# BRITISH CHEMICAL ABSTRACTS

# **B.**—APPLIED CHEMISTRY

SEPTEMBER 9, 1927.

# I.—GENERAL; PLANT; MACHINERY.

Ball and tube flowmeter suitable for pressure circuits. J. H. AWBERY and E. GRIFFITHS (Proc. Physical Soc., 1927, 39, 296—299).—A robust form of the Ewing ball-and-tube flowmeter employing an inclined tube and suitable for the metering of fluids under pressure, *e.g.*, ammonia in a refrigerating plant, is described. The behaviour of the instrument under conditions of pulsating flow is examined.

J. S. G. THOMAS.

Carbon crucible for very high temperatures. M. K. HOFFMANN (Z. Elektrochem., 1927, 33, 200-202). —A carbon crucible is dried at 150° and then soaked for 1 hr. at ordinary temperature in a 1.5% sol of silica or vanadium pentoxide. This process is repeated several times. On heating at high temperatures the corresponding carbide is produced and the durability of the crucible is thereby greatly increased. The outside of the crucible may be painted with "Thermonit," a preparation of alumina. The process is compared with that of Ruff and Konschak (A., 1927, 102). H. J. T. ELLINGHAM.

Contamination of samples ground in iron mortars. O. HACKL (Chem.-Ztg., 1927, 51, 442).— After grinding a sample of pure borax glass in an iron mortar it contained 0.79% Fe; the same sample after treatment with a magnet still contained 0.11% Fe. To avoid contamination of samples in this way coarse crushing in a stone rock-breaker, followed by fine crushing by pounding in a "diamond" steel mortar, is recommended. Very fine grinding should be done in an agate mortar. A. R. POWELL.

#### PATENTS.

Separation of liquids and solids of low melting point by centrifuging. BERGEDORFER EISENWERK A.-G. (G.P. 437,482, 4.9.23. Conv., 4.9.22).—The solid of low m.p. is heated and separated in a viscous or liquid state without interrupting the operation of the centrifuge. The heating is carried out conveniently by the addition of a quantity of material of the same composition as the solid to be separated, which has been heated above its m.p. A. B. MANNING.

Condensation of vapours and extraction of non-condensable gases. R. MERCIER (E.P. 265,199, 26.1.27. Conv., 26.1.26).—The gas and a liquid are passed together through a centrifugal rotor which is provided with two kinds of liquid jets; one set of small area and/or larger radius produces a high velocity to transport the gas, and the other jets have larger area and lower velocity to produce a larger volume of spray to scrub the gas. B. M. VENABLES.

Apparatus for cooling by the expansion of gases. V. CREMIEU (U.S.P. 1,625,712, 19.4.27. Appl., 9.1.26. Conv., 12.5.25).—The gas is expanded through narrow passages formed through and along a tapered seat on the end of the supply pipe. The first tapered seat is surrounded by another attached to a tube surrounding the supply tube, and having slight freedom of movement. Outwardly again are another fixed tube and a final movable tube. B. M. VENABLES.

Apparatus for condensing vapours. E. W. Isom and J. E. BELL, Assees. to SINCLAIR REFINING CO. (U.S.P. 1,627,544, 3.5.27. Appl., 23.6.21).—A number of condensing tanks arranged in series are each arranged to contain both vapour and liquid condensate, and are each provided with a cooling coil. In any tank, vapour taken from the one before (or from the supply) is sprayed into the mass of already-formed condensates. The several condensates are drawn off separately.

B. M. VENABLES.

Sub-cooling condenser. R. J. PABODIE, Assr. to GRISCOM-RUSSELL CO. (U.S.P. 1,625,821, 26.4.27. Appl., 5.5.21).—The vapour and condensate pass through tubes arranged in a number of sections, the total crosssectional area of the tubes being constant in each section, but varying section by section inversely as the density of the vapour-liquid medium.

#### B. M. VENABLES.

Machinery for separating particles from air by centrifugal action [cyclone separators]. T. ROBIN-SON & SON, LTD., C. J. ROBINSON, and W. N. ROBINSON (E.P. 267,580, 19.11.25).-Two cyclone separators are operated in tandem with a fan between them, so that the first operates under partial vacuum and the second under positive pressure. They are each of cylindroconical construction and of the same diameter, but the cylindrical part of the second is deeper. The central outlet pipes are carried down inside to below the cylindrical part, and the lower end of these pipes is perforated (in addition to the open end) preferably by cutting and raising portions so as to act as collecting scoops for the air rotating in the central part of the B. M. VENABLES. vortex.

Apparatus for cleansing air and gases. E. C. R. MARKS. From DE VILBISS Co. (E.P. 268,163, 3.5.26).— An arrangement of discs and baffles within a casing to effect, *e.g.*, the removal of oil from compressed air. B. M. VENABLES.

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

**Reactivity of coke.** S. W. PARR and W. D. STALEY (Ind. Eng. Chem., 1927, 19, 820—822).—The "ignition temperature" of coke, *i.e.*, the temperature at which it commences to react with oxygen, rises with the coking temperature. Cokes from an Illinois coal carbonised at temperatures ranging from 500° to 1000° showed a progressive rise in ignition temperature from 144° to above 600°. This variation cannot be explained by graphitisation. Amorphous carbon prepared from sugar by the action of sulphuric acid shows a similar progressive fall in reactivity after being heated at successively higher temperatures. It is concluded that for each advance in temperature carbon assumes a definite form which directly affects its reactivity. A. B. MANNING.

Uniform heating of coke ovens. H. KUHN (Fuel, 1927, 6, 267-277, 318-331).-With the increased height of modern coke ovens the older systems of heating lead to excessive vertical temperature gradients in the oven. The longer time then required to complete the coking of the upper part of the charge, and the consequent overheating of the lower part, have serious disadvantages, e.g., increased heat consumption, nonuniformity of the coke, lower yield of by-products, etc. The advantages and disadvantages of modern heating systems are summarised. Those based on single-flame heating are unsatisfactory. Multiflame systems, in which the air is admitted in several stages from the sole flue upwards, e.g., that used in the Still oven, solve the problem of uniform heating without introducing any new defects. The coking period is about 20% less, the heat consumption about 30% less, and the yields of tar, ammonia, and benzene correspondingly higher, than in a similar single-flame heated oven. The system permits of easy regulation; it has been applied with success to ovens 4 m. high, and a further increase of height to 5 or 6 m. is probable in future designs. A. B. MANNING.

Modern carbonising economics exemplified by results and working costs at Portsmouth Gas Works. T. CARMICHAEL (Gas World, 1927, 86, 604-622).—Detailed results and working costs obtained from tests carried out on different types of gas-making plant at Portsmouth Gas Works are given. The following figures are the minimum costs of gas production per therm for each type of plant, excluding capital and maintenance charges, and allowing credit for the residual products and for the steam produced from the waste-heat boilers with which each type of plant is equipped :-vertical retorts (Durham coal), 1.25d.; horizontal retorts (Durham coal), 1.34d.; blue water-gas, 2.44d.; carburetted water-gas, 3.60d. Economy in working is claimed by screening the coal and using the fines in horizontal retorts and the screened coal in verticals. Information is given regarding the behaviour of the refractory materials used in the construction of the vertical retorts. H. D. GREENWOOD.

Ovens as a gasworks carbonising plant. G. M. GILL (Gas World, 1927, 86, 633-646).—A survey of the different types of carbonising ovens which might be

adapted for use in gas works, typical results and comparative economics of each type being discussed. It is suggested that in the case of small works a horizontal oven with a coal capacity of 1—2 tons/12 hrs. might replace horizontals, whilst a slightly larger oven unit carbonising 4—6 tons/12 hrs. might be adapted for use in works of medium size. In the case of larger works a choice is available between horizontal, inclined, or vertical intermittent ovens, the latter two types being favoured on the Continent. It is considered that the coke from these ovens is very suitable for central heating, and the rapid development of the coke boiler should remove one of the main objections to the use of large carbonising units. H. D. GREENWOOD.

Cleaning of blast-furnaces gases. Kling-Weidlein gas cleaner. G. W. HEWSON (Fuel Econ., 1927, 2, 251-253).—Some details and working results are given for two Kling-Weidlein gas-cleaning plants, each capable of dealing with the output of gas from one blast furnace (cf. B., 1924, 209). In one 5 hr. test the dust content of the gas was reduced from  $1 \cdot 03$  to  $0 \cdot 184$  grain/cub. ft., and the average drop in temperature of the gas on passing through the apparatus was  $50-60^{\circ}$ . The power required to operate the cleaner and cost of cleaning the gas are indicated. A. B. Manning.

Modern design of carbon dioxide recorders and indicators. A. GROUNDS (Fuel Econ., 1927, 2, 233-235).—A brief account is given of three modern types of carbon dioxide recorder, the Cambridge Instrument Co.'s (cf. Engineering, 1925, 119, 53), the "Ranarex" (cf. Power User, 1922, 302), and the "Union" (cf. B., 1922, 235 A; 1924, 814), to illustrate the improvement in design due to utilising the physical properties of gases in such instruments.

A. B. MANNING.

Coking of oil shales. W. L. FINLEY and A. D. BAUER (U.S. Bur. Mines, Tech. Paper, 1926, No. 398, 11 pp.).-In order to examine the progress of the coking exhibited by some shales, a coking shale was heated in a standard assay retort until 10% of the oil had been produced; the retort was then cooled and opened. In the upper part of the retort the shale was unchanged, the bottom of the retort held dense, hard coke, and in between was a layer of semi-plastic shale. The shale apparently melts below its temperature of decomposition. By mixing a non-coking shale with a coking variety, satisfactory removal of the spent shale from the retort was accomplished. Thus, addition of 40% of a non-coking shale produced a distinctly softer coke than when a coking shale alone was used, whilst a mixture of 20% of coking with 80% of non-coking shale gave a spent shale which stuck together, but which could be easily broken apart. Mixing of a coking shale with a spent shale produced somewhat similar results as regards the resulting spent shale. The effect of steam was examined ; external heat raised the temperature of the shale to 250°, saturated steam was then blown in, and the temperature gradually raised to 555°. A coke was obtained neither so hard nor so dense as when no steam was used. Steam at 650° blown through the shale without application of external heat produced coke just as hard and dense as when external heat alone is

applied. After 100 hrs. roasting at 165-186° practically no oil was obtained on retorting. Preliminary oxidation at 125-135° destroyed the coking properties of a shale, increased both the amount of water produced and the fixed carbon in the spent shale, and decreased the oil yield by nearly 55%. The oil so produced had a higher sp. gr. and a lower setting point than the oil from raw shale, and yielded on distillation a more unsaturated distillate giving a highly carbonaceous residue. Extraction of powdered shales with carbon tetrachloride, pyridine, or xylene gave no indication of the coking properties of the shale. Extraction with pyridine removes some of the coking constituents from the shale, but the amount of extract cannot be determined accurately owing to the interaction of the pyridine with organic matter in the shale. W. N. HOYTE.

Composition of petroleum (kerosene and other) fractions, with the standardisation of miscibility and optical tests. J. A. CARPENTER (J. Inst. Pet. Tech., 1926, 12, 518-567) .- Solubility of aniline in hydrocarbons is greatly affected by the presence of water, and yields useful information as to the composition of hydrocarbon fractions. The research concerns the correlation of this method with optical and other methods; the study of the liquid systems resulting from aniline and o-toluidine with hydrocarbons or water; the separation of hydrocarbons in petroleum fractions; and the study of fractions above 300°. Details are given of the examination of fractions of Burmah crude, American kerosene, and Badarpur crude fractions. The nature of the unstable compounds in kerosene fractions is discussed, and an attempt made to identify them. In determining unsaturateds and aromatics the losses of hydrocarbon on acid treatment are much less accurate than the aniline point determinations. H. MOORE.

Burkheiser ammonium sulphite-bisulphite process. TERRES and HAHN.—See VII.

#### PATENTS.

Dust removal from carbonisation gases containing tar vapours. KOHLENSCHEIDUNGS-GES.M.B.H. (G.P. 439,251, 2.12.21).—The gases pass through a filter which can be heated at a temperature above the dewpoint of the highest-boiling constituent of the tar vapours. Or, by working at a lower temperature, the dust, together with some of the higher-boiling constituents of the tar, is deposited on the filter mass, which consists of gascoke, semi-coke, or other distillation residues, in such a manner that the latter can be briquetted without further addition. A. B. MANNING.

Conversion of heavy into lighter hydrocarbons. DEUTSCHE ERDÖL A.-G. (E.P. 244,107, 3.12.25. Conv., 3.12.24).—Oil is preheated under pressure to the temperature of its conversion into lighter hydrocarbons and then transferred to a reaction chamber, the temperature of which can be controlled independently of the preheaters, in which the reaction is completed. The product is discharged into a low-pressure rectifying column from which the desired light hydrocarbons pass as vapours and are condensed, whilst the heavier fractions are returned to dilute the hydrocarbons to be treated. S. PEXTON.

Separating a mixture of a large number of volatile liquid and gaseous hydrocarbons. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, and J. H. C. DE BREY (E.P. 255,428, 3.6.26. Conv., 14.7.25).-Casing-head gasoline, rich in constituents volatile at ordinary temperatures and pressures, is fed to the middle of the rectifying column of a still which operates under 4 atm. pressure. The boiling and the reflux are regulated so that the top fraction, which usually amounts to 20% of the crude gasoline, has a boiling range of 0° to below 100°. This fraction is discharged in the gaseous condition, and can be used as fuel. The bottom fraction, of boiling range 0-150°, flows under its own pressure to the middle of a fractionating column working under 2 atm. pressure, where it is divided into fractions boiling at 0-30° and 30° to above 150°, respectively. The first fraction is suitable as a fuel for blending with heavy gasoline, the other is run to a fractionating column working at 11 atm. pressure where it is resolved into a stable motor fuel boiling at 30-100° together with a heavy fraction which can be blended with the more highly volatile fractions. S. PEXTON.

Treating hydrocarbons. W. R. HOWARD, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,436, 3,5.27. Appl., 9.6.22. Renewed, 3.3.27).—Oil is vaporised and the vapours are passed through a supply of oil contained in a closed shell within the still. The uncondensed vapours are subjected to reflux condensation, the condensate being returned to and re-treated in the shell. The operation is carried out under pressure.

C. O. HARVEY.

Treatment of hydrocarbons. O. G. MESSENGER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,623,061, 5.4.27. Appl., 6.1.21).—Refinery waste oils are vaporised and passed at low pressure through a cracking tube at about 400° into which chlorine is introduced. Lowboiling chlorohydrocarbons of value as solvents are obtained. T. S. WHEELER.

Condensing, treating, and washing hydrocarbon vapours. C. L. FREELAND (U.S.P. 1,627,431, 3.5.27. Appl., 9.9.21).—The vapours are passed through the hollow passageway formed in a volume of water by spraying it into a tube in such a manner as to impart a whirling spiral movement. C. O. HARVEY.

Treating petroleum and other hydrocarbons. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,159, 3.5.27. Appl., 30.11.17. Renewed 16.7.23).—The oil is cracked under pressure by passage through a series of chambers maintained at progressively increasing temperatures, the oil supply to the chamber of lowest temperature being continuous and the vapours from this chamber being condensed and collected. The condensable vapours from the other chambers are returned thereto by means of reflux condensers, and the remaining vapours are forced back through the pool of oil contained in the chamber of next lower temperature. Uncondensable gases formed are collected and passed through the oil. C. O. HARVEY.

Treating oil shale. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,162-3, 3.5.27. Appl., 10.1.21. Renewed, 4.2.27).—(A) The crushed shale is heated in a vessel consisting of an upper and a lower drum connected by a number of vertical tubes heated externally, and also by means of superheated steam in actual contact with the shale, which is fed to the upper drum and passes downwards through the tubes. The evolved vapours are condensed. (B) Aluminous shale is destructively distilled under pressure in a similar apparatus, and chlorine is passed through the hot shale in countercurrent to its direction of movement.

C. O. HARVEY.

Cracking hydrocarbon oils. CHEMICAL RESEARCH SYNDICATE, LTD., Assees. of J. B. WEAVER (E.P. 253,562, 15.6.26. Conv., 15.6.25).—See U.S.P. 1,601,786; B., 1927, 6.

Cracking of hydrocarbons. E. C. HERTHEL and H. L. PELZER, ASSTS. to SINCLAIR REFINING CO. (U.S.P. 1,634,666, 5.7.27. Appl., 8.4.24).—See E.P. 232,178; B., 1925, 907.

Apparatus for removing [coke] deposits from stills and the like. A. E. WHITE. From TEXAS Co. (E.P. 273,159, 29.11.26).

Process and apparatus for the gasification of solid fuels. L. CHAVANNE (E.P. 247,571, 10.2.26. Conv., 11.2.25).

Coke-extracting mechanism for vertical retorts for the distillation of carbonaceous materials. F. J. West, E. West, and West's Gas Improvement Co., LTD. (E.P. 272,691, 28.6.26).

[Charging device for use in the] low-temperature distillation of fuel. J. PLASSMANN (E.P. 260,569, 11.10.26. Conv., 27.10.25).

Apparatus for carburetting water-gas. [Automatic] apparatus for making water-gas and other cyclical gas-making processes. HUMPHREYS & GLAS-GOW, LTD., Assees. of H. G. TERZIAN (E.P. 266,680—1, 24.12.26. Conv., 1.3.26).

Apparatus for removing and quenching hot coke discharged from retorts or furnaces. H. J. Toogood, and R. DEMPSTER & Sons, LTD. (E.P. 264,013, 13.1.26).

Manufacture of air gas from inflammable volatile liquids. H. FOERSTERLING (E.P. 248,770, 5.3.26. Conv., 5.3.25).

# IV.—DYESTUFFS AND INTERMEDIATES. Patents.

Manufacture of azo dyes containing metal. Soc. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 249,884, 26.3.26. Conv., 28.3.25).—A sulphonated 2-diazo-1-naphthol-8-sulphonic acid is coupled with any second component and the product boiled with a chromium or copper compound. Further, 1-naphthol-8-sulphonic acid is coupled with an o-diazophenol and boiled with a chromium compound, or with an o-diazonaphthol and boiled with a copper compound. From 2-amino-1-naphthol-4:8 (or 3:8)-disulphonic acid, diazotised and coupled with 1-phenyl-3-methyl-5-pyrazolone, the copper compound dyes wool red, the chromium

compound blue-red. The same diazo compound is coupled with 1-naphthol-4:8-disulphonic acid (copper, violet), with  $\alpha$ - or  $\beta$ -naphthol (chromium, navy blue), (with 2:3-hydroxynaphthoic or 2-naphthol-4:5-dicarboxylic acid (chromium, blue), with resorcylic acid (chromium, violet), with 1-naphthol-3:8-disulphonic acid (chromium, blue). 1-Naphthol-4:8(or-3:8)-disulphonic acid is coupled with diazotised 4-nitro-2-aminophenol (chromium, bordeaux), with diazotised 4-chloro-2aminophenol (chromium, red-blue or violet), with diazotised picramic acid (chromium, black). If carboxyl groups are present, the dye is suitable for chrome printing on cotton. C. HOLLINS.

Manufacture of anthraquinone derivatives. BADISCHE ANILIN U. SODA-FABR., Assees. of FARBEN-FABR. VORM. F. BAYER & Co. (E.P. 244,462, 8.12.25. Conv., 19.12.24).-The labile compounds from 4-substituted 1-aminoanthraquinones and formaldehyde condensed as described in G.P. 123,745 (A., 1902, i, 119) are further condensed in acid media (especially 65-85%) sulphuric acid) to give new products of unknown constitution. The condensation is facilitated in certain cases by addition of boric acid. Two series of products, a and b, are formed according as 1 mol. or 2 mols. (or more) of formaldehyde react. Moderated oxidation of the b-compounds gives further new products, from which formaldehyde may be split off, with formation of a-compounds; further oxidation yields the products of E.P. 244,463 (cf. following abstract). To 1-amino-4-hydroxyanthraquinone sulphuric acid, heated at 90° and cooled to 25°, paraformaldehyde is added and the bluish-violet solution of the intermediate product is kept for 12-36 hrs. at 15° or is heated at 70-75° for a short time. The colour changes to green and a product of the b-type separates. With less paraformaldehyde at 40° the aproduct is obtained. A mixture of a- and b-products gives blue-grey shades on cotton from a hyposulphite vat. Similar products result when 1:4-diaminoanthraquinone is used. 4-Amino-1-methoxyanthraquinone gives a bluish-grey to black vat dye. C. HOLLINS.

Manufacture of anthraquinone derivatives. BADISCHE ANILIN U. SODA FABR., Assees. of FARBENFABR. VORM. F. BAYER & CO. (E.P. 244,463, 8.12.25. CONV., 9.12.24).— $\alpha$ -Aminoanthraquinones are condensed with formaldehyde as described in E.P. 244,462 (cf. preceding abstract) to give compounds of series *a* or *b*, which are then oxidised (more energetically than in the earlier patent), forming new quinone-like vat dyes. For the oxidation, manganese dioxide and sulphuric acid are used. From 1-amino-4-hydroxyanthraquinone a greenishblue vat dye is obtained. C. HOLLINS.

Violet vat dye of the 1-thionaphthen-2-indoleindigo series. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,631,865, 7.6.27. Appl., 5.8.26. Conv., 6.11.24).—The process of U.S.P. 1,590,685 (B., 1926, 816) is modified in that 3-alkyl derivatives of the hydroxythionaphthen compounds therein claimed are employed. *E.g.*, 4:6-dichloro-2-hydroxy-3-methylthionaphthen, m.p. 135°, from 4:6-dichloro-m-toluidine through 4:6-dichloro-m-tolylthioglycollic acid, m.p. 106°, using the methods of the patent cited, is condensed with 2:5:7trichloroisatin to give a dye, yielding on cotton and wool in a hyposulphite vat, bluish-violet shades fast to washing, light, and chlorine. T. S. WHEELER.

Manufacture of water-soluble condensation products. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,294, 23.4.26. Conv., 23.4.25).—An aromatic sulphonic acid or a derivative thereof other than a hydroxy-sulphonic acid is condensed with an aromatic hydroxy-sulphonic acid is condensed with an aromatic hydroxy-alcohol, a derivative thereof, an aliphatic, aromatic, or hydroaromatic alcohol, a phenol, or a compound containing a replaceable halogen atom. In place of the aromatic sulphonic acid may be used aldehyde or other condensation products of an aromatic sulphonic acid of high mol. wt., or an aromatic hydrocarbon with subsequent sulphonation. Condensation occurs at moderate temperatures, and, if desired, in the presence of mineral acid.

S. S. WOOLF.

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

Viscose. XI. G. KITA, R. TOMIHISA, K. SAKURADA, Y. NAKAMURA, and H. KONO (J. Cellulose Inst., Tokyo, 1927, 3, 117-125).-Viscose containing 6% of cellulose was spun into a coagulating bath containing 17.5% of ammonium sulphate, 8% of sulphuric acid, and 7.5% of dextrose, the influence of the time of ageing of the alkalicellulose and ripening of the viscose and the alkalinity of the viscose being studied. The viscose from alkalicellulose aged only for a short time spins more easily under a high pressure, whereas that from more seasoned alkali-cellulose spins under a lower pressure. The viscose spins more easily as its ripening advances. The ease of spinning is measured by the speed ; a speed of 57 m./min. is not easily obtainable when the alkali-cellulose has been aged for too short or too long a time, but is readily obtained with viscose from moderately aged alkali-cellulose. With the same viscose, differences in tenacity and elongation of the thread may be produced by differences in spinning speed and pressure owing to variations in the coagulation conditions, *i.e.*, the velocity of the action of the bath on filaments of different thickness. The tenacity and elongation increase with the times of ageing of the alkali-cellulose and ripening of the viscose up to a certain point, beyond which they remain constant or decrease slightly. It would not be right, however, to conclude that the increase in tenacity and elongation is dependent on the decrease in viscosity of the viscose which takes place at the same time.

#### J. F. BRIGGS.

Comparing the cleanliness of sulphite pulps. T. L. CROSSLEY (Pulp and Paper Mag., 1927, 25, 771).— The comparative cleanliness of sulphite pulps is judged from their content of readily-reducible sulphur compounds, as determined by reduction with zinc and hydrochloric acid in a vessel, the outlet of which is covered with a lead acetate paper. 0.25-0.1 g. of the sample is used for the test, and standard stains are made from 0.0001N-sodium thiosulphate. By this method bleached pulps usually show from 0.00008% to 0.1% S. D. J. NORMAN.

#### PATENTS.

Production of artificial fibres from viscose. H. JENTGEN (G.P. 439,359, 17.1.25).—Ammonium salt solutions, used as the first precipitation bath in the twobath process, contain aldehydes, with or without sugars, vegetable extracts, etc. L. A. Coles.

Preparation of spinning baths for viscose. VER-EINIGTE GLANZSTOFF-FABR. A.-G. (G.P. 438,461, 11.5.19). —Viscose threads on leaving the coagulating bath are washed with dilute sulphuric acid whereby salts are removed as readily soluble bisulphates. The wash liquor is then concentrated, if necessary, and further quantities of sulphuric acid are added.

D. J. NORMAN.

Manufacture of fine viscose filaments. VER-EINIGTE GLANZSTOFF-FABR. A.-G. (G.P. 438,460, 24.1.19). —By increasing the concentration of the acid in the coagulating bath the thread becomes progressively finer. D. J. NORMAN.

[Non-inflammable] cellulose composition. W. G. LINDSAY, ASST. to CELLULOID Co. (U.S.P. 1,630,752, 31.5.27. Appl., 25.4.22. Cf. U.S.P. 1,233,374; B., 1917, 1004).—The composition comprises pyroxylin (100 pts.), tricresyl phosphate (75 pts.), and hexachloroethane (125 pts.). T. S. WHEELER.

Cellulose acetate composition [film] of low inflammability. S. J. CARROLL, Assr. to EASTMAN KODAK CO. (U.S.P. 1,631,468, 7.6.27. Appl., 27.4.25).— Cellulose acetate film composition is mixed with pure s-tribromophenol or similar compound.

T. S. WHEELER.

Production of weighted artificial silk. G. RIVAT, Assr. to LYONS PIECE DYE WORKS (U.S.P. 1,631,062, 31.5.27. Appl., 15.10.25).—Cellulose acetate silk can be weighted in the same manner as natural silk, if, before treatment with stannic chloride and sodium phosphate solution, it is gelled by soaking in cold 15—20% aqueous formic or acetic acid. T. S. WHEELER.

Manufacture of hollow [artificial silk] fibres. W. O. SNELLING (U.S.P. 1,631,071, 31.5.27. Appl., 11.5.25).—Molten sugar is forced through a jet to form a fibre, which is coated with a solution of a cellulose ester, e.g., nitrocellulose or viscose, the soluble core being removed by treatment with water after coagulation and the hollow fibre treated in any usual way, e.g., denitrated. T. S. WHEELER.

Manufacture of paper pulp. J. B. BEVERIDGE (U.S.P. 1,631,789, 7.6.27. Appl., 13.4.25).—Cellulosic material is digested with an aqueous solution containing sodium hydroxide, sodium sulphide, and the waste liquor from a pulping process in which a solution of sodium acid sulphite has been used. D. J. NORMAN.

Manufacture of a paper product. J. McINTOSH, Assr. to DIAMOND STATE FIBRE CO. (U.S.P. 1,631,750, 7.6.27. Appl., 2.3.26).—A cellulose ester is added to the pulp before it reaches the Fourdrinier machine. The resulting paper is parchmentised, and is then subjected to heat and pressure to cause the cellulose ester to flow and permeate the sheet. D. J. NORMAN.

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

Burkheiser ammonium sulphite-bisulphite process. E. TERRES and E. HAHN (Gas- u. Wasserfach, 1927, 70, 309-312, 339-343, 363-367, 389-395).-- The ternary system ammonia-sulphur dioxide-water has been studied from the point of view of the practicability of the Burkheiser process. Solubility curves, showing the variation of the composition of the solution with varying excess of ammonia or of sulphur dioxide, have been plotted for 0°, 20°, 40°, and 60°. They exhibit a number of discontinuities which do not correspond with any definite change in the solid phase; the only solid phases in equilibrium with the solution are NH<sub>4</sub>HSO<sub>3</sub> and (NH<sub>4</sub>)<sub>2</sub>SO<sub>3</sub>, H<sub>2</sub>O, in the region of excess sulphur dioxide, and (NH4)2SO3,H2O, in the region of excess ammonia. The solubility of ammonium sulphite in water rises regularly from 32.9% at 0° to 50.0% at 60°, and that of the bisulphite from 72.8% at 0° to 86.1% at 60°. The partial pressures of ammonia and of sulphur dioxide over solutions of ammonium sulphite are almost linear functions of the concentration; the values for ammonia are much higher than the corresponding values for sulphur dioxide, but both are sufficiently small, even at 40° and over concentrated solutions, to permit a practically quantitative removal of either constituent from a gas by washing. The partial pressures at 20°, 40°, and 60° have also been determined over saturated solutions in the presence of excess of ammonia and of sulphur dioxide. The results definitely demonstrate the possibility of the successful operation of the Burkheiser process, and indicate the most favourable conditions under which it should be carried out.

A. B. MANNING.

Commercial sodamide. A. GUNTZ and F. BENOIT (Bull. Soc. chim., 1927, [iv], 41, 434—438).—Commercial sodamide of dark colour contains metallic iron and up to 16% of sodium hydride; the presence of the latter substance is probably the cause of the accidents sometimes attending the use of sodamide. The accion of hydrogen on sodamide and of ammonia on sodium hydride is investigated. In order to prepare pure sodamide the sodium must be heated in a current of ammonia in the region of 300° until it is completely transformed and the greenish liquid decolorised; the melt is then poured on to a cold surface.

# S. K. TWEEDY.

Calcium sulphate. II. Supersaturated solutions of calcium sulphate, pure, and mixed with other salts. L. CHASSEVENT (Ann. Chim., 1926, [x], 6, 313-351; cf. A., 1926, 1217).-The effects of changes of temperature and of the addition of varying quantities of the di- and hemi-hydrates on the velocity of crystallisation of saturated solutions of the hemihydrate (prepared from calcium sulphate dehydrated below 300°), which are, therefore, supersaturated with regard to gypsum, have been studied by means of conductivity measurements. Even when special precautions are taken to exclude seeds of the dihydrate, the solutions of the hemihydrate crystallise spontaneously after about 26 min. at 16.5°, the period before crystallisation sets in being longer the higher the temperature. Addition of the dihydrate induces crystallisation at all temperatures, the velocity being greater the larger the proportion of dihydrate added. The velocity of crystallisation of solutions becomes very small above 60°, even in the presence of a large quantity of the dihydrate.

The effect of a rise in temperature on solutions saturated with respect to the hemihydrate is to cause immediate crystallisation of the dihydrate. The addition either of anhydrous calcium sulphate (dehydrated at 430°, 590°, 900°, or 1000°), or of the hemihydrate, has no effect in the velocity of crystallisation. The influence of salts, bases, and acids on the velocity of hydration of calcium sulphate has been studied calorimetrically. In general, substances which increase the concentration of the sulphate ions in the solution, either by the direct addition of soluble sulphates or by double decomposition with the calcium sulphate, decrease both the period preceding crystallisation and the duration of the hydration. Thus the addition of potassium chloride increases the velocity of hydration, the increase being proportionately greater for small concentrations of potassium chloride than for larger, and at concentrations of less than  $18 \cdot 2$  g./litre the total amount of heat evolved is the same, and the separated crystals consist entirely of gypsum, but at higher concentrations the total heat evolved is greater, and the separated crystals have composition approximately corresponding with a 2KCl, CaSO<sub>4</sub>, 0.5H<sub>2</sub>O. A similar effect is obtained by adding potassium sulphate, only gypsum separating up to a concentration of 24 g./litre. Aluminium sulphate also increases the velocity, only gypsum separating up to a concentration of 160 g./litre, but its action is much greater on solutions of commercial calcium sulphate containing 3% of lime and calcium carbonate, this being due to the action of the lime on the aluminium sulphate, since the addition of a saturated solution of lime to a solution of pure calcium sulphate decreases the velocity of crystallisation. Alum also increases the velocity of crystallisation. Thus the crystallisation of the gypsum is probably induced by a secondary crystallisation, in the solution, of a compound of the hemihydrate or the gypsum with other salts present (either added or as impurities in a commercial sample). Addition of sodium hydroxide to a solution of pure calcium sulphate in the absence of carbon dioxide decreases the velocity of crystallisation, the decrease being greater the larger the concentration of the base, but, conversely, with a commercial sample of calcium sulphate in air, the velocity of crystallisation is increased by the addition of sodium hydroxide. Sulphuric acid increases the velocity of crystallisation up to a maximum at about 4-6N, greater concentrations of acid then causing a decrease; a similar effect is observed with hydrochloric acid. Rohland's view (A., 1904, ii, 33) that an increase in the solubility of the gypsum increases the velocity of setting of the plaster cannot be upheld, because the solubility of gypsum is increased in the presence of potassium chloride but decreased in the presence of potassium and aluminium sulphates, whilst all these substances increase the velocity of hydration of the J. W. BAKER. hemihydrate.

Dehydration of gypsum. R. STUMPER (Z. anorg. Chem., 1927, 162, 127—140).—The heating curve of gypsum shows that the temperature of transition from dihydrate to hemihydrate rises with increase in the rate at which the temperature of the mass is raised. The change from the hemihydrate to the anhydrous salt takes place in all cases at about 190°. Following the dehydration gravimetrically yields similar results. Unless the rate at which the temperature is raised is 3-25°/min., the first stage in the dehydration may not be observed. R. CUTHILL.

Production of magnesium sulphate from Manchurian magnesite. R. MATSUURA (J. Soc. Chem. Ind. Japan, 1927, 30, 190-194).-Magnesite is converted into magnesia by light burning, then into magnesium sulphite by the action of sulphur dioxide, and finally into sulphate by means of oxygen in the presence of cobaltous sulphate. Manchurian magnesite used has the following composition: 44.65-47.95% MgO, 0.43-4.13% CaO, 0.09-0.19% Al<sub>2</sub>O<sub>3</sub>, 0.33-1.14% Fe<sub>2</sub>O<sub>3</sub>, 0.28-5.50% of matter insoluble in hydrochloric acid, and 49.04-51.60% burning loss. The calcined magnesite yielded magnesia of 91.32% purity. 48 kg. of the product are charged with 270 litres of water into a wooden barrel having a conical bottom. After closing the barrel and converting the oxide into sulphite by passing in sulphur dioxide, 50 g. of cobalt sulphate and 300 litres of water are added, together with sufficient magnesia (about 1.6 kg.) to neutralise the solution, and compressed air is introduced to oxidise the sulphite. After filtration, the solution is evaporated, yielding about 200 kg. of magnesium sulphate crystals. A semi-commercial experiment was successfully carried out with a unit producing 1 ton of pure sulphate crystals from 225 kg. of oxide. K. KASHIMA.

# PATENT.

**Production of sodium fluoride.** E. P. STEVENSON, Assr. to A. D. LITTLE, INC. (U.S.P. 1,634,122, 28.6.27. Appl., 8.10.21. Renewed 1.12.26).—Sodium fluosilicate is decomposed into sodium fluoride and silicic acid by treatment with a carbonate evolving carbon dioxide in the reaction. The silicic acid is dissolved in alkali, separated from the sodium fluoride, and then treated with carbon dioxide to decompose the silicate and form a bicarbonate suitable for conversion into carbonate for use again in the process. W. G. CAREY.

Purification of alkali-metal cyanide solutions containing sulphides. I. G. FARBENIND. A.-G. (E.P. 262,456, 3.12.26. Conv., 4.12.25).—The solution is treated with bismuth hydroxide or a bismuth salt whereby bismuth sulphide is precipitated without any secondary reactions. W. G. CAREY.

Manufacture of stable peroxidised composition. J. A. L. VAN DER LANDE, ASST. to NOVADEL PROCESS CORP. (U.S.P. 1,631,903, 7.6.27. Appl., 3.5.26. Conv., 18.10.24. Cf. E.P. 234,163; B., 1925, 613).—Hygroscopic flour improvers, *e.g.*, ammonium persulphate, are mixed with non-poisonous, anhydrous salts, *e.g.*, calcium sulphate, which can form stable crystals containing water of crystallisation. T. S. WHEELER.

Production of copper oxychloride. MOUNT LYELL MINING & RAILWAY Co., LTD., Assees. of E. W. TREND (Austral. P. 22,518, 1.4.25).—Copper oxychloride precipitated by treating cupric chloride solution with potassium or magnesium carbonate has good adhesive properties and covering power, and is of uniform colour.

L. A. COLES.

#### VIII.—GLASS; CERAMICS.

Measurement of the softening temperature of glasses. J. T. LITTLETON, JUN. (J. Amer. Ceram. Soc., 1927, 10, 259-263).-The softening temperature, at which the glass possesses a certain definite viscosity, is that temperature at which a thread  $(22 \cdot 9 \text{ cm. in length})$ and of uniform thickness, 0.65-1.0 mm.) elongates under its own weight at the rate of 1 mm./min. Since both surface tension and sp. gr. affect the force tending to elongate the specimen, such a conception is only approximate. In the experimental determination the thread, which is formed to a needle point and bent at right angles at one end to serve as a pointer, is suspended from the other end in an electric furnace, at 50-100° below the softening point. The temperature is then raised at the rate of 5-10° per min. Measurements of temperature and elongation are taken every 30 sec., and the results reported graphically. From the graph the softening temperature is easily computed. The control data so obtained also serve to indicate differences in the viscosity of glasses of varying composi-A. T. GREEN. tion.

Antimony white enamel. H. MELZER (Keram. Rundsch., 1926, 34, 801–803; Chem. Zentr., 1927, 1, 785).—The mixture for producing the enamel must be easily fusible as compared with the foundation; the enamel must have the correct coefficient of expansion, high tensile strength, and good turbidity, and must contain not more than 8% Sb<sub>2</sub>O<sub>3</sub> and 7% Al<sub>2</sub>O<sub>3</sub>, whilst the remaining constituents must be carefully selected.

L. A. COLES. Cause of the white ring on the edge of glasses melted by coal gas. G. OFFE (Gas- u. Wasserfach, 1927, 70, 435-437).—The ring produced when the edges of glassware are heated to soften in the coal-gas flame is due to the formation of sulphurous acid by combustion of organic sulphur compounds in the gas. This partly oxidises to sulphuric acid, which, by union with the lime of the glass forms calcium sulphate, a molten ring of which adheres to the glass. With glasses containing little lime the ring is nearly or quite absent, whilst with lime glasses the effect increases with increase of flame temperature. The trouble was greatly reduced by provision of a simple device which largely destroyed the sulphur compounds in the gas. A. COUSEN.

Effect of various sodium silicates and other electrolytes on clay slips. S. J. McDowell (J. Amer. Ceram. Soc., 1927, 10, 225-237).-Commercial sodium silicate varies widely in chemical composition, constitution, and physical properties. One variety may have two and a half times as much silica as another, consequently differences in the deflocculating effect of different varieties may be anticipated. Six clays, including china clays and ball clays, were sampled. A portion of each was dried for 24 hrs. at 70° after having been passed through 8-mesh, and made into a slip with twice its weight of water. The slips were well shaken in a ball-mill and passed through 65-mesh, after which they were placed in sealed jars. The sp. gr. of the slips was  $1.25 \pm 0.01$ . Controlled quantities of the different silicates (increments of 0.005% Na2O based on the weight of the clay in the slip) were added, and (a) the

rate of flow through an "efflux" viscosimeter; (b) the  $p_{\rm H}$  value by the method of Randolph and Donnenwirth (B., 1926, 823); and (c) the amount of settling over a period of 16 hrs. determined. The results are reported graphically. In all cases of silicate additions a maximum rate of flow is produced when the slip is acid, and those silicates high in silica have the most pronounced deflocculating effect for a given  $Na_2O$  content. The  $p_{\rm H}$ value at which clay particles attain maximum charge is lower for sodium silicate solutions than for sodium hydroxide or carbonate solutions. In this connexion the influence of colloidal silica is discussed. The use of sodium silicate rich in silica in preference to sodium carbonate seems desirable where soda blisters are a Possible explanations of the mechanism of difficulty. deflocculation are put forward. A. T. GREEN.

Particle size distribution of typical felspars and flints. E. SCHRAMM and E. W. SCRIPTURE, JUN. (J. Amer. Ceram. Soc., 1927, 10, 264—267).—The results of tests on the size of the particles of felspars and flints, ground by the continuous tube mill, the batch mill, and the Hardinge mill with air separator, are presented. The method of testing has been previously reported (B., 1925, 500). The data show the misleading character of screen tests and indicate the advantage of carrying the analysis beyond the range of screens. Whilst the continuous systems with air separation are most efficient in producing material to pass the screens, the batchground product of the same screen test has the smaller average size. A. T. GREEN.

Glazes of low fusibility containing rutile. M. M. FRENCH and C. M. HARDER (J. Amer. Ceram. Soc., 1927, 10, 268—269).—A very fusible lead glaze containing rutile was used as the base, to which oxides of cobalt, copper, manganese, uranium, and iron were added as colorants. When the amount of rutile was increased, felspar was added to check the fluidity. A number of glazes were formed from this base, giving at cone 09—08 very good matts in dull mottled colours. At cone 04—03 the rutile crystals formed in stripes, the colour depending on the oxide used. These glazes give an interesting variety of colours and textures, and will fit a "Monmouth Clay" body modified with 10—20% of flint. A. T. GREEN.

# IX.—BUILDING MATERIALS.

PATENTS.

Wood preservative. K. H. WOLMAN, F. PETERS, and H. PFLUG (U.S.P. 1,622,751, 29.3.27. Appl., 30.1.26. Conv., 2.1.26).—Complex salts of tartaric acid with arsenic and an organic base, *e.g.*, aniline or quinoline, are dissolved in a mineral oil, and used to impregnate wood. T. S. WHEELER.

Wood preservative. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAGET (Danish P. 34,370, 28.11.23).—The phenols and phenol ethers used in the preservative described in E.P. 190,074 (B., 1923, 271 A) may be partly replaced by sulphonic acids or chlorine substitution products. Formaldehyde or paraformaldehyde may also be added to diminish still further any loss of preservative by washing out; this addition, moreover, increases the antiseptic action of the preservative. A typical mixture contains, e.g., sodium fluoride  $67\%_0$ , phenols or phenol ethers 15%, phenol sulphonic acids 10%, and paraformaldehyde 8%. A. B. MANNING.

Impregnating wood. A. DESSEMOND (G.P. 437,131, 29.12.25. Conv., 5.3.25).—After each impregnation the wood is subjected to a partial vacuum at a pressure and for a length of time determined by measurement of the liquid extracted, in order to determine the quantity which finally remains in the wood. The autoclave used for the purpose is connected with a graduated container, in which the liquid removed under reduced pressure from the impregnated wood can be measured, and from which it can then be transferred into the main tank containing the impregnating liquid. The distribution of the liquid remaining in the wood is quite homogeneous. A. B. MANNING.

Manufacture of acid-proof cementing compositions. I. G. FARBENIND. A.-G. (E.P. 267,396, 13.8.26. Conv., 25.3.26. Addn. to E.P. 256,258; B.,1927, 333).—A silicofluoride is substituted for silica as a cement powder in the prior patent. B. W. CLARKE.

Manufacture of cement. T. RIGBY (U.S.P. 1,628,000, 10.5.27. Appl., 28.11.25. Conv., 28.7.24).—See E.P. 243,410; B., 1926, 129.

Treatment of wet raw materials in the manufacture of cement. J. S. FASTING, ASST. to F. L. SMIDTH & Co. (U.S.P. 1,627,553, 10.5.27. Appl., 9.4.25). —See E.P. 227,977; B., 1925, 232.

Treatment of cement raw materials in rotary kilns. M. VOGEL-JORGENSEN, ASST. to F. L. SMIDTH & Co. (U.S.P. 1,627,585, 10.5.27. Appl., 25.4.25. Conv., 10.5.24).—See E.P. 255,569; B., 1926, 825.

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

Theory of overheating phenomena in grey cast iron. H. HANEMANN (Stahl u. Eisen, 1927, 47, 693-695).—On the assumption that the solidification of grey cast-iron takes place according to the equilibria of the iron-graphite system, and that no change takes place in the amount of combined carbon present during further cooling to the ordinary temperature, it is shown that there must be a stable saturation limit for the combined carbon dependent on the total carbon content of the metal; this limit is depressed by a rise in the silicon content. If the amount of combined carbon exceeds this limit the solidification takes place partly according to the iron-iron carbide equilibria, and if it is below the limit solidification ensues wholly according to the graphite-iron system. In the latter case true equilibrium is obtained only if the metal is so far overheated that the graphite is entirely dissolved in the molten iron, and if subsequent cooling is sufficiently slow to allow the separation of completely saturated mixed crystals to take place. The higher the temperature to which the metal is heated the finer is the graphite that is precipitated during cooling at normal rates. Hence there are two opposed reactions taking place during overheating, one accompanied by a decrease in the amount of combined carbon owing to decomposition of the solid solution induced by the fine dispersion of graphite, and the other accompanied by an increase in

the combined carbon by reason of the closer approach of the system to equilibrium. This accounts for the increase followed by a decrease in the combined carbon during overheating of grey cast iron without assuming any change in the molecular condition of the liquid metal, as suggested by Pivovarsky (B., 1925, 805). A. R. POWELL.

Influence of the blast-furnace conditions on the total carbon content of pig iron. A. MICHEL (Stahl u. Eisen, 1927, 47, 696—698).—Tests on a number of blast furnaces smelting different classes of iron ores have shown that the carbon content of the pig iron produced with any given charge is dependent solely on the temperature in the furnace, being smaller the lower the temperature. Hence a very basic slag requiring a high temperature for melting results in a high-carbon iron. A low-carbon content is also obtained by the use of a higher ratio of ore to coke, *e.g.*, in the so-called "cold" blast-furnace process. A. R. POWELL.

Properties of high-silicon constructional steel. E. H. SCHULZ and H. BUCHHOLTZ (Giessereiztg., 1926, 23, 615-622; Chem. Zentr., 1927, I, 644).—Addition of 1% Si to mild steel for rolling increases its elastic limit considerably, but its effect on the ultimate strength is dependent on the other constituents of the steel and on the degree of rolling. The steel must be annealed at a higher temperature than a similar plain carbon steel owing to the effect of silicon in displacing the upper transformation point. A. R. POWELL.

Importance of cementite. R. G. GUTHRIE (Trans. Amer. Soc. Steel Treat., 1927, 11, 341-354).-Microexamination of a 0.09-0.15% carbon steel billet for wire-drawing showed that the core contained an appreciable amount of pearlite, whilst the rim showed a large amount of cementite spheroids. The rim showed greater hardness, higher tensile strength, less ductility, less resistance to corrosion, and more rapid etching. In low carbon steels not subject to hardening it is desirable to have the cementite in the spheroidal condition, in which they are more stable, take a more uniform electroplated coating, and are more readily weldable. Photomicrographs are given which show that in highcarbon steels, however, the cementite should be in the lamellar form, which is more readily soluble at the hardening temperature, and the steels give a higher degree of hardness and greater homogeneity.

## T. H. BURNHAM.

Corrosion-fatigue of metals as affected by chemical composition, heat treatment, and cold working. J. D. McADAM (Trans. Amer. Soc. Steel Treat., 1927, 11, 355–390).—Stress-cycle endurance curves are given for a series of 26 steels, tested in air, in fresh water, and for interrupted exposure to corrosion. The corrosion-fatigue limit may be less than one third of the endurance limit. Specimens that failed in water showed corrosion-fatigue spots with transverse cracks passing through non-metallic inclusions. Spots were not, however, visible on stainless iron unless tested considerably above the corrosion-fatigue limit. For steels containing up to 0.24% C the endurance limit was greater in the quenched and tempered than in the annealed condition. Above 0.36% C the reverse held good, as

also for nickel steels. Decrease in the cycle frequency decreased the number of cycles to fracture, but the total time of the test increased. For high-chromium steels the corrosion-fatigue limit was higher than for carbon and nickel steels in both fresh and salt water, and rose with increasing chromium content. Heat treatment had little effect. In salt water the limits were considerably lower than for fresh water. The corrosion-fatigue limits of nickel-chromium steels were higher still. High chromium content was more effective than high nickel content in both fresh and salt water. Increase in corrosion resistance caused increase in corrosion-fatigue limit, but though the effects of variation in tensile strength and ductility were insignificant, there appeared to be an undetermined strength factor. There were slight indications that it might be the notch-toughness of the steel. T. H. BURNHAM.

Decomposition of austenite in liquid oxygen. O. E. HARDER and R. L. DOWDELL (Trans. Amer. Soc. Steel Treat., 1927, 11, 391—398).—The scleroscope hardness of six austenitic steels after immersion in liquid oxygen increased as follows :—Cobalt-chromium steel, 80.7%; high carbon-chromium steel, 65.3%; 22% nickel steel, 34.4%; hyper-eutectoid carbon steel, 26.1%; high-speed steel, 5.2%; manganese steel, 0%. A typical martensitic structure was developed except in the case of the nickel steel and manganese steel, which showed little or no change. Decomposition of austenite was accompanied by decrease in sp. gr., which was greatest in those steels which showed the highest increase in hardness. T. H. BURNHAM.

Transformation of retained austenite into martensite by stress. K. HONDA and K. IWASE (Trans. Amer. Soc. Steel Treat., 1927, 11, 399-410).-On hammering steel rods containing 1.4% C and 5% Cr quenched from 1100° in oil, the intensity of magnetisation increased with the strokes. The transformation of austenite into martensite was probably due to the impulsive shock resulting in an atomic agitation. Rockwell and magnetometric hardness determinations on strips of steel containing 0.84% C and 2.96% Cr showed that in thick specimens a greater amount of martensite was produced by water quenching than by oil quenching, but the reverse in thin specimens, confirming Bain's view that internal stress in quenched austenite steels promotes the martensite transformation. Magnetometric measurements demonstrated that the rise of hardness on immersion of the oil-quenched steels in liquid air increases as the diameter of the specimens decreased. T. H. BURNHAM.

Non-corroding steel and iron. A. C. YORKE (Metal Ind., 1926, 29, 419—420; Chem. Zentr., 1927, I, 174).—A short description of the composition, physical and mechanical properties, and methods of making rustless steel and iron is given. The most favourable temperature for rolling is 1050—1100°, for forging, 900— 1150°, and for the subsequent annealing, 750—800°. Forging must be done quickly and, after annealing, cooling must be slow. To remove the oxide film the metal is pickled for 10 min. in 50% commercial hydrochloric acid, then for 25 min. in 20% nitric acid.

A. R. POWELL.

British	Chemical	Abstracts-B.					
680		CL.	XMETALS ;	METALLURGY,	INCLUDING	ELECTRO-METALLUBGY.	

Silicon steel as a constructional material and for castings. K. VON KERPELY (Zentr. Hütten- u. Walzw., 1926, 30, 411—414, 425—427, 437—440, 455— 459; Chem. Zentr., 1927, I, 174).—Silicon steel containing less than 0.04% P has a tensile strength of 51.8 kg./mm.<sup>2</sup>, an elastic limit of 39.8 kg./mm.<sup>2</sup>, an elongation of 26.4%, an impact strength of 8.5 kg./cm.<sup>2</sup>, and a hardness number of 150—155. These properties, combined with its good resistance to repeated impacts and alternating stresses, make the alloy suitable for most types of constructional work in place of the usual steels. The alloy may be welded satisfactorily by the resistance method, but arc welding gives somewhat unsound joints. A. R. POWELL.

Location of the carbon atom in boydenite. H. A. SCHWATZ (Trans. Amer. Soc. Steel Treat., 1927, 11, 277-283).-The author considers that there may be two types of solution of carbon in iron, viz., additive and substitutional, corresponding to austenite and boydenite, the solid solution of the stable system. This is supported by the observation of Osawa that in austenitic steels part of the carbon atoms is substituted for metal atoms. From dilatation data determined in Honda's laboratory the author calculates that the densities of austenite and boydenite at 900° are 7.55 and 7.393 respectively, which difference is of an order consistent with the view expressed. The density of a nickel-iron alloy containing 19.93% Ni and 3.9% of total carbon shows that the solid solution T. H. BURNHAM. in this case is austenite.

Detection of small inclusions in crystallites with the aid of the production of lines of slip. G. TAM-MANN and H. H. MEYER (Z. Metallk., 1927, 19, 85).— If an indentation is made in a crystallite of a polished surface of metal by means of a gramophone needle the slip lines are regularly oriented around the impression if there are no inclusions in the crystallites. In the presence of such impurities the slip lines are curved round the inclusion, *e.g.*, in meteoric iron, or run towards it, *e.g.*, in copper containing particles of oxide.

# A. R. POWELL.

Metals and alloys for thermocouples for the measurement of high temperatures. W. ROHN (Z. Metallk., 1927, 19, 138-144).-A review of the composition, preparation, working range, and properties of commercial thermocouples. For temperatures above 1000° only alloys of the platinum metals are available, and efficient service is obtained only when metals of the highest degree of purity are used. Traces of calcium, silicon, or carbon derived from the materials used in melting may produce serious errors in the readings, whilst contamination with even 0.02% Fe or Ir produces considerable deviation in the calibration during prolonged use. Of the various base metal couples manufactured for use at temperatures up to 1000° those comprising nickel and chromium with relatively little iron give the most constant readings after prolonged use. Protection tubes of 30% chromium-iron alloy are suitable for use with thermocouples in copper and brass foundries as this alloy remains completely unattacked in molten copper and its alloys. A. R. POWELL.

Reduction of mineral oxides. B. BOGITCH (Compt. rend., 1927, 184, 883-885).—The solubility of carbon in mineral oxides containing silicates and silico-aluminates of calcium, and small amounts of copper, nickel, and iron, fused rapidly below 1600°, varies from 0.09 to 0.15%, the fused mineral separating from the reducing coke. Hence the difficulty of obtaining complete reduction of a mineral insufficiently reduced at the moment of fusion. The solubility of the carbon increases with temperature, and calcium carbide is formed. The addition of a base (chalk) increases the rate of reduction when fusion occurs rapidly, and the yield of metal rises to a maximum and falls as the amount of base is increased, owing to a diminution of the fluidity of the medium.

J. GRANT. Gaseous reduction of tungsten and molybdenum oxides. E. W. ENGLE (Trans. Amer. Electrochem. Soc., 1927, 51, 345—349).—The reduction of tungsten trioxide by hydrogen commences at 620° and is rapid at 950°, the metal produced by reduction at the latter temperature being most suitable for sintering and subsequent working. The hydrogen consumption per kg. of tungsten powder varies from 900 to 2800 litres. Molybdenum trioxide commences to reduce at 300° in hydrogen, and reduction can be completed at 625°, but it is preferable to carry the reduction nearly to completion at 850°, cool and powder the product, and finish the reduction at 1100°. A. R. POWELL.

Gaseous nature of carbon reduction of tin concentrates. C. J. FINK and C. L. MANTELL (Trans. Amer. Electrochem. Soc., 1927, 51, 371-381).--Solid carbon does not reduce stannic oxide in an atmosphere of nitrogen below 950°, but, in the presence of a small quantity of air, reduction by graphite commences at about 800° even if the two substances are not mixed together, showing that carbon monoxide is the true reducing agent. A brief description with flow-sheet is given of Bolivian tin-smelting practice.

A. R. POWELL.

Reduction of tin oxide and cassiterite concentrates. E. F. KERN (Trans. Amer. Electrochem. Soc., 1927, 51, 403-412).—Reduction of stannic and stannous oxides by coal gas starts at about 500°, and is rapid at 700°; by bituminous coal it starts about 600°, and is rapid above 750°, and by charcoal it starts at about 750°, and is rapid above 810°. For the reduction of cassiterite, hydrogen is a much more rapid and efficient reducing agent than carbon monoxide. At a given temperature the percentage reduction by hydrogen-nitrogen mixtures increases with an increase in the hydrogen content, but the efficiency of the reduction decreases; mixtures of nitrogen and carbon monoxide behave similarly. A. R. POWELL.

Gaseous reduction of zinc [oxide]. C. J. MAIER and O. C. RALSTON (Trans. Amer. Electrochem. Soc., 1927, 51, 429—446).—From thermodynamical considerations it is shown that hydrogen and carbon monoxide are almost equally efficient as reducing agents for zinc oxide, and that if carbon monoxide is used it is much more economical to effect the reduction in two stages, (a)  $ZnO + CO = CO_2 + Zn$ , and (b)  $CO_2 + C =$ 2CO, separately. Very rapid reduction of zinc oxide by carbon monoxide takes place at 1200°, but on cooling the gases to condense the zinc vapour, rapid reversion of the reaction takes place; this could, however, be overcome by subjecting the hot zinc-carbon dioxide mixture to the producer gas-reaction (b) directly, but trouble would then be experienced owing to dilution of the zinc vapour and consequent difficulty in effecting condensation. Re-oxidation by water vapour in the case of hydrogen reduction is even more serious than re-oxidation by carbon dioxide. Methane has the advantage over both hydrogen and carbon dioxide that complete reduction of zinc oxide is effected by it below 900°, thus,  $ZnO + CH_4 = Zn + CO + 2H_2$ , which is not a reversible reaction, but the maximum proportion of zinc vapour in the issuing gases is only 25% by volume using pure methane. A. R. POWELL.

Nickel-silver alloys. C. ROTHERT and G. DERN (Z. Metallk., 1927, 19, 158-161).—The colour of nickelsilver alloys is governed almost entirely by the nickel content, the whitening effect of zinc being relatively small. Nickel also has a much greater effect on the hardness than zinc. The most satisfactory alloy out of a series of 28 melts of varying composition was found to be that containing 18% Ni, 20% Zn, and 62% Cu, the structure of which resembles  $\alpha$ -brass in being a homogeneous solid solution, and in having a pronounced tendency to the formation of twinned crystals on annealing. This alloy, after cold rolling, commences to recrystallise just below 600°, but the best structure is obtained at 800-850°. The work-hardened alloy has a tensile strength of 71.5 kg./mm.<sup>2</sup> with an elongation of 6.6%; after annealing the corresponding figures are 51 kg./mm.<sup>2</sup> and 33%. The depth of penetration of an annealed sheet 2 mm. thick in the Erichsen test is 13.0 mm. A high annealing temperature causes a very rapid growth of the crystals with a corresponding reduction in the tensile strength and the danger of the formation of inclusions of zinc oxide along the grain boundaries.

A. R. POWELL.

Influence of molybdenum and silicon on the properties of non-rusting chromium steels. W. OERTEL and K. WÜRTH (Stahl u. Eisen, 1927, 47, 742-753).-The hardness of plain chromium steels with 14-15% Cr increases rapidly with the carbon content from 0.1 to 0.3% C, then only relatively slowly; in all cases maximum hardness is obtained by annealing at 1050-1100°, above which temperature the hardness begins to fall again, especially in the case of the steel containing 0.5% C. Silicon has practically no effect on the hardness of low-carbon chromium steels, but reduces the hardness of those containing more than 0.3% C. Molybdenum has little effect on the hardness of any of the steels, but it tends to overcome the softening effect of silicon in the higher-carbon steels. Chromium steels containing more than 3% Si do not undergo the A1 and A3 transformations on heating or cooling; they become very coarse-grained during annealing, and have a relatively low tensile strength and tenacity. With up to 1% Si and a low carbon content, chromium steels are characterised by a high Erichsen value, hence they are suitable for the manufacture of hollow vessels. The steel with 0.1% C, 0.32% Mo, and 14-15% Cr becomes very soft and ductile on annealing and has a relatively high elastic limit. Non-rusting steel containing less than 0.3% Mo is highly resistant to corrosion by acetic acid

or sea-water, and is more resistant to 5% hydrochloric acid than any other steel so far tested. The silicon steels, on the other hand, are most resistant to nitric acid and to scaling on heating when more than 1% Si is present. After 48 hrs. at 900° the surface of a steel containing 0.15% C, 4.7% Si, and 14.5% Cr was grey and smooth, without any sign of oxide scale.

A. R. POWELL.

Uses of pure nickel. R. J. McKAY (Trans. Amer. Electrochem. Soc., 1927, 51, 457—461).—The properties of pure nickel show that the pure metal possesses few commercial advantages over the best samples of nickel which can be produced on the technical scale. The advantages of nickel as a non-corrosive material in engineering design and in food handling processes are pointed out. L. M. CLARK.

Determination of the gas content of molten metals. A. WÜSTER and E. PIVOVARSKY (Stahl u.Eisen, 1927, 47, 698—702).—An apparatus is described and illustrated by means of which the gas content of metals melted in an electric resistance furnace in a vacuum may be determined, and examples of its use in the examination of the gas content of cast iron poured at varying temperatures are given. In general, the gas content of cast iron increases with the temperature at which the metal is heated before casting up to 1500—1600°, then decreases again ; hydrogen is usually the predominant gas, followed by carbon monoxide, carbon dioxide, nitrogen, and methane. A. R. POWELL.

Magnesium and its alloys. W. R. D. Jones (Metal Ind., 1926, 29, 433-435, 459-461, 463; Chem. Zentr., 1927, I, 645).—Commercial magnesium contains 0.001-0.02% Cu, 0.051-0.01% Si, and 0.059-1.08% Fe, and, in the cast state, has a higher tensile strength than commercial aluminium. Its tensile properties are considerably improved by forging, especially if the castings are free from blowholes, which may be ensured either by adding a small quantity of calcium just before casting or by adopting a special melting process. A magnesium alloy with 8% Al ("Dow metal ") has an elastic limit of 1 ton/in.2, a tensile strength of 11.1 tons/in.2, an elongation of 4%, and a Brinell hardness of 55 in the cast state; for the same alloy after forging the corresponding values are  $4 \cdot 6$ ,  $21 \cdot 4$ , 7%, and 71. The normal oxide film on magnesium and its alloys is an efficient protection against corrosion, as well as an electrical insulator, a film 0.1 mm. thick withstanding 220 volts. The thickness of the film may be increased and its insulating properties improved by heating the metal at 320-330° in an atmosphere of A. R. POWELL. high-pressure steam.

Melting of alloys. M. FOURMENT (Rev. Mét., 1927, 24, 179-193).—An account of modern electric melting furnace practice.

Protective value of nickel plating. II. C. T. THOMAS and W. BLUM (Trans. Amer. Electrochem. Soc., 1927, 51, 515—527; cf. B., 1925, 810).—In continuation of earlier work on the behaviour of nickel-plated steel in accelerated corrosion tests, samples of the platings that gave the most satisfactory results in the laboratory were exposed to the atmosphere for 1 year. The behaviour of the specimens showed that the time required to produce initial corrosion in the accelerated tests was no criterion of the protective value of the coating against atmospheric corrosion. An approximate idea of the relative protective value of nickel coatings is, however, obtained by expressing the porosity of the coating, as determined by the ferricyanide test, in terms of the reciprocal of the number of perforations in a given area. Deposits having a high content of iron turn yellow very quickly on exposure to the atmosphere of a town, and frequently the deposit peels off. The best protection is afforded by nickel deposits having an intermediate copper layer between the nickel and steel. A. R. POWELL.

Inductive heating. NORTHRUP.-See XI.

Protective coatings for light-weight alloys. GARD-NER.—See XIII.

Painting galvanised iron pipes. GARDNER.—See XIII.

#### PATENTS.

Making fluxing materials.—J. LUND (U.S.P. 1,626,292, 26.4.27. Appl., 23.10.20).—A blast-furnace is operated so as to produce molten iron and a slag which contains an alkali carbonate. Superheated steam is directed on to the surface of the slag as it floats on the iron in a heated chamber, and the purified slag is withdrawn. C. A. KING.

Manufacture of pure iron. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 269,345, 10.5.26).—Iron formed by the decomposition of iron carbonyl, and containing carbon and oxygen, is melted or sintered in a high-frequency induction furnace *in vacuo*, and is afterwards exposed to an inert gas. C. A. KING.

**Protecting iron and steel articles against corrosion.** S. G. S. DICKER. From METALS PROTECTION CORP. (E.P. 269,305, 15.3.26).—As a protection against corrosion iron or steel articles are coated with a metal electropositive to iron, *e.g.*, cadmium or zinc, on which a second coating of a metal, *e.g.*, copper, electronegative to the first and preferably to chromium also, is applied. A final coating consists of chromium. The middle coating may be omitted, or the iron may be given an overcoating of chromium and an undercoating of a metal which is electronegative to both iron and chromium.

## C. A. KING.

Apparatus for the production of metal [zinc] dust. R. B. FINN, Assr. to J. FINN METAL WORKS (U.S.P. 1,622,433—4, 29.3.27. Appl., [A], 28.4.26; [B], 12.6.26).—In an apparatus of the usual type for the production of zinc dust, the bottom of the condensing chamber is fitted with a scraping element to remove zinc oxide which first collects there. T. S. WHEELER.

Treatment of zinciferous materials. P. C. RUSHEN. From NEW JERSEY ZINC Co. (E.P. 269,324, 10.4.26).—A briquetted charge of zinciferous material and a reducing agent is fed into an externally heated vertical retort of greater cross-sectional dimension than the length of the briquettes. The centre of the charge is heated mostly by convection of gases in the spaces between the briquettes. Zinc is volatilised and the retort is so worked that, without sintering, the briquettes disintegrate and may be drawn off at the bottom of the retort as a pulverulent residue. C. A. KING.

**Treatment of zinc-gold slimes.** C. E. MEYER (U.S.P. 1,622,960, 29.3.27. Appl., 10.4.22. Conv., 16.4.21).—Zinc-gold slimes from the cyanide process for the recovery of gold are treated with potassium nitrate and steam, and the zinc oxide formed is extracted with ammonium carbonate solution and then recovered by removing ammonium carbonate from the solution with steam under pressure. T. S. WHEELER.

Nickel alloy. T. S. FULLER, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,623,948, 5.4.27. Appl., 16.2.25). —An alloy containing 60-75% Ni, 25-40% Cu, 0.5-5% Al, 0.4-0.03% C, 1-5% Mn, and about 2.5% Fe when forged below 1000° has a high tensile strength and elasticity. The higher aluminium content is associated with the lower carbon content and vice versa. T. S. WHEELER.

**Resistor alloy.** N. B. PILLING, Assr. to WESTING-HOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,626,691, 3.5.27. Appl., 2.9.22).—An alloy containing 35—55 pts. of nickel, 65—45 pts. of copper, and up to 0.1% of magnesium is described, which has a negative coefficient of resistance. F. G. CROSSE.

Treatment of metallic oxides. ALUMINUM CO. OF AMERICA, Assees. of B. T. HORSFIELD (E.P. 248,360, 16.2.26. Conv., 2.3.25).—A stream of molten alumina, magnesia, or calcium oxide, free from silica, is blown with a gas at a velocity sufficiently high to form the oxide into hollow globules. In the preparation of alumina for electrolytic reduction to aluminium the natural raw material after electro-thermal reduction for the removal of associated oxide impurities contains an excess of carbon which is removed by disintegrating the molten stream by means of an air-blast. The granulated alumina is notably tougher than when solidified in the ordinary manner, and the product has superior properties as an abrasive. C. A. KING.

Centrifugal amalgamator and concentrator. F. D. LEWIS (U.S.P. 1,623,421, 5.4.27. Appl., 6.3.25).— A centrifuge for the treatment of gold-bearing gravels consists of three cylinders, which rotate about a common vertical axis. The gravel mixed with water is introduced into the innermost cylinder, and in passing through the apparatus to the external cylinder comes into contact with mercury maintained by centrifugal force in a number of annular grooves in the inner walls of the cylinders. The free gold particles amalgamate with the mercury. Means are also provided for separating and discharging through separate orifices light gravel and heavy concentrates containing mineral values other than free gold. T. S. WHEELER.

Amalgamator. N. S. KEITH (U.S.P. 1,627,181, 3.5.27. Appl., 21.4.24).—A machine for effecting amalgamation consists of a rotating vertical shaft carrying a disc clamped to the shaft by a boss to which is fixed a vertical cylinder covered by, but insulated from, a lead casing. Concentric with this cylinder is a cup bolted to, but raised from, the disc. The cup contains a removable cylinder with an inner surface of copper and a serrated bottom. The whole is rotated, pulp containing mercury is supplied to the annular space between the cylinders, and an electrical current is passed from the inner to the outer cylinder.

C. A. KING.

Apparatus for washing refuse containing precious metal. G. UNRATH (U.S.P. 1,627,129, 3.5.27. Appl., 18.5.26. Conv., 17.3.25).—Sweepings containing particles of precious metal are washed in a vessel of hemispherical shape having radial grooves on the inner side converging towards a central opening through which the heavier particles are discharged. A hollow stirring device agitates the muddy liquor in the cup, and lighter material is carried over into an outer container from which it flows into a settling tank. Clear water from the settling tank is lifted into the stirring arm for continuing the process with a fresh supply of refuse.

C. A. KING,

Operating cupolas. F. K. VIAL and G. S. EVANS, Assrs. to GRIFFIN WHEEL Co. (U.S.P. 1,627,536, 3.5.27. Appl., 19.1.25).—Combustible gases formed inside a cupola are withdrawn and used to preheat air to be supplied to the cupola. C. A. KING.

Separating magnetic from non-magnetic material. L. J. INGOLFSRUD and W. H. SOULE (U.S.P. 1,625,257, 19.4.27. Appl., 9.2.25. Conv., 31.3.24).— In a magnetic separator consisting of an endless belt which travels over a series of magnets, the belt is given a gradually increasing inclination lateral to the direction of motion. C. A. KING.

**Electrolytic refining of copper.** SIEMENS & HALSKE A.-G. (E.P. 264,116, 6.3.26. Conv., 6.1.26).—In the electrolytic refining of impure copper, *e.g.*, copper bronzes and brasses, aqueous cuprous chloride is used as the electrolyte, and the liquor flowing from the bath is oxidised by means of air or chlorine to cupric chloride. The latter is reduced to the monovalent form again by bringing it into contact with a further quantity of the impure copper, thus maintaining the concentration of copper in the bath. After some time concentration of nickel, zinc, tin, or other impurity necessitates preferential electrolytic purification of the solution. Hydrochloric acid and colloids may be added to assist the process. C. A. KING.

# XI.—ELECTROTECHNICS.

High speed-high frequency inductive heating. E. F. NORTHRUP (Trans. Amer. Electrochem. Soc., 1927, 51, 497-513; cf. B., 1926, 283).-The construction of a high-frequency induction furnace suitable for melting 300 lb. of iron-nickel alloy in 20-25 min. is described with reference to illustrations and diagrams. Power is supplied by a 150 kw. generator at 900 volts and 1920 cycles, and the efficiency of the furnace is over 80%. For melting a charge of 250 lb. of nickel-iron alloy (casting at 1320°) the current consumption is about 53 kw.-hr. The crucibles of refractory sand are packed inside the copper induction coil with loose sand, which gradually builds up the crucible on the outside, whilst the melting operation wears away the inside; the average life of a crucible is 5-6 melts. Owing to the rapid rate of heating practically no impurities are introduced into the metal, and, in the case of alloys, efficient mixing of the constituents is obtained by the rotating action of the current on the molten metal. A. R. POWELL.

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

Protective coatings for duralumin and similar light-weight alloys for exposed construction. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 296, 1-26).-A comprehensive series of exposures (accelerated roof tests and sea-spray tests) on panels of duralumin, other light-weight alloys, and, for comparison, steel, covered with various types of protective coatings are fully described, and the results obtained over a period of 6 months are tabulated. The need for protection against the rapid form of surface- and intercrystalline corrosion to which the light-weight alloys are prone is stressed, and, owing to the greater degree of expansion and contraction of these metals compared with that of iron and steel, elastic linseed-oil paints are shown to be very suitable for the purpose. The use of basic pigments is, contrary to earlier opinions, highly desirable. Clear nitrocellulose or clear varnish coatings are not durable, but improvement is achieved by pigmentation and the use of a long-oil varnish vehicle. Aluminium powder has also given satisfactory results.

S. S. WOOLF.

Painting galvanised iron conductor pipes: accelerated corrosion test. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 298, 51-64). —The causes of corrosion of galvanised iron rain pipes etc. are discussed. Pipes made from two grades of galvanised iron were painted with various described protective coatings, and semi-submerged at an angle in water, water containing resinous wood shingles of a corrosive nature, dilute sulphuric acid, and salt water, alongside untreated pipes in each case. The tabulated results of these accelerated corrosion tests indicate that painting the inside and outside surfaces of such pipes greatly increases their life. The salt-water test is regarded as too severe. S. S. WOOLF.

**Penetration tests on paste paints.** E. F. HICKSON (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 300, 124—134).—The penetration test for asphalt (A.S.T.M. Standards, 1924, 944) as modified for lubricating greases (Proc. A.S.T.M., 1924, I, 518) is applied to paste paints ranging in consistency from stiff white lead-in-oil to soft runny pastes. Trials carried out with the 45° cone that replaced the needle in the original asphalt test, and with the later developed double-taper cone (90° cone with 30° point) show that a satisfactory classification into very stiff pastes, stiff pastes, medium stiff pastes, semi-pastes, and runny (thin) pastes is afforded.

S. S. WOOLF.

Rapid method for the evaluation of Schweinfürth Green. D. KÖSZEGI and S. GERÖ (Chem.-Ztg., 1927, 51, 303).—1 g. of the substance is dissolved in dilute sulphuric acid, the solution is made alkaline with potassium hydroxide solution, and heated, when the copper is reduced to cuprous oxide with the oxidation of the equivalent proportion of arsenite, which is determined by collecting and washing the cuprous oxide precipitate, dissolving it in ferric sulphate solution, and titrating with potassium permanganate solution. The filtrate is neutralised with hydrochloric acid, excess of sodium bicarbonate added, and the arsenite remaining is determined by means of iodine and sodium thiosulphate. The total arsenic thus obtained is in good agreement with that determined by the distillation method. F. R. ENNOS.

Laboratory experiments on ethylene and propylene glycol ethers; their use in "odourless" brushing lacquers. H. A. GARDNER and A. W. VAN HEUCKEROTH (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 302, 149-166).-A number of nitrocellulose lacquers, clear and pigmented, were made up, the nature and amount of resin, solvent, diluent, and plasticiser being varied in order mainly to examine the possibility of (a) replacing the strongly smelling butyl acetate type of solvent by ethers of propylene and ethylene glycols, (b) introducing rosin in place of more expensive resins, and (c) using light petroleum ("gasoline") as diluent in place of toluene, which has powerful solvent action on previously applied varnish etc. Satisfactory results were obtained on these lines, propylene glycol monoethyl ether being the best of the three ethers of this type investigated. The results of physical tests on the experimental lacquer films are tabulated and discussed. S. S. WOOLF.

Darkening of shellac solutions : effect of metals and solvents. H. A. GARDNER and S. LEVY (U.S. Paint Manufrs. Assoc., Mar., 1927, Circ. No. 304, 199-202).-Four types of shellac-clean dry bleached, superfine, fine orange, and T.N.-were each dissolved in five grades of denatured alcohol, and each solution was divided into four parts, to three of which were added bright iron, sheet tin, and granulated zinc, respectively, the fourth being used as a blank. The four sets of tests, in glass jars with paper-insulated caps, were shaken daily for 5 min. over a period of 6 months. The presence of metal was shown to cause a gradual darkening, tin being worse than iron, and zinc being the least harmful in this respect. The grade of alcohol seemed to affect the colour to some extent, particularly that containing pyridine, but the darkening effect was less marked than that due to metal. An increase of viscosity was also noted in the solutions containing tin, and to a lesser but still considerable extent in those containing iron and zinc. It is advisable that shellac be "cut" in apparatus containing little, if any, metal, and be stored in glass containers. S. S. WOOLF.

Influence of driers on the properties of some ester gum and synthetic resin varnishes. G. G. SWARD and H. A. GARDNER (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 301, 135—148).—Various amounts and combinations of lead, manganese, and cobalt linoleates were added to varnish bases prepared by cooking equal pts. by wt. of oil (3 linseed oil : 1 tung oil) and resin at 264°, until requisite body was reached. The time required for this was 9 hrs. when ester gum was the resin used, and  $3\frac{1}{2}$  hrs. in the case of a synthetic resin derived from phenol, formaldehyde, and colophony. The varnishes were thinned to 50% non-volatile content, and tested for drying time, viscosity, colour, elasticity, hardness, resistance to solvents, keeping qualities, durability, and behaviour when made up into enamels. The tabulated results indicate the influence exerted by kind and concentration of drier on the type of varnish under consideration. Large amounts of driers tend to produce brittleness, and do not accelerate drying. Manganese driers reduce the elasticity of varnish films, and induce skinning on storage of the varnish to a greater extent than do lead or cobalt driers, which are preferred for enamels. The advantages accruing from entire omission of driers might be obtained in varnishes intended for any exterior uses where slow attaining of final hardness is not a drawback. S. S. WOOLF.

Pigment finishes. VOGEL.-See XV.

#### PATENTS.

Converting into soluble form insoluble condensation products of carbamide or a derivative thereof and formaldehyde. Soc. CHEM. IND. IN BASLE (E.P. 246,127, 4.1.26. Conv., 17.1.25).—Hard condensation products of carbamide or its derivatives and formaldehyde, particularly waste splinters or shavings from the working of these materials, are converted into soluble form by heating them under pressure at above 100° with excess of formaldehyde. The latter is distilled off, or further condensed with a fresh quantity of carbamide etc. S. S. WOOLF.

Preparation of alcohol-soluble, bromine-containing condensation products. A. JÜDEFEIND (G.P. 436,819, 9.10.23).—Tannin, brominated before or after the condensation, is condensed with carbamide or its derivatives and formaldehyde in the presence of a condensing agent, the product being used for dermatological purposes. S. S. WOOLF.

**Production of phenol-aldehyde resins.** BAKELITE G.M.B.H. (E.P. 246,834, 22.1.26. Conv., 27.1.25).— Phenol-aldehyde condensation products are heated with suitable quantities of alkali in aqueous solution (amounts to be determined by preliminary experiments). They are then "salted out" in gel form with sodium chloride, alkali carbonates, magnesium sulphate, alums, etc. In addition, hydrotropic salts, preferably so selected as to form soluble or insoluble compounds with the salts used for precipitating the resins, may be present. Typical hydrotropic salts are sodium salicylate, alkali metal soaps, or salts of fatty acids, of sulphonated-, chlorinated-, or hydroxy-fatty acids, or of resin acids. After washing and drying, the resins salted out in this way are powders having m.p. higher by 100° or more than the original phenol-aldehyde resins.

S. S. WOOLF.

Hardening phenol-aldehyde condensation products under heat and pressure. G. M., and N. G. HICK (E.P. 269,037, 12.8.26).—A sealed pressure-tight vessel containing phenol-aldehyde condensation products (moulded in plaster of Paris) and a volatile liquid, *e.g.*, alcohol, is immersed in a boiling liquid, *e.g.*, a saturated solution of sodium chloride, the temperature and pressure inside the vessel being thus determined. Gas under pressure may be substituted for the volatile liquid. Different types of pressure vessels are detailed.

S. S. WOOLF.

# XV.—LEATHER; GLUE.

Tanning, dyeing, and finishing formaldehydetanned leathers and skins. J. W. LAMB (Halle aux Cuirs, 1926, 6-16; Chem. Zentr., 1926, II, 2650-2651). -Every 15 min., 7.2 kg. of sodium carbonate and 4.5 litres of 40% formaldehyde in 36 litres are added in 4 portions for every 100 kg. of pelt in 225-270 litres of water at 35°. The tannage is complete in 3-6 hrs. with light skins, and in  $\frac{1}{2}$  days with heavy hides. The temperature may be increased to 50° towards the end of the tannage. The leather is then treated with a 1% ammonium sulphate solution, horsed up, and then drummed with a solution of  $2 \cdot 25$  kg. of soft or neutral soap and  $2 \cdot 25$  kg. of sodium chloride at 50° for 2—3 hrs., giving a longer time for heavy leather. An emulsion of fish or olive oil with soap can also be used, and then the salt solution must be given afterwards. The addition of alkali during the tannage prolongs the process; an excess of formaldehyde causes brittleness. For dyeing with basic dyes, the leather is mordanted with 6% chrome alum and 0.75% sodium carbonate (on dry weight) and dyed with addition of sodium carbonate and 5-10% of sodium chloride or sulphate. Chrome leather dyes (direct) can be used if sodium bisulphate is added to the dye liquor. Acid dyes can be applied in the drum or with a brush if soap and formaldehyde are mixed with the dye solution. For deeper shades sodium chloride is added. The fastness to washing can be increased by dipping the dyed leather in a solution of potassium dichromate, copper sulphate, or acetic acid for a short time. Unfixed dye is removed by washing and soap. Previous treatment with ammonium acetate or formate is recommended when dyeing with acid dyes.

## D. WOODROFFE.

Pigment finishes. W. VOGEL (Collegium, 1926, 560-562).-Pigment finishes consist of three ingredients chiefly, viz., pigment, binding agent or carrier, and the solvent. Both inorganic pigments and organic pigments (colour lakes) are used. Fats, proteins, and carbohydrates are used as carriers. Linseed oil and varnishes are used as carriers in patent leather manufacture. Aqueous solutions of glue, egg albumin, and casein are used for this purpose; also aqueous solutions of gums, dextrin, and starch. There are two kinds of pigment finishes used in leather finishing, viz., albumin finishes containing egg albumin in aqueous solution and the collodion finishes, with nitrocellulose as the binding agent in organic solvents. The albumin finishes cover the leather well and give an even colour without detriment to the appearance or feel of the leather. The waterproofness leaves much to be desired, and where it is essential, the collodion finishes must be used. Collodion finishes are made by dissolving collodion wool in organic solvents, grinding with pigments usually together with a small amount of castor or olive oil to make it elastic. Both types of finish can be used for making splits into imitation grain leathers. The practical value of these finishes can be tested by applying them to leather, drying out, and testing for fastness to light, rubbing, and water, also the feel. The fastness to light is exceptionally good in most of the commercial finishes. Collodion-finished leathers are satisfactory to rubbing and to water. They have not such a smooth feel as leathers finished with the albumin finishes.

D. WOODROFFE.

# XVII.—SUGARS; STARCHES; GUMS.

Luminescence of sugars and of sugar factory products. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, 51, 237—245).—Luminescence in sugar factory products is due, not to the ash nor to the colouring matter, but to a colourless or but slightly coloured substance, soluble in ether and chloroform, one of the first products of the caramelisation of sugar or of the destruction of invert sugar. Beet products exhibit it to a greater extent than cane products. An apparatus is described for its measurement. J. P. OGILVIE.

Determination of hydrogen-ion concentration of sugar factory products. O. SPENGLER and F. TÖDT (Z. Ver. deut. Zuckerind., 1927, 115—118).— Wulff's method (Chem.-Ztg., 1926, 50, 732) applied to a beet molasses at 30° and a liquor at 50° Brix required 5 and 10 min., respectively, before the final indication could be obtained, and is considered suitable only for liquids that are not more viscous than water. For routine practice in the sugar factory the colorimetric drop method is preferred. J. P. OGILVIE.

Determination of amides in sugar factory products. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1927, 51, 261—271).—As Schulze's method gives results lower than the truth in the presence of sugar, a process is recommended depending on the principle that amides, ammonium salts, and most of the amino-acids are entrained by the precipitate formed on the addition of sodium carbonate to mercuric acetate. In determining the original ammonia and the amides, respectively, the mercuric precipitate is distilled in the presence of magnesia, magnesium sulphate, and sodium thiosulphate before and after hydrolysis by acid. J. P. OGILVIE.

Decolorising carbon [in sugar] work. S. KÜHN (Z. Zuckerind. Czechoslov., 1927, 51, 271—272).—A consideration of the question whether it is advantageous to filter the liquors previous to their treatment with decolorising carbon, leads to the following figures for the insoluble matter in liquor taken directly from the blow-ups being given: organic matter, 3.77, and inorganic, 7.12 kg. per 1000 quintals of sugar introduced in 24 hrs. J. P. OGILVIE.

Decolorising carbons and their action on molasses. M. GARINO and G. BENVENUTO (Giorn. Chim. Ind. Appl., 1927, 9, 169—175).—Of a number of samples of commercial decolorising carbons, carboraffin gives the best results for decolorising either molasses solutions or methylene-blue solutions. Molasses contains different colouring matters, each of which exhibits a definite degree of affinity for any particular adsorbent carbon. Adsorption of the various colouring substances obtainable from caramel differs greatly from that of the substances formed by the action of lime on invert sugar, these being, according to Peligot, mainly glucic and *apoglucic acids.* T. H. POPE.

Invertase present in [beet] sugar factory wastewaters. C. L. MATOUŠEK (Z. Zuckerind. Czechoslov., 1927, 51, 213—220).—Invertase resulting from the micro-organisms introduced by insufficiently washing the roots may, when the water used in the diffusion battery is impure, and especially in the case of returned waste waters, give rise to a relatively considerable invert sugar formation. At a temperature of 36° the invertase present in a very dirty water, formed, in a 5% sucrose solution, invert sugar to the amount of 1.36—1.95% in 24 hrs. J. P. OGILVIE.

Uniformity in sugar beet analysis. A. W. LING (Int. Sugar J., 1927, 29, 86-88).—Recommendations as to sampling are made. Dirt is determined by the usual method of washing and re-weighing, and in order to obtain the sample of pulp for the sugar determination the whole of the 25 roots should be taken. A circular saw revolving at 1000 r.p.m. is used to obtain the pulp, this being driven so that it rotates away from the beet, the resulting fine pulp being collected in a hopper beneath the saw, and well mixed. In determining the sucrose content, the Krüger method may be used, but if a less efficient method of pulping has been employed (viz., a mincing machine) it is then necessary to apply a hot water digestion process, e.g., the Pellet. J. P. OGILVIE.

Content of sulphur dioxide in German consumption sugars. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zuckerind., 1927, 167—173).—Direct titration with 0.005N-iodine solution affords a useful sorting-out test, the end-point generally, though not always, being sharp. Useful results were obtained by the sulphidestain method (cf. B., 1927, 312), slightly modified. Of 39 samples of white consumption sugars examined only 5 gave over 10 pts. per million of sulphur dioxide, the highest being 30 p.p.m. Ultramarine when present does not affect the iodine titration. A sample of sugar free from sulphur dioxide on being strongly blued with ultramarine, showed 17 p.p.m. by the sulphide-stain method. J. P. OGILVIE.

# XVIII.—FERMENTATION INDUSTRIES.

Detection of fruit wine in grape wine. A. HEI-DUSHKA and C. PYRIKI (Chem.-Ztg., 1927, 51, 442).—Röttgen's reaction for the detection of fruit wine in white wine is uncertain if any quantity of blue grapes has been used in its manufacture or if the wine is adulterated with decolorised red wine. A negative result in the test, however, indicates the certain absence of fruit wine, but there is still no sufficiently sharp reaction for the certain detection of the latter in white wines (cf. B., 1927, 264). A. R. POWELL.

Spectrometric detection of fruit wine in wine. O. E. KALBERER (Z. Unters. Lebensm., 1927, 53, 208— 221).—The absorption spectra of various red and white wines, with and without added fruit wine, have been measured photographically and represented graphically by plotting the thickness of the layer against the number of waves per mm. There are characteristic differences between grape and fruit wines. The former show an increase in general absorption with decrease of wavelength, with distinctive absorption bands between 34,000 and 38,000, the greatest absorption occurring at about 36,800—38,100. Some grape wines also show an absorption band between 29,000 and 31,000. With fruit wines, the latter band is strongly marked at 27,000, and these wines are further distinguished by a band in the region 40,800—43,000, which is scarcely noticeable with grape wines. A formula has been deduced from which the extinction coefficients of wines can be calculated, and the curves obtained by plotting logarithms of these coefficients against reciprocal of wave-lengths agree closely with those determined experimentally. It is shown that by comparison of the extinction curve of a mixed wine with that of a pure wine, it is possible to detect and determine the amount of any addition of fruit wine. Y

Determination of degree of acidity of wines with diazoacetic ester. G. BREDIG and K. SIEBENMANN (J. pr. Chem., 1927, [ii], 116, 118-128).-Preliminary experiments were carried out on the hydrolysis of diazoacetic ester (cf. Bredig and Fraenkel, A., 1905, ii, 692; Fraenkel, ibid., 1907, ii, 746) with 0.0021N-nitric acid and 0.0025M-tartaric acid, at 25°, in aqueous-alcoholic solution (alcohol content, 3-18% by vol.). The values for the ratio of the velocity constant (k), and hydrogenion concentration in mol./litre (h) are tabulated, and they are found to decrease in magnitude with increasing amount of alcohol. The inversion of sugar, catalysed by 0.002N-hydrochloric acid and 0.0025M-tartaric acid, at 75.86°, in various aqueous-alcoholic solutions has also been studied, values for k and h being determined. The percentage decrease of hydrogen-ion concentration  $(dh/h \times 100)$  of dilute solutions of malonic, tartaric, and acetic acids, on adding alcohol, has been determined by the following methods: (a) electrometrically at 18°, (b) diazoacetic ester, (c) sugar inversion. The results from methods (a) and (b) are in good agreement; those from (c) are distinctly higher. The degree of acidity (no. of mg. of hydrogen ion per litre  $= h \times 10^3$ ) has been determined for four wines-white Gaillard (1913 and 1922), white Burgundy (1921), and Portuguese red (1923)—by methods (a), (b), and (c). In method (a)a hydrogen wine saturated calomel cell was used. The results from (a) and (b) are in close agreement, whilst H. BURTON. those from (c) are slightly lower.

Determination of methyl alcohol in alcohol and alcoholic beverages using the immersion refractometer. J. F. WILLIAMS (Ind. Eng. Chem., 1927, 19, 844-845; cf. Leach and Lythgoe, A., 1905, ii, 655).-The distilled sample is accurately diluted to 20% of total alcohol by volume, and determinations made of the sp. gr. and refractive index, the % of total alcohol indicated by the sp. gr. of the diluted solution being slightly greater than 20% if methyl alcohol is present, since the densities of the two alcohols on dilution do not run From the difference between the strictly parallel. refractometer scale reading and the % of total alcohol the amount of methyl alcohol is obtained by reference to a table. The results are accurate to within 0.5%with solutions containing 1% or more of methyl alcohol, but smaller quantities are best determined by Wright's colorimetric method (cf. A., 1927, 687).

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