

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 26, 1927.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. VI. Diameters of irregularly shaped crushed sand particles lifted by air currents of different speeds and different temperatures. G. MARTIN. VII. Efficiency of grinding machines and grinding media, with special reference to ball and tube mills. G. MARTIN, F. B. TURNER, and F. LINSTAD. VIII. Variation in sp. gr. of quartz sands on prolonged grinding. G. MARTIN, W. WATSON, and E. BOWES. (Trans. Ceram. Soc., 1927, 26, 21—33, 34—44, 45—58).—VI. Previous work on this subject (cf. B., 1927, 543) is discussed. Two series of experiments were carried out, the air in the first case being in turbulent flow, and in the second in stream-line flow. The lifting power of the air in stream-line flow was much greater than in turbulent flow; in each case the air exhibited the same laws, but with different constants. For very small particles, Stokes' law is obeyed, the constant depending upon the flow of the gas and upon the shape and nature of the particles. At some larger critical diameter Stokes' law becomes unstable, and is replaced by the parabolic law, $V = k\sqrt{d}$, in which k again probably depends upon the same three factors. The temperature of the gas has a marked influence upon the size of particle supported. The method of calculating the correction for temperature is explained. The diameter and weight of quartz particles lifted by air at speeds varying from zero to 328 ft./sec., and the corresponding mesh apertures of industrial sieves are tabulated. VII. The efficiency of a grinding machine is defined as the work performed in ft.-lb. in increasing the surface of standard Leighton Buzzard sand by 1 sq. ft. The work done in grinding is calculated from the formula: $W = B(S_2 - S_1)$, in which S_1 and S_2 are the original and final surfaces of the powder, and B is a constant measuring the efficiency of the machine (work required to increase the surface by 1 sq. ft.). The most efficient grinding media were $\frac{3}{4}$ -in. steel balls and 1-in. steel balls. Flints were very inefficient. Maximum efficiency of a mill is obtained when the surface of the sand exceeds that of the balls by from 100 to 500 times. The optimum conditions regarding speed of revolution, percentage load, and volume of the balls and the charge, and the influence of the size of the machine on the efficiency were investigated. Grinding in a current of air did not appreciably increase the efficiency. VIII. The sp. gr. of standard Leighton Buzzard sand and of 17 grades of crushed sand prepared by air-elutriation were determined by the pycnometer method. The maximum increase in sp. gr. in these grades was about 0.2%; hence they contain no amor-

phous silica. Reducing the grain-size of quartz from 0.75 mm. to 0.30 mm. has no effect on the sp. gr.; further reduction from 0.30 to 0.03 mm. causes an increase of 0.2% in sp. gr. Still further reduction below 0.03 mm. causes a fall in density of 0.07%. Formation of amorphous silica is, therefore, confined to the grade having a particle size below 0.03 mm. The amount of amorphous silica formed by prolonged grinding varies directly as the time of grinding, after the initial increase in sp. gr. has ceased. The amount of amorphous silica formed after grinding standard sand for $3\frac{1}{2}$ hrs. was 0.91%. F. SALT.

Recovery of volatile materials by condensation. G. WEISSENBERGER (Kautschuk, 1927, 162—163).—Methods for the recovery of volatile liquids such as acetone by cooling are considered. Although self-contained drying systems, such as those with internal or external circulation of the drying gases, enable a greater recovery by condensation than those operated with a continuous draught, they are less desirable on grounds of safety. The yield possible with any condensation process is naturally lower than that attainable with an absorption or adsorption process.

D. F. TWISS.

PATENTS.

Centrifugal separator. V. E. METCALFE (U.S.P. 1,630,201, 24.5.27. Appl., 16.8.23. Renewed 6.4.27).—A rotary conveyor is arranged within and co-axial with an annular rotary screen, and an electro-inductive drive is provided with means for varying the relative speeds of the conveyor and the screen. H. HOLMES.

Apparatus for the cooling or heating of liquids. H. WILKINSON (E.P. 270,537, 18.8.26).—In apparatus of the type where one liquid trickles down over a vertical stack of tubes and the other liquid zig-zags through them, a method is described of making the joints between the tubes and the end connexions so as to give flexibility.

B. M. VENABLES.

Distillation apparatus. W. K. LEWIS, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,626,346, 26.4.27. Appl., 5.12.23).—An apparatus for fractional distillation comprises a still and a fractionating column, and means for leading the vapour passing from the still to the intermediate portion of the column through a coil in the bottom of the latter, to supply the heat necessary for fractionation. T. S. WHEELER.

Apparatus for deodorising fluids. TE AROHA DAIRY CO., LTD., and H. L. MURRAY (E.P. 270,509, 11.6.26).—The hot fluid is admitted through a float

chamber to a closed cylindrical container which communicates through a goose-neck with an ejector-condenser to produce a vacuum. The container is fitted with beaters, and its lower portion is steam-jacketed so that intense ebullition of the liquid is caused, and the vapours are expelled through the ejector. A centrifugal pump discharges deodorised liquid continuously from the container.

W. G. CAREY.

Stabilisation of colloidal systems. CONSORT. FÜR ELEKTROCHEM. IND., Assees. of W. O. HERRMANN and W. HAEHNEL (U.S.P. 1,629,161, 17.5.27. Appl., 1.7.26. Conv., 17.7.25).—Sols, *e.g.*, hydrosols containing metals, metallic oxides, metallic sulphides, etc., can be stabilised by addition of polymerised vinyl alcohol, which is obtained as a white powder, soluble in water, by saponifying polymerised vinyl acetate. T. S. WHEELER.

Absorption refrigerator. E. WIRTH, Assr. to SULZER FRÈRES SOC. ANON. (U.S.P. 1,630,648, 31.5.27. Appl., 18.11.24. Conv., 22.12.23).—See E.P. 231,240; B., 1925, 385.

Kiln and other heat-treatment furnace. C. W. SPIERS, Assr. to MORGAN CRUCIBLE CO., LTD. (U.S.P. 1,630,587, 31.5.27. Appl., 21.1.26. Conv., 9.9.25).—See E.P. 261,866; B., 1927, 76.

Determining critical temperature. G. F. GERDTS (U.S.P. 1,630,485, 31.5.27. Appl., 12.12.24. Conv., 26.8.24).—See E.P. 240,636; B., 1925, 945.

Driving device for centrifugal separators. AKTIEBOLAGET SEPARATOR (E.P. 267,842, 19.1.27. Conv., 18.3.26).

[Rotary valve for] pulp thickeners or filters. R. HADDAN. From OLIVER CONTINUOUS FILTER CO. (E.P. 270,616, 11.6.26).

Separation of gases (E.P. 248,377).—See VII.

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

Netherlands East Indian coals. J. VAN DER WAERDEN (Fuel, 1927, 6, 252—256).—The coal deposits in the East Indian Archipelago belong to the eocene or younger tertiary formation; they are brown coals of high volatile matter content. Their moisture content is, in general, lower than that of the European brown coals, and their ash content frequently lower also. Some of the coals, *e.g.*, the Lematang, are of bituminous or anthracitic type. Their caking power is small, and is entirely lost on exposure of the coal to air. No positive conclusions with regard to their behaviour on carbonisation, and in particular their capacity to yield a good coke, can be drawn from a comparison with other coals of similar proximate analysis, or from their content of "oil bitumen" and "solid bitumen." The most satisfactory method of utilising these coals would appear to be by blending with coals of high caking power before carbonisation.

A. B. MANNING.

Gasifying fuels by [a mixture of] oxygen and steam. T. CERASOLI (Gas- u. Wasserfach, 70, 508—510).—The methods of calculation used by the author for determining the composition of mixed gas (B., 1924, 4) have been extended to a study of the gasification of

carbon by a mixture of oxygen and steam. The oxygen requirement, equilibrium temperature, quantity, and composition of the resulting gas have been calculated for different values of the steam consumption per kg. of carbon. The method has also been applied to the complete gasification of a typical coal by a mixture of oxygen and steam. The gas yield per kg. of coal is equivalent to 6005 kg.-cal. (1.643 m^3 of gross calorific value $3654 \text{ kg.-cal./m}^3$), as compared with 4996 kg.-cal. when air is used, the oxygen requirement being 0.387 m^3 .

A. B. MANNING.

Reactions in the fuel bed of a gas producer.

II. Effect of depth of fuel bed and rate of firing.

R. T. HASLAM, R. F. MACKIE, and F. H. REED. **III.**

Effect of steam-coal ratio. R. T. HASLAM, J. T.

WARD, and R. F. MACKIE (Ind. Eng. Chem., 1927, 19,

119—124, 141—144).—II. Tests were made in an

experimental gas producer burning anthracite to determine the effect of depth of fuel bed and rate of firing on the reactions in the fuel bed. Steam was admitted at a constant rate of 0.4 lb. per 1 lb. of fuel, and samples were drawn through water-cooled sampling tubes from different points in the bed. Runs were made with beds of 1.5, 3.0, and 4.5 ft. depth, with firing rates of 10, 40, and 70 lb. of coal per sq. ft. of grate area per hour. The heating value of the gas, cold gas efficiency, and % of steam decomposed increased with the depth of fuel bed and the rate of firing. With increasing depth of bed and rate of firing, the temperature of the primary reduction zone is increased, as also is the proportion of carbon monoxide and hydrogen in the gas, at the expense of carbon dioxide and steam. The increased time of contact with low rate of firing does not compensate for the resulting low temperature. The thickness of the primary reduction and the oxidation zones is not affected by increase in depth of fuel bed or rate of firing. The combination of oxygen and carbon is independent of the time of contact, but is greatly affected by the velocity of the oxygen past the carbon face. Steam passes through the oxidation zone without appreciable decomposition. **III.** Under the same conditions as above, the effect of increasing the steam per lb. of coal from 0.366 to 1.03 lb. was tested, with a constant depth of fuel bed of 3 ft. and a constant rate of firing of 40 lb./sq. ft. of grate area. The heating value of the gas produced and the cold gas efficiency increased to a maximum, and then decreased, the optimum ratio being 0.7—0.8 lb. of steam to 1 lb. of coal. This value is higher than those obtained by other experimenters, owing to the high rate of firing employed, and the ratio, it appears, increases with the rate of firing. The best results are obtainable from producers by high rates of firing and steam consumption.

H. MOORE.

Measurement of bloom of lubricating oils.

L. M. HENDERSON and H. C. COWLES, JUN. (Ind. Eng. Chem., 1927, 19, 74—76).—The bloom was measured by a modified Ives tint photometer, in which a septum prevented the light from the photometer lamp striking the oil, a beam from an auxiliary (75-watt Mazda) lamp being cast on the surface of the oil at 45° . The oil container was a crystallising dish sand-blasted inside and out, and painted black outside. Colours were

reflected from an oil-air interface, and differed from those reflected from oil-glass. The layer of oil was 35 mm. thick and the temperature about 35°. At higher temperatures oils reflect less, but assume their bloom on cooling. The reflected colours of asphalt and white oil are similar. Deblooming increases the absorption of blue light. Debloomed and fatty oils have similar reflected and transmitted colours.

H. MOORE.

Determination of oil in paraffin wax: direct refractometer method. S. H. DIGGS and C. C. BUCHLER (Ind. Eng. Chem., 1927, 19, 125—127).—The indices of refraction for wax-free oil and oil-free wax were determined. Pressed distillate of the lowest cold test obtainable represented the oil component, and had a cold test of -22° , and n_D^{60} 1.4762. An oil-free wax was obtained by repeated treatment of a wax-oil mixture with filtering clay, and had m.p. 54° , n_D^{60} 1.4361. The wax does not suffer fractionation by filtration, but the values of n of waxes vary with their m.p. The curves of indices of refraction are based on the assumption that the index for 100% of oil constituent is a fixed point, that for 100% of the wax constituent being determined. The curve indicates the percentage of constituents for a certain refractive index, and a chart is given to facilitate its determination. For waxes containing less than $2\frac{1}{2}\%$ of oil, the index is determined at 60° on crude wax, and then on wax filtered through clay. For waxes with more than $2\frac{1}{2}\%$ of oil, the index at 60° is alone determined, use being made of the established value for the oil-free wax component. The percentages of oil determined by this method are generally higher than by the press method with waxes containing small amounts of oil.

H. MOORE.

Petroleum waxes. C. C. BUCHLER and G. D. GRAVES (Ind. Eng. Chem., 1927, 19, 718—722).—Zalozecki's theory (B., 1888, 428) that the change from amorphous to crystalline paraffin wax is due to a change from *iso*- to normal paraffins and Gurwitsch's views to the contrary are reviewed. Samples of ordinary paraffin wax, slop wax from higher-boiling distillates, petrolatum from residual stocks, and rod wax from petroleum wells were freed from oil by recrystallising from ethylene dichloride to constant m.p., and were fractionally distilled under a pressure of less than 1 mm. of mercury. That decomposition or rearrangement did not occur was shown by recombining aliquot parts of the fractions and still residuum, when this mixture had in each case the same m.p. and refractive index as the undistilled material. After removal by recrystallisation of a portion designated "soft wax" from all the fractions, a straight-line relationship between refractive indices and m.p. was found to hold, this relationship being identical for all four types of wax. Mol. wts. were determined cryoscopically and confirmed by combustion analyses, and the m.p.-mol. wt. curves were found to be in close agreement with that of Kraft for synthetic normal paraffins. It is concluded that all four types of wax are composed of members of the normal paraffin series ($C_{18}H_{38}$ to $C_{43}H_{88}$), and, as all the purified fractions crystallised in the form of plates of the same type, it is believed that the "soft wax" which was removed is

in some way responsible for the needle form of ordinary crystalline wax.

C. O. HARVEY.

Mechanism of coking. E. AUDIBERT and L. DELMAS (Chim. et Ind., 1927, 17, 707—722).—See B., 1927, 383.

Bone black. KNOWLES.—See XVII.

PATENTS.

Separating coal from dirt and like substances. W. H. BERRISFORD (E.P. 265,341, 11.12.25).—Coal containing dirt is fed on to the upper of a series of smooth inclined plates which are spaced. The lower end of each plate is turned upwards slightly, whilst an adjustable slide also turned upwards is attached to the upper end of each plate. The coal slides down the plates and leaps from plate to plate, describing a parabolic path. By turning up the lower and upper edges of each plate the paths of the coal and the dirt are made relatively more divergent. The gaps between the successive plates can be easily adjusted so that the dirt falls between the plates and the coal passes forward. S. PEXTON.

Coal washing apparatus. C. MARCHANT (E.P. 264,460, 22.11.26. Conv., 16.1.26).—Coal is fed into a vertical conduit, where it meets a succession of upward currents of water fed into the conduit at different levels. Corresponding to each water inlet there is an auxiliary conduit branching from the opposite side and at a higher level than the water inlet. As the coal meets the streams of water the lightest fractions are removed into the auxiliary conduits, and only the heavy scale passes through to the bottom. S. PEXTON.

Manufacture of carburetted water-gas. A. E. SHIPPEE, Assr. to STONE & WEBSTER, INC. (U.S.P. 1,630,316, 31.5.27. Appl., 18.1.21).—In a plant consisting of a generator and a carburettor, a process is operated consisting of three steps—an air-blast run, a steam run, and an independent run of free air through the carburettor.

R. A. A. TAYLOR.

Producing air-gas from inflammable liquids. H. FOERSTERLING (U.S.P. 1,628,135, 10.5.27. Appl., 5.3.25).—The liquid is vaporised with air in such proportions that the mixture burns without further addition of air, the oxygen content being slightly above that required for complete combustion, further dilution with air rendering the mixture non-combustible.

C. O. HARVEY.

Manufacture of air gas. H. FOERSTERLING (U.S.P. 1,624,046, 12.4.27. Appl., 28.3.25).—Air impregnated with a mixture of methyl alcohol and acetone vapour at ordinary temperature contains sufficient combustible gas to combine with all the oxygen present, and can be ignited at a jet to give light and heat.

T. S. WHEELER.

Gas production. T. F. RINTZE (F.P. 611,236, 13.2.26).—High-boiling hydrocarbons are cracked, the condensed light products converted by distillation into light or slightly higher-boiling secondary products, and the heavier condensation products vaporised by heating, permanent gases being formed thereby.

A. B. MANNING.

Gas generators and the like. PATENT RETORTS, LTD., and T. M. DAVIDSON (E.P. 270,429, 12.2.26).—A

retort in which coal is carbonised is superposed on a generator so that the coke which is formed in it may pass straight in to be gasified. A pair of such retorts is arranged so that the hot gas passes down one generator and then up through a flue into the top of the retort on the second generator. The connecting flues may be placed vertically in the setting of the generators and connected with the top and the bottom of the generators by means of radially-disposed passages, or arranged externally to the generators, extending cross-wise from the bottom of one generator to the top of the other retort.

R. A. A. TAYLOR.

Removal of hydrogen sulphide from coal- or water-gas. W. E. LEUCHTENBERG (U.S.P. 1,629,396, 17.5.27. Appl., 20.7.25).—An alkali carbonate solution is used to absorb hydrogen sulphide from the gas in question. The solution is then revived by being passed counter-current to a flow of air, the contaminated air being subsequently freed from hydrogen sulphide by passing it through iron oxide moving counter-current to it.

R. A. A. TAYLOR.

Low-temperature carbonisation. G. MARS (G.P. 436,918, 3.10.23).—The material is carbonised in a shaft furnace, the heat being supplied by the combustion of part of the material itself. The position and temperature of the combustion and carbonisation zones can be accurately controlled by suitable regulation of the air supply, which is distributed over the whole cross-section of the shaft.

A. B. MANNING.

Prevention of sticking of the material in continuous carbonisation processes. I. G. FARBEENIND. A.-G. (Swiss P. 116,958, 11.11.25).—The material is compressed into briquettes, which are then powdered with the dry material before being charged into the retorts.

A. B. MANNING.

Working up the volatile products of wood distillation. V. FREUND (Aust. P. 104,143, 6.12.24).—The vapours from the distillation of wood are freed from tar and the acetic acid then absorbed by passage through a tube heated externally at 100°, through which at the same time quick or slaked lime is continuously passed. The residual vapours, consisting chiefly of water and wood spirit, are separated by dephlegmation and condensed. Dry, solid calcium acetate and highly concentrated wood spirit are thus obtained directly.

A. B. MANNING.

Production of gasoline. H. B. SETZLER, Assr. to NATIONAL REFINING Co. (U.S.P. 1,629,810, 24.5.27. Appl., 9.6.16).—Carbon is separated from gas-oil by distilling at 370–400° a volume of the oil, maintained substantially constant and approximately filling the still, at 50–100 lb. pressure. Means are provided for scouring the bottom of the still and for advancing the hydrocarbons to the point of withdrawal during the distillation.

H. HOLMES.

Refining petroleum. W. E. LUMMUS (U.S.P. 1,628,252, 10.5.27. Appl., 21.5.23).—The vapours of the lighter fractions of the oil are fractionated by passage through a series of condensers, the uncondensed vapours being absorbed in higher-boiling condensate.

C. O. HARVEY.

Treatment of impure petroleum oils. J. D. BRADY, Assr. to BRADY PROCESS Co. (U.S.P. 1,628,574, 10.5.27. Appl., 29.8.25).—Emulsified petroleum is treated in a vertical tank containing a funnel head and conductor tube down which the oil passes to a chamber containing baffles and heated from below. Valves are provided for the withdrawal of oil and gases.

C. O. HARVEY.

Separating metallic base reaction products from other derivatives in the treatment of hydrocarbon oils. LITHARGE RECOVERY CORP., Assees. of A. KINSEL (E.P. 263,730, 8.3.26. Conv., 26.12.25).—An apparatus which facilitates the recovery of lead sulphide formed during the treatment of oils with sodium plumbite (cf. U.S.P. 1,525,301; B., 1925, 276, and Kinsel, B., 1926, 37) consists of a steam-heated chamber, into which the alkaline sulphide wash from the agitator is run. Coagulation of the sulphide is effected by heating with or without the addition of coagulants such as ferric sulphate, and any hydrocarbon material volatilised during heating or steam-blowing is condensed and collected. The oily and aqueous alkaline layers which separate in the chamber are drawn off through suitably placed cocks, and the lead sulphide residues pass to a filter, and may be regenerated by treatment with sulphuric acid followed by dissolution of the lead sulphate in a concentrated solution of caustic alkali.

C. O. HARVEY.

Continuous [bleaching] treatment of oils. O. J. SALISBURY (U.S.P. 1,628,747, 17.5.27. Appl., 11.4.24).—Oil is introduced into a treating chamber, to which partially spent fuller's earth is added. After agitation, the oil is passed to filters through a series of treating and settling chambers, in which it is treated with fresh fuller's earth. Means are provided for returning some of the oil carrying partially spent fuller's earth from the settling chambers to the initial treating chamber.

T. S. WHEELER.

Furnace for the treatment of spent fuller's earth. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,630,044, 24.5.27. Appl., 23.2.24).—An inclined rotary shell is provided with means for internal heating and for supplying fuller's earth into its upper end. An air conduit leading into the shell communicates with a distributing pipe leading from the conduit to the lower side of the shell, and thence close to the wall of the shell in the direction of rotation. The portion of the pipe normally covered by the fuller's earth is perforated.

H. HOLMES.

[Cracking] treatment of hydrocarbons. W. S. HADAWAY, JUN., Assr. to TEXAS Co. (U.S.P. 1,628,143, 10.5.27. Appl., 15.9.23).—The oil enters a vertical cylindrical cracking chamber through a hollow rotatable spraying mechanism actuated by the oil.

C. O. HARVEY.

Conversion of oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,628,236, 10.5.27. Appl., 8.10.21).—Oil, cracked under pressure in a heating zone, passes to an insulated vapour chamber in which a pool of liquid oil collects. The vapours pass to a dephlegmator and condenser, the reflux condensate is returned to the cracking zone, and the uncondensable gases are heated and passed through a conduit immersed

in the oil in the vapour chamber, and are finally discharged into this oil. C. O. HARVEY.

Cracking petroleum oil. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,628,270, 10.5.27. Appl., 6.12.20. Renewed 6.10.26).—The oil (under pressure) is cracked, substantially vaporised, and subjected to reflux condensation. The condensate is distilled at atmospheric pressure in a still heated by the flue gases from the furnace of the cracking zone, and the high-boiling still residues are recracked.

C. O. HARVEY.

Preparation of bituminous emulsions. UNION FRANÇ. DE CRÉDIT (F.P. 611,479, 19.2.26).—The molten bitumen is mixed with 3–5% of an animal or vegetable oil, the latter being brought into solution by the addition of a strong acid or an oxidising agent without permitting the temperature to fall; finally a dilute boiling alkali solution, in volume between once and twice that of the bitumen, is added.

A. B. MANNING.

Manufacture of agglomerated fuel. L. LIAIS (U.S.P. 1,630,662, 31.5.27. Appl., 5.4.26. Conv., 30.4.25).—See E.P. 251,605; B., 1926, 907.

Motor fuel. J. LA RIBOISIÈRE (E.P. 257,613, 25.8.26. Conv., 26.8.25).—See U.S.P. 1,557,257; B., 1925, 950.

IV.—DYESTUFFS AND INTERMEDIATES.

Vat dyestuffs of the anthraquinone [pyrazole-anthrone] series. A. HOLL, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,627,738, 10.5.27. Appl., 8.12.25. Conv., 22.12.24).—Pyrazoleanthrone-yellow, or a derivative, is treated with paraformaldehyde in presence of concentrated sulphuric acid, to yield *dyes*, which give on cotton from a hyposulphite vat, reddish- to greenish-yellow shades, fast to light and alkali. The shades obtained can be varied by varying the concentration of the acid and the temperature of condensation.

T. S. WHEELER.

Manufacture of benzanthrone. R. G. CASWELL and E. G. MARSHALL, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,626,392, 26.4.27. Appl., 31.7.20).—Anthraquinone (3 pts.) dissolved in concentrated sulphuric acid (44 pts.) is treated at 38–42° with freshly precipitated copper (2 pts.), and when dissolution of the copper is complete, glycerol (4 pts.) and water (4 pts.) are added, and the mixture is heated at 120° for 3 hrs. The yield of benzanthrone is about 90%.

T. S. WHEELER.

Azo dyes from aminodiphenylene oxide. E. GLIETENBERG, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,624,944, 19.4.27. Appl., 2.12.25. Conv., 11.12.24).—Diazotised 3-aminodiphenylene oxide is coupled with 2-naphthol-3-carboxy- α -naphthylamides to give *dyes* yielding clear claret shades on cotton, which, when directly produced on the fibre, are very fast to light and kier boiling.

T. S. WHEELER.

Production of indophenol. J. G. DINWIDDIE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,628,534, 10.5.27. Appl., 18.3.24).—In the production of indophenols by the condensation of derivatives of carbazole with derivatives of nitrosophenol in presence of sulphuric

acid, products of increased purity are obtained by performing the condensation at below –4°.

T. S. WHEELER.

Separation of tertiary from secondary and primary aromatic amines. BRITISH DYESTUFFS CORP., LTD., E. H. RODD, and R. W. EVERATT (E.P. 270,930, 21.8.26).—The dry mixture of amines ordinarily obtained by alkylation etc. of arylamines is treated with chlorosulphonic acid, whereby the primary and secondary amines are converted into sulphamic acids. On steam distillation from alkali, the tertiary amine is obtained pure. The sulphamic acids are hydrolysed with dilute sulphuric acid, and secondary and primary amines recovered. A mixture containing only secondary and tertiary amines gives each in pure condition. If the original mixture contains more than 15% of secondary amine, it should be diluted with tertiary amine or a solvent. The separation of mono- and di-ethylanilines (12% of the former, 88% of the latter) is described.

C. HOLLINS.

Manufacture of dihydroxyperylene. A. ZINKE and A. KLINGLER, Assrs. to F. BENSA (U.S.P. 1,629,194, 17.5.27. Appl., 4.10.22).—See E.P. 191,363; B., 1924, 34.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Provision of cellulose for viscose silk factories and utilisation of waste lyes from the manufacture. F. GIORDANI and A. CITTADINI (Giorn. Chim. Ind. Appl., 1927, 9, 165–167).—The extraction of cellulose by the Italian gaseous chlorine method and the manufacture of viscose artificial silk are conveniently worked in conjunction, the waste lye from the pressing of the alkali-cellulose being used for the preliminary alkali soaking of the vegetable fibre. Under suitable conditions this procedure exerts no deleterious effects on the yield or quality of the viscose silk, and does not increase the amounts of reagents used.

T. H. POPE.

Evaporation of sulphite[–cellulose] waste liquor. W. L. BADGER (Ind. Eng. Chem., 1927, 19, 677–680).—An evaporator, in which the liquor is mechanically circulated, thus minimising the formation of scale on the heating surfaces, is described. The liquor is pumped through vertical, steam-heated nickel tubes enclosed in a tower, issues from the upper ends of the tubes in the form of spray, and returns to the pump down the sides of the tower. After a run of 150 hrs., during which the test conditions (steam pressure etc.) were adjusted to imitate those in the several stages of a quadruple-effect evaporator, no scale was formed inside the tube, and the heat transfer coefficients were several times as high as those obtained in a standard vertical or horizontal tube evaporator.

W. J. POWELL.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Conditioning [bleaching] textiles. W. L. CONRAD (U.S.P. 1,630,786, 31.5.27. Appl., 25.5.23).—The material, with the same end always foremost, is passed in a continuous process through a vat or kier containing a boiling cleansing solution, through a washing machine, a bleaching bath, a piling machine, a washing machine, a hypochlorite or similar bath, a second piling device,

and then through a final washing machine. After leaving each bath, the excess liquor is squeezed out before the material passes to the next, and the latter is submitted to air-bleaching during its passage through the piling devices.

B. P. RIDGE.

Reserving animal fibres. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 248,007, 22.2.26. Conv., 20.2.25).—Wool or silk in union fabrics resists direct cotton dyes when certain synthetic tanning materials are applied to the fibre, or are added to the direct dye-bath; two-colour effects are thus obtainable. The substances used are aromatic, or partly hydrogenated aromatic, sulphonic acids condensed through a carbon, sulphur, or oxygen atom with a sulphonated or unsulphonated residue, and suitable examples are sulphonated ditolyl methylene ether, sulphonic acids of benzylated and alkylated naphthalenes, the salts of sulphonic acids obtained by condensing naphthalene with sulphur chloride in sulphuric acid or by oxidation of a sulphurised phenol resin, or by condensing a phenol-aldehyde resin with sulphobenzyl chloride. Thus half-wool, dyed with Naphthol Yellow SEL or Amidonaphthol Red BB, is immersed in a bath containing Dianil Pure Blue PH or Dianil Green BBN and 3% of the reserving substance; or half-silk, dyed with Ponceau G, is immersed in a bath containing Dianil Green GN and 3% of the reserving substance.

C. HOLLINS.

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

Rotary kilns for pyrites burning. C. P. DEBUCH (Papierfabr., 1927, 25, 365—379).—The principle of the rotary kiln has not hitherto been applied to pyrites burning owing to the very large proportion of air required for the reaction and the necessity for close temperature-control if sintering is to be avoided. The Kauffman kiln avoids these difficulties by distributing the air between a number of ports along the kiln. This arrangement incidentally causes the cinders to be discharged at a much higher temperature than usual. To complete the burning off, the latter part of the kiln is fitted with scrapers. These also improve the mixing of the gases and are equivalent to a lengthening of the kiln. They are constructed by the insertion of special projecting blocks in the internal brick lining. The pyrites is charged and the cinders are withdrawn by devices arranged to exclude air-leaks. The air-ports consist of cast-iron sockets, leading well to the interior of the kiln, protected with an outer brick lining and easily removed. They are constructed for either suction or pressure supply, and also serve as observation holes. The gas exit pipe passes downwards through a bend luted into the flue so as to allow for the play necessary consequent on the rotation of the kiln. Earlier forms of this apparatus failed owing to the length being insufficient, but the kiln described has been in constant use since 1924, on pyrites fines. The capacity is 10 tons per 24 hrs. The residue is well burnt; the gases leave at 500—800° and average 10% SO₂. From 4% to 5% of the sulphur used is burnt to sulphur trioxide. The dust produced and the reduction in size of the pyrites particles during combustion do not seem to be very

different from corresponding results with a shelf burner. The power required is 5 h.p., and in spite of the need of careful control, labour requirements are on the whole less than with the shelf burner. Whilst high-class constructional materials must be used, the capital outlay does not exceed that for the best shelf burners. The life of the lining of the kiln is estimated at 3—6 years.

C. IRWIN.

Calcium nitrate. McCANDLESS and BURTON.—See XVI.

PATENTS.

Treatment of brine. A. K. SMITH and C. F. PRUTTON, Assrs. to DOW CHEMICAL CO. (U.S.P. 1,627,068, 3.5.27. Appl., 29.9.23).—Brine containing calcium and magnesium chlorides is concentrated to *d* 1.38, and filtered from separated sodium chloride. The filtrate is evaporated under reduced pressure, when tachydrate separates until the greater portion of the magnesium chloride is removed. The precipitate of tachydrate is washed with 25% of water at 22—50°, and with saturated magnesium chloride solution. Relatively pure magnesium chloride remains.

T. S. WHEELER.

Manufacture of ammonium nitrate. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,625,807, 26.4.27. Appl., 26.5.26).—Air is passed up a tower in counter-current to a descending stream of aqueous ammonia, and the mixture of air and ammonia gas thus obtained is led through ammonium nitrate solution, to which nitric acid is simultaneously added. The air freed from ammonia and water vapour is returned to the tower. The heat of reaction between ammonia and nitric acid is used to concentrate the solution of ammonium nitrate as it is formed.

T. S. WHEELER.

Preparation of alkali metal cyanides. R. W. POINDEXTER, JUN., and P. T. DOLLEY, Assrs. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,624,147, 12.4.27. Appl., 17.4.26).—Hydrogen cyanide is introduced at about 600° into a molten mixture of sodium carbonate and sodium cyanide, and, when reaction is complete, carbon is added, the mixture being heated at 900° to reduce any sodium cyanate and sodium cyanamide, and to decompose any sodium ferrocyanide present. The product, while still molten, is filtered through a perforated iron plate, and contains about 95% of sodium cyanide.

T. S. WHEELER.

Production of alkali hyposulphites. I. G. FARBENIND. A.-G. (Austr. P. 104,397, 19.10.25. Conv., 14.2.25).—Alkali bisulphites are reduced with alkali amalgams under such conditions that the alkali sulphite content of the reaction mixture is kept as low as possible.

L. A. COLES.

Production of ammonium polysulphide. D. F. WILHELM (Dutch P. 15,635, 6.11.24).—Liquid ammonia is stirred with an excess of sulphur heated above its m.p.

L. A. COLES.

Manufacture of sodium azide. F. WILCOXON and B. GROTTA, Assrs. to ATLAS POWDER CO. (U.S.P. 1,628,380, 10.5.27. Appl., 27.10.24).—Hydrazine hydrate solution (1 pt., 50% N₂H₄), sodium hydroxide (7.49 pts.) in absolute alcohol (7.5% solution), and ethyl nitrite (1.76 pts.) react to give sodium azide in 90% yield.

T. S. WHEELER.

Manufacture of magnesium oxide and calcium pentasulphide. V. DREWSSEN, Assr. to WEST VIRGINIA PULP AND PAPER CO. (U.S.P. 1,628,311, 10.5.27. Appl., 11.12.23. Renewed 8.10.26).—Dolomitic lime is slaked and the aqueous suspension resulting is heated with sulphur under pressure at 120°. A solution of calcium pentasulphide and thiosulphate is obtained, leaving a residue of magnesium hydroxide.

T. S. WHEELER.

Treatment of alunite. T. A. MITCHELL, Assr. to L. M. HUGHES (U.S.P. 1,628,174, 10.5.27. Appl., 9.8.22).—Ground alunite (100 pts.) is calcined at 1200° with calcium carbonate (80 pts.), and the product extracted with sodium carbonate solution to give a solution of sodium and potassium aluminates, carbonates, and sulphates, which is treated with carbon dioxide to precipitate aluminium hydroxide, concentrated to separate alkali sulphates, and then returned to the process. In a modified method, by using a mixture of calcium and barium carbonates, no sulphate passes into solution on treatment of the calcine with sodium carbonate solution.

T. S. WHEELER.

Production of arsenic compounds. E. R. RUSH-TON (U.S.P. 1,624,281, 12.4.27. Appl., 28.5.24).—Arsenic trioxide vapour is passed with air over calcium oxide at about 900°, when tricalcium orthoarsenate, which is of value as an insecticide, is rapidly formed in satisfactory yield.

T. S. WHEELER.

Separation of crystals from solution and the regeneration of heat in connexion therewith. P. H. MÜLLER (E.P. 269,068, 18.10.26).—Heat is recovered from hot solutions or lyes from which salts are deposited on cooling, by passing the hot solution through a series of closed vessels at diminishing pressures, the vapours formed thereby being used to heat liquid for the treatment of further crude salt. The vacuum vessels are connected by horizontal or inclined pipes, and the inlet pipe for hot liquid terminates so far below the liquid level that the pressure at the mouth of the pipe is equal to or greater than the vapour tension of the liquid, whilst downwardly extending pipes are connected with pumps to draw off the salt which settles out, the salt from the first two vessels being removed separately. In the upper part of the vacuum vessels are placed mixing or surface condensers at different levels in each vessel, and joined by siphon pipes through which the cold lye circulates in the reverse direction to the hot liquid passing into the vessels, air being drawn off from these condensers by ejectors actuated by steam or lye.

W. G. CAREY.

Dispersion of matter into a finely-divided form. H. L. GLAZE, Assr. to A. R. MAAS CHEMICAL CO. (U.S.P. 1,625,203, 19.4.27. Appl., 30.9.24).—Sulphur (1 pt.) is mixed with soda ash (3 pts.), and the mixture is heated at 120°, cooled, and extracted with water. The sulphur is obtained in a finely-divided form. T. S. WHEELER.

Separation of gases. GES. FÜR LINDE'S EISMASCHINEN A.-G. (E.P. 248,377, 24.2.26. Conv., 25.2.25).—The process applies particularly to the production of mixtures of hydrogen and nitrogen from gases which contain also carbon monoxide and other gases. Partial condensation is effected under pressure by cooling the

compressed gases by heat exchange from counter-current flow. The components less volatile than nitrogen are thus partially liquefied, so that the hydrogen in the residual gas reaches a concentration approximating to that desired. The residual gas is then washed with liquid nitrogen under pressure, whereby the constituents not required are replaced by nitrogen. The washing is effected at nearly constant temperature; the washed gas is then heated by heat exchange with the unwashed gases, and is expanded, the resultant cooling effect being utilised for liquefying nitrogen; or this refrigerating effect may be transferred indirectly to the nitrogen by means of the gas mixture remaining after liquefaction.

R. A. A. TAYLOR.

VIII.—GLASS; CERAMICS.

Microstructure of earthenware. H. INSLEY (J. Amer. Ceram. Soc., 1927, 10, 317—326).—Samples of glazed earthenware bodies, varying in composition and in firing treatment, were examined microscopically. The development of mullite and the solution of the quartz, both within the body and at the contact of the body and glaze, were particularly noted. In general, the greater the heat treatment of the unglazed ware the longer the mullite crystals within the body and at the point of contact of body and glaze. Mullite crystals at the contact grow out from the body into the glaze normally to the plane of contact. Such crystals are, in general, larger than those found within the body.

A. T. GREEN.

[Terra cotta] colour problem. H. SPURRIER (J. Amer. Ceram. Soc., 1927, 10, 330—333).—Pieces of white enamelled terra cotta became contaminated by a chromium compound during particular kiln firings, the colour being impaired. Microscopic examination of pieces of the enamel which had flaked off from the body showed that the colour effect (pinkish) was entirely superficial, and was located in small spots of rather greater opacity than the contiguous areas. These spots, which were the nuclei for the colour formation, proved to be rich in tin oxide. The coloured enamel contained chromium in amounts greater than those found in the original constituent ingredients. Further, the kiln atmosphere carried chromium. By passing carbon dioxide over heated charcoal in a Hoskins furnace and allowing the products of the reaction to act on the pinkish-coloured ware for a short time in the furnace, the colour was removed. A test for chromium was achieved by the use of a solution of diphenylcarbazine in glacial acetic acid, the solution being made up with methyl alcohol. Use of complementary colour effects is also developed in the detection of these spots.

A. T. GREEN.

Ageing of enamels. H. G. WOLFRAM and R. H. TURK (J. Amer. Ceram. Soc., 1927, 10, 334—338).—The ageing of an enamel as a suspension causes the clay and other particles to assume effective colloidal properties, thus increasing its power of cohesion to the metal base. It is shown that where the necessity for rapid production does not allow time for ageing, soaking of the clay is an excellent substitute. The mobility and yield value of enamel suspensions are discussed.

A. T. GREEN.

Solubility of enamel frit in mill water. H. L. COOK (J. Amer. Ceram. Soc., 1927, 10, 339—343).—Periodic occurrences of "pinholing" were proved to be due to the fusion of borax crystals formed as the sprayed enamel dried slowly. A 600 lb. batch of enamel was milled and 25 lb. samples were taken at intervals during the grinding. The fineness of the particles was determined and the liquors were analysed. The solubility of the frit increases with increasing fineness. After dry-grinding, the enamel frit is in such a state that an appreciable portion of the soda and boric acid immediately dissolves. Further experiments suggest that the operations of mixing, smelting, puddling, and resmelting have little effect upon the solubility. A. T. GREEN.

Effect of various electrolytes when added to enamel suspensions made with and without clay. H. L. COOK (J. Amer. Ceram. Soc., 1927, 10, 344—346).—A clay slip, an enamel suspension, a finely-ground clay-sand slip, and a finely-ground sand suspension were subjected to the action of the electrolytes, magnesium sulphate, magnesium hydroxide, barium chloride, ammonium carbonate, calcium hydroxide, calcium chloride, borax, sodium hydroxide, and sulphuric acid. The anticipation that the "setting-up" or stiffening of water suspensions is dependent on the presence of clay was not realised, for suspensions without clay showed marked action. It is suggested that, in the circumstances of the experiments, the fineness of grinding and degree of solution of the suspended material are factors of greater importance than the colloidal properties of the clay. A. T. GREEN.

Some dryer considerations. R. S. TROOP (Trans. Ceram. Soc., 1926, 25, 352—360).—A *résumé* of the problems involved in formulating drying schedules and determining the efficiencies of drying plants, together with a consideration of the methods in vogue for drying refractories, clay wares, moulds, and cores. A. T. GREEN.

PATENT.

Composition for coating metal surfaces. R. R. DANIELSON (U.S.P. 1,629,072, 17.5.27. Appl., 1.12.23. Free of use in U.S.A.).—A mixture of silica (64.9 pts.), borax (36.96 pts.), sodium nitrate (6.95 pts.), and triplumbic tetroxide (8.17 pts.) is sintered at 800—900°, the product being ground and mixed with enamel clay (93 pts.) and water to form a paste which is employed to protect metallic surfaces in selective carburisation. The surfaces are pickled in acid before application of the paste. T. S. WHEELER.

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

Direct production of iron [from its ores]. F. WÜST (Stahl u. Eisen, 1927, 47, 905—915, 955—964).—The production of sponge iron from iron ores by the methods of Edwin, Wiberg, Hornsey, and the U.S. Bureau of Mines is discussed at length, and the chemical equilibria and heat-balance of the various methods are compared. Direct reduction with carbon may be effected at temperatures between 700° and the sintering point of the ore without the use of an excess of reducing agent and with the minimum loss of heat energy; the reaction is strongly endothermic, and the reduced metal

usually contains more or less phosphorus and sulphur. On the other hand, reduction with carbon monoxide commences at 800—850°, is slightly endothermic, and requires a large excess of gas, entailing greater heat losses, but the product contains relatively little sulphur and phosphorus. In the Edwin process the gases from the reduction are cleaned by passage through a lime-tower, heated at 1600° together with some vaporised oil in a high-tension arc, and regenerated by passage through coke to which sufficient lime is added to slag the ash. The ore is reduced at 800—850° and the product separated magnetically into sponge iron for briquetting, an intermediate product, which is returned to the reducer, and a non-magnetic product, comprising the greater part of the gangue. For an ore containing 44% Fe the energy consumption per ton of iron was 7300 kw.-hrs., the coke consumption 312 kg., and the oil consumption 44.3 kg. The sponge iron produced contained 0.31% C, 0.16% Mn, 0.032% P, 0.013% S, 1.57% SiO₂, and 1.37% O, and the recovery was over 90%. The analyses of soft iron and mild steel obtained by melting the briquettes are given, together with details of their mechanical properties. In the Wiberg process the reduction is carried out in shaft furnaces, into which the gas, heated at 1100° in the regenerator, is passed; part of the gas is withdrawn from the middle of the shaft, cleaned from suspended dust, and passed through a coke layer in the regenerator, whilst the remainder passes up to the cooler parts of the shaft and effects a preliminary reduction of the freshly charged ore. The consumption of reducing agent and of electrical energy in this process is very similar to that of the Edwin process, but the rate of production of sponge iron is much lower. Methods involving reduction with solid carbon require a much simpler apparatus, and hence entail smaller heat losses, and consequently the costs of production are much lower. A. R. POWELL.

Graphitising behaviour of iron carbide in pure iron carbon alloys in the critical range. H. P. EVANS and A. HAYES (Trans. Amer. Soc. Steel Treat., 1927, 11, 691—710).—Pure iron-carbon alloys containing 2.34% C were made by melting together Armco iron and pure Acheson graphite. When heated in a slightly oxidising mixture of carbon monoxide and dioxide at a pressure of 5 atm. at a temperature of 700—1080° graphitisation occurred, showing that iron carbide was metastable. The density fell during treatment from 7.80 to 7.66. The graphitisation at normal pressure was less than at 5 atm. T. H. BURNHAM.

The A3 stable transformation. H. A. SCHWARTZ (Trans. Amer. Soc. Steel Treat., 1927, 11, 767—780).—From observation of the stable A1 point of an iron-carbon-silicon alloy it was found that 1% of silicon raised it 17.6° compared with 16.6° for metastable alloys. By extrapolation the A3 stable point approaches 799° as a limit. Experiments with addition of nickel and other elements indicate the same temperature with sufficient accuracy. The A3 stable point is so close to that of A2 in pure iron that the β -phase is thought not to exist in the stable system. It is suggested that whilst austenite contains carbon in solution in the form of one atom of carbon in each molecule, boydenite contains

more than one and probably three atoms of carbon per molecule.

T. H. BURNHAM.

Effect of stress on the decomposition of austenite. R. L. DOWDELL and O. E. HARDER (Trans. Amer. Soc. Steel Treat., 1927, 11, 781—790).—Bars of alloy steels quenched to give an austenitic-martensitic structure were tempered at 100—200° under a bending stress less than their yield point. A permanent stress resulted but no difference of microstructure on the tension and compression sides. On upsetting specimens of austenitic structure at room temperature prominent slip lines were produced, but not the characteristic acicular martensitic structure. Tensile stress produced by forcing rings on a taper rod produced characteristic martensite. Austenitic specimens deformed in tension and subjected to -57° were transformed to martensite, whilst unstressed specimens were unchanged. Hammered austenitic specimens were transformed more readily on heating than unstressed ones. It is concluded that deformed austenite is rendered less stable on heating or on cooling.

T. H. BURNHAM.

High-silicon structural steel. H. W. GILLET (U.S. Bur. Stand. Tech. Paper No. 331, 1926, 21, 121—143).—A number of analyses of "Freund" steel showed a content of about 0.12% C, 0.5—0.8% Mn, and 0.8—1.2% Si. This steel is at present made in the Bosshardt furnace in Germany, but the physical properties (high yield-point and high ductility) are purely functions of the chemical composition, samples made in other furnaces giving similar results. The content of phosphorus, sulphur, and oxygen does not differ from that of other open-hearth steels. American high-strength structural steel on the contrary usually contains 0.3% C and about 1.0% Mn. The specimens of Freund steel (d 7.78) examined unetched showed much finely-divided sulphide, but few silicate inclusions. The combination of yield-point and ductility was better than that found in high-carbon steels of normal manganese content. The properties were, however, closely matched by reducing the carbon content and increasing manganese. Silicon in quantities above 0.5% is considered to be a true alloying element, and may be looked upon as equivalent to manganese, the choice between the two depending on economic considerations.

C. IRWIN.

Temperature measurements in liquid iron and steel. M. WENZL and F. MORAWE (Stahl. u. Eisen, 1927, 47, 867—871).—The temperature of a bath of molten steel or iron in the reverberatory or blast furnace may be measured by means of a platinum-platinum-rhodium thermocouple enclosed in a thin quartz tube inside a "silite" tube; for temperatures up to 1,350° a nickel-iron couple may also be used. Temperatures taken with a Holborn-Kurlbaum optical pyrometer during casting are approximately 10° lower than those taken with a thermocouple, provided that the bright spots in the metal stream are focussed. The temperature of a stream of molten steel usually appears to be lower than that of the metal in the mould after removal of the slag layer, if the measurements are taken with an optical pyrometer; this is probably due to the presence of an oxide skin on the molten stream.

A. R. POWELL.

Macro- and microstructure of blowhole segregations in steel. A. WIMMER (Stahl u. Eisen, 1927, 47, 781—785).—The recurrence and mode of formation of blowholes in steel and iron are discussed with reference to the composition and microstructure as revealed by various etching reagents. The results appear to indicate that sulphide and oxide segregations are the chief cause of blowholes as, in all cases, the sulphide inclusions are much more numerous around the blowholes than in the remainder of the metal. On the basis of observations on the distribution and arrangement of the various slag inclusions in steel and iron, a diagram of the ternary system, iron-ferrous oxide-ferrous sulphide, has been constructed in which the possibility of the formation of a ternary eutectic is suggested.

A. R. POWELL.

Magnetic analysis of high-speed steel. T. SPOONER (Proc. Amer. Soc. Testing Materials, 1927, 26, ii, 116—147).—The results of resistivity and various magnetic tests of high-speed steel are plotted against the quenching temperatures. Quenching temperatures between 1211° and 1296° gave similar microstructure and hardness, but different magnetic results. The most effective quench was obtained after 3—4 min. at the high temperature. The quenched bars were drawn in a salt bath at 538—621°, and the results of magnetic tests at once and after ageing are recorded.

CHEMICAL ABSTRACTS.

Treatment of metallic surfaces with aluminium. E. NEUMANN (Zentr. Hütten- u. Walzwr., 1926, 30, 554—556; Chem. Zentr., 1927, I, 945).—Treatment of the surface of iron and steel articles with aluminium renders them immune from scaling up to 1000°; nickel or nickel-chromium alloys may similarly be protected from oxygen and gases containing sulphur compounds up to the m.p. of the metal. The coating process involves heating the article at a high temperature in a mixture of aluminium turnings, alumina, and ammonium chloride in a closed container.

A. R. POWELL.

Analyses of copper-refining cell voltages. E. W. ROUSE and P. K. AUBEL (Amer. Electrochem. Soc., April, 1927, 51. Advance copy. 9 pp.).—In the electrolysis of a copper-refining electrolyte containing 185.5 g. of sulphuric acid, 42 g. of copper, 14.8 g. of nickel, 3.8 g. of arsenic, and 0.5 g. of iron per litre with current densities of 1.6 and 1.9 amp./dm.² at 57° using two copper electrodes 4 cm. apart, measurements have been made of the polarisation at anode and cathode, the voltage drop in the electrolyte, the total cell voltage, and the effect on these quantities of varying separately the temperature, the concentrations of sulphuric acid, copper, nickel, arsenic, and iron, and of adding small quantities of chloride (as hydrochloric acid) or glue to the electrolyte. The results are summarised by a series of graphs. The total cell voltage at given current density is mainly determined by the temperature and the concentrations of sulphuric acid and copper. Decreasing the copper content decreases the resistance, but increases the cathode polarisation, the latter effect becoming very considerable when the concentration falls below about 15 g./litre. Decreasing the nickel concentration reduces the voltage drop in the electrolyte, but causes an appreciable increase in cathode polarisation;

as a result of these opposing effects, the total cell voltage has a minimum value for about 13 g. of nickel per litre. Very small concentrations of glue raise the cathode polarisation considerably, so that addition of 10 mg./litre raises the total cell voltage by 60%. Added chloride acts as a cathodic depolariser. H. J. T. ELLINGHAM.

Addition agents in electro-deposition. G. FUSEYA and M. NAGANO (Amer. Electrochem. Soc., April, 1927, 51. Advance copy. 15 pp. Cf. Fuseya and Murata; B., 1926, 950).—The effect of addition of glycine on the electrodeposition of copper from a *M*-copper sulphate solution has been investigated. Cathodic deposits from such solutions weigh more than the quantity of copper corresponding to Faraday's law, and assuming that the excess is due to glycine in the deposit, the concentration, c_s , in mols. of glycine per mol. of copper in the deposit can be determined. With neutral solutions at 17° and 0.04 amp./cm.², c_s increases from 0.015 to 0.05 when c , the concentration of glycine in the solution (mol./mol. of copper), is increased from 0.00125 to 0.10, c_s being greater than c until c exceeds 0.01. When $c = 0.01$, increasing the current density from 0.01 to 0.1 amp./cm.² causes first a rapid and then a slower increase in c_s . Increasing the temperature of such a solution from 17° to 35° at 0.04 amp./cm.² causes an almost linear fall in c_s from 0.0236 to 0.001. Acidifying this solution with sulphuric acid causes only a slight fall in c_s until the acid concentration exceeds about 0.001 *M*, when there is a rapid fall, and with *M*-sulphuric acid concentration the effect of the glycine on the weight of the deposit is entirely eliminated. Under all conditions the physical character of the deposit seemed to be determined solely by the magnitude of c_s ; size of crystals diminishes with increase of c_s . In an attempt to relate the above phenomena to the concentration of complex ions in the solution, measurements were made of the *E.M.F.* of cells of the type, Cu/*M*-CuSO₄/*M*. CuSO₄ + glycine (concentration c)/Cu. The glycine lowers the copper-ion concentration, c_o , to an apparent value, c_o' , such that $(c_o - c_o')/c_o = 1.3$ *c*, approximately. H. J. T. ELLINGHAM.

Reversed potentials in the corrosion of tin plate. C. L. MANTELL and W. G. KING, JUN. (Amer. Electrochem. Soc., April, 1927, 51. Advance copy. 7 pp.).—In a study of the conditions for the corrosion of the tin plate of cans containing food products, measurements were made of the *E.M.F.* between iron and tin electrodes in freshly-opened cans of various food products (Mantell and Lincoln, Canadian Chem. Met., Feb., 1927). In foods preserved in saline solutions, the *E.M.F.* soon fell to zero, and then increased to a maximum in the reverse direction, the iron becoming nobler than the tin, apparently owing to the formation of a passivity film. This *E.M.F.* reversal did not occur with foods put up in sugar solutions. *E.M.F.s.* have now been measured between iron and tin electrodes in various simple solutions, and their variation followed during a period of 5 min. In strong electrolytes, the *E.M.F.* reverses rapidly, the iron being ennobled and thus protected against corrosion. In organic acids, such as formic, acetic, and citric, the *E.M.F.* falls rapidly at first, but a partial recovery occurs, any reversal of *E.M.F.* being temporary. In

sucrose or carbamide solutions the small initial *E.M.F.* falls away to zero, and no reversal occurs unless a strong electrolyte is added to the solution. Solutions in which reversal of *E.M.F.* occurs are those in which perforation of the container by electrolytic action is rare. Foods put up in sugar solutions are most liable to give rise to perforation troubles. H. J. T. ELLINGHAM.

Age-hardening aluminium alloys; replacement of silicon with germanium. W. KROLL (Metall. u. Erz., 1926, 23, 684—685; Chem. Zentr., 1927, I., 1061).—Germanium may replace silicon in alloys of the type of duralumin, aludur, and lantal, but its age-hardening action is not nearly so powerful. The mechanism of the action appears to be similar to that of the silicon alloys, but the ageing must be effected at 150°.

A. R. POWELL.

System germanium-aluminium. W. KROLL (Metall. u. Erz., 1926, 23, 682—684; Chem. Zentr., 1927, I., 1061).—A thermal and microscopic examination of aluminium-germanium alloys with 0—60% Ge indicates the absence of compounds and solid solutions, and the presence in all alloys of a eutectic with 55% Ge, m.p. 423°. The presence of magnesium in the alloys leads to the formation of Mg₂Ge, which forms a solid solution with aluminium at high temperatures.

A. R. POWELL.

PATENTS.

Steel alloy. W. OERTEL, Assr. to GLOCKENSTAHL-WERKE A.-G. VORM. R. LINDENBERG (U.S.P. 1,630,448, 31.5.27. Appl., 31.7.24. Conv., 9.1.22).—The alloy, which is highly resistant to chemical action and of great strength, consists of 8—25% Cr, 0.1—1.2% C, 0.2—6.0% Mo, 0.5—2.0% Ni, and remainder iron. The addition of the nickel renders the alloy more ductile when hot. F. G. CROSSE.

[Copper-iron] alloys. T. D. KELLY (E.P. 270,553, 22.9.26).—Alloys containing 10—90% Cu, 90—10% Fe, and up to 10% of nickel, chromium, or other metal commonly alloyed with iron are prepared by rapidly melting the charge of metal or, in certain cases, of metallic oxide in an electric furnace in the presence of non-oxidising fluxes, e.g., cryolite, fluorspar, etc.

C. A. KING.

Solder for fixing in position lead-bearing metal containing alkali or alkaline-earth metals. A. WERNER (G.P. 438,392, 4.12.24).—The solder comprises 2 pts. of lead, 2 pts. of tin, and 0.2% P. The latter is preferably added as phosphor-tin. A. R. POWELL.

Separation of zinciferous pyrites into its constituents. H. SCHUMACHER (G.P. 437,891, 29.2.24).—Pyritic iron ore containing zinc is roasted and the product mixed with an oxidised iron ore or with the residue from leaching roasted cupriferous pyrites with sulphuric acid. The mixture is sintered by roasting with a small proportion of the original pyrites, and the product is smelted in a blast furnace to obtain pig iron and zinc oxide fumes. A. R. POWELL.

Recovery of zinc oxide from furnace gases. Manufacture of zinc oxide. J. F. CREGAN, Assr. to AMERICAN SMELTING AND REFINING Co. (U.S.P. 1,628,952—3, 17.5.27. Appl., [A], 12.8.22; [B], 25.10.24).—(A) The

zinc fume formed in a reverberatory furnace is subjected to a reducing atmosphere at a high temperature by means of a reducing gas; the resulting metallic fume is subsequently oxidised. (B) The vapour distilled from the smelting of material containing zinc is passed into a settling chamber to discharge impurities, after which it is removed and mixed with air and converted to the oxide within a combustion chamber.

H. ROYAL-DAWSON.

Platinum metal alloys for tipping the nibs of fountain pens. W. C. HERAEUS G.M.B.H., Assees. of E. HAAGN (G.P. 437,173, 17.9.25).—The alloys consist of 40–60% Ru, 35–50% Os, and 5–15% Pt.

A. R. POWELL.

Concentration [flotation] of ores. C. H. KELLER, ASST. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,628,151, 10.5.27. Appl., 24.11.25).—Potassium ferri-cyanide is added to sodium xanthate solutions used in ore flotation.

T. S. WHEELER.

Ore flotation process. J. HERMAN, A. W. ALLEN, and H. R. NEWITT (U.S.P. 1,628,046, 10.5.27. Appl., 25.11.25).—Ore pulp containing oxygen compounds of metals is subjected to the usual flotation process, but in place of air, a reducing gas, *e.g.*, coal-gas, is employed to provide an ascending stream of bubbles.

T. S. WHEELER.

De-watering flotation slimes. F. KRUPP GRUSONWERK A.-G. (G.P. 435,890, 29.3.23).—Flotation slimes are filtered on a bed of coarse-grained material which is given a shaking motion in order to force the coarse particles into the froth and break the bubbles.

A. R. POWELL.

Concentration of minerals from ores by the use of sulphones as flotation reagents. F. LAIST and F. F. FRICK (U.S.P. 1,629,080, 17.5.27. Appl., 13.4.26).—Sulphones, *e.g.*, diphenylsulphone, are of value as froth-flotation reagents.

T. S. WHEELER.

Manufacture of plated articles. E. G. BEK (E.P. 244,487, 11.12.25. Conv., 11.12.24).—Copper, bronze, brass, or nickel-silver alloys are plated with gold or silver from any of the well-known plating baths, the deposit is washed and coated with a strong solution of boric acid and/or borax, and the article is then heated at such a temperature above 450° and for such a time (2–5 min.) that the desired colour is imparted to the surface of the article. If desirable, in order to obtain a modified colour, the article may be given a preliminary coating, by electrodeposition of one or more layers of a metal, such as copper or nickel, which will diffuse into the gold during the heating operation. A. R. POWELL.

Coating metal surfaces (U.S.P. 1,629,072).—See VIII.

XI.—ELECTROTECHNICS.

Law of alternating-current electrolysis and the electrolytic capacity of metallic electrodes. J. W. SHIPLEY and C. F. GOODEVE (Amer. Electrochem. Soc., April, 1927, 51, Advance copy. 25 pp.).—Previous work (Eng. J. Canada, 1927, 10, 3) showed that, in A.C. electrolysis using given electrodes under given conditions, there is a certain critical current density which must be exceeded before gas evolution begins, but at higher current densities the excess of current

above that corresponding to the critical density generates electrolytic gas in quantity required by Faraday's laws. Investigations have now been made at 30° in 0.5*N*-sodium hydroxide using 60-cycle A.C. and electrodes of various metals held between rubber insulators so as to avoid exposure of points and edges at which the current density might vary. The above conclusions were generally supported, except that, at current densities just above the critical, deviations from the quantitative law were found with platinum and silver and especially with nickel. The observed critical current densities were: Pt, 0.7; Ag, 1.6; Cu, 1.9; Fe, 3.8; steel, 4.5; Ni, 4.6 amp./cm.² For aluminium the value, if existing, must be less than 0.01 amp./cm.² In many cases it was found possible to exceed the critical density without gas evolution if the current were very gradually raised, but when gas evolution did begin its rate rose sharply to the normal value for that current density. In such cases of delayed gas evolution, oxide films were observed, especially with copper, but they disappeared when gas was evolved. With an oxidised copper electrode the apparent critical current density was 10 amp./cm.² With corroded steel there was a true critical current density of 8 amp./cm.² Assuming that the existence of a critical current density is due to an "electrolytic capacity" of the electrodes for storing up the products of electrolysis, values of this capacity are calculated for various metals, and the figures so obtained agree with those furnished by direct measurement of the maximum quantity of electricity obtainable by completely discharging an electrode immediately after breaking the electrolysis circuit. The electrolytic capacity represents the average of the quantities of electricity per cm.² which must pass after the theoretical decomposition voltage is reached before gases are liberated from each of two electrodes; the value for iron was about 0.014 coulomb/cm.² Preliminary work on the effect of frequency and temperature on electrolytic capacity is recorded. The relation between electrolytic capacity and "polarisation capacity" (Kohlrausch) is discussed, and it is concluded that the latter is negligible compared with the former in these experiments. The relation of the back *E.M.F.* to the quantity of electricity passed was determined for platinum electrodes and related to the voltage wave of the A.C. The curve of resultant voltage is distorted and out of phase with the current wave, the current leading the voltage. The experiments were undertaken to find the conditions under which production of explosive gases in electric boilers can be avoided. Current densities used in such boilers are well below the critical value for steel, and explosion risks only arise from concentration of current on small areas. In the previous paper (*l.c.*) designs of electrode chambers leading to conditions of uniform current density were given. H. J. T. ELLINGHAM.

Miguet electrode and the Miguet furnace. M. ARROUET (Amer. Electrochem. Soc., April, 1927. Advance copy. 4 pp.).—For the manufacture of ferro-alloys, calcium carbide, etc. three-phase furnaces have been preferred, but, when properly designed, large single-phase furnaces are practical and efficient. The design of the Miguet furnace enables the current to be led to a single

electrode through a number of separate circuits arranged symmetrically round the furnace. The current in each circuit being relatively small, though the total may reach 240,000 amp. The bus-bar interlacing is brought very close to the furnace so that the loop is exceptionally small and the power factor higher than has hitherto been attained with high-power single-phase furnaces. The Miguet continuous electrode is built up of previously baked carbon segments assembled above the furnace, and is fed into the furnace by releasing the pressure on the bronze supporting plates which also conduct the current to the electrode. A reducing agent such as coke is found to give maximum efficiency if the furnace is fed with alternate layers containing excess of carbon and excess of material to be reduced. The current is thus spread over a larger reaction zone, and heating is entirely by resistance. The bottom of the electrode is kept about 8 in. above the bath, so that the current passes only at the edge of the circular reduction zone, the area under the electrode being available for special refining reactions. A 5000 kw. unit operated in France during 1926 required only 3100 kw.-hrs. per metric ton of calcium carbide produced, and a saving of raw materials as compared with three-phase furnaces is claimed.

H. J. T. ELLINGHAM.

Chemical method for the standardisation of ultra-violet light. J. E. MOSS and A. W. KNAPP (Brit. J. Actinotherapy, May, 1927. Reprint. 4 pp.).—The standard Uroxameter apparatus cannot be used with vertical carbon arcs because it cannot be placed close enough to the arc. A modified apparatus is described using a circular flat-bottomed dish 15 cm. in diameter and 1.8 cm. high. The ratio of uranium salt to oxalic acid in the solution was increased 20 times, and contained 25 c.c. of a 2% aqueous solution of uranium acetate, 10 c.c. of a 2% aqueous solution of oxalic acid, and 15 c.c. of water. The depth of the solution, which must be constant in all tests, was 0.23 cm., and the dish was placed 12 in. from the arc in a direction 45° to the vertical. After a 15 min. exposure the excess of oxalic acid was titrated with permanganate. Comparative results on different types of arc are given.

C. J. SMITHELLS.

Progress of electrometric control methods in industry. H. C. PARKER (Ind. Eng. Chem., 1927, 19, 660—667).

Corrosion of tin plate. MANTELL and KING.—See X.

Copper-refining cell voltages. ROUSE and AUBEL.—See X.

Addition agents in electrodeposition. FUSEYA and NAGANO.—See X.

PATENTS.

Electric incandescence lamp [getter]. W. C. SPROESSER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,624,077, 12.4.27. Appl., 3.12.21).—A getter mixture containing silica (40%), cryolite (50%), and phosphorus (10%) is applied to the filament before flashing.

T. S. WHEELER.

Electric incandescence lamp. D. MACRAE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,624,109, 12.4.27.

Appl., 2.12.21).—A method of eliminating flashing of the filament in the manufacture of incandescence lamps, comprises exhausting the lamp sufficiently to render the residual gas non-conducting at the working voltage, and employing as a getter a substance, e.g., cryolite, which on burning is completely vaporised without evolution of sufficient permanent gas to render conducting the atmosphere in the lamp. T. S. WHEELER.

Getter for controlling crystal growth. Introduction of clean-up agents into evacuated vessels. Application of getters to electric incandescence lamps and the like. W. SPROESSER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,626,637—9, 3.5.27. Appl., [A] 6.12.22; [B] 5.5.23; [C] 21.8.23).—(A) A getter for application to thoriated tungsten filaments comprises phosphorus (13 pts.), cryolite (75 pts.), and thorium oxide (25 pts.). (B) Phosphorus pentoxide (1 pt.) is fused with cryolite (1 pt.), and the product, which does not absorb moisture, is mixed with an equal weight of powdered aluminium, and employed as a getter. On flashing, free phosphorus is liberated. (C) The getter is placed on an auxiliary filament of platinum, which is temporarily sealed in the lamp and flashed. The auxiliary filament is then replaced by the actual filament.

T. S. WHEELER.

Evacuation of [electric lamp] bulbs and the like. W. C. SPROESSER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,626,640, 3.5.27. Appl., 7.9.23).—Electric lamp bulbs are evacuated by passing through them at about 400° a current of the vapour of aluminium chloride, or like substance possessing an inappreciable vapour pressure at ordinary temperatures.

T. S. WHEELER.

Optical pyrometer. SIEMENS BROTHERS & Co., LTD., and L. G. SALMON (E.P. 270,901, 12.5.26).—In optical pyrometers of the disappearing filament type, a circular scale extending over a comparatively large arc, e.g., one extending over 240°, is employed in the indicating instrument. Preferably the portion of the scale corresponding to the non-luminous condition of the filament is masked.

J. S. G. THOMAS.

Positive electrode for electric batteries. R. OPENHEIM, Assr. to SOC. ANON. LE CARBONE (U.S.P. 1,631,642, 7.6.27. Appl., 3.1.25. Conv., 19.11.24).—See E.P. 243,300; B., 1926, 446.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Crystallising-out" of solutions of driers. ANON. (Farben-Ztg., 1927, 32, 2020—2021).—The "throwing out" of driers from solutions in light petroleum, benzene, and turpentine, etc. either by precipitation (lead driers in general) or by true crystallisation (cobalt linoleate etc.) is described in detail. The change is shown to proceed from the surface in all cases, and is attributed to the production of insoluble substances by atmospheric action. The influence of composition and of the method of preparation of the driers (i.e., fusion or precipitation), both of which have been advanced as causes of this "crystallisation," are shown to affect concentration of solution only, and to afford no true explanation of the phenomena in question.

S. S. WOOLF.

Vanadium compounds as driers. F. HEBLER (Farben-Ztg., 1927, 32, 2077—2078).—Polemical with Swehten (B., 1927, 259) and with Rhodes and Chen (B., 1922, 335 A). Vanadium driers accelerate the rate of oxygen-absorption of linseed oil films, but retard their actual drying. Boiled oil films containing 0.1% and 0.2% of vanadium remained tacky for more than 1 week, whilst the original raw linseed oil dried in 5 days. The wrinkling of films reported by Swehten is also not confirmed, and his experimental conditions are queries. The field of application of vanadium as a commercial drier is considered to be problematical.

S. S. WOOLF.

Nitrocellulose for use in lacquers. E. VON MÜHLENDahl and H. SCHULZ (Farben-Ztg., 1927, 32, 2021—2022).—Stability, solubility, and viscosity determinations to be carried out on nitrocellulose are described.

S. S. WOOLF.

Investigation of resins with X-rays. S. VON NÁRAY-SZABÓ (Biochem. Z., 1927, 185, 86—87).—The behaviour of various resins towards X-rays indicates that they exist in all grades of conditions from the amorphous to the crystalline. Crystalline resins, particularly of the benzoin type, show well-marked differences corresponding to their different chemical compositions.

C. R. HARRINGTON.

PATENTS.

Manufacture of condensation products of crotonaldehyde. W. CARPMAEL. From I. G. FARBEIND. A.-G. (E.P. 270,433, 15.2.26).—The resinous condensation product of crotonaldehyde and an aromatic amine in the absence of an acid condensing agent is claimed. A suitable solvent or diluent may be present, *e.g.*, alcohol, benzene, acetone.

S. S. WOOLF.

Production of coloured [transparent] polymerised styrene and its homologues. I. OSTROMISLENSKI, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,627,195, 3.5.27. Appl., 29.1.25. Cf. E.P. 233,649; B., 1926, 451).—Colouring materials soluble in polymerised and unpolymerised styrene, stable about 175° and unaffected by formaldehyde, which is formed in the polymerisation of styrene, are dissolved in styrene, which is then heated at 140—175° to induce polymerisation. *E.g.*, anthracene (0.5%) in styrene gives a violet fluorescence to the polymerised material, without affecting its transparency.

T. S. WHEELER.

Manufacture of resin-like substances from cracked hydrocarbon products. J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,054, 3.5.27. Appl., 27.4.23).—The coloured resinous substances which are removed by adsorbents from cracked petroleum distillates are treated with air at a slightly elevated temperature, and with polymerising agents, *e.g.*, the salts of heavy metals. Hard resins of value in the manufacture of varnishes are obtained.

T. S. WHEELER.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ultramicroscopic studies on theory of vulcanisation by H. Dannenberg. New effect of light in the system rubber-sulphur by H. Pohle. F. KIRCHHOF (Kautschuk, 1927, 184).—The formation of

nuclei of colloidal sulphur in solutions of sulphur in rubber under the influence of light of short wave-length is attributed to the occurrence of ionised atoms or positive ions which arise from the action of the radiation on the neutral sulphur atoms. The effect is regarded as analogous to that of the ionisation of gases by α - or β -rays in moist air with formation of mist. An analogous phenomenon is observable in a solution of rubber and phosphorus in benzene. The marked increase in absorption of light by rubber on vulcanisation is ascribed to the formation of the colloidal sulphur.

D. F. TWISS.

Measurement of abrasion-resistance of rubber. I. WILLIAMS (Ind. Eng. Chem., 1927, 19, 674—677).—The standardised conditions of surface contact, load, and degree of slippage in most abrasion-testing machines do not ensure the expenditure of a comparable amount of work with different samples. A machine is described enabling the measurement of the power consumed, the results being expressed in volume loss per unit of work done. The machine comprises a rotating disc with an abrasive surface against which two test blocks of the rubber are pressed with known force; measurement is also made of the force necessary to keep the blocks stationary. Results are quoted for tensile and abrasion tests on five mixtures of comparable composition, but containing new and "reclaimed" rubber in the proportions, 100:0, 75:25, 50:50, 25:75, and 0:100, the relative values for abrasion resistance at an equal rate of work being 1.0, 0.77, 0.55, 0.37, and 0.21 respectively.

D. F. TWISS.

Tables of comparison for steam pressures and temperatures in vulcanisation. W. ESCH (Kautschuk, 1927, 156—158).—Steam-pressure gauges calibrated to give a reading of 1 kg./cm. at 100° show a discrepancy of 0.5 lb./sq. in. relative to the value of 14.7 lb./sq. in. for the pressure of 1 atm. Tables are given for the inter-conversion of steam pressures and the corresponding temperatures. A table is also given showing equivalent periods of vulcanisation at different temperatures, its value being illustrated by the vulcanisation behaviour of several mixtures.

D. F. TWISS.

Physiological considerations on the latex-flow of rubber plants. A. ZIMMERMANN (Kautschuk, 1927, 95—98, 118—121, 147—149).—A critical survey of the knowledge of the character and features of latex-flow under various conditions.

D. F. TWISS.

Lamp-black in rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (Kautschuk, 1927, 166—174).—A comparative examination of several grades of lamp-black and one of carbon black prepared from natural gas, as to their chemical composition, sp. gr., relative bulkiness, behaviour on sieving, appearance under the microscope, and their effect on the strength, extensibility, energy of resilience, ageing, and abrasion-resistance of vulcanised rubber in which they have been incorporated.

D. F. TWISS.

PATENTS.

Regeneration of rubber and particularly that contained in the skeletons of disused tyres. C. DANIER (E.P. 269,127, 22.9.26. Conv., 8.4.26).—

Waste material with a small content of desirably pure rubber and a larger proportion of canvas (*e.g.*, pieces of pneumatic tyre) is impregnated with the liquid obtained by the destructive distillation of otherwise worthless rubber scrap (*e.g.*, beads of disused motor covers, old rubber tubing, etc.). The impregnated material is then immersed (for from 15 to 20 days) in concentrated hydrochloric acid at the ordinary temperature, which causes destruction of the canvas and also effects the polymerisation of the absorbed distillate into synthetic rubber. After being washed and crêped the product can be used instead of new rubber for all purposes except for making rubber solution.

D. F. TWISS.

Preservation of goods of rubber or like substances. B. D. PORRITT, T. R. DAWSON, and RESEARCH ASSOC. OF BRIT. RUBBER & TYRE MANUFACTURERS. (E.P. 269,745, 25.5.26).—In the manufacture of goods of rubber there are added small proportions of substances which protect the rubber simultaneously against the effects of oxidation and of actinic light. The former effect is produced by antioxidants such as polyphenols and aminophenols, whilst the latter is produced by dyestuffs capable of absorbing ultra-violet light.

D. F. TWISS.

Applying liquids [by means of porous rubber]. H. BECKMANN (E.P. 270,374, 27.1.26).—Rubber, or even vulcanite with microscopically small pores, is used for making rollers, *e.g.*, for use in printing machines, or pads or wicks to apply oil to bearings. Such rubber with capillary pores can be produced by coagulating a mixture of latex and sulphur with magnesium sulphate, and vulcanising the resultant firm jelly.

D. F. TWISS.

XV.—LEATHER; GLUE.

[Polarisation microscope for controlling] tanning and the penetration of the tannin. J. JOUANOVITS (Collegium, 1927, 226—236).—Pieces of limed South American hides were washed, delimed with formic acid, and used for the experiments. Collagen fibres 1—2 cm. long were isolated. They consisted of bundles of fine fibrils 5—8 μ in diameter with blunt bulbous ends, and showed the property of double refraction. A smaller number of other coarser fibres were found, thickness 8—15 μ , which were like corkscrew bands, and closely resembled cotton fibres in appearance, and were strongly refractive. These are possibly the so-called "elastic fibres." Heat gelatinises the collagen fibres mounted in water, the change occurring suddenly with the fibre bundle turning on its own axis, contracting towards the middle, and finally forming a short swollen cylinder, the power of double refraction being lost. The elastic bands are not affected by the heat. Acids and alkalis are both similar to gelatinisation in their effects. During tannage the fibre bundles become much darker in colour as seen through the microscope, and then later the polarised colour of the fibre bundles becomes weaker and is finally lost, and an even dark brown colour remains. With pyrogallol tannins, the first phase of the tannage causes a reversal of the double refraction, but with the progress of the tannage this diminishes and finally fails leaving an even dark brown colour. The physical properties of the elastic fibres are not affected by tannage.

The polarisation microscope can be used to follow the progress of the tannage from liquor to liquor, and to determine whether the leather is sufficiently tanned. Then sections of insufficiently tanned leather, mounted in water or glycerin-gelatin medium, show the brightly illuminated interference colours of the raw hide fibre when examined under the instrument. Complete penetration of the tannin is revealed by a few isolated, intense green, fibre bundles in the positive position with sole leather.

D. WOODROFFE.

Determination of moisture in leather. [American Leather Chemists' Association's Committee Report.] F. P. VEITCH and T. D. JARRELL (J. Amer. Leather Chem. Assoc., 1927, 22, 265—274).—The moisture was determined in samples of chrome- and vegetable-tanned, stuffed and unstuffed leathers respectively, by the official method and the toluene distillation method (B., 1926, 926). It was found that there was a small continued loss in weight with any kind of leather upon repeated drying by the official method. The results by the latter method were affected by the relative humidity of the air unless a current of dry air was passed through the drying bottle during the drying. Variations in the temperature of the drying ovens caused variations in the results. The highest moisture figures were obtained with the vacuum oven at 105°. The results obtained by the toluene distillation method and the official method did not agree well, difference in types of ovens, in temperature, and humidity within the ovens causing large differences between the results of different analysts. Greater concordance was evidenced by the toluene distillation method than by the official method.

D. WOODROFFE.

Action of trypsin on unlimed calfskin. H. B. MERRILL and J. W. FLEMING (J. Amer. Leather Chem. Assoc., 1927, 22, 274—278).—Samples of limed and unlimed calfskin from the same pelt were prepared, treated with a 0.01% solution of commercial trypsin in a buffer solution of p_H 8.0 at 40°, and the nitrogen content was determined after 24 hr. periods up to 8 days. Both the limed and unlimed skin were hydrolysed more rapidly during the first two or three days, the rate of hydrolysis gradually falling off to a constant value. Limed calfskin contained a greater amount of material easily digested by trypsin, but after its removal the rate of hydrolysis of the collagen was the same as for the unlimed skin. The rate of digestion of collagen by trypsin is independent of whether or not it has previously been limed.

D. WOODROFFE.

Badan. R. W. FREY and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1927, 22, 243—244).—A sample of badan root (*Saxifraga crassifolia*) obtained from Russia analysed as follows, on a moisture-free basis:—Tans, 26.8%; non-tannins, 23.7%; insolubles, 2.4%; sugars before hydrolysis, 6.6%; after hydrolysis, 13.8%. The presence of a mixture of pyrogallol- and pyrocatechol-tannins was indicated. A cow-grain skiver tanned in the aqueous extract had a fawn colour.

D. WOODROFFE.

Insoluble constituents of myrobalan extract. P. CHAMBARD (Le Cuir Technique, 1926, 18, 372—373; Chem. Zentr., 1926, II., 3132).—Insoluble residues

obtained by centrifuging the extracts contain microscopic crystals soluble in alcohol and in alkalis, but precipitated from solution in the latter by acidification. The crystals, which in neutral solution give a black coloration with iron salts and a precipitate with gelatin, are probably a crystalline form of the active tanning agent in the extract, resembling chebulinic acid.

L. A. COLES.

XVI.—AGRICULTURE.

Plant analysis as a means for determining the content of nutrients in soils. A. JACOB (*Ernährung d. Pflanze*, 1926, 22, 300—302; *Chem. Zentr.*, 1927, I., 792).—A discussion of the method proposed by Hoffer and Trost (*Purdue Univ. Agric. Exp. Stat., Bull.* 298) for detecting a shortage of potassium in soil by determining the extent to which accumulation of iron has taken place in the tissues of the stem of maize plants grown on the soil in question. The relative iron content is indicated by the depth of colour obtained on placing a few drops of a 10% aqueous solution of potassium sulphocyanide and dilute hydrochloric acid on the cut surface of the stem. If the majority of the plants show a high content of iron, the soil lacks potassium. Application of lime to the soil increases the iron content of the plants; kainit diminishes it, even if lime also is given.

C. T. GIMINGHAM.

Manuring trials with potassium nitrate. F. FROWEIN (*Chem.-Ztg.*, 1927, 51, 341—343).—Field trials with potatoes indicated that potassium nitrate was as efficient as a mixture of sodium nitrate and potassium sulphate, both as regards total yield and starch content of tubers. Slightly greater yields of leaves of tobacco resulted from the use of potassium nitrate in comparison with the mixed fertiliser. Inferior yields were obtained with oats when potassium nitrate was used. Laboratory experiments with the Neubauer method showed that the intake of phosphorus by the plants was greater when potassium nitrate was applied than when mixtures of potash salts and the usual nitrogenous fertilisers were used.

A. G. POLLARD.

Analysis of calcium nitrate. J. M. McCANDLESS and J. Q. BURTON (*J. Assoc. Off. Agric. Chem.*, 1927, 10, 216—219).—The analysis of synthetic calcium nitrate presents difficulty owing to the extreme hygroscopicity of the salt. It is recommended that lumps should be crushed and, without sieving, placed in stoppered bottle. 15 g. weighed rapidly in tightly-covered dish are at once brought into solution and diluted to 1 litre, of which 25 c.c. are treated with 0.4 g. of sodium carbonate, and evaporated to dryness to remove traces of ammonium nitrate, the residue being then treated with salicylic-sulphuric acid mixture and analysed by the Kjeldahl-Gunning method. Analysis of the salt by the reduced iron method gave low results, probably owing to evolution of nitric oxide with the hydrogen. The determination of moisture presents difficulty, since drying at 130° removes part of the water of crystallisation. Distillation with toluene (cf. Bidwell and Sterling; *B.*, 1925, 268) and with xylene gave inconsistent results. The following method is recommended: Mix 10 c.c. of calcium nitrate solution (10 g. in 250 c.c. of water) with 10 c.c. of sodium carbonate solution (250 c.c. contain sufficient

sodium carbonate to combine with all the calcium nitrate, factor 0.646), evaporate the mixture to dryness, dry at 130° for 3 hrs., cool, and weigh. The difference between the combined weights of salts taken and the weight of the dried residue is the weight of water in the calcium nitrate, allowance being made for the ammonium nitrate driven off.

H. J. DOWDEN.

Suggested modification of the official method for potash [determination] in mixed fertilisers. L. D. HAIGH (*J. Assoc. Off. Agric. Chem.*, 1927, 10, 220—222).—The presence of phosphoric acid and soluble phosphates in fertilisers leads to a low assay in the determination of potash by the official method. Removal of phosphates by magnesium chloride may give a high assay (cf. Bible; *B.*, 1925, 606). They can, however, be satisfactorily removed by heating the solution with calcium carbonate. Heat at the b.p. for 2—3 min. 2.5 g. of the fertiliser, 2 g. of calcium carbonate, and 75 c.c. of water. Evaporate down to 25 c.c., filter, wash, and to the filtrate add 1—2 c.c. of strong ammonia solution and sufficient ammonium oxalate solution to precipitate all the calcium. Cool, dilute to 250 c.c., and, after filtering, determine potash in the filtrate by the usual Lindo-Gladding method. H. J. DOWDEN.

Determination of the replaceable bases of soils, in either presence or absence of alkali salts. P. S. BURGESS and J. F. BREAZEALE (*Arizona Agric. Exp. Sta. Tech. Bull.*, 1926, 9, 187—203).—The methods are applicable to acid, neutral, or calcareous soils. 0.1*N*-Barium chloride solution is employed as the replacing agent, the barium being removed as chromate in the determination of calcium and magnesium, or as sulphate in that of sodium and potassium. A proposed method for the determination of replaceable bases in black alkali soils involves the determination of calcium only.

CHEMICAL ABSTRACTS.

Reaction between calcium sulphate and sodium carbonate, and its relation to the reclamation of black alkali lands. J. F. BREAZEALE and P. S. BURGESS (*Arizona Agric. Exp. Sta. Tech. Bull.*, 1926, 6, 125—129).—Soils containing black alkali contain no free carbon dioxide. When gypsum is applied as a corrective, the amount of black alkali cannot be reduced below about 25 pts. per million; the soluble salts should then be leached out. Application of organic matter and aeration permit the aerobic soil bacteria to produce carbon dioxide necessary to convert the small remaining quantity of sodium carbonate into the innocuous sodium hydrogen carbonate.

CHEMICAL ABSTRACTS.

XVII.—SUGARS; STARCHES; GUMS.

Electrical conductivity of solutions of granulated sugars. [Determination of ash electrically.] A. R. NEES (*Ind. Eng. Chem.*, 1927, 19, 225—226).—The apparatus used consisted of a 1 : 1 transformer (utilising current from a 100-volt, 60-cycle lighting circuit), an alternating-current galvanometer, a dial-type Wheatstone bridge, and a conductivity cell, it being possible for one operator with assistant to make 150 conductivity determinations in 8 hrs. Determinations were carried out at 25°, using solutions containing 25 g. of sugar per 100 c.c.,

the results being expressed in terms of the sp. conductance (as multiples of 10^{-5}), this divided by the factor 231.5 in the case of beet sugars produced in Colorado, Nebraska, and Montana giving the % of ash. Variation of the conductance with the concentration of the sugar solution is very slight, being near the maximum at a concentration of 25 g. per 100 c.c. J. P. OGILVIE.

Use of the isoelectric point as guide to the neutralisation of converter liquor in starch-glucose manufacture. H. S. PAINE and M. S. BADOLLET (Facts about Sugar, 1926, 21, No. 51).—Ultramicroscopic cataphoresis measurements were made of a number of samples of "converter liquors," and it was found that, on addition of sodium carbonate for neutralisation, maximum flocculation occurred in all cases at a definite p_H value (the isoelectric point), but that this value varied with different samples, and was apparently dependent on a number of factors, such as character of the raw material, conditions of hydrolysis, etc.

J. P. OGILVIE.

Determination of reducing sugars volumetrically. M. VAN DE KREKE (Archief Suikerind. Nederl.-Ind., 1926, 411—419; Int. Sugar J., 1927, 29, 110).—Schoorl's iodine method is modified for application to cane molasses as follows: 50 c.c. of Fehling's solution are mixed with 50 c.c. of the solution of the sample, heated to b.p. in 3 min., and ebullition is continued for 2 min. longer. After cooling, 25 c.c. of 20% potassium iodide solution and 35 c.c. of sulphuric acid (1 : 5 pts.) are added, the iodine thus liberated being titrated with 0.1*N*-sodium thiosulphate, using starch indicator. A "blank," using water in place of sugar solution, is carried out, the figure found being deducted from that actually obtained.

J. F. OGILVIE.

Bone black [animal charcoal]. H. I. KNOWLES (Ind. Eng. Chem., 1927, 19, 222—225).—Shank and knuckle bones were carbonised, and the following percentage results found for the chars respectively: carbon, 7.26, 16.37; insoluble in hydrochloric acid, 0.12, 0.96; calcium carbonate, 9.49, 7.48; apparent sp. gr., 0.662, 0.502; colour removed (from a solution containing 1000 colour units), 96.9, 98.4; ash removed, 64.1, 60.0%. Carbon content is not always a criterion of char value, there being a difference in the quality of the carbon. There appears to be an optimum carbon content, depending on the available bone surface area, and for shank char this seems to be about 10%. Ash removal by char is selective, and, like colour removal, is an adsorptive phenomenon, from which it follows that tests for measuring colour and ash must take into consideration the concentration of these constituents. The Brix and p_H values are also important. A hardness test is described in which the char was subjected to the impact and abrasion of steel balls in a small cast-iron ball mill, the increase in "fines" passing both 24- and 50-mesh being measured.

J. P. OGILVIE.

Decomposition of the invert sugar by lime [in the defecation of beet raw juices]. V. ČTYROKÝ (Z. Zuckerind. Czechoslov., 1927, 51, 230—236).—In the decomposition of invert sugar by lime, the rate of decomposition depends firstly on the temperature, and secondly on the amount of lime. Under the conditions of prac-

tice almost the total invert sugar is destroyed during the time of contact of the juice with the milk of lime in defecation and saturation. This time practically suffices for the complete decomposition of amounts of invert sugar as high as 1%.

J. P. OGILVIE.

XIX.—FOODS.

Rate of drying of wheat flour, starch, and gluten.

E. A. FISHER (Cereal Chem., 1927, 4, 184—206).—The rates of drying of different varieties of flour, starch, and crude powdered gluten were studied by exposing them over sulphuric acid at a constant temperature in an apparatus which allowed of their being weighed at intervals without removing them from the drying vessel. The curves obtained by plotting the rates of drying against moisture content are all of the same type. The rate of drying is a linear function of the moisture content down to about 6% of the latter. Between 6% and 2½% the curve is again linear, but different in direction, and below 2½% the rate of drying becomes extremely slow, the curve bending round to the origin. There is some evidence to suggest that the intercepts on the moisture axis cut off by the extension of the two straight-line portions of the curve are characteristic of each flour and connected with the amount of gluten present. The rate of drying is probably controlled largely by convection currents set up by differences of temperature between the drying material, the acid, and the parts of the apparatus in contact with the outside air.

F. R. ENNOS.

Use of Ostwald viscosimeters for flour suspensions. H. J. DENHAM, G. W. S. BLAIR, and G. WATTS (Cereal Chem., 1927, 4, 206—220).—The Ostwald type of viscosimeter has been found satisfactory for measurements on flour suspensions of concentrations up to 30%, provided the instrument is calibrated with solutions of known viscosity and the measurements are expressed in absolute units. Varying the dimensions of the capillary within wide limits does not affect the accuracy of the results, provided the flow is laminar and the critical velocity is not exceeded. Viscosity "fall-back," or the tendency of the viscosity of flour suspensions to decrease on keeping, is shown to be a colloidal phenomenon, and is probably due to syneresis occurring in the suspensoid. From a study of the change of viscosity of flour suspensions with temperature, an empirical equation relating temperature coefficients with concentration has been put forward.

F. R. ENNOS.

Vitamin-C content of fresh and canned pear.

V. C. CRAVEN and M. M. KRAMER (J. Agric. Res., 1927, 34, 385—392).—The minimum dose of raw pear required to protect guinea-pigs from scurvy is 10—15 g. per day. The antiscorbutic factor in pear is destroyed by the "open-kettle" method of home canning, involving boiling the fruit in water for 15 min., and is partly destroyed by the "cold-pack" method, in which the jars containing the fruit are kept in boiling water for 20 min. Storage of fresh pears seemed to make no difference in the content of vitamin-C.

C. T. GIMINGHAM.

Corrosion of tin plate. MANTELL and KING.—See X.