pass 60% of the sand to that which will pass 10%. The sand also should have a low "effective diameter," i.e., the screen opening, expressed in millimetres, which will allow 90% of the sand to pass through.

W. G. CAREY.

Physico-chemical analyses by seeded precipitation : applications to the determination of lime in waters and tartaric acid in wine. M. DUBOUX (Ann. Chim. analyt., 1926, 8, 257-264).-The electrometric titration of substances by a precipitating agent is a slow process in dilute solutions (N/300 to N/3000) owing to the time taken for the precipitate to form. By "seeding" the solution with the appropriate precipitatewhich has been previously prepared by the usual analytical procedure, thoroughly washed, and preserved in the moist condition-the time required for titration is considerably curtailed. In potable waters calcium is determined, after decomposition of bicarbonates, by electrometric titration with sodium ammonium racemate, which does not precipitate magnesium, the solution being seeded with calcium racemate. For the determination of d-tartaric acid in wine, an excess of ammonium *l*-tartrate is added and the solution titrated with calcium acetate after the addition of calcium racemate. A correction factor must be applied in this case owing to the entanglement of calcium *l*-tartrate with the precipitated racemate. The results compare favourably with those obtained by the bitartrate method.

F. R. ENNOS.

Determination of cresols. DANCKWORTT and SIEBLER.—See III.

PATENT.

[Apparatus for] preserving sea-water for biological purposes. J. KUNSTLER (E.P. 241,893, 16.10.25. Conv., 25.10.24).