BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 28, 1926.

I.—GENERAL; PLANT; MACHINERY.

Theory of shaft furnaces. H. LE CHATELIER (Compt. rend., 1926, **182**, 735—739).—The theory of heat exchanges in furnaces consisting of a vertical cylinder at the top of which the solid substances to be worked up enter continuously, and hot gases at the bottom, is discussed. Assuming that the heat exchange is not affected by secondary factors and is instantaneous, then if the specific heat of the gases exceeds that of the solids, the latter will leave the furnace at the temperature of the incoming gas. If the reverse is the case, the gases will leave at the temperature of the cold solids. When the specific heats are equal, the behaviour is variable. According as the heat capacity of the solids entering is greater or less than that of the gases entering in the same time, the whole furnace will be at the temperature of the cold solids or of the hot gases.

R. CUTHILL.

Solubility of calcium sulphate at boiler-water temperatures. R. E. HALL, J. A. ROBB, and C. E. COLEMAN (J. Amer. Chem. Soc., 1926, **48**, 927—938).—The solubilities in water of anhydrite and soluble anhydrite were determined under conditions obtaining in boilers. The values in g. of $CaSO_4$ per 1000 g. of solution, fell from 0·810 at 100° to 0·065 at 207° in the former case, and from 1·124 at 115° to 0·264 at 185° in the latter. The figures for soluble anhydrite agree with those of Melcher (B., 1910, 150), and of Boyer-Guillon (Ann. Conserv. Arts Métiers, 1900, [iii.], **2**, 189); those for anhydrite agree with Melcher's results at higher temperatures, but do not agree so well at lower temperatures. S. K. TWEEDY.

Sodium bicarbonate in dry fire extinguishers. C. GENTSCH (Z. angew. Chem., 1926, **39**, 450—451).—Sodium bicarbonate is not instantaneously decomposed at a red heat, as has been stated, for the decomposition requires 3 min. in a crucible heated over a powerful flame, and only reaches 10% in the first 15 sec. The carbon dioxide produced by this decomposition cannot therefore be relied upon for the instantaneous extinguishing of a fire.

B. W. CLARKE.

Apparent paradox of plasticity. A. DE WAELE (Kolloid-Z., 1926, **38**, 257—259).—The method of testing the plasticity of "stand oil," prepared by diluting oils which have been polymerised to the gelation point, in the plastometer may lead to smaller values of ϕ, the volume proportion of the viscous phase, than those obtained by the use of a viscosimeter. It is found that ϕ increases with the radius of the capillary, when the radii are small. An explanation is suggested in terms of the fact that the size of particle of the disperse phase becomes comparable with the radius of the tube. It is therefore necessary to use sufficiently wide tubes for this test.

C. H. D. CLARK.

Adsorption from solutions. SABALITSCHKA and ERDMANN.—See XX.

PATENTS.

Vertical drying and dehydrating apparatus. BAECHLER, KISER & CO. (E.P. 241,901, 20.10.25. Conv., 21.10.24).—A vertical cylindrical casing contains a number of inverted truncated conical hoppers, through which the material drops in succession on to flat plates arranged below the small ends of the cones. Between each cone and plate rotates a runner, comprising a number of curved (involute) blades which work the material from the centre to the circumference of the plate, one blade at least being long enough to push the material over the edge of the plate, whence it drops into the next cone. The drying is effected by hot air admitted to the bottom and exhausted at the top of the casing. The rate of treatment can be adjusted by varying the distance between the cones and plates. B. M. VENABLES.

Centrifugal dryer. A. MCKAY (U.S.P. 1,576,528, 16.3.26. Appl., 18.10.24).—A number of dished plates are mounted on a vertical rotating shaft inside a casing through which a drying medium is passed, a fixed, inverted conical hopper being provided between each pair of plates. Each plate has a raised centre and periphery, and is flatter at the centre and turned up to a greater height at the periphery than the one above it. A. GEAKE.

Centrifugal machines. AKTIEBOLAGET SEPARATOR (E.P. 244,737, 23.11.25. Conv., 19.12.24).—In a centrifugal purifying or separating device for liquids, at least one constituent of which is volatile, and may form an explosive or noxious mixture with air, means are provided whereby a current of air automatically flows outwards through the outlet for one constituent (preferably the most volatile one) and inwards through the outlets for the other separated constituent and for excess feed. The volatile constituent and air are conducted away together through a pipe leading to a room apart from the separator room, and enter a closed tank above the level of the liquid therein, the liquid being drawn off through an inverted siphon pipe forming a liquid seal, and the noxious air being allowed to exhaust through a high-level outlet, preferably including a condenser which will recover some of the volatile liquid. The current of air is produced either by fan blades running with the separating bowl, or by having the final outlets from the bowl for the volatile liquid at a greater radial distance from the centre than the outlets for the other constituents. B. M. Venables.

Centrifuges. Aktiebolaget Separator (E.P. 246,831, 22.1.26. Conv., 2.2.25).—A centrifugal separator which is provided with an upper feed vessel and a collector with outlet for excess feed, is also provided with an overflow, adjustable as regards height, from the stationary feed vessel into the collector, so that the untreated liquor or pulp is fed at a head not exceeding a definite value. B. M. Venables.

Centrifugal machines. L. Salcines (E.P. 248,022, 26.9.24).—The apparatus comprises a perforated outer rotating basket through which the filtered liquor passes and an inner conveying and scraping drum which rotates at a slightly different speed to the basket. The inner drum serves to convey the deposited solid matter to outlets at the bottom of the basket, the bulk of the material being conveyed by a spiral worm on the surface of the drum; the outer edges of the spiral are some distance from the filtering surface of the basket, but scraping projections are pushed outwards to within a short distance of the filtering surface by means of compressed air supplied at intervals to the interior of the drum. After the scrapers have removed as much as possible of the deposited solids the filter surface is finally cleaned by jets of steam or compressed air projected through nozzles also situated on the outer edges of the conveying worm. The rate of feed of material is regulated by pistons in the feed shoot, which control electrical contacts, which in turn control a feed valve operated by a solenoid. B. M. Venables.

[Ring and roll] crushers. Aciéries Réunies de Burbach-Eich-Dudelange Soc. Anon. (E.P. 244,073, 17.11.25. Conv., 8.12.24).—In a crushing machine of the ring and roller type the shaft of the top roller is supported by fixed bearings on one side only of the crushing zone and is driven by the source of power. The ring is driven by the top roll only, and the other two rolls are driven by the ring. The bearings for the two lower rolls are mounted entirely on springs, a system of three springs being used near the rollers to produce the grinding pressure, and five springs at the outer ends of the roll shafts, so arranged that the bearings can give in any direction, radial or axial, but the rolls are always pressed outwards against the ring. B. M. Venables.

Ball or tube mills for grinding or crushing. F. W. Davis (E.P. 249,045, 27.11.25).—For drying materials undergoing the process of grinding, a ball mill is provided with air-inlet holes in the shell, near the end at which the material is admitted. Such holes may be guarded on the outside by hoods facing in the direction of rotation and on the inside by baffles sloping in the direction of rotation and interlocking with each other to form steps, with spaces at each step for the inlet of air. The air is exhausted with the ground material and if desired may be heated before entering. B. M. Venables.

Recovery of the solid constituents of liquids containing them. H. N. McLeod (E.P. 248,081, 28.11.24).—The liquid to be treated is pumped first through one tube of a three-tube regenerative system and is then heated above its boiling point (*e.g.*, by passing it through the jacket of an internal-combustion engine which works the pump) and sprayed into a chamber which may be heated by passing the exhaust gases of the engine through a jacket around it. The dry powder is removed from the spray chamber by mechanical means and the vapour from the chamber and the exhaust gases from the jacket are passed through the other two tubes respectively of the regenerator, the vapour being thus condensed to a liquid, which has been rendered innocuous by the use of a sufficient degree of heat or by the addition of a chemical agent, or both. The process is particularly applicable to waste products from butter and cheese factories. B. M. Venables.

Crystalliser. M. J. Kermer (U.S.P. 1,575,584, 2.3.26. Appl., 2.4.24).—A heating or cooling agent passes through the outer of two concentric chambers or conduits and the material to be crystallised is conveyed through the inner one by means, such as a double screw conveyor, which moves the material in both directions, but to a greater extent in one direction. Both sets of conveyor blades are mounted on the same shaft, which is supported at intervals by bearings inside the inner conduit. B. M. Venables.

Dialysing and evaporating membranes [from nitrocellulose]. J. M. Looney, Assr. to P. A. Kober (U.S.P. 1,576,523, 16.3.26. Appl., 23.11.21; cf. B., 1922, 271 a).—Permeable membranes are prepared by mixing nitrocellulose and ethyl alcohol, dissolving the nitrocellulose by agitating the mixture with ether, adding a water-soluble ester, spreading, and removing the solvent. A. Geake.

Liquefaction of gases. Badische Anilin- & Soda-Fabr. (F.P. 600,028, 24.6.25. Conv., 20.6.24).—The gases are dried and cooled by treatment with calcium chloride and liquid ammonia, and are then freed from the last traces of moisture by passing through porous non-vegetable matter such as silica gel, permutit, or silicates, prior to introduction into the liquefaction apparatus. E. S. Kreis.

Drying of road-making aggregates and other broken or granular materials. J. F. Wake (E.P. 249,279, 23.9.24).

See also pages 397, **Securing intimate contact between liquids** (U.S.P. 1,575,116). 400, **Filters** (E.P. 248,044). 405, **Heat interchangers** (E.P. 248,999). 408, **Furnaces** (E.P. 248,036). 409, **Rotary kilns** (E.P. 248,079).

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

Composition of coal: plant cuticles in coal. V. H. Legg and R. V. Wheeler (Safety in Mines Res. Bd. Paper No. 17, 1926, 10 pp.).—A comparison has been made of the fossil plant cuticles found in bituminous coal with cuticles from modern plants (cf. B., 1925, 619). Fossil cuticles can be separated from bituminous coal by mild oxidation followed by extraction with alkali, but, a sufficient supply being more readily obtainable from Russian "paper coal," this has been used as the main source of material. Geological considerations show that the cuticles from both sources are probably closely allied. When freed from ulmin material and from a small quantity of waxy constituents that they contain, the cuticles are attacked only to a slight extent by alkalis, differing in this respect from modern cuticles (*loc. cit.*). The difference is due probably only to the elimination of the external acid groupings of the cuticle rather than to any profound change in structure. On distillation in a vacuum the fossil cuticles yield almost as high a proportion of tar (40%) as do the modern cuticles. The chemical composition of the cuticles differs considerably from that of the ulmic ground mass of the coal in which they are embedded.

A. B. MANNING.

Fundamental study of Japanese coal. III. Relation between microscopic structure and chemical properties. C. IWASAKI (Tech. Rep. Tôhoku, 1926, 6, 1—84; cf. B., 1925, 194).—Soya, Miruto, and Miike coals were examined under the microscope by transmitted and reflected light and by means of *X*-rays. The Soya coal is a black lignite with 20% of moisture; plant tissues such as bark, cork, bast-fibres, resins, stone cells, cuticles, and bark-parenchyma were detected. Miruto coal is a low-grade bituminous coal with only traces of plant tissues. Miike coal is a sub-cannel coal consisting of 'dull' coal with a homogeneous structure. The conversion of the plant substances to humic matter is traced by photographs of the various coals. More sulphur was found in the 'bright' than in the 'dull' coal and the Miike coal was particularly rich in 'sulphate' sulphur, viz., 0·56%. The ash from the 'bright' coal in the Soya coal contained 27·99% Fe_2O_3 as compared with 6·32% in the ash from the 'dull' coal; the silica content was 58·43% in the ash from the 'dull' coal, and 7·13% in the ash from the 'bright' coal. Curves showing the volatile matter evolved every 50° from 150° to 1100° gave peaks at 250°, 400°, and 550°. The first and second peaks are due to the woody and plant substances present and the third to the transformed humic matter.

A. C. MONKHOUSE.

Fungi which grow on coal. T. IWASAKI (Tech. Rep. Tôhoku, 1926, 6, 85—94).—The growth of fungi on Yoshioka, Horonubu, Soya, and Machida coals was examined and the following fungi were detected:—*Aspergillus minimus*, *A. oryzæ*, and *Citromyces pfefferianus* (cf. B., 1925, 350). The growth of fungi is less as the geological age of the coal increases, the maximum growth being obtained on the brown lignite. The fungi are richer in growth on the "dull" than on the "bright" coal and for the particular coals examined maximum growth was obtained with *Aspergillus minimus*.

A. C. MONKHOUSE.

Recent investigations on the origin of coal. F. FISCHER (Z. deuts. Geol. Ges., 1925, A., 77, 534—550; Chem. Zentr., 1926, I, 2081).—Wood breaks down under the action of fungi and enzymes into cellulose and lignin. The former is completely decomposed by bacteria and fungi, and disappears, while the latter does not support the growth of these organisms. The lignin only loses its acetyl and methoxyl groups, with formation of humic acids and by loss of water, anhydrides of these acids. These form the humins of brown coal. Under the influence of pressure and a higher temperature carbon dioxide and methane are given off, and a process occurs resulting in the ultimate formation of bituminous coal, the humins of which are insoluble in sodium hydroxide even on boiling. The humins of "pitch coal" become soluble on boiling with sodium hydroxide, this forming a member of the coal series intermediate between bituminous and brown coal, the humins of the latter being directly soluble in alkali.

A. B. MANNING.

Examination and evaluation of coals for carbonising purposes. G. WEYMAN (Gas J., 1926, 173, 601—603, 677—679, 746—747).—From coal to coal the quantities and qualities of the same products may vary, and even different products may result. The size of the coal affects the time of carbonisation, dust impeding the passage of gases and hence of heat through the charge. Hence hardness is an influential factor. Sieving tests are of little value in this connexion; a better index is the bulk density of a 10-ton truck-load. Ultimate analysis does not yield information of much use, but knowledge of the amount and composition of the ash is of greater value. Extraction with solvents is of little practical value. The calorific value does not indicate how the coal will behave when carbonised, unless it is considered in conjunction with the volatile matter. Proximate analysis gives indefinite and incomplete results. Ordinary methods of assay, in which large-scale practice is imitated, do not give sufficient of each product for thorough examination The performance of large-scale tests generally repays for the effort expended. The best method of test, however, is one which is as small as is compatible with yielding sufficient products for analysis. A plant carbonising 1/1000 ton of $\frac{1}{2}$-in. coal has accordingly been devised. It consists of a metal retort, gas-heated in a muffle furnace under conditions permitting of accurate temperature control. The off-take pipe passes from the side of the retort (so that tar will not reflux and crack) to air- and water-cooled condensers which in turn lead the gas through a water and an acid washer, oxide purifier boxes, and a meter fitted with an attachment for recording the rate of gas evolution, into a holder. The yields and the behaviour of the products when tested in the usual way are ascertained. As a basis of evaluation a standard coal is selected and the yield of each product per ton of coal carbonised is multiplied by its basic price in pence per unit. The values of all the products per ton of coal thus obtained are summated and the value of each calculated as a percentage of the total. This percentage value for each product, divided by the yield, gives a unit value which is used as a basis to compare subsequent coals with the standard. The total value of all the products will then amount to more or less than 100, a figure indicative of the return to be expected from the coal used. This is gross valuation. Nett valuation is obtained by allowing a 1% difference for 1% difference in the moisture (which, in the standard coal is 2%), a correction for difference in ash (standard, 8%) at twice this rate, and a further correction for rate of carbonisation as measured by average rate of gas evolution. R. A. A. Taylor.

Path of the gases in the distillation of coal. T. P. L. Petit (Het Gas, 1926, **46**, 50—51; Chem. Zentr., 1926, I, 2161).—It is now generally accepted that as the coking of the coal in the oven proceeds from the wall towards the centre the gases evolved escape through the pores of the coke already formed in the direction of the nearer oven wall, and not, as earlier theories have stated, through the coal core. According to Hilgenstock (Gas- u. Wasserfach, **49**, 617) the gases at first pass through the coal core until this is cemented together by the tar produced, then, with the advance of the coked layer, the tar is gradually gasified and the gases escape again through the core. The author has already refuted Hilgenstock's theory (Polytechn. Weekblad, 1921, Nr. 1) on the basis of the results of Teune and Kummel (Het Gas, **40**, 168). A simple apparatus is now described which provides indisputable proof that the evolved gases pass exclusively through the pores of the coked layer. A. B. Manning.

Behaviour of a coal of recent formation on prolonged heating at 110°. D. J. W. Kreulen (Chem. Weekblad, 1926, **23**, 186—187).—After the first hour, the coal increased in weight, the increase continuing but becoming gradually slower for 1275 hrs., after which an intermediate period supervened, followed by a diminution of weight. S. I. Levy.

Analysis of mixtures of unsaturated hydrocarbon gases. Determination of erythrene in gas mixtures. A. Dobrjanski (Neftjanoe i slancevoe Chozjajstvo, 1925, **9**, 565—577; Chem. Zentr., 1926, I., 2220—2221).—A method for the determination of the proportions of different olefines in gas mixtures depends upon the varying rates of absorption of members of the olefine series by sulphuric acid of different concentrations. Mixtures containing the gases were treated with sulphuric acid in a glass cylinder of 19·9 sq. cm. cross section, the diminution in volume being measured in the cylinder to avoid transference from one vessel to another and to ensure exposure of the gas to a constant surface of the acid. The rates of absorption of the olefines by sulphuric acid of different strengths are approximately as follows:—ethylene, 0·7 c.c. and 0·007 c.c. per min. by 101·2% and 87·87% acid; propylene, 2·2 c.c. and 0·009 c.c. per min. by 85% and 67·2% acid; *iso*butylene, 3·0 c.c. and 0·013 c.c. per min. by 67·2% and 22·3% acid, respectively. The proportions of these gases in mixtures are determined by bringing the gas successively in contact with 63—64% acid, which absorbs practically nothing but the *iso*butylene, with 83—84% acid, which absorbs the propylene, and with 100—102% acid, which absorbs the ethylene. In a modified procedure for use when the mixture contains erythrene, *iso*butylene is absorbed by 45—46% acid, propylene and erythrene together by 83% acid, and ethylene by weak oleum; a separate sample of the gas is then shaken with bromine water, formed *in situ* by the addition of potassium bromate and potassium bromide to sulphur dioxide solution in the reaction vessel, and excess bromine is titrated with sodium thiosulphate in the presence of potassium iodide and starch. Erythrene absorbs 4 atoms and *iso*butylene only 2 atoms of bromine under these conditions. L. A. Coles.

Composition of Swedish generator shale oils. G. Hellsing and J. S. W. Troedsson (Ark. Kem. Min. Geol., 1925, **9**, [22], 1—12; Chem. Zentr., 1926, I., 2064).—The oil obtained from a Swedish shale used as fuel in a generator is considerably richer in aromatic hydrocarbons than the oil obtained by the usual dry distillation of the shale (cf. B., 1921, 536 A). It is also rich in dissolved sulphur. The latter acts as a dehydrogenating agent at high temperatures, increasing the content of aromatic hydrocarbons at the cost of the naphthenes and other substances present. Both crude oils are probably similar in composition to begin with, their difference arising only from the difference in the distillation process employed. One of the principal constituents of the fraction 155—185° of the generator oil is ψ-cumene, which was isolated as the sulphonamide. The residue after sulphonation consists of a mixture of naphthenes ($C_{10}H_{20}$ and $C_{11}H_{22}$), as was verified by nitration. The presence of β-decanaphthene in the residue was shown by bromination. According to Bruhn (Chem.-Ztg., 1898, 900) the relatively high content of aromatic hydrocarbons in Swedish shale oil is explained first by the formation of naphthenes from oleic acid (set free from esters present in the oil), by loss of carbon dioxide, and rupture at the double bond, and then by their dehydrogenation due to the action of the sulphur at high temperatures. A. B. Manning.

Examination of spirit produced by the hydrogenation of coal. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, **12**, 77—78).—The spirit examined was derived by the "Berginisation" of Orgreave Washed Slack. By a long series of fractionations, observations of refractive indices and iodine values, and preparation of nitro-derivatives of the aromatic compounds present, the authors arrive at the following composition for the spirit:—Unsaturated compounds, 3·1%; benzene, 2·7; toluene, 1·9; xylenes etc., 2·9; butanes, *iso*pentanes, etc., 10·8; *n*-pentane, 10·5; *iso*hexane etc., 6·3; *n*-hexane, 10·0; *iso*heptane etc., 2·9; *n*-heptane, 6·5; *cyclo*pentane, 0·8; methyl*cyclo*pentane, 4·5; *cyclo*hexane, 10·3; methyl*cyclo*hexane, 11·4; residue, 15·4% by weight. The spirit examined formed approximately 10% of the weight of the coal treated. W. N. Hoyte.

Action of sulphuric acid on petrol. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, 12, 68—76).—The authors treated two petrols with sulphuric acid in a series of washes, the strength of the acid being steadily increased at each wash, from 79·1% to 100·5%. Determinations of percentage loss of weight, density, refractive index, and aniline point, following this treatment indicated that polymerisation and removal of unsaturated compounds take place with acid of strength below 88%, and that with acid above that strength removal of aromatic compounds takes place. W. N. Hoyte.

Symposium on determination of unsaturated hydrocarbons (J. Inst. Petrol. Tech., 1926, 12, 48—67).—J. S. S. Brame critically reviewed current methods, and suggested the use of 84·5% sulphuric acid for the removal and polymerisation of unsaturated compounds.

A. E. Dunstan pointed out that the assumption that iodine values referred exclusively to unsaturated compounds was probably incorrect.

E. L. Lomax and E. S. Pemberton showed that treatment with 80% sulphuric acid is inadequate for the complete removal of olefines and advised the splitting of the spirit to be examined into separate fractions by distillation prior to treatment with acid.

H. Moore described experiments using various strengths of sulphuric acid from 80% to 95% and stated that unsaturated compounds were not removed sufficiently completely by an acid weaker than 90%. A rise in the aniline test of 1° corresponded to a loss by acid absorption of 2·59% of unsaturated compounds.

F. H. Garner stated that under certain conditions combination between unsaturated compounds and aromatic compounds occurs in the presence of 93% sulphuric acid. W. N. Hoyte.

Vapour pressure of [motor] fuel mixtures. II. J. S. Lewis (J. Inst. Petrol. Tech., 1926, 12, 32—47; cf. B., 1925, 487).—The vapour pressures of the mixtures, alcohol-*cyclo*hexane, alcohol-hexane, alcohol-blend of hexane and heptane, benzene-*cyclo*hexane-alcohol, at temperatures between 20° and 50°, have been studied. Experiments with three motor spirits having equal vapour pressures, showed that the greatest increase in vapour pressure occurred on addition of alcohol to the paraffinoid spirit; a smaller increase occured on adding alcohol to the naphthenic spirit; and the least increase when the benzol spirit was used. Tables are given showing the vapour pressure at temperatures between 20° and 50° of the ternary systems alcohol–*cyclo*hexane–water and alcohol–*cyclo*hexane–benzene and the boiling points of all the possible constant-boiling point mixtures, binary, ternary, and quaternary, of the components alcohol, benzene, *cyclo*hexane, and water. In blending hydrocarbons of the paraffin series the vapour pressure can be calculated approximately from the mixture formula. The actual value of the vapour pressure is higher than the calculated value and the difference increases with increasing differences between the vapour pressures of the components. The difference between the actual and calculated values is greater when a naphthene and still greater when an aromatic hydrocarbon is dissolved in a paraffin. When naphthene and aromatic hydrocarbons are mixed the actual value far exceeds the calculated value and in the case of benzene and *cyclo*hexane a mixture of maximum vapour pressure is obtainable. W. N. Hoyte.

Effect of water on aniline points [of hydrocarbons]. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, 12, 89—91).—The water content of an approximately dry sample of aniline was determined from observations on the freezing point of the sample. Using this sample as starting point, aniline of known water content, varying from 0·07% to 1·75%, was used for the determination of the aniline point of heptane, *cyclo*hexane, and a commercial motor spirit. The authors find that 1% of water in the aniline causes a rise in aniline point of 6·2° for heptane and the spirit examined and 5·6° for *cyclo*hexane. They suggest the use of a similar method for determining the water content of petroleum spirit. W. N. Hoyte.

See also pages 399, **Determination of carbon disulphide and hydrogen sulphide** (Hegel). 404, **Ammonia synthesis from coke-oven gas** (Claude); **Ammonium sulphate** (Bateman). 405, **Determination of carbon monoxide in hydrogen** (Schuftan). 410, **Sulphur in producer-gas** (Brown).

Patents.

Artificial fuel. L. L. Summers (U.S.P. 1,576,253, 9.3.26, Appl., 29.9.21).—Raw coal is extracted with a solvent, and the carbonaceous residue, after compression and distillation, is covered with the extract. W. T. K. Braunholtz.

Pulverisation of coal. H. Kreisinger, Assr. to Combustion Engineering Corp. (U.S.P. 1,576,335, 9.3.26. Appl., 16.1.25).—Coal is pulverised in a mill in a system including a separator and air as carrier, and is preheated to such a degree that its sensible heat suffices to dry the coal during pulverisation in the presence of the carrying air. W. T. K. Braunholtz.

Carbonisation of coal. Illingworth Carbonization Co., Ltd., and S. R. Illingworth (E.P. 248,077, 27.11.24).—A succession of blows are struck by means of hammers or by the coal-feeding tubes on the upper part of a vertical retort during carbonisation. The hammers can be connected with the coal feed valve in order to control the rate of striking. A coal with 22% of volatile matter carbonised at 600° produced a fuel of apparent density 1·09 when the retort was vibrated as compared with 0·832 with no vibration. A. C. Monkhouse.

Cooling of coke. Illingworth Carbonization Co., Ltd., and S. R. Illingworth (E.P. 248,078, 27.11.24).—The sensible heat of the coke is used to dry the raw coal which is to be carbonised. The coke is discharged into a cooling wagon containing a measured amount of non-caking coal and the wagon is rotated; caking coal is then added and after further mixing the contents of the wagon pass to the coke cutter and are then discharged on to a screen where the smaller coal and breeze pass into a hopper and thence to the disintegrator and retort hoppers, whilst the coke is conveyed to the coke store. The heat in low-temperature coke is sufficient to dry the corresponding charge of coal from 10% to 3% of moisture. The proportion of caking to non-caking coal can be adjusted and the process made continuous by combining with the cooler a conveyor working in a closed chamber beneath the retorts. A. C. Monkhouse.

Destructive distillation [of maize cobs etc.]. F. C. Atkinson, Assr. to J. W. Garland, Inc. (U.S.P. 1,572,510, 9.2.26. Appl., 20.8.19).—Maize cobs or like waste material are crushed, extracted with water at a pressure of 3 atm. for 3 hrs., dried, and distilled out of contact with air to 440°. Charcoal of good quality free from mineral matter is obtained as a residue. The distillate separates into two layers on cooling, the upper being an aqueous layer containing acid substances. The lower oily layer is fractionated when cresols distil at 150—250° and are purified by treatment with sodium hydroxide solution. A creosote similar to beech wood creosote is obtained. T. S. Wheeler.

Decolorising and defecating product. G. J. Esselen, jun., Assr. to United Fruit Co. (U.S.P. 1,575,561, 2.3.26. Appl., 6.10.22).—The product consists of a charcoal derived principally from the pith of the sugar-cane, intimately associated with an alkaline material. R. A. A. Taylor.

Oven for the distillation of solid material. O. Lasche and C. Geissen, Assrs. to Kohlenveredlung G.m.b.H. (U.S.P. 1,576,916, 16.3.26. Appl., 6.3.35).—The oven comprises an outer cylindrical shell and an inner, thick-walled, hollow body of refractory material pierced with holes. There is an annular space between the shell and inner body, and heating gas is introduced axially into the latter. W. T. K. Braunholtz.

Manufacture of water-gas. G. Patart (F.P. 599,589, 25.5.25).—A mixture of oxygen, steam, and pulverised fuel is blown into a generator of the usual type. A. B. Manning.

Flameless combustion of vapours and gases by means of catalysts. H. Schimmel (G.P. 424,209, 7.10.24).—The vapours or gases are forced under pressure in extremely fine streams through the catalyst, whereby they are completely burnt. A. B. Manning.

Using low-compression fuels [in internal-combustion engines]. T. Midgley, jun., Assr. to Gen. Motors Corp. (U.S.P. 1,575,436–444, 2.3.26. Appl., 18.9.23).—A fuel which detonates at low compressions in internal-combustion engines is mixed with a volatile compound of selenium tellurium, arsenic, antimony, phosphorus, cadmium, bismuth, titanium, or tin, in order to diminish the tendency to detonate. W. N. Hoyte.

Treating oil-bearing shales. N. H. Freeman (E.P. 248,014, 4.9.24).—The finely crushed shale mixed with lime is subjected to a temperature of about 180° for 1 hour; by this means the shale oil is rendered soluble. This is followed by extraction of the treated shale with kerosene, not volatile below 150°. The shale is separated from the oil either by a centrifuge or a filter. The shale oil is then separated by distillation from the kerosene, which is used again. It is claimed that the addition of lime preliminary to the heat treatment lowers the sulphur content of the resulting oil. W. N Hoyte.

Production of light hydrocarbons. Allgem. Ges. für Chem. Ind. (E.P. 244,697, 6.5.25. Conv., 19.12.24).—A liquid catalyst is prepared by the action of anhydrous aluminium chloride at 30—90° on a purified oil of high boiling-point such as gas oil. The purification is effected by washing the oil with liquid sulphur dioxide, or methyl, ethyl, or amyl alcohol, or by treatment with sulphuric acid or silica gel. About 10% by volume of the catalyst is introduced into the cracking stock maintained at a suitable temperature (about 200°). Cracking proceeds smoothly, and the process is characterised by the small quantity of gas and the large proportion of saturated hydrocarbons formed. W. N. Hoyte.

Cracking of liquid hydrocarbons. V. L. Oil Processes, Ltd., O. D. Lucas, and E. L. Lomax (E.P. 248,115, 9.12.24).—The process is based on the observation that the percentage of cracked spirit obtainable in one operation is increased by maintaining a low concentration of lighter oils, produced by cracking, in the residues. The oil is preheated and pumped under a suitable pressure through a pipe still where it is heated to cracking temperature. It passes to a reaction vessel maintained at cracking temperature. The lighter oils produced pass to a refluxing chamber situated above the reaction chamber and maintained at a temperature at least 200° lower than that of the latter chamber. The condensed oil from this chamber passes back to the reaction chamber, either being sprayed in at the top or introduced at the bottom with the main charge. The vapours from the reflux chamber pass through an expansion chamber to a dephlegmator. The vapours from the dephlegmator pass via a vapour refining apparatus to the main condenser. The liquid deposited in the expansion chamber and dephlegmator runs through the preheater above mentioned to the residue tanks. The liquid residue from the reaction chamber passes through coils in the base of the dephlegmator to the expansion chamber. Suggested temperatures and pressures are as follows:—Oil exit from pipe still, 600°; reaction chamber, 450—550° and 600—1000 lb. per sq. in.; refluxing chamber, 360—400° and 600—1000 lb. per sq. in.; expansion chamber, 75 lb. per sq. in. and 350°; dephlegmator, 75 lb.

per sq. in.; outlet for gas and vapour from the column, 25 lb. per sq. in. W. N. Hoyte.

Fractional distillation of hydrocarbons. C. de Ganahl and F. C. Koch (E.P. 248,186, 16.3.25).——The apparatus comprises substantially a pipe still and a dephlegmator. When sharp fractionation is desired the preheated charge enters the dephlegmator at a suitable point, the light fractions are removed, the residue is pumped through the pipe still into a vaporising chamber, and the vapours from this pass up the dephlegmator where they undergo fractionation. The vapours from the top of the dephlegmator pass via the preheater mentioned above through a condenser, thence a portion of the condensate is run to stock, and the remainder is pumped to the top of the dephlegmator to assist fractionation. The liquid deposited in the vaporising chamber is pumped to a second unit similar to the one described, and is introduced as before into the dephlegmator at a suitable point. Sufficient units are used to give the degree of fractionation desired. When only rough fractionation is required the preheated charge is pumped direct to the pipe still instead of being introduced into the dephlegmator. W. N. Hoyte.

Operation of oil stills. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,574,547, 23.2.26. Appl., 10.8.23; cf. E.P. 240,355; B., 1926, 120).—Cracking stills are heated by hot products of combustion from a furnace and a portion of the hot waste gases is passed again over the oil heating surfaces, in admixture with fresh hot gases from the burners. W. N. Hoyte.

Pressure still for oil refining. D. Pyzel, Assr. to Simplex Refining Co. (U.S.P. 1,574,935, 2.3.26. Appl., 13.3.22).—In a steel pressure still for cracking oil a number of thermocouples are provided to indicate the temperature of various portions of the still. Any deposition of carbon causes local overheating owing to its insulating properties, and this is shown by the corresponding thermocouple. The still is then cooled for cleansing purposes. T. S. Wheeler.

Cracking oils under pressure. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,575,031, 2.3.26. Appl., 1.4.24).—The oil is raised to cracking temperature under pressure, the resulting vapours are condensed fractionally under pressure, and the amount of condensation is increased or decreased with increase or decrease of pressure. Uncondensed vapours escape through a fixed orifice and are condensed at atmospheric pressure. W. N. Hoyte.

Apparatus for condensing [hydrocarbon] vapours. E. W. Isom and J. E. Bell, Assrs. to Sinclair Refining Co. (U.S.P. 1,575,269, 2.3.26. Appl., 27.5.21).—A vessel fitted with cooling coils is kept about half full of the liquid condensate, and the mixture of hydrocarbon vapours and gases under treatment is introduced through nozzles situated below the liquid level so as thoroughly to agitate the liquid. W. N. Hoyte.

Securing temporary intimate contact [without emulsification] between immiscible liquids. L. D. Jones, Assr. to Sharples Specialty Co. (U.S.P. 1,575,116, 2.3.26. Appl., 3.9.21).—An apparatus for causing intimate contact between immiscible liquids without emulsification, *e.g.*, for treating viscous lubricating oils with sulphuric acid or alkali-refined oils with water, consists of a rotating vertical cylinder to the bottom of which the interacting liquids are introduced. A number of superposed horizontal discs arranged centrally in the cylinder may be used, so that as the liquids rise in the cylinder the lighter passes in a thin film within the heavier around the edges of the discs. Rings may be fitted to the inside of the cylinder, or a combination of peripheral rings and central discs may be used. At the top of the cylinder the liquids are centrifugally separated and withdrawn. T. S. Wheeler.

Refining and decolorising bitumens. G. W. Acheson (U.S.P. 1,574,742, 2.3.26. Appl., 8.10.24).—The bitumen is treated with a solid reflocculated absorbent material (cf. U.S.P. 1,563,713, B. 1926, 224) in conjunction with an acid reagent. The refined bitumen is separated from the sludge and is then treated with an anhydrous oxide to decolorise it. W. N. Hoyte.

Method of treating particles of matter [coal]. B. E. Eldred and R. N. Graham, Assrs. to Nat. Carbon Co. (U.S.P. 1,578,274, 30.3.26. Appl., 10.3.22).—See E.P. 199,753; B., 1923, 812 a.

Production of coke and tar from coal. F. Seidenschnur (U.S.P. 1,578,376, 30.3.26. Appl., 2.10.24).—See E.P. 228,494; B., 1925, 700.

Gas off-take pipes for fuel distillation retorts or coke ovens. W. Mueller (E.P. 245,728, 12.10.25. Conv., 8.1.25).

Sulphur from gases containing hydrogen sulphide (G.P. 423,395).—See VII.

Bituminous emulsions (E.P. 248,859).—See IX.

III.—TAR AND TAR PRODUCTS.

Patent.

Fractional distillation of hydrocarbons (E.P. 248,186).—See II.

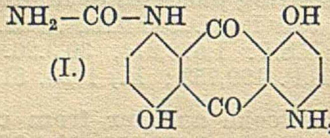
IV.—DYESTUFFS AND INTERMEDIATES.

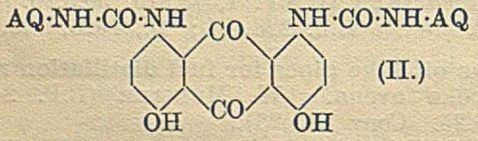
Vat dyes and some recent developments. R. F. Thomson (J. Soc. Dyers and Col., 1926, 42, 124—126).

See also A., April, 403, **Colour and chemical constitution** (Moir). 407, **Nitro-derivatives of 1-hydroxy-3-methylanthraquinone and related products** (Eder and Manoukian). 408, **Anthraquinonylamides of substituted anthraquinonecarboxylic acids** (Rollett). 412, **Benzene-4-hydrazo- and -azo-pyridine derivatives** (Koenigs and others). 415, **Synthesis of substitution derivatives of indigotin** (Overmyer). 420, **Determination of nitro-compounds** (Kolthoff and Robinson).

PATENTS.

Manufacture of new anthraquinone dyestuffs possessing affinity for acetyl [cellulose acetate] silk. Brit. Dyestuffs Corp., Ltd., W. H. Perkin, and C. Hollins (E.P. 248,858, 16.12.24).—New carbamides of the anthraquinone series containing two or three anthraquinonyl groups linked together by the chain —NH·CO·NH—, and having affinity for cellulose acetate silk, are obtained by condensing α-anthraquinonylethyl carbamate or α-anthraquinonylcarbamic chloride with equimolecular proportions of α-aminoanthraquinones or derivatives, preferably those having two amino-groups in α-positions, such as 1 : 4-, 1 : 5-, or 1 : 8-diaminoanthraquinones, diaminoanthrarufin, or diaminochrysazin. For example, 13·5 kg. of diaminoanthrarufin when heated with 14·5 kg. of α-anthraquinonylethyl carbamate (or an equivalent proportion of the corresponding carbamic chloride) at the m.p. of the mixture for about ½ hr., form a product which dyes cellulose acetate silk a brown shade and is probably the monocarbamide (I.) :—


(I.)


(II.)

(AQ=an anthraquinonyl residue).

Diaminochrysazin and α-anthraquinonylethyl carbamate condensed in the same way give a product which is probably the dicarbamide (II.) and which gives a steel-grey shade. A. Coulthard.

Manufacture of anthraquinone derivatives. H. Dodd, W. C. Sprent, and United Alkali Co., Ltd. (E.P. 248,874, 29.12.24).—Iron and nickel have a specific action in directing the condensation of aromatic amines with quinizarin. In an iron vessel the simple condensation product is obtained free from the double one, whilst in a nickel vessel the double condensation takes place if iron is absent. For example, 60 g. of quinizarin, 170 g. of dry *p*-chloroaniline, and 30 g. of boric acid are stirred and gently heated in an iron vessel. After gradually raising the temperature to 130° for about 2 hrs., the excess of *p*-chloroaniline is removed by dilute hydrochloric acid. The residue is the simple condensation product, $C_{14}H_6O_2(OH)\cdot NH\cdot C_6H_4Cl$, in 80% yield. 60 g. of quinizarin, 200 g. of *p*-chloroaniline, and 30 g. of boric acid, heated for 2 hrs. in a nickel vessel at a temperature not above 150°, give a yield of 81 g. of the double condensation product as a blue powder. The two dyes after sulphonation give totally different colours on wool. A. Coulthard.

Manufacture of dyestuff intermediates [2-chloroanthraquinone]. J. Thomas, and Scottish Dyes, Ltd. (E.P. 248,411, 8.9.24).—2-Chloroanthraquinone made by heating *p*-chlorobenzoylbenzoic acid with sulphuric acid of 85—90% strength is only slightly soluble in the residual acid and crystallises out on cooling. The acid filtrate may be revivified by the addition of oleum or sulphur trioxide and used repeatedly. For example, 100 pts. (by weight) of *p*-chlorobenzoylbenzoic acid are dissolved in 1000 pts. of 90% sulphuric acid. The mixture is well stirred and heated to about 170°, kept at this temperature for about 1 hr., and then cooled. The 2-chloroanthraquinone crystals are filtered off and after washing and drying have m.p. 206—208°. 850 pts. of the acid filtrate are then mixed with 150 pts. of 65% oleum and the process is repeated, and so on, until the accumulation of impurities renders the filtrates unfit for further use. A. Coulthard.

Preparation of aminodianthrimides. Brit. Dyestuffs Corp., Ltd., H. M. Bunbury, and R. Robinson (E.P. 248,866, 19.12.24).—Anthraquinonemono-oxamic acids (obtained in good yields direct from diaminoanthraquinones and oxalic acid) of the type $NH_2\cdot AQ\cdot NH\cdot CO\cdot CO_2H$, where AQ=a bivalent anthraquinone residue, condense with halogenated anthraquinones to form mono- and dianthrimides. During the condensation the oxalyl group is converted wholly or partly into the formyl group by loss of carbon dioxide. Hydrolysis of the dianthrimide derivative removes the oxalyl (or formyl) group, leaving an aminodianthrimide. For example, 4 : 5′-diamino-1 : 1′-dianthrimide is prepared by heating 11·4 pts. of 4-benzamido-1-chloroanthraquinone, 10 pts. of 1 : 5-diaminoanthraquinonemono-oxamic acid, 3 pts. of fused sodium acetate, 0·3 pt. of copper powder, and 100 pts. of nitrobenzene for 15 hrs. at a gentle boil. The 5 : 5′- and 4 : 5′-diamino-1 : 1′-dianthrimides are prepared in a similar way. They are valuable intermediates for vat dyestuffs. A. Coulthard.

Soluble products from vat dyes (E.P. 247,787 and 248,802).—See VI.

Phenolphthalein (U.S.P. 1,574,934).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Swelling] action of caustic soda on cellulose pulp. J. D'Ans and A. Jäger (Kunstseide, 1925, 7, 252—256).—The swelling action of caustic alkalis in the presence or absence of neutral salts on bleached and unbleached soda- and sulphite-celluloses was determined by observation of the increase in weight of the cellulose in the form of paper after immersion in the alkaline solutions and drainage from excess of liquor, correction being made for the density of the liquor. The maximum swelling effect was produced by solutions containing 11—12% of caustic soda and 15% (wt./vol.) of caustic potash, the swellings being about 11% and 5·5% respectively. Swelling is considerably reduced by the presence of neutral salts and other substances, *e.g.*, sodium chloride, sodium carbonate, ammonia, alcohol, sugar, tannic acid, and gelatin. For example, a 12% solution of caustic soda containing 10% of sodium chloride produces a swelling of only 4·9%. Swelling is also reduced by rise of temperature of the caustic liquor, but this effect is less evident with highly concentrated alkaline solutions. For example, a 10% solution of caustic soda produces swellings of 14·6 and 6·6% at temperatures of 13—16° and 23—25° respectively, whereas under similar conditions the swellings produced by an 18% solution of caustic soda are 6·6% and 5·9%. Dry cellulose liberates 2·6 cal. per g. when moistened with 6% of water. Over a period of 20 hrs. alkali-cellulose saturated with 17% caustic soda liberates 0·417 cal. per hr. per g., whereas under similar conditions during 24 hrs. dry alkali-cellulose (as prepared for the manufacture of artificial silk) liberates 0·27 cal. per hr. per g., the rate of liberation of heat not being affected by the presence of oxygen. Alkali-cellulose heated to 60° and then cooled liberates no heat on keeping (cf. B., 1925, 984). A. J. HALL.

Determination of the degree of swelling of cellulose by Schwalbe's hydrolysis number method. G. BERNARDY (Z. angew. Chem., 1926, 39, 259—261).—As usually carried out this method (Schwalbe, B., 1910, 689) yields irregular results. Reproducible results are obtained if care is taken in neutralising the acid after the first (hydrolytic) boil. An excess of even a few drops of the concentrated sodium hydroxide results in the cuprous oxide, subsequently produced by reduction of the Fehling's solution, being brownish-yellow instead of red, and in such cases high results are obtained. The apparatus is modified to facilitate exact neutralisation. A. GEAKE.

Scientific foundations of the viscose process. E. HEUSER and M. SCHUSTER (Cellulosechem., 1926, 7, 17—55).—The chemical mechanism of the process of ripening of viscose as originally established by Cross and Bevan is upheld, and the data on which recent investigators have based a purely colloidal explanation are shown to be unreliable owing to imperfect purification of the xanthates examined. Viscose is the sodium dithiocarbonic ester of cellulose or alkali-cellulose. Preparations precipitated by alcohol contain variable amounts of sodium hydroxide depending on the conditions of dilution; xanthates precipitated and purified with sodium chloride are free from sodium hydroxide. Ripening is simply a process of hydrolysis of the ester with elimination of sodium hydroxide and carbon disulphide, these products then undergoing secondary reactions, principally with formation of sodium trithiocarbonate. The primary product of complete xanthogenation is the C_{12} xanthate in the ratio $(C_6H_{10}O_5)_2 : CS_2 : NaOH$; no evidence could be obtained of the formation of a C_6 xanthate nor of any higher degree of xanthogenation. When the proportion of carbon disulphide is restricted to the amount theoretically necessary to produce the C_{12} xanthate (23·6% of the cellulose), there is difficulty in preparing a homogeneous solution and the stability of the viscose does not become normal until the C_{24} stage has been reached. A properly soluble viscose is not obtained unless the concentration of the mercerising sodium hydroxide has been sufficient to produce the alkali-cellulose compound $(C_6H_{10}O_5)_2,NaOH$; in practice a slightly higher concentration is desirable. The same law holds in the case of the hydroxides of the other alkali metals, lithium, potassium, and rubidium, all of which give "viscoses" if employed at equivalent concentrations. The solvent powers of *N*-solutions of the hydroxides of the alkali metals decrease as the atomic weights increase. For comparative purposes the products of xanthogenation may conveniently be dissolved in *N*-sodium hydroxide. For instance, a rubidium xanthate gave an imperfect viscose when dissolved in *N*-rubidium hydroxide but a perfectly smooth viscose when dissolved in sodium hydroxide; the latter viscose, however, yielded a pure rubidium xanthate when neutralised and precipitated by salt. Viscose solutions are most stable against coagulation when they contain 8% of free sodium hydroxide; this also corresponds with the condition of minimum viscosity. The effect of by-products (salts) and of higher concentrations of sodium hydroxide is to hasten coagulation. The complexity of the cellulose molecule, or its degree of dispersion, as measured by the viscosity of the regenerated product in cuprammonium, undergoes a quick change after the immersion in the mercerising liquid, the viscosity falling in 6 hrs. from 75 to 25. During the ageing of the alkali-cellulose the fall in viscosity proceeds more slowly to 12·3 after 3 days. During the xanthate reaction another quick drop in viscosity to 8·3 is recorded, after which, during the ripening of the viscose, very little change in the cellulose complex can be detected. The changes in the viscosity of the viscose itself are initially influenced by colloidal physical conditions. The dispersion of the cellulose xanthate requires an appreciable time or vigorous mechanical treatment. The viscosity of freshly prepared viscose is fairly high and falls, as dispersion becomes more complete, to a minimum after 24—48 hrs. Subsequently there is a steady rise in viscosity during ripening, attributable to loss of ester groups from the xanthate and accumulation of saline by-products in the solvent; the rise in viscosity immediately before coagulation is very rapid. J. F. BRIGGS.

Determination of carbon disulphide and hydrogen sulphide in gaseous mixtures [from decomposition of viscose] K. HEGEL (Z. angew. Chem., 1926, 39, 431).—The mixture of gases, containing carbon disulphide and hydrogen sulphide, evolved when viscose (10 g.) is heated with 50 c.c. of *N*-sulphuric acid, is swept by a stream of nitrogen through calcium chloride at 55°, and then through a freshly prepared, dry, fat-free, 1% ether solution of triethylphosphine cooled to —10°. Carbon disulphide is completely absorbed by the phosphine with formation of the double compound, Et_3P,CS_2, which is precipitated quantitatively from ether solutions of triethylphosphine. The crystalline compound is dried at room temperature *in vacuo* before weighing. Hydrogen sulphide is determined by passing the gas mixture through 0·1*N*-iodine solution, the excess of which is titrated with thiosulphate.

L. M. Clark.

Naphthenic acid esters of cellulose. G. Kita, T. Mazume, T. Nakashima, and I. Sakurada (Cellulose Ind., Tokyo, 1926, 2, 31—38).—Naphthenic acid fractions having acid values between 199 and 250, and prepared free from hydrocarbons by suitable treatment of petroleum fractions, were used in the forms of acid anhydrides and acid chlorides (cf. B., 1926, 45). Cellulose esters are obtained with the naphthenic acid anhydrides but the degree of esterification is low. With the acid chlorides in the presence of pyridine, esters containing up to 2·5 mols. of naphthenic acid to 1 mol. of cellulose are produced; unmodified cellulose reacts less readily than hydrocellulose. The esters resemble those produced with the higher fatty acids; those with high naphthenic acid content show definite melting points. Naphthenic esters of unmodified cellulose are substantially insoluble, although a portion is soluble in benzene; the solubility depends not only on the naphthenic acid content but also on the condition of the cellulose. The di-naphthenic ester prepared from hydrocellulose is completely soluble in benzene, chloroform, carbon tetrachloride, and ether. The originally insoluble ester also becomes soluble in these media after fusion. Naphthenic esters containing up to 1 mol. of naphthenic acid to 4 mols. of cellulose are obtained by treating alkali-cellulose with the acid chlorides. J. F. Briggs.

Production of straw-board pulp. J. D. Rue and W. Mousson (Paper Trade J., 1925, 81, [15], 52—53, [20], 49; Papier-Fabr., 1926, 24, 169—172).—Pulp made from straw boiled with milk of lime imparts an undesirable odour to eggs packed in it. A product with a much milder odour is obtained by boiling with sodium sulphite, with the addition of sodium carbonate to prevent corrosion of the boiler. The proportions recommended are 1·5 lb. of sodium sulphite, 7·5 lb. of sodium carbonate, and 20 galls. of water to 134 lb. of straw. The temperature is raised to 145° during 2 hrs., and is maintained for 6 hrs. The product contains 80% of cellulose, instead of 70% when lime is used. The cost of chemicals is greater but may be partly or wholly compensated by increased yield and better quality of product. A. Geake.

Removing printers' ink from old paper. K. Stephan (Z. angew. Chem., 1926, 39, 269—270).—The process of Berl and Pfannmüller (B., 1926, 286) is stated to be inefficient and too expensive.

A. Geake.

Tall oil, a by-product of sulphate-cellulose manufacture (Dittmer).—See XII.

Patents.

Manufacture of artificial silk. E. Lunge, and Courtaulds, Ltd. (E.P. 248,043, 20.11.24).—In order to control the ripening of viscose the pipes and pumps conveying the solution from the chambers in which it is prepared to the spinning nozzles, are immersed in baths the temperature of which is controlled. To prevent corrosion of the pumps and hardening of viscose adhering to their moving parts, the bath surrounding them contains, preferably, a 1% solution of sodium hydroxide or carbonate. This is covered with a layer of oil to prevent absorption of sulphur fumes, which would lead to corrosion. At any stoppage, such as week-ends, the viscose may be cooled to such a temperature that ageing practically ceases. A. Geake.

Filters [for viscose]. E. Lunge, and Courtaulds, Ltd. (E.P. 248,044, 20.11.24).—The filtering medium covers the top of a long trough and the filtered liquid is received in a similar trough inverted over the filter. The feed and delivery pipes are at opposite ends of the pair of troughs. The filter is designed especially for viscous liquids, such as viscose; it secures a large filtering surface, all of which is equally and effectively used, and avoids the possibility of stagnation at any part. This latter is inportant in the case of viscose since stagnant liquid will ripen, and the product will then lack uniformity. In the preferred form of construction the troughs consist of channels in two discs bolted together; the channels are zig-zagged so as to be as long as possible, and arranged so as to register throughout their lengths. Either end of the one channel may be connected to the feed pipe, and the corresponding opposite end of the other channel is simultaneously opened to the delivery pipe; the possibility of stagnation is thus further reduced. The filtering medium is a suitable fabric or other material, and may be supported by a perforated plate. A. Geake.

Manufacture of new cellulose compounds [thiourethanes]. L. Lilienfeld (E.P. 248,246, 9.7.25. Conv., 30.5.25. Addn. to 231,801, cf. B., 1925, 955).—Instead of the cellulose xantho-fatty acid derivatives mentioned in the original specification, other compounds of cellulose containing the CSS-group may be brought into reaction with a primary or secondary amine to form new compounds of *N*-substituted thiourethane type. For instance, cellulose xanthate (viscose) or cellulose xanthic acid, or the products which are obtainable from them by the action of oxidising agents, such as iodine, copper salts, potassium ferricyanide, and the like, or products of the reaction between esters of chlorocarbonic acid and cellulose xanthate or cellulose xanthic acid, or the esters of cellulose xanthic acid (*e.g.*, compounds prepared by the action of alkyl sulphates or iodides on viscose) are treated with primary or secondary aliphatic or aromatic amines or hydroxy-amines in the manner described in the original patent. J. F. B.

Manufacture of fibres, tape, etc. from viscose. A. Kämpf (U.S.P. 1,575,210, 2.3.26. Appl., 7.5.23).—Products are formed from completely unripened viscose prepared from completely unripened alkali-cellulose by spinning the unripened viscose in an acid bath having a temperature of about 4°, the bath containing no added organic matter or salts other than those unavoidably introduced by the operation of spinning. J. F. B.

Increasing the durability of lacquers, threads, and other products from cellulose. P. Le Play (F.P. 595,208, 16.3.25).—Resins or rubber or rubber latex are incorporated in cellulose solutions. For example, artificial silk threads resistant to tensile strain and moisture are prepared from solutions of nitrocellulose or cellulose acetate containing rubber or from viscose or cuprammonium solutions of cellulose containing rubber latex. A. J. Hall.

[Manufacture of] cellulose xanthate. P. Moro (F.P. 597,336, 31.3.25).—Alkali-cellulose is pressed free from excess of caustic lye, then disintegrated and mixed with a solution of sulphur in carbon disulphide in the usual type of jacketed mixing machine for a prolonged period at 30—40° until a clear solution is obtained ; after keeping for 12—24 hrs. the product is filtered and spun into artificial silk. Alternatively, the alkali-cellulose is distributed over sieves arranged in one or more towers through which carbon disulphide containing sulphur is drawn by means of a pump which also communicates with a condenser for the recovery of excess carbon disulphide. A. J. Hall.

Separation of vegetable fibres in the manufacture of cellulose. Chem. Fabr. Griesheim-Elektron (F.P. 599,540, 15.6.25. Conv., 16.6 and 3.7.24).—Raw fibrous materials are subjected for one or several hours' digestion with alkali sulphites in presence of dilute acid (sulphur dioxide) at temperatures not below 100°. A. J. Hall.

Treating bagasse for fibre-making purposes. T. B. Munroe (U.S.P. 1,574,254, 23.2.26. Appl., 7.7.24).—Piled bagasse is subjected to an acetic acid fermentation until the fibre bundles are partially resolved, but without deterioration of the strength of the fibres. The fermented bagasse is mixed with a sufficient quantity of fibres derived from wood to assist the drying of the final board ; the mixture is cooked, refined, and separated from the products of the cooking process, a fire-resisting material is incorporated with the pulp, and the mass is formed into a heat-insulating fibre board. J. F. B.

Apparatus for making sulphite liquor. F. J. Babcock, Assr. to Stebbins Engineering & Manuf. Co. (U.S.P. 1,576,705, 16.3.26. Appl., 24.7.24).—In a tower system for making acid sulphite liquor two separate reversible limestone towers and a tank for receiving liquor from either of the towers are combined with means for strengthening the acid liquor in the tank. This consists of a supplementary tank in communication with the first tank for receiving relief gas, and a supplementary limestone tower. Liquor may be circulated through the supplementary tank and tower while gas is supplied to the latter. A. Geake.

Manufacture of safety paper. M. Reinheimer (G.P. 416,302, 13.7.23).—Safety paper is prepared by the introduction of nickeled iron powder into the paper pulp in the hollander. The genuineness of the paper can then readily be tested at any time by an electro-magnetic device. A. B. Manning.

Recovery of soda from solutions [used in paper manufacture]. W. M. Wallace (E.P. 248,864, 18.12.24. Addn. to 217,468).—Complete burning of the calcined ash, issuing from the furnace on a travelling grate as described in the chief patent (B., 1924, 707), is assisted by supplying air from above, in addition to that supplied from below. A. Geake.

Regeneration process for black liquor [from sulphate pulp process]. A. H. White, Assr. to J. E. Alexander and E. G. Goodell (U.S.P. 1,575,473, 2.3.26. Appl., 26.12.24. Renewed 6.11.25).—The black liquor from the sulphate pulp process is concentrated and subjected to destructive distillation ; the resulting solid residue is calcined together with calcium carbonate to form a mixture of calcium oxide, sodium carbonate, and sodium sulphate ; the calcined residue is caused to react with carbonaceous material at a temperature above 300° but below 900° in the absence of air until the sulphate is reduced ; the soluble sodium salts are then recovered by leaching. J. F. B.

Manufacture of chlorinated products from cellulose waste lyes. Königsberger Zellstoff-Fabr. & Chem. Werke Koholyt A.-G. (G.P. 419,815, 11.5.22).—Cellulose waste liquor is chlorinated in an electrolytic cell the anodes of which are constructed of materials which allow of varying overvoltage towards chlorine and oxygen respectively so that chlorination and oxidation may be effected simultaneously. A high degree of chlorination may be effected when electrodes of magnetite are used. Chlorination is preferably carried out at a moderately high temperature, and the anode and cathode are separated by a diaphragm ; or the process may be made continuous by causing the electrolyte to flow from the cathode to the anode, the diaphragm being omitted. For example, from sulphite-cellulose waste liquors chlorinated products containing 25·4—22·7% Cl, easily soluble in water with a brown colour and containing no free hydrochloric acid, are obtained by using a solution of sodium chloride at 40—50° as electrolyte, with a magnetite anode, an earthenware diaphragm, and a graphite cathode, or a graphite anode and an iron cathode. A. J. Hall.

Utilisation of gases from cellulose digesters. Metallbank u. Metallurgische Ges. A.-G. (G.P. 423,198, 7.11.23).—The hot gases containing sulphur dioxide are led through an electrical precipitation apparatus whereby their heat value is utilised and they are freed from organic, particularly aromatic and organic acid impurities, and are rendered suitable for re-use. Successive falls of temperature between the units of the precipitation apparatus allow of the fractional separation of constituents of the impurities. A. J. Hall.

Manufacture of viscose. H. Dreyfus (U.S.P. 1,578,588, 30.3.26. Appl., 28.1.22).—See E.P. 183,882; B., 1922, 748 a.

Dialysing membranes from nitrocellulose (U.S.P. 1,576,523).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Theory of dyeing on wool and silk. P. Pfeiffer and O. Angern (Z. angew. Chem., 1926, **39**, 253—259).—Benzeneazophenol forms crystalline compounds with 1 mol. of phenylalanine and of sarcosine, and benzeneazoresorcinol with 2 mols. of sarcosine. The methyl ether of benzeneazophenol does not form such compounds, and it is concluded that salt-like combination takes place between the amino-group of the amino-acid and the phenolic hydroxy-group. Similar compounds with basic dyes could not be obtained. Sarcosine anhydride (dimethyldiketopiperazine) forms crystalline compounds with 2 mols. of benzeneazophenol, benzeneazoresorcinol, and benzeneazosalicylic acid. Azobenzene and the methyl ether of benzeneazophenol do not form such compounds, and it is probable that molecular combination takes place between the carbonyl oxygen of the amino-acid anhydride and the phenolic hydroxyl. Sarcosine anhydride also combines with *p*-aminoazobenzene, *o*-tolueneazo-*o*-toluidine, *o*-phenylenediamine, *m*-phenylenediamine, and β-naphthylamine, but not with *p*-dimethylaminoazobenzene or dibenzyl-β-naphthylamine. These compounds are generally decomposed by suitable solvents. It is concluded that the dyeing of wool and silk is due, in general, to the formation of molecular compounds, which may, in special cases, be of a salt-like character. If combination takes place only on the surface of the fibre this theory becomes similar to the adsorption theory. A. Geake.

Dyes not fast to ironing. R. Haller (Kolloid-Z., 1926, **38**, 248—253).—An investigation of the nature of the dyeing process when blue and violet benzidine dyes are used on cotton, wool, and acetate silk fibres, with special reference to the reversible colour change (blue to corinth-red) when the dyed cotton is touched with a hot iron. The experiments confirm the view that these dyes form colloidal solutions of different degrees of dispersion, the larger particles colouring cotton fibre blue, and the smaller corinth-red (cf. A., 1921, ii, 28, 576). Solutions with a low degree of dispersion are particularly sensitive to temperature changes or to variations in the medium employed. Thus, in hot dye baths, wool and cotton are dyed red by aqueous solutions, but on cooling the colour on the cotton becomes blue-violet; alcoholic solutions hardly affect wool, but dye cotton a permanent corinth-red. The effect of touching the dyed cotton with heated metal is to increase the degree of dispersion of the dye on the fabric, with change of colour from blue to red. The addition of hydrazine hydrate to Diamine Blue 3R causes a similar change, and the solution will then dye cotton corinth-red. The adsorption of the dyes by fibrous alumina and barium sulphate was also investigated. With 1% Diamine Blue 3R and alumina, a dark blue deposit and a corinth-red solution were obtained; using barium sulphate, selective adsorption was clearly demonstrated. The largest particles, which are colourless, are first deposited, then a layer of smaller violet particles, and lastly a small zone of corinth-red particles. This indicates a fixed relationship between the degree of dispersion of the colouring matter and that of the adsorbent. The surface of the adsorbent plays a decisive part also when fibres are used, for swelling causes inner, micellar surfaces to come into play, the difference in the sizes of these accounting for the different behaviour of different adsorbents. Wool and acetate silk after swelling have larger inner surfaces than cotton. Thus an alcoholic solution of Diamine Blue 3R causes swelling of acetate silk and dyes corinth-red. After saponification with sodium hydroxide, washing the fibre, and acidifying, the colour changes to blue, showing that in saponification the inner structural conditions are changed. The reddening of blue-dyed cotton is also produced by desiccation over sulphuric acid, but to a less extent than by heating. The hypothesis of Justin-Mueller that two forms of the dye exist, one containing water and the other water-free, whilst not consistent with all experimental facts, is applicable to the portion of the dye existing in a low degree of dispersion. The observed colour changes on heating and drying may thus be connected with dehydration and simultaneous increase in the degree of dispersion. C. H. D. Clark.

[Printing] coloured discharges on indigo grounds by means of vat dyes. C. Sunder and R. Solbach (Sealed Note 2171, 22.4.12. Bull. Soc. Ind. Mulhouse, 1925, 91, 755—757. Report by O. Michel, *ibid.*, 757—758).—Coloured discharges on an indigo ground are obtained by printing with a paste containing an indanthrene or helindone dye, Rongalite, anthraquinone (catalyst), and stannous hydroxide (instead of the more commonly used zinc oxide), and then passing the material, after steaming for the discharge of the indigo, through caustic soda, whereby the vat dyes are reduced and fixed. For example, for red discharges, non-mercerised indigo-dyed cotton is printed with a paste containing 70 g. of Helindone Scarlet S, 70 c.c. of glycerin of 28° B. (*d* 1·24), 160 c.c. of water, 200 g. of Rongalite C, 250 g. of a 50% solution of British gum, 40 g. of anthraquinone (paste), 30 g. of Leucotrope W, and 180 g. of 60% stannous hydroxide, then dried, steamed for 4 min., passed during 20 sec. through caustic soda of 19° B. (*d* 1·15) at 75°, washed in cold water, and soaped hot. Superior results are obtained on mercerised cotton, a paste containing 80 g. of Indanthrene Yellow G. powder, 100 c.c. of glycerin, 150 c.c. of water, 360 g. of Rongalite C, 360 g. of a 50% solution of British gum, 60 g. of anthraquinone (paste), 120 g. of Leucotrope W, and 270 g. of 60% stannous hydroxide being used for yellow discharges and a paste containing 100 g. of Indanthrene Blue RS, 40 c.c. of glycerin, 150 c.c. of water, 200 g. of Rongalite C, 260 g. of a 50% solution of British gum, 40 g. of anthraquinone, 60 g. of Leucotrope W, and 150 g. of 60% stannous hydroxide for blue discharges. Michel draws attention to the related later processes of the Badische Anilin- & Soda-Fabrik and the investigations of Haller (B., 1923, 1127 A). A. J. HALL.

PATENTS.

Machines for mercerising fabrics. C. A. GRUSCHWITZ A.-G. (E.P. 247,909, 26.6.25. Conv. 17.2.25).—The usual stenter type of machine for mercerising cotton fabric is provided with a tank through which the stretched mercerised fabric passes in a to-and-fro motion while subject to a constant weft tension and is simultaneously freed from mercerising liquor by means of water sprays. A. J. HALL.

Bleaching textiles by means of oxygen or ozone baths. R. MOHR (G.P. 421,906, 9.9.24 Addn. to 410,106; B., 1925, 541).—The bleaching apparatus described in the chief patent is connected through a control valve with a storage vessel to which excess of pressure liquid is supplied by a special pump. The maintenance of pressure during the process is thus assured by the pressure liquid without a separate supply of compressed air or gas. A. J. HALL.

Wetting-out means [product] for carbonisation, dyeing, etc., in which textile materials containing animal fibres are treated with a mineral acid. CHEM. FABR. POTT & Co., and F. POSPIECH (E.P. 248,814, 12.11.24).—The wetting-out of textile materials with acid liquids is assisted by the presence of comparatively small quantities of the sulphonic acids of naphthalene and tetrahydronaphthalene or their alkyl derivatives together with hydrocarbons, alcohols, or ketones; such assistants are completely stable to acids and are thus superior to those containing the salts of fatty acids. Sodium *iso*propylnaphthalenesulphonate and a mixture of sodium tetrahydronaphthalene-β-sulphonate and hexahydrophenol are suitable products. A. J. HALL.

Dyes and dyeing. [Soluble products from vat dyes.] J. MORTON, J. I. M. JONES, B. WYLAM, J. E. G. HARRIS, J. WILSON, and MORTON SUNDOUR FABRICS, LTD. (E.P. 247,787, 11.8, 8.9, and 16.10.24, and 11.6.25).—Vat dyes in solution or suspension in a suitable organic base, are treated with an alkylsulphuric acid halide in the presence of a metal. The leuco-derivatives thus obtained are, in general, soluble in hot water and alkalis and sometimes also in acids, and may be used for dyeing animal or vegetable fibres or artificial silk. The parent dye may be regenerated on the fibre by treatment with a mild acid oxidising agent, such as acid ferric chloride. The dye may or may not be refluxed with the base and metal and cooled before the addition of the alkylsulphuric acid halide. An acid or an acid salt may be added to the mixture of dye, metal, and base. The process is applicable to indigoid dyes, such as indigo and dichlorodibromoindigo, and to anthraquinone dyes, such as benzanthrone, indanthrone, flavanthrone, pyranthrone, and anthraquinone-acridone dyes. The metal is usually zinc dust, but iron, cobalt, copper, aluminium, cadmium, tin, copper bronze, and a mixture of zinc and iron may be used. Methylsulphuric acid chloride is usually employed, but ethylsulphuric acid chloride is also suitable. Tertiary organic bases are used, usually pyridine; dimethylaniline, quinaldine, and quinoline may be employed, and diluents, such as chlorobenzene or carbon disulphide, may be added. Suitable acidic substances are hydrochloric and acetic acids, zinc and stannous chlorides, sodium bisulphite, and pyridine hydrochloride. In general, cold water is added to the reaction mixture and the product obtained as an undissolved powder. The nature of the primary products is unknown, but there is no evidence that they are alkyl sulphuric esters; that from indigo is a methylpyridinium derivative of indigo-sulphuric acid. Condensation is usually effected at the ordinary temperature, but with flavanthrone a temperature of about 80° is used and the metal is copper or copper bronze powder. A. GEAKE.

Dyes and dyeing. J. I. M. JONES, B. WYLAM, J. MORTON, and MORTON SUNDOUR FABRICS, LTD. (E.P. 248,802, 25.7.24 and 25.5.25).—Stable leuco-derivatives of vat dyes capable of application to textile materials from simple aqueous solutions, the shade being developed by subsequent oxidation, *e.g.*, with ferric chloride or a dichromate, are obtained by treating the leuco-derivatives with phosphorus oxychloride or an alkylphosphoric halide in the presence of a suitable diluent such as carbon disulphide, chlorobenzene, pyridine or other tertiary amine. For example, a dark blue product slightly soluble in water and more readily soluble in dilute caustic soda and suitable for dyeing is obtained when 8 pts. of phosphorus oxychloride are added during 1½ hrs. to 3 pts. of leucoflavanthrone suspended in 8 pts. of pyridine and 25 pts. of carbon disulphide, the mixture is heated for 15 min. at 70°, then shaken with a limited quantity of water, filtered, and the residue washed free from acid. Or a reddish-violet paste containing a stable leuco-derivative of indanthrone is prepared by adding 12½ pts. of leucoindanthrone to a mixture of 30 pts. of methylphosphoric dichloride and 120 pts. of pyridine contained in a closed vessel in which air has been displaced by carbon dioxide, the mixture being maintained at 55° for 30 min. and then diluted to 2500 pts. by addition of water and filtered. A. J. HALL.

Finishing textile materials. RADUNER & Co. A.-G. (G.P. 423,858, 12.5.25).—Textile materials are impregnated with carbamide whereby they acquire a soft moist handle and are rendered partially fireproof; the carbamide may be used in conjunction with various soaps. A. J. Hall.

Treating silk. H. B. Smith (U.S.P. 1,578,933, 30.3.26. Appl., 8.3.23).—See E.P. 212,517; B., 1925, 37.

Degreasing agent [for textiles]. G. Zimmerli Chem.-Tech. Fabr. (E.P. 236,209, 20.6.25. Conv., 27.6.24).

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

Ammonia synthesis from coke-oven gas. G. Claude (Compt. rend., 1926, 182, 877—881).—An illustrated article in which the present position of the process is briefly considered. Hydrogen is obtained from the coke-oven gas by partial liquefaction (cf. B., 1924, 933). Nitrogen is obtained by burning some of the hydrogen in air and is then combined with hydrogen under 900 atm. in the usual Claude apparatus. S. K. Tweedy.

Neutralisation of sulphate of ammonia and notes on manufacture. C. Bateman (Gas J., 1926, 173, 748—751).—Ammonium sulphate prepared by the ordinary methods may, after "whizzing," still contain 0·3% of free acid. Washing in the centrifugal dryer with water did not leave the salt neutral, probably because part of the acidity is due to ammonium hydrogen sulphate. However, as the water did reduce the acidity, the salt was always washed with a minimum of hot water. Experiments were made with various neutralising agents and the best results were obtained with solid ammonium carbonate. After washing the ammonium sulphate in the whizzer, it was dropped into a chamber and mixed with the calculated amount of ammonium carbonate. The resulting salt contained the maximum percentage (21·10) of ammonia. On storing, it did not cake or dissociate No trouble due to pyridine was observed in the saturation process. For the removal of hydrogen sulphide from the waste gases from sulphate manufacture, oxide from the purification of oil-gas may be used. Its high tar content may be reduced by heating it by means of hot waste gases from the sulphate plant. The advantages of the semi-direct process of ammonia recovery, in which acid washers are combined with the ammonia scrubbers, are emphasised. A saving in water for removing the last traces of ammonia from the gas, less steam for the distillation of the liquor (which is less in quantity and more highly concentrated), and less effluent liquor from the still are among the economies effected. R. A. A. Taylor.

Determination of perchlorate in Chile saltpetre. G. Leimbach (Z. angew Chem., 1926, 39, 432).—The formation of the insoluble nitron salt is used as a means of determining perchlorate in Chile saltpetre. 10 g. of the sample are dissolved in a small volume of water and the greater part of the sodium is precipitated as chloride by the addition of excess of concentrated hydrochloric acid. The filtrate from the sodium chloride is concentrated: the solid which separates is dissolved in water, and nitrate and any chlorate present are decomposed by repeated evaporation with hydrochloric acid. A final evaporation to dryness suffices to drive off excess hydrochloric acid. The residual solid is dissolved in water and a little sulphuric acid added. 10 c.c. of nitron acetate (10% solution) are added to the hot solution and the mixture is slowly cooled. After keeping for 1 hr. at the ordinary temperature, the nitron perchlorate is removed, washed with a saturated aqueous solution of nitron perchlorate, and dried at 100°. The method requires modification in the presence of much potassium.

L. M. Clark.

Detection and determination of small quantities of perchlorate in Chili saltpetre and in chlorates. F. L. Hahn (Z. angew Chem., 1926, 39, 451—454).—Small quantities of perchlorate in sodium nitrate solutions can be determined accurately by the colour produced by the addition of a methylene-blue solution containing a considerable quantity of zinc sulphate, the dark blue colour changing to greenish-blue and reddish-violet with increasing perchlorate concentration. By comparison with standard coloured solutions of known perchlorate content, up to 1% of perchlorate can be determined to within 0·05%. For stronger perchlorate solutions, dilution with sodium nitrate is necessary; with less than 0·1% of perchlorate, a strong methylene-blue solution is to be preferred. The process can be completed in about 1 hr., and may be applied to the determination of perchlorates in chlorate solutions.

B. W. Clarke.

Detection of perborates in the presence of borates. L. Rossi (Quim. et Ind., 1926, 3, 4—5; Chem. Zentr., 1926, I., 2219).—An alkali metavanadate or pyrovanadate is reduced in aqueous solution to a vanadyl salt by treatment with sulphur dioxide, excess sulphur dioxide is expelled by boiling, and the solution is acidified with hydrochloric or sulphuric acid; on heating the solution with the borate under test, an orange-yellow coloration is produced if perborates are present, due to oxidation of the vanadyl salt to vanadate or polyvanadate. The presence of 0·01 g. of perborate in 2 g. of the borate can be detected by the reaction.

L. A. Coles.

Deterioration of commercially packed chlorinated lime [bleaching powder]. C. C. McDonnell and L. Hart (U.S. Dept. Agric. Bull. 1389, Feb., 1926, 19 pp.).—The average loss of available chlorine in samples of bleaching powder, packed commercially in various containers, was 1·08% per month, over a period of 12—15 months. The rate of loss is affected by the temperature, averaging 1·44% per month during May to September and 0·61% during September to May; it is only slightly affected by the type and size of vessel in which the material is stored, deterioration being somewhat slower when 5—10 lb. were stored in metal containers than when smaller quantities were stored in waxed fibre vessels. The rate of deterioration was lowest, however, when samples were stored in tightly stoppered, glass bottles. The visible rays of light and the presence of moisture have a slight accelerating effect upon the loss of available chlorine. Practically the whole of the loss of available chlorine is due to its conversion into " chloride " chlorine, changes in the chlorate content of the material and losses of chlorine by volatilisation being slight. L. A. Coles.

Determination of mercuric iodides. J. Sandilands (Pharm. J., 1926, 116, 357—358).—The mercuric iodide is dissolved in cold sodium thiosulphate solution, the mercury precipitated with hydrogen sulphide, and the mercuric sulphide filtered off, washed, dried, and weighed. For the determination of the iodine, the mercury is liberated by the addition of either zinc dust, aluminium, or magnesium and dilute sulphuric acid to a suspension of the iodide in water. The amalgam is filtered off and the iodine in the filtrate determined by Volhard's method.

E. H. Sharples.

Determination of carbon monoxide in hydrogen. P. Schuftan (Z. angew. Chem., 1926, 39, 276—278).—When hydrogen containing carbon monoxide is led over a nickel-aluminium oxide catalyst at 200—300°, methane and water are formed according to the equation $CO+3H_2=CH_4+H_2O$. When the gas is free from oxygen and is saturated with water vapour, the carbon monoxide content may be determined by measuring the volume contraction, which is 3 times the volume of carbon monoxide. If the reaction temperature is too high or the carbon monoxide content is over 10% by volume, an error is caused by decomposition of carbon monoxide to the dioxide and carbon. For 10% of carbon monoxide the temperature should not be above 205°, but may be higher when the carbon monoxide content is lower. A. Geake.

See also A., April, 368, **Silicides of calcium and magnesium** (Wöhler and Schliephake); **Decomposition on heating of sulphides, carbides, silicides, phosphides, silicates, and spinels with alkaline-earth oxides** (Hedvall); **Decomposition of alkaline-earth sulphates** (Zawidzki and others); **Volatility of compound of barium sulphate with sulphuric acid** (Krauss). 370, **Ammonium carbonates** (Bonnier); **Oxidation of combined nitrogen to potassium nitrate and reduction of potassium nitrate to cyanide** (Hofmann and others). 371, **Bettendorf's reaction for arsenic** (Zwicknagl). 373, **Separation of hafnium and zirconium** (de Boer); **Preparation of pure nickel monoxide** (Le Blanc and Sachse). 374, **Analysis of iodides, and standardisation of permanganate and thiosulphate solutions** (Alsterberg). 375, **Test for iodate ion** (Bicskei); **Reactions for nitrate and hydroxylamine** (Blom); **Iodometric determination of arsenic acid** (Ormont). 376, **Titration of thallous salts by potassium iodate** (Berry); **Potentiometric evaluation of titanous chloride solutions** (Kolthoff, Tomicek, and Robinson).

Solubility of calcium sulphate at boiler-water temperatures. Hall, Robb, and Coleman.—See I.

Determination of carbon disulphide and hydrogen sulphide. Hegel.—See V.

Recovery of tin salts from tin-plate scrap. Mantell.—See X.

Patents.

Acceleration of the oxidising or condensing reactions occurring in the preparation of nitric acid from nitrous gases. N. Caro and A. R. Frank (G.P. 424,728, 17.8.24).—When most of the nitrous gases have been condensed the cooled gases are treated with ozone, and the nitrogen pentoxide produced is converted into nitric acid.

A. B. Manning

Apparatus for producing and recovering reaction gases [hydrogen cyanide]. M. J. Brown, Assr. to Pacific R. and H. Chemical Corp. (U.S.P, 1,569,171, 12.1.26. Appl., 26.3.25).—Two liquids which produce gas on mixing, *e.g.*, acid and cyanide solution, are separately stored in reservoirs connected by conduits with a reaction chamber. The liquids are introduced through control valves into a vessel suspended from the sides of the reaction chamber and are mechanically mixed. Hydrocyanic acid gas is immediately evolved and escapes at the top of the chamber into a gas reservoir. The liquid overflows from the vessel and flows from the bottom of the chamber through an outlet chamber externally heated by steam. Further hydrocyanic acid gas is produced which passes up into the main gas exit tube. The main feature of the process is that hydrolysis is prevented by quick evolution of the gas. R. B. Clarke

Heat interchangers for carrying out catalytic gas reactions [ammonia synthesis]. Synthetic Ammonia and Nitrates, Ltd., and F. H. Bramwell (E.P. 248,999, 22.7.25).—In an apparatus for the synthetic production of ammonia the cylindrical reaction chamber is surrounded by a heat exchanger in which the outgoing gases give up their heat to the ingoing gases, the whole combination being surrounded by a pressure-tight casing. To minimise the pressure loss due to friction, the gases flowing in one direction are passed in parallel through a very large number of tubes of small bore, which are made U-shaped and situated in annular passages formed by annular heat-resisting walls alternately depending and upstanding from the top and bottom of the apparatus. The limbs of each U are in adjacent annuli and all the tubes in one annulus are connected to a common header. The other stream of gases passes zig-zag up and down around the U-tubes. B. M. Venables.

Apparatus for the synthesis of ammonia. G. Cicali (F.P. 599,768, 17.6.25. Conv., 16.2.25).—The apparatus comprises three concentric cylinders, the innermost one being the catalyst chamber.

In the annular space between the outer and the intermediate cylinder there is a serpentine tube up which the hot reaction gases pass. The entering gases pass down the outer annular space, up through the next space containing the serpentine tube, where heat exchange takes place, and down through the last space into the catalyst chamber. By-passes permit the admixing of hot or cold gases with the mixture in the last annular space to bring it to the combining temperature. E. S. KREIS.

Fixation of solar or ultra-violet energy for producing catalytic phenomena from solar or ultra-violet rays. E. F. ROUSSEAU (E.P. 226,534, 13.12.24. Conv., 17.12.23).—Salts or solutions of salts of uranium, manganese, iron, or nickel are subjected to the action of solar or ultra-violet radiation, and then added to liquids in which photocatalysis or the sterilisation or activation of ferments is to be effected. J. S. G. THOMAS.

Manufacturing colloidal compounds of arsenic. A. CHWALA, Assr. to ODERBERGER CHEM. WERKE A.-G. (U.S.P. 1,573,375, 16.2.26. Appl., 20.8.24).—The arsenic compound is dispersed in the presence of protective colloids, and the salts of metallic acids, *e.g.*, calcium plumbate, sodium antimonate, with or without the addition of compounds of high molecular weight forming compounds of a complex nature with the insoluble arsenic compound, *e.g.*, protalbinic acid. E. S. KREIS.

Double salts of thiocyanic acid. H. FRIEDENTHAL (F.P. 598,766, 25.5.25).—A solution of a thiocyanate and the bromide or iodide of a volatile base, such as ammonia or hydrazine, is added to an alkaline-earth salt of a volatile weak acid, and the ammonium or hydrazine salt of the volatile acid which is formed is driven off. The double salts produced find application for photographic, disinfectant, and other purposes. A. B. MANNING.

Preparation of cyanides from cyanamide salts. N. CARO and A. R. FRANK (Austr. P. 101,316, 14.8.24).—A powdered mixture of a cyanamide salt and carbon, *e.g.*, commercial calcium cyanamide, together with a flux such as sodium chloride, falls through an arc directly on to a cooled surface under such conditions that the molten material solidifies immediately in as thin layers as possible.
A. B. MANNING.

Manufacture of pure alumina. J. D. RIEDEL A.-G. (G.P. 424,701, 31.12.19).—A solution of alumina containing iron is obtained by decomposing iron-bearing aluminosilicates with ammonium sulphate and lixiviating. From this solution, either directly or after removal of the iron, by addition of ammonium sulphate and ammonia, solid aluminium sulphate or insoluble basic sulphate is prepared. These compounds are converted by the action of ammonium sulphate and ammonia or of ammonia into the solid hydroxide. The pure hydroxide, then obtained directly by the action of steam, or, if iron is present, after a previous purification by the Bayer process, is finally calcined. A. COUSEN.

[Manufacture of] sodium peroxide. VEREIN FÜR CHEM. U. MET. PRODUKTION (Swiss P. 112,963, 16.10.24).—In an apparatus for producing sodium peroxide by burning sodium in one chamber and oxidising the sodium oxide so produced in a second chamber, the sodium oxide is obtained as a porous mass so that it can be easily oxidised, by preventing undue rise of temperature in the first chamber by regulating the amount of air admitted.
E. S. KREIS.

[Manufacture of] sulphuryl chloride. R. M. MCKEE and C. M. SALLS (Can. P. 251,586, 25.6.24; cf. B., 1924, 378).—Chlorine and sulphur dioxide are passed into a mixture of sulphuryl chloride and an inert diluent containing active charcoal in suspension. The mixture is cooled to prevent rise in temperature. E. S. KREIS.

Liquefaction and rectification of air or other gaseous mixture into its components. G. CICALI (E.P. 248,977, 16.6.25).—The purified air is compressed to a few atmospheres in a low-pressure cylinder surrounded by cooling water. One portion of the issuing air passes to a heat exchanger and is cooled by cold, rectified nitrogen passing in the opposite direction. Thence it passes into the bottom of the rectifying column. The other portion is conveyed to water-cooled compressors and compressed to the maximum pressure used; it then enters the intermediate tube of a three-pipe heat exchanger, the outer tube of which contains cold, rectified nitrogen, and the inner tube, cold, rectified oxygen. The air issuing from this exchanger is subdivided. One part is expanded (and employed to do work) and then introduced into the foot of the column; the other part is liquefied, without altering its pressure, in a coil surrounded by liquid nitrogen. The air passing up the column meets a baffling device which causes oxygen to condense at the foot.
R. B. CLARKE.

Continuous separation of sulphur from sulphur solutions. E. LEGELER (E.P. 249,044, 26.11.25. Conv., 6.7.25).—The sulphur solution is introduced at the top of a heated cylindrical vessel, and drips on to a heating device situated about half-way up the vessel. The device is in the form of a segment of a sphere, or conical, and is heated to the boiling point of the solvent and at least to the melting point of sulphur. Sulphur melts on the plate, falls to the bottom of the vessel, and is siphoned off. It is entirely free from solvent. The rising solvent vapours are withdrawn and condensed.
R. B. CLARKE.

Producing purified sulphur. C. MARX, Assr. to UNION SULPHUR CO. (U.S.P. 1,574,989, 2.3.26. Appl., 19.12.22; cf. following abstract).—Sulphide ores, which may also contain free sulphur, are distilled under a high vacuum and the sulphur vapours are condensed in a vacuum receiver. In the case of iron pyrites the reaction, $FeS_2 = FeS + S$, takes place, and any free sulphur originally present is also isolated. R. B. CLARKE.

Producing purified sulphur from impure sulphur or ores containing it in elemental form. C. Marx, Assr. to Union Sulphur Co. (U.S.P. 1,574,988, 2.3.26. Appl., 19.12 22 ; cf. preceding abstract).—The sulphur-containing material is distilled under a high vacuum, and the sulphur vapours are condensed in a vacuum receiver The physical nature of the sulphur may be modified by varying the conditions of temperature and pressure ; thus it may be obtained either molten or as a fine granular powder. R. B. Clarke.

Separation of sulphur from gases containing hydrogen sulphide, and free from ammonia. F. Muhlert (G.P. 423,395, 18.3.24).—The gases are washed with a solution containing a mixture of cupric chloride with ammonium chloride, or an alkali, alkaline-earth, or earth metal chloride, in the proportion of at least 1 mol. of the former to two of the latter. To separate the sulphur from the copper sulphide sludge so obtained, the latter is heated with an amount of the original cupric solution (or similar copper solution) at least equivalent to the cupric sulphide. The solution then remaining, which contains a double salt of cuprous chloride, is oxidised by air, oxygen, or chlorine, and the cupric chloride solution thereby regenerated. A. B. Manning.

Extraction of hydrogen from gaseous mixtures. Soc. d'Etudes et de Constructions Métallurgiques (F.P. 599,895, 27.9.24).—The gases containing the hydrogen are passed along one side of a permeable partition under a slight pressure, whilst an indifferent gas, such as sulphur dioxide or ammonia, which is easily separated from the hydrogen, is led along the other side, under a slightly reduced pressure. E. S. Kreis.

Manufacture of hydrogen by partial liquefaction of gaseous mixtures. G. Claude, Assr. to Lazote, Inc. (U.S.P. 1,579,348, 6.4.26. Appl., 7.1.22).—See E.P. 174,327 ; B., 1922, 463 A.

Recovering soda from solutions (E.P. 248,864).—See V.

VIII.—GLASS; CERAMICS.

Preparation and properties of light-dispersing glasses. G. Schott (Glastech. Ber., 1925, 3, 315—329).—Two chief types of such glasses may be distinguished, (1) transparent glasses with specially shaped or treated surface, (2) glasses inhomogeneous throughout. In the first class are "Holophane" (glass with parallel ribbing on the inner surface), sand-blasted, and etched ware. The matt etch of hydrofluoric acid varies according to the method of procedure and is, in general, less coarse than the sand etch. In the second class are alabaster, opal, and milk glasses. Alabaster glass is formed by devitrification, usually in a potash-lime-silicate of low lime content. Stannic oxide also gives a crystalline deposit on cooling the glass in which it is dissolved, and recently zirconia, titania, and sodium chloride have similarly been used for producing opal glass. Opal effects are obtained by using fluorine-containing materials or by phosphates which form emulsions on cooling. The composition of the disperse phase has not been definitely determined, but aluminium fluoride or sodium silicofluoride has been suggested. Wo. Ostwald gives three causes for light scattering, viz., refraction, reflection, and diffraction, usually two or more being operative at the same time. In the first two cases determinative factors are the refractive index of the glass, the difference of refractive index of the two phases, and the size and number of the particles. Dispersion of light by refraction and reflection results in chromatic resolution, but with a large difference of refractive index of the two phases the colour phenomenon is masked. When the size of the particles is of the order of a wave-length of visible light, diffraction becomes the most important factor, the scattering thereby increasing with the degree of division until the particle size becomes less than that of a light wave-length, when scattering once more decreases. Colour effect due to opalescence is more important than that due to absorption, and it gives usually violet-blue reflection and yellowish-red transmission. Opalescent colours are most marked with a highly disperse phase, though colour effects may occur through refraction when large particles are present. The author has prepared a phosphate glass which in thin layers allows light to pass unchanged in colour. The glass contains relatively few particles which, in consequence of their large size, give good scattering and little absorption. Absorption and reflection losses are small, the following being percentage light loss on transmission through various glasses :—clear glass 5·5, etched glass 7·5, sand-blasted glass 11·2, casing glass (*a*) 9·3 (thin), (*b*) 11·5 (thick), (*c*) 27·0, Jena milk glass (massive) 19·5, Peters glass 25·2. The light-distribution curves of six glasses are given. The efficiency of a light-dispersing glass may be numerically represented by the formula $E =$ scattering power/absorption.

A. Cousen.

Nomenclature and classification of optical glasses. P. Nicolardot (Rev. gén. Colloïd., 1926, 4, 9—13, 39—50).—Methods of classification of optical glasses suggested by Zschimmer, Zschokke, Morey, Peddle, and Montgomery were examined. The schematic diagram of Montgomery (B., 1920, 572) is modified, as a basis of classification, by plotting the logarithm of dispersive power against the index of refraction. Since the region richest in glasses is then cramped a division into three diagrams is proposed, corresponding to visual ($x = n_D$, $y = \log V_D$, $V_D = [n_F - n_C]/[n_D - 1]$), photovisual ($x = n_F$, $V_F = [n_G - n_D]/[n_F - 1]$), and photographic ($x = n_G$, $V_G = [n_H - n_F]/[n_G - 1]$). The method of classifying recently adopted by the firm of Schott is commended. A. Cousen.

Patents.

Apparatus and process for making glass. R. R. Shively, Assr. to B. F. Drakenfeld & Co. (U.S.P. 1,575,514, 2.3.26. Appl., 12.4.24).—The batch is placed within the furnace chamber so as to cover only a minor portion of the floor thereof, at a point near one end. It is heated uniformly over the surface by suitable arrangement of the heating medium, and fusion takes place with a progressive and uniform reduction of the bulk. As the glass is formed it is led away to an outlet at the opposite end of the chamber. A. Cousen.

Glass batch. Corning Glass Works, Assees. of W. C. Taylor (Can. P. 249,088, 26.7.24).—Essentially, the material is free from silica, and contains up to 65% of boric oxide, at least 15% of alumina, up to 10% of potassium oxide, and the oxide of an element of the second group of the periodic system. A. Cousen.

Furnaces. R. Malvos and M. Crozemarie (E.P. 248,036, 19.11.24).—A furnace in which a temperature of 1800° is obtainable from solid fuel, such as hard coal, and which is suitable for burning refractory products such as abrasives, comprises three or more intermittent burning chambers in conjunction with a common regenerator. The burning chambers are charged with the goods through holes in the roof which are then sealed up and the goods not touched till cool again at the end of the burning. Each chamber has several U-shaped furnaces, each with two grates and water troughs forming ash-pits. The furnaces are first fired slowly using cold air, but later hot air from the regenerator is used. After the burning the charge in any one chamber is progressively cooled by drawing out hot gases from the chamber with admission of an equivalent quantity of cold air. B. M. Venables.

Manufacture of refractory material. L. Longchambon and A. Travers (F.P. 599,517, 13.6.25).—The material is obtained by heating to 900—1470° quartzite rock in the presence of 0·5—5% of an alkali salt, whereby the quartz is converted into tridymite or cristobalite, or into a mixture of these. L. A. Coles.

Enamelling metal. W. J. Beck and J. A. Aupperle, Assrs. to Amer. Rolling Mill Co. (U.S.P. 1,578,706, 30.3.26. Appl., 16.11.23).—See E.P. 239,902 ; B., 1925, 962.

Annealing furnaces [lehrs]. C. E. Frazier (E.P. 249,636, 9.2.25).

IX.—BUILDING MATERIALS.

Aluminous cements. H. Eisenbeck (Chem.-Ztg., 1926, 50, 165—167, 202—204, 239—240, 245—248).—The chemical properties and constituents, and therefore to a large extent the value as a building material of aluminous cements, are represented by their position in the lime-silica-alumina system plotted on the Gibbs triangular co-ordinate method. Aluminous cements develop a strength in 24 hrs. equal to that of Portland cement in 28 days ; this rapid hardening is accompanied by a slow setting, which is of great importance in building operations. The hardening process is accompanied by a considerable rise in temperature which enables the cement to be used in cold weather with the minimum of protection. Aluminous cement offers much greater resistance than Portland cement to attack by waters containing alkalis, acids, sulphates, or magnesium salts and is therefore very valuable in sea water work. The price of the cement is double that of Portland cement, but a much leaner mix can be used and the rapid hardening properties permit of economy in time and in initial outlay on forms, shuttering and the like. B. W. Clarke.

Producing a hydraulic cement from copper slag. G. Agde and P. Assmann (Z. angew. Chem., 1926, 39, 271—276).—Slag remaining from the recovery of copper from its alloys with base metals, and containing 42% SiO_2, 13·2% FeO, 1·7% Fe_2O_3, and 23·5% CaO in addition to copper, aluminium, zinc, and magnesium, can be used for the production of a valuable cement by an ordinary oxidising burning with the addition of lime ; the most suitable proportion must be determined empirically. The presence of cupric and cuprous oxides is not detrimental. Phosphoric acid greatly diminishes the strength if more than 0·6% P_2O_5 is present in the finished cement. If 3% of gypsum is added setting begins more slowly but complete setting is accelerated. A. Geake.

Rapid determination of silica and lime in the raw mixture for blast-furnace cement. F. Strumpf (Z. angew. Chem., 1926, 39, 278—279).—Silica is determined by evaporating the powder with concentrated hydrochloric acid, treating the residue with dilute acid, filtering, and weighing. The filtrate is made alkaline with ammonia and then acid with acetic acid. Iron and aluminium are precipitated as phosphates and, without filtering, calcium is precipitated as oxalate. The combined precipitates are purified by redissolving and precipitating, and are finally dissolved in sulphuric acid and calcium determined by titrating the oxalic acid with permanganate. A. Geake.

Curing concrete in a semi-arid climate. C. L. McKesson (Eng. News-Rec., 1926, 96, 452—453 ; cf. Gonnerman and McKesson, B., 1926, 14).—Further tests on concrete pavement laid down during the dry season in California, with a day humidity of 15—40%, show that watering for seven days after laying is sufficient to give the concrete maximum strength at 90 days ; in an emergency, watering for three days would give satisfactory results, both from the points of view of strength and of surface hardness. Protecting the surface from evaporation by dry earth gives a concrete which does not increase much in strength after about 14 days, as the original water used for mixing is not sufficient for the final hydration process on which the ultimate strength depends. Covering the surface with flake calcium chloride gives fairly good results and its use is justified where water is not available. B. W. Clarke.

Wood impregnation by the Cobra process. R. Nowotny (Z. angew. Chem., 1926, 39, 428—431).—Inspection of cross-sections of spruce poles, which have been treated previously for immunity from rotting and wood diseases by injecting an aqueous solution of sodium fluoride, sodium dinitrophenoxide and a little zinc chloride, shows that after several months, complete diffusion of sodium fluoride across the section has occurred. Sodium dinitrophenoxide diffuses more slowly, but after some months, a section shows that a complete annulus of this reagent has spread around the circumference of the pole, and that in general, the depth of penetration is determined by the depth of the injection puncture. In both cases, diffusion takes place more readily in the direction of the "annual" rings than radially. The Cobra method of wood preservation (injection of the preservative solution through boreholes drilled to a depth of several cm. in the neighbourhood of the danger zone) offers advantages over older processes, since it is not necessary to use seasoned poles, as the presence of moisture in the wood renders diffusion more rapid, while the necessity for storing the timber during seasoning is consequently avoided. The danger that water-soluble impregnating materials may be slowly dissolved out of the timber by long contact with moist soil, is negatived by the fact that a pine pole impregnated sixteen years previously with sodium fluoride still had this salt diffused throughout its cross-section. L. M. Clark.

Patents.

Kiln for burning cement and for similar purposes. C. Naske (E.P. 227,444, 6.1.25. Conv., 8.1.24).—An automatic shaft furnace is constructed so that the raw material together with the fuel is blown in from underneath, and the completely burnt material, owing to its increased specific gravity, falls and leaves the furnace at the inlet for the mixture, while the incompletely burnt material leaves at the top of the furnace together with the combustion gases and is collected in a dust chamber, and returned to the furnace to complete the burning process. B. W. Clarke.

Rotary kilns for burning cement, ore and similar materials. Vickers, Ltd., and L. D. Parker (E.P. 248,079, 27.11.24).—In a rotary kiln, a heat-recuperating device is attached to the lower or clinker outlet end of the main rotating cylinder, and is so constructed that the clinker moves in circuitous passages counter-current to the incoming air for combustion. B. M. Venables.

Manufacture of aluminous cements. E. Martin (F.P. 597,978, 30.4.25).—To the initial materials small amounts of alkali salts (as chlorides, sulphates, carbonates, silicates, or aluminates), calcium chloride, calcium fluoride, or cryolite are added, and the mixture is heated to fusion, sintering, or to a temperature at which neither fusion nor sintering occurs. The addition facilitates the formation of hydraulic products which set more rapidly than the untreated material. A. Cousen.

Manufacture of cement. H. Loescher (F.P. 599,104, 4.6.25. Conv., 15.10.24, and 599,507, 12.6.25). —Clinker rich in lime is quenched with water, the powdery portion is separated, and the lumpy portion ground and again mixed with the powder. Blast-furnace slag is added, and the mixture dried, or completely slaked as desired. Alternatively, clinker rich in lime or hydraulic lime may be slaked by means of moist, granulated, blast-furnace slag. A. Cousen.

Manufacture of cement. Soc. d'Exploit. des Proc. Ind. Canalot (F.P. 599,286, 9.6.25).—Cement is manufactured in vertical kilns, a portion of the raw material being moistened with a paste made by mixing raw material with water. L. A. Coles.

Cooling and hydrating cement clinker from rotary kilns. F. Krupp Grusonwerk A.-G., Assees. of C. Mittag (G.P. 424,065, 20.3.25. Addn. to 410,325).—Additional steam for hydrating the clinker in the process described in the chief patent (cf. B., 1925, 633), is generated in the cooling chamber and is added therein to the mixture of air and steam. The waste heat of the gases withdrawn from the apparatus, is utilised in heat-exchangers. L. A. Coles.

Manufacture of lime-silica bricks. A. A. Coutant (F.P. 599,918, 22.4.25).—The mixture of lime, silica, and water used for the production of the bricks is treated with steam in a rotating autoclave in order to ensure complete slaking of the lime. L. A. Coles.

Bituminous emulsions. G. S. Hay (E.P. 248,859, 16.12.24).—The stability of an aqueous bituminous emulsion is improved by mixing therewith, after manufacture, up to 10% of a stabilising agent comprising one or more compounds soluble in or miscible with water and which have the property of lowering the freezing point of the emulsion without causing disruption. Suitable compounds are monohydric, dihydric, or polyhydric alcohols of the paraffin, olefine, or acetylene series, *cyclo*hexanol or hydrogenated phenols ; glucose and other sugars ; dextrin and soluble carbohyrates. B. W. Clarke.

Laying the dust on roads. Allchemin Allgem. Chem. Ind. A.-G., and R. Lichtenstern (F.P. 599,497, 12.6.25. Conv., 7.8.24).—Sodium or potassium naphthenate is added to oil for spraying on roads to lay the dust. Calcium naphthenate is formed by interaction with lime or calcium carbonate in the surface material, and prevents subsequent oxidation of the oil. L. A. Coles.

Product for impregnation of wood. P. R. Chambige (E.P. 249,698, 25.5.25).

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

Effect of the physical properties of ore and coke on the capacity of the blast furnace. T. L. Joseph, P. H. Royster, and S. P. Kinney (Proc. Eng. Soc. W. Pa., 1926, 41, 428—459).—The relations existing between the physical properties of coke and ore and the capacity of the blast-furnace as investigated previously by the U.S. Bureau of Mines (cf. Sherman and Blizard, B., 1923, 436 A; Royster and Joseph, 1924, 470) are correlated with blast-furnace practice. Variation in the combustibility of coke or in the quantity of air blown did not alter the shape of the combustion zone, which, however, was shortened by decrease in size of the coke. The capacity of the furnace is believed to depend on the rate of gasification of carbon at the tuyère, but this rate is limited by the speed of reduction in the upper part of the shaft, which itself is dependent on the degree of contact between the ore particles and the reducing gas. C. A. King.

Diminution and behaviour of the sulphur contained in producer-gas in the Siemens–Martin furnace. J. Bronn (Stahl u. Eisen, 1926, **46**, 78—80).—Experiments were made by adding 2·3% of burnt lime to the coal containing 1·4% S to be gasified in a Morgan gas producer, and the effect on the sulphur content of the slag formed without fluorspar additions in a Siemens–Martin furnace was determined. After addition of the lime 2—4% of metallic iron was found in the producer slag or ash, the iron being the principal carrier of the sulphur. The sulphur content was increased from 1·54% to 2·79% and diminished in the Siemens–Martin slag from 0·27% to 0·188%. The gas producer slag per 100 kg. of coal gasified had retained more than twice the amount of sulphur, and the sulphur in the Siemens–Martin slag was diminished by roughly a third. The sulphur in the bath remained approximately the same, so that the amount taken up thereby from the gases was negligible. A lime addition of 3·5% to the coal produced no further decrease in the sulphur content of the Siemens–Martin slag and freshly slaked lime was not so effective as burnt lime. The Siemens–Martin slag does not take up sulphur as sulphur dioxide but in the form of sulphur compounds not containing oxygen for which it has a much greater affinity. The lime additions in the producer diminished the tendency of the coal to clinker, and the slag was more friable. Even with lignite it is advantageous to add lime. T. H. Burnham.

Pre-melting pig-iron for use in the scrap-pig iron process in Siemens-Martin furnaces. E. Herzog (Stahl u. Eisen, 1926, **46**, 357—361).—Consideration of published data (cf. Ber. Stahlw.-Aussch. V. d. Eisenh., 1925, [98], and Iron Age, 1921, **108**, 403—405), indicates that the use of cupolas for premelting pig-iron for the manufacture of steel by the Siemens-Martin process is more economical than direct melting of pig-iron in the Siemens-Martin furnace and slightly more economical than the use of molten pig-iron direct from a blast furnace. The economy is ascribed chiefly to two factors, (*a*) a shortening in the time required for melting, (*b*) a decreased loss of iron in the slag. Moreover, since most of the silicon is removed as calcium silicate during the premelting process, pig-irons of high silicon content, which otherwise could not be used in a Siemens–Martin furnace, are made available. Premelting cannot be used economically to obtain steels of less than 0·04% sulphur content. L. M. Clark.

Smelting experiments with Fricktaler and Gonzen iron ores. R. Durrer (Stahl u. Eisen, 1926, 46, 328—332).—The experiments were carried out at the Oehler Iron and Steel Works in a one-phase electric furnace with a capacity of 450 kw. The furnace was lined with waste material from the electrodes. The charge was broken up into pieces the size of the fist. Both coke (small) and charcoal were used as reducing materials with spathic iron ore as flux. A record of 20 tappings is given, also analyses of the materials used and the iron and slags produced. In the earlier batches, using Fricktaler ore, and coke, the iron produced had a normal carbon content, the phosphorus passed almost entirely into the iron, and desulphurisation was considerable, the slag containing less sulphur than that introduced by the flux. When charcoal was used the iron showed a lower carbon and sulphur content but was high in silicon (up to 11·5%). On charging with Gonzen ore, which is more difficult to reduce, the silicon content of the iron fell to 3—6%, the phosphorus decreased, and the sulphur increased in accordance with the composition of the ore. Analysis of the slags showed less than 0·7% of carbon when coke was used but 2·2 to 15% with charcoal as reducing material. This is attributed to the higher temperature and consequent formation of calcium carbide. In large-scale practice, the higher temperatures which occur bring about the same effects when using coke as only happen in small furnaces when charcoal is used. A. Coulthard.

Nitridation of ordinary and special steels. L. Guillet (Compt. rend., 1926, **182**, 903—907).—A new method of case-hardening steel, which is being applied technically, consists in heating the material at 500—510° for 6 hrs. in a current of ammonia. Investigations conducted with a number of special steels show that an increase in surface hardness occurs with alloys containing chromium, silicon, molybdenum, and aluminium. The increase in hardness is particularly marked in the case of a steel with a high content of aluminium, and this promises to have technical developments. The surface-hardening effect is attributed to the influence of the added elements in decreasing the diffusion of nitrogen by the formation of nitrides. C. J. Still.

Utilisation of chlorine in recovery of tin and tin salts from tin-plate scrap. C. L. Mantell (Trans. Amer. Electrochem. Soc., 1926, **49**, 87—95. Advance copy).—A historical survey of the development of the chlorine process for detinning tin-plate scrap is given together with a brief discussion of its economics and commercial adaptation. Essentials for successful work are perfectly dry and clean scrap free from grease, varnish, and other organic matter, efficient cooling to keep the temperature in the reaction vessel below 38°, and thorough washing of the residual iron to remove traces of ferric chloride. The cost of the process is almost completely covered by the sale of the detinned scrap, so that the value of the tin or stannic chloride recovered is almost entirely profit. A. R. Powell.

Examination of badly corroded brass condenser tubes. M. von Schwarz (Korrosion u. Metallschutz, 1926, **2**, 9—17; Chem. Zentr., 1926, I,. 2406).—After less than 1 year's use brass condenser tubes from an electricity generating station were badly corroded; the insides of the tubes were covered irregularly with numerous bright patches arranged along the lines caused by drawing and extensive dezincification had taken place. The deposits contained 54·9% ZnO, 21·6% CO_2, 7·9% SiO_2, and 6·5% H_2O, whilst slightly corroded portions of the tube assayed 60·46—62·04% Cu, 36·20—38·83% Zn, 0·33—0·78% Pb, 0·14—0·93% Fe, and a trace of arsenic. The cause of the corrosion appeared to be the use of water containing sodium hydrogen carbonate formed during softening by the permutit process; this decomposed on heating giving the normal carbonate which attacked the β solid solution, without seriously affecting the α constituent. It is recommended that brass condenser tubes should consist entirely of α, should contain a small proportion of arsenic, and should be annealed to obtain a coarsely crystalline structure. A. R. Powell.

Phosphor-bronze. Thermal analysis of the system copper-phosphorus-tin. L. C. Glaser and H. J. Seemann (Z. techn. Physik, 1926, **7**, 42—46; Chem. Zentr., 1926, I, 2049).—The part of the system Cu-Sn-P which is of technical importance, namely, the corner in the neighbourhood of copper, has been investigated thermally and microscopically up to a content of 25% Sn and 8% P. The existence of a ternary eutectic of m.p. 628° at 80·7% Cu, 4·5% P, 14·8% Sn, has been established. The exact position of the saturation limits of the Cu-mixed crystals, could not be determined by this method, owing to the marked liquation phenomena.

A. B. Manning.

Determination of chromium in chrome iron ore. E. Dittler (Z. angew. Chem., 1926, **39**, 279).—Chromium has been determined in 19 samples of chromite from Uskub by the iodometric and permanganate methods. Of the three empirical factors, 0·310, 0·3109, and 0·3165 (theoretical 0·3105), recommended for the latter method, 0·3109 gives results agreeing best with the iodometric method.

A. Geake.

See also A., April, 364, **Electrochemical precipitation of copper from its salts by zinc** (Galecki and Orlowski; Galecki and Kuczynski). 374, **Determinations of carbon in steel based on measurement of gaseous volume** (Hackspill and d'Huart). 376, **Precipitation of magnesium with aluminium hydroxide** (Lassieur); **Time factor in de Haen-Low method of determining traces of copper** (Dunncliff and Ram). 377, **Detection of titanium and uranium** (Tananaev and Pantschenko); **Separation of germanium** (Wada and Kato); **Determination of iron by dichromate method** (Besombe); **Determination of gold dissolved in mercury** (Tammann and Kollmann).

Cement from copper slag. Agde and Assmann.—See IX.

Tin-plate for containers for food products. Serger.—See XIX.

Patents.

Open hearth furnaces. E. Bosshardt (E.P. 247,509, 9.7.25. Conv., 10.2.25. Addn. to 223,870).—In mild steel produced by furnaces of the type described in the main patent (B., 1925, 456), blowholes and blisters are caused by the fact that gas from the generators is forced under pressure into the melting chamber, rendering it difficult to free the molten metal from gas. To overcome this difficulty the furnace is modified so that gas is drawn into the melting chamber by the suction of the furnace chimney, causing a partial vacuum about the molten charge. The furnace heads and gas generators are air-tight, and an adjustable inlet mechanism is provided below the grate to admit the air necessary for combustion of the fuel. Steel with a carbon content of 0·05%, and practically free from oxygen, has been produced during continuous working, while fuel consumption is reduced by 40%. L. M. Clark.

Treating silicon steel sheets. Brit. Thomson-Houston Co., Ltd. From Gen. Electric Co. (E.P. 248,140, 6.1.25).—Rolled steel sheets, with a silicon content of 3—5%, for use in transformer cores, have their magnetic permeability increased by about 15% at the working density of magnetic flux and the hysteresis watt loss decreased by 20% when they are annealed at 800—850° under conditions favourable to the removal of deleterious gases, followed by treatment with a pickling solution of dilute acid, which removes a superficial surface layer of the sheet in addition to the scale. Somewhat less effective is a preliminary pickling treatment with subsequent annealing in hydrogen. L. M. Clark.

Apparatus for reducing iron ore and iron cinders from pyrites to metal and for other purposes. F. L. Duffield (E.P. 248,418, 3.10.24).—Iron ore or pyrites cinder is introduced into and passes through a rotary furnace having the shape of a truncated cone. From this furnace the calcined material is conveyed by means of a shoot into a second similar furnace situated at a lower level in which the ore is reduced. Means are provided for introducing air and fuel into the respective furnaces, which are both mounted so that they can be tilted.

C. A. King.

Heat treatment of steels and other metals. F. B. Dehn. From E. G. Budd Manuf. Co. (E.P. 248,801, 15.9.24).—Excessive grain growth in low-carbon steels and metals of the so-called ductile metal class, occasioned by annealing the metals after cold working, is prevented by maintaining the cold-worked metal for a suitable period at a temperature below that at which grain growth develops and then raising the temperature to the normal annealing point. C. A. King.

Heat treated alloy steels containing zirconium. H. E. Potts. From Electro Metallurgical Co.

(E.P. 248,996, 14.7.25).—The properties of steels containing alloying elements of the class which form structurally free carbides in the fully annealed alloy steels, but other than or in addition to chromium, *e.g.*, manganese, molybdenum, tungsten, vanadium, are enhanced if zirconium (*e.g.* 0·03—0·5%) is incorporated in the alloy, particularly when the Brinell hardness is greater than 300. C. A. King.

Metal chip briquette. G. E. Gail, jun. (U.S.P. 1,574,878, 2.3.26. Appl., 2.2.25).—A briquette adapted for use in cupolas is composed of a dried compacted block of metallic chips with a binder consisting solely of diluted water-glass. The briquette withstands high temperatures without disintegration. L. M. Clark.

Beneficiation of [iron] ores. C. P. McCormack (U.S.P. 1,575,852, 9.3.26. Appl., 5.5.25).—Oxidised iron ores containing aluminium are roasted with sodium carbonate in order to convert a definite amount of the alumina into soluble sodium aluminate which is then removed by leaching. A. R. Powell.

Direct production of steel or malleable iron from ore. F. Burgers and K. Kintzinger (G.P. 424,184, 29.10.21).—Formation of slag and loss of iron by oxidation in the direct production of steel from ore are reduced to a minimum by passing the reducing gases through a shaft connected with the hearth, and packed with an agglomerated mixture of ore and flux. L. A. Coles.

Decarbonisation of ferrochromium. Siemens & Halske A.-G., L. Frederich, and W. Rodenhauser (F.P. 600,079, 25.6.25. Conv., 26.6.24).—Ferrochromium is decarbonised by treatment while heated to 1400—1475° in an induction furnace, with air, air enriched with oxygen, steam, or peroxides. Absorbed nitrogen is removed by treatment with, *e.g.*, titanium, calcium, or aluminium. L. A. Coles.

Alloys and alloy articles. Western Electric Co., Inc. From Western Electric Co., Ltd. (E.P. 248,458, 3.12.24).—Alloys are prepared in such a manner that a supersaturated solid solution is produced. For example a lead alloy containing 2·5% Sb is heated at a temperature below the eutectic fusing temperature, quenched, and aged at atmospheric temperature. Articles of such an alloy may be cast, rolled, or forged and afterwards quenched and aged. C. A. King.

Treating [pickling] metal. W. C. Kronquest, Assr. to Acme Steel Co. (U.S.P. 1,574,823, 2.3.26. Appl., 27.5.25).—Metal is pickled by the electrolysis of a solution of acid using the metal as anode and a material not affected by the acid as cathode. Adsorption of hydrogen by the metal is thereby diminished. C. A. King.

Refining gallium metal. S. Boyer. Assr. to Gen. Electric Co. (U.S.P. 1,576,083, 9.3.26. Appl., 12.8.25).—A film of halogen compound is formed on the surface of molten gallium and the temperature is raised in a vacuum until this film is completely volatilised. A. R. Powell.

Forming a protective coating on metals. Metallbank u. Metallurgische Ges. A.-G. (F.P. 599,974, 22.6.25. Conv., 9.1.25).—Metals such as aluminium, magnesium, or calcium are coated by electrolytic, chemical, or mechanical means with a protective layer consisting of cadmium (preferably), selenium, lead, chromium, tungsten, molybdenum, manganese, cobalt, bismuth, zinc, antimony, or tin, or alloys of these metals. L. A. Coles.

Recovery of zinc from fine ores or other material containing it. Metallbank u. Metallurgische Ges. A.-G. (G.P. 421,384, 11.5.16).—The material, *e.g.*, burnt pyrites containing zinc, is sintered and desulphurised, *e.g.*, in a Dwight–Lloyd furnace, and the zinc is volatilised and recovered by heating the product in a shaft furnace. L. A. Coles.

Welding electrodes. Brit. Thomson-Houston Co., Ltd., Assees. of R. T. Gillette (E.P. 237,902, 23.7.25. Conv., 30.7.24).—See U.S.P. 1,539,810; B., 1925, 598.

Purified sulphur from sulphide ores (U.S.P. 1,574,987).—See VII.

Rotary kilns (E.P. 248,079).—See IX.

XI.—ELECTROTECHNICS.

Recent improvements in the lead accumulator. C. Féry (Bull. Soc. d'Encour., 1926, 125, 49—60; cf. B., 1925, 997).—It is shown that local action and the secondary reaction previously proposed quantitatively account for the discrepancies between the results of Chèneveau's experiments and the results predicted by the author's theory of accumulator discharge. The sulphation accompanying the storage of discharged cells is attributed to the oxidation of the plumbous sulphate formed in the negative grids during discharge: $Pb_2SO_4+O+H_2SO_4=2PbSO_4+H_2O$. An accumulator which will not sulphate is described; the negative grid is placed horizontally underneath the positive so that it is not accessible to atmospheric oxygen or to the oxygen which may be produced at the positive plate by spontaneous decomposition of the Pb_2O_5. S. K. Tweedy.

See also A., April, 362, **Electrochemical behaviour of chromium** (Grube, Heidinger, and Schlecht). 365, **Water and metals under the influence of electrolysis** (Saxon). 366, **Electrolysis of aqueous solutions of hydrogen chloride** (Hollard); **Oxidation of manganous ion to permanganate** (Campbell). 374, **Errors in electro-analysis** (Boehm). 376, **Use of water-alcohol mixtures in electrometric study of precipitation reactions** (Athanasiu). 378, **Simple hydrogen electrode** (Sideris).

Patents.

Electrodes for the purification of gases. Siemens-Schuckertwerke Ges. m. b. H. (E.P.

235,582, 11.6.25. Conv., 12.6.24).—Precipitation electrodes which resist shock and corrosion and which may be sawn and nailed are made from fibrous asbestos materials, slag wool, hair, or wood, with a semi-conducting binder, such as cement, plaster, magnesium oxide or chloride. Graphite or oxides, or other conducting compounds of metals may be added to increase the conductivity. J. Grant.

Electrical precipitation of suspended particles from gases. Siemens-Schuckertwerke G.m.b.H. (Swiss P. 112,962, 14.11.24. Conv., 30.11.23).—The potential and the gas velocity are so regulated that the particles are not precipitated on the electrodes, but pass away with the gases and are subsequently removed through baffled openings in the channel walls. E. S. Kreis.

Electric incandescence lamps. Brit. Thomson-Houston Co., Ltd., Assees. of G. R. Fonda (E.P. 246,516, 25.1.26. Conv., 24.1.25).—The resistance to disintegration of the filament of a gas-filled electric incandescence lamp is reduced by the addition of a few hundredths of one per cent. of carbon monoxide to the inert gas, *e.g.* argon, contained in the lamp bulb, at two-thirds of an atmosphere pressure. J. S. G. Thomas.

Electrodes for electric arc lamps. Gen. Electric Co., Ltd., W. Singleton, and A. Hare (E.P. 248,187, 17.3.25).—Carbon electrodes are heated to about 700° for about 1 hr. in a stream of silicon fluoride vapour, and then cooled. When in use, an adherent, non-oxidisable coating forms on the hot parts of the carbon, and the rate of consumption is reduced. If required, the electrodes may be preheated under reduced pressure. J. Grant.

Composition for storage-battery positive electrodes. R. C. Benner, Assr. to Prest-O-Lite Co., Inc. (U.S.P. 1,572,512, 9.2.26. Appl., 6.11.23).—Compositions containing lead peroxide and lead sulphate suitable for preparing active material for the positive electrodes of storage batteries, especially those of the type which initially do not require to be charged, yield when treated with alkali hydroxide solutions, *e.g.* 20% sodium hydroxide solution, crystalline compounds which serve as bonding compounds and facilitate the application of the material to the grids. The paste obtained hardens well when dried at 65°. T. S. Wheeler.

Electrolyser with stirring device. A. Barth (G.P. 423,801, 15.1.24).—A horizontal diaphragm perforated in the centre has a cylindrical diaphragm fixed on it above the perforation, the joint being watertight. The reaction liquid can be circulated from the cathode space to the anode space, or in the reverse direction, by a pump, the diaphragm being used as a filter. A. B. Manning.

Electrolytic cell. F. Petz, Assr. to Elektrizitäts-A.-G. vorm. Schuckert & Co. (U.S.P. 1,579,138, 30.3.26. Appl., 18.7.24).—See E.P. 222,856; B., 1925, 511.

Process for producing ozone. A. Starke and H. von Wartenberg, Assrs. to Ozonhochfrequenz G.m.b.H. (U.S.P. 1,579,162, 30.3.26. Appl., 2.2.23).—See E.P. 193,422; B., 1923, 986 a.

See also pages 401, **Chlorinated products from cellulose waste lyes** (G.P. 419,815); **Utilising gases from cellulose digesters** (G.P. 423,198). 406, **Cyanides from cyanamide salts** (Austr. P. 101,316). 417, **Preservation of nitrogen compounds in manure** (G.P. 424,667). 419, **Lactose from whey** (E.P. 248,998).

XII.—FATS; OILS; WAXES.

Analysis of soya-bean oil. H. Pfahler (Chem. Umschau, 1926, 33, 65—70).—A freshly prepared sample of soya-bean oil was found to have the following composition: linolenic acid 1·9%, linoleic acid 29·2%, *iso*linoleic acid (including a little *iso*linolenic acid) 24·3%, oleic acid 30·8%, stearic acid 7·0%, palmitic acid 2·35%, glyceryl residue (as C_3H_2) 3·88%, and unsaponifiable matter 0·5%. These numbers do not differ much from those of Baughman and Jamieson (B., 1923, 149 a), and yield a theoretical iodine value of 130, which is in close agreement with the value 132·4 obtained experimentally. To prepare linoleic acid the mixed fatty acids were brominated and the tetrabromide was separated and converted into the methyl ester of linoleic acid. The acid liberated from the ester had iodine value 172·4 (theoretical 181·4). This linoleic acid was again brominated; the resulting tetrabromide (m.p. 113°) corresponded to only 33% of the original linoleic acid. It appears that in debrominating the bromide isomerisation of the acid takes place so that what is re-brominated is a mixture of isomeric linoleic acids leading to a solid bromide and to a liquid one. H. M. Langton.

China wood [tung] oil. H. Wolff (Chem. Umschau, 1926, 33, 70—72).—A critique of the work of Fonrobert and Pallauf (B., 1926, 371), and a restatement of the author's view that the thickening or gelatinisation of tung oil is colloidal in character. The results of molecular weight determinations are regarded as unimportant since such determinations in systems of high molecular weight are uncertain and high values may be due to the formation of molecular aggregates, and not necessarily proof that polymerisation has occurred. Marcusson has shown that tung oil in benzene solution thickens under the action of ferric chloride. A similar effect is produced by hydrochloric acid and sulphurous acid. The author allowed tung oil to thicken to the point of gelatinisation in the one case by heating at 200° and in the other by acting on it with 0·1% of hydrochloric acid solution (*d* 1·125). The viscosity and the iodine value of the two samples of oil were taken from time to time and the results plotted against time. The viscosity curves were nearly coincident, but the curves indicating fall in iodine value became widely divergent. Owing to the relatively long time occupied in the change from α- to β-elæostearin it appears unlikely that β-elæostearin crystals are formed in quite a short time during drying. Moreover the characteristic phenomena associated with tung oil occur equally in strongly diffused light and in darkness where a change into the β-form cannot yet be assumed. H. M. Langton.

Tall oil, a by-product of sulphate-cellulose manufacture. M. Dittmer (Z. angew. Chem., 1926, 39, 262—269).—Tall oil, obtained by hydrolysing the soap separating from black liquor from sulphate-cellulose manufacture, probably contains decomposition products of lignin and oxidised acids, in addition to the resin from the wood. The boiling process hydrolyses esters and the oil contains, therefore, only acids and unsaponifiable substances. A commercial sample from Sweden contained 12·4% of unsaponifiable substances, 30·4% of resin acids, 54·9% of fatty acids, and 2·2% insoluble in petroleum ether. This last substance imparts the dark colour to the oil and may be removed by dissolving the oil in petroleum ether, decahydronaphthalene, hexahydrotoluene, or hexahydroxylene. The unsaponifiable substances consist of a phytosterol ($C_{22}H_{38}O$) and hydrocarbons. The acids were separated into two classes by esterification in the presence of sulphuric acid; the usual procedure with hydrochloric acid cannot be used because the resin acids are slowly attacked by this acid. The resin acids are abietic acids ($C_{20}H_{30}O_2$); pinabietic acid (cf. Aschan, A., 1921, i, 669) is only obtained from the distillate from tall oil. The iodine value (Wijs) of the fatty acids is not constant, but depends on the time of treatment and the excess of halogen taken. This is due to polymerisation of unsaturated acids during the cellulose boiling process and to gradual depolymerisation during the determination of the iodine value; the molecular weight (284) calculated from the saponification value is less than that (369) found cryoscopically by Rast's camphor method. Similar abnormalities were observed when the fatty acids from herring oil were heated with alkali under pressure (Persapol process). The density of the fatty acids was normal (0·907—0·910). By catalytic hydrogenation only stearic acid was obtained, and the saponification value of the original acids also indicated a chain of 18 carbon atoms. No saturated acids were found in the original acids, but oleic, linoleic, and linolenic acids were identified. A. Geake.

Semi-micro-method for rapid determination of the iodine value [of fats and oils]. B. M. Margosches and E. Neufeld (Chem.-Ztg., 1926, **50**, 210—211).—The sample (0·05—0·075 g. for fatty oils, and 0·1—0·2 g. for solid fats) is dissolved in 5 c.c. of alcohol at 50°, 10 c.c. of 0·2*N*-alcoholic iodine solution are added, followed by 10 c.c. of water, and, after shaking for 5 min., the excess of iodine is titrated with 0·05*N*-thiosulphate. The results compare favourably with those obtained by the macro-method. A. R. Powell.

See also A., April, 358, **Equilibria underlying soap-boiling processes** (McBain and Elford). 381, **Hydrogenation of organic substances at high temperature under pressure** (Kling and Florentin).

Patents.

Increasing the durability of pure salad or sweet oils. H. Bollmann, Assr. to M. F. Foster (U.S.P. 1,575,529, 2.3.26. Appl., 28.5.25).—A small quantity of lecithin is added to the oil directly after purification. H. M. Langton.

Preparation of highly viscous lubricating oils. Continentale A.-G. für Chemie (G.P. 423,702, 21.4.21).—Lubricating oils are prepared from mobile animal oils by heating them in a vacuum to about 300°, and maintaining them at that temperature for a long time. A. B. Manning.

Oxidation of linseed oil (Swiss P. 101,617).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Evaluation of white pigments. A. Eibner (Z. angew. Chem., 1926, 39, 261—262).—A criticism of Wolski's paper (cf. B., 1925, 930) on this subject. A. Geake.

Transformation and oxidation of chromium sesquioxide. Blanc and Chaudron.—See A., April, 372.

Apparent paradox of plasticity. De Waele.—See I.

Tall oil. Dittmer.—See XII.

Patents.

Resinous condensation products. Brit. Thomson-Houston Co., Assees. of J. G. E. Wright and W. J. Bartlett (E.P. 235,589, 12.6.25. Conv., 13.6.24).—The initial fusible resinous product from phthalic acid and glycerol is dispersed in a suitable medium, such as ethyl phthalate or benzyl benzoate, and heated to 210—290°. After this treatment the resinous product, recovered, *e.g.*, by precipitation, is non-corrosive to metals, and when further heated passes readily into the final insoluble condition without liberation of phthalic anhydride. D. F. Twiss.

Manufacture of water-insoluble resin soaps. R. F. Uzac (F.P. 595,497, 20.3.25).—An alcoholic solution of magnesium chloride or other alcohol-soluble metal salt is added to an alcoholic solution of an alkali resinate or an alcoholic solution of a resin neutralised with sodium alkoxide. The product is separated from sodium chloride by filtration and the alcoholic solution of a metal resinate thereby obtained is suitable for use as a lacquer. A. J. Hall.

Manufacture of staining solutions and lacquers from soft lignite. C. Ehrenberg, H. Wiederhold, C. Krug, M. G. Holsboer, K. Fischer, and Studienges. f. Ausbau der Ind. (G.P. 422,739, 28.12.21).—Staining solutions for wood are obtained by extracting lignite with acetone or its homologues containing hydrochloric acid; the resulting stains have the advantage over those obtained by extraction with alkaline solutions in being fast to washing with water. Cellulose acetate may be dissolved in the acetone solutions, and the products used as paints or lacquers. A. J. Hall.

Oxidation of linseed oil. P. W. H. J. V. de Wael (Swiss P. 101,617, 2.5.22).—Oxidising gases are led through linseed oil, the temperature of the gases being controlled so that the temperature of the oil does not exceed 300°. After passage through the oil, the gases are led through a condenser to recover vaporised fatty acids. A. J. Hall.

Increasing the durability of lacquers from cellulose (F.P. 595,208).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Utilisation of clotted latex for making sheet [rubber]. H. P. Stevens (Bull. Rubber Growers' Assoc., 1926, 8, 135—136).—The use of sodium silicofluoride (2 g.) with clotted latex (3000 c.c.) prevents further decomposition and enables coagulum and sheet to be prepared free from bubbles. In spite of the retarding effect of sodium silicofluoride the finished sheet rubber from clotted latex vulcanises more rapidly than the normal. D. F. Twiss.

Patent.

Increasing the durability of products from cellulose (F.P. 595,208).—See V.

XV.—LEATHER; GLUE.

Solvents for tannins. Michel-Durand (Compt. rend., 1926, 182, 937—939; cf. A., 1924, i, 477).—Boiling water is a very satisfactory solvent for extracting tannins from wood, and extracts as much tannin-material as combined extractions by means of acetone, alcohol, ether, water, etc. Only a portion of the water-soluble tannins is soluble in acetone. L. F. Hewitt.

Patents.

Drying gelatin. H. W. Luscher, Assr. to United States Glue Co. (U.S.P. 1,575,348, 2.3.26. Appl., 30.3.22).—Filtered air is, in part, passed through an ozoniser and the resulting air is mixed with the remainder which has been heated; the substantially sterile gelatin is submitted to the drying action of this mixture. D. F. Twiss.

XVI.—AGRICULTURE.

Nitrogen-fixing capacity of soils. Winogradsky (Compt. rend., 1926, 182, 907—910).—The nitrogen fixation by soils from widely different sources has been investigated by the method described previously (cf. B., 1925, 328). In each case approximately the same amount of nitrogen (about 10 mg.) was fixed per 1 g. of mannitol consumed. With the most active soil 120 hrs. were required for the complete decomposition of 2 g. of mannitol under the conditions described. L. F. Hewitt.

Determination of the adsorption capacity and "degree of saturation" of soils. E. Bobko and D. Askinasi (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 25, pp. 39; Chem. Zentr., 1926, I, 2045).—In place of the method of Gedroiz (leaching with ammonium chloride solution and analysis of the filtrate), the authors displace the bases originally present by means of barium chloride and then extract the adsorbed barium with 0·05N-hydrochloric acid or, in the case of chalky soils, with N-sodium chloride; barium only need then be determined. The results obtained are somewhat higher than by the ammonium chloride method. Repeated determinations on the same sample proved that the adsorptive capacity of a Podsol soil was not changed by this treatment; five experiments reduced it by about 12% only. On the other hand, the adsorptive capacity of black soils is greatly reduced—about 42% after four experiments. Ammonium chloride decomposes the soil in much smaller amount. C. T. Gimingham.

Injurious action of heavy dressings of calcium carbonate on Podsol soils in connexion with the special character of the biological processes occurring therein. A. Tjulin (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 26, pp. 39; Chem. Zentr., 1926, I, 2043).—Heavy dressings of calcium carbonate indirectly affect the biological processes in the soil unfavourably, influencing the nitrogen cycle in particular. In presence of harmful amounts of calcium carbonate, soluble nitrogen compounds, including nitrites, are produced in greater amounts and denitrification follows more rapidly than under other conditions. The alkalinity and the amount of humus soluble in water are increased in such soils. Addition of ammonium bicarbonate intensifies this action of calcium carbonate, whereas sodium or potassium bicarbonate tends to reduce it. Light "unsaturated" soils are specially sensitive towards calcium carbonate. C. T. Gimingham.

Influence of calcium carbonate on the decomposition of organic substances in soil. A. Tjulin (Trans. Inst. Fertilisers [Russ.], 1923, Lief. 14, pp. 12; Chem. Zentr., 1926, I, 2042).—A comparison of the humus content of a slightly acid soil after treatment with different amounts of dung, green manure, and calcium carbonate. The soils receiving calcium carbonate were poorer in humus than the control soils. The loss in "humic acids," *i.e.*, that part of the humus which is extracted by 2·5% caustic soda solution but not by 0·5% sulphuric acid, was particularly marked. The material soluble in 0·5% sulphuric acid was increased by manuring with calcium carbonate. C. T. Gimingham.

Liming and the nitrate of soils. N. Remesov (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 30, 105—117; Chem. Zentr., 1926, I, 2042).—Changes in the nitrate content of soils manured with different amounts of calcium carbonate and dung have been followed from July to October. On Podsol soils, neither calcium carbonate nor dung alone had much effect, but together they caused a large increase in nitrate. On light sandy soils, dung alone increased the nitrate content more than when calcium carbonate was also added. C. T. Gimingham.

Fertilising action of calcium carbonate. E. Truninger (Landw. Jahrb. Schweiz, 1925, 39, 807—842; Chem. Zentr., 1926, I, 2230).—The addition of calcium carbonate to acid soils has an injurious effect upon the action of simultaneously applied phosphatic fertilisers, but has no effect on their action when the soil has a neutral or alkaline reaction. The different action of calcium carbonate upon acid and non-acid soils is caused by the different adsorptive properties of the two types of soil, calcium carbonate having an injurious effect only when the soil is capable of adsorbing or decomposing it. Calcium carbonate should never be added in conjunction with phosphatic fertilisers to acid soils. The higher the acidity, the greater the risk of injurious effect by the addition of calcium carbonate. The high adsorption of hydroxyl ions by the soil colloids, and the presence of buffer substances protect plants from the harmful effects of excessive alkalinity of the soil solution.

L. A. COLES.

Trials with powdered phosphorite on different soils. K. KALININ (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 30, 118—127; Chem. Zentr., 1926, I, 2041).—In pot experiments with oats, weathered black soils, which showed little response to manuring with nitrogen and potassium, gave increased yields of 50—70% with powdered phosphorite used at the rate of 0·15 g. of phosphoric acid per kg. of soil. On Podsol soils, where potassium and nitrogen give large increases of crop, manuring with phosphorite was ineffective. C. T. GIMINGHAM.

Influence of manuring with calcium chloride on some crops. O. DAFERT and F. G. ERDÖDY (Fortschr. Landw., 1, 69—78; Chem. Zentr., 1926, I., 2041).—In pot experiments with mustard, oats, and beans, addition of calcium chloride increased the green weight but not the dry weight of mustard and oats and hastened the absorption of nutrients by mustard but not by oats. The yield of dry matter was reduced by calcium chloride in the case of beans. There was an increase in the chlorine content of the tissues of all plants.

C. T. GIMINGHAM.

Utilisation of the nitrogen of peat by plants. P. KUPREENOK (Trans. Inst. Fertilisers [Russ.], 1923, Lief. 19, pp. 15; Chem. Zentr., 1926, I., 2042).—Peat (Hochmoortorf) acted as a source of nitrogen and increased the yield of oats when given with potassium phosphate and chloride. Its utilisation was facilitated by addition of calcium carbonate and the introduction of nitrifying bacteria with dung. The plants utilised 20—28% of the nitrogen of the peat. C. T. GIMINGHAM.

Effect of straw on plant growth. R. C. COLLISON and H. J. CONN (New York Agr. Exp. Stat., 1925, Tech. Bull. 114, 35 pp.).—Vegetation experiments with wheat, barley, and buckwheat confirmed the injurious effect upon the plants of addition of straw and other highly carbonaceous plant residues to the soil or sand. The effect is especially marked if the supply of available nitrogen is low and it can be corrected by addition of nitrate. In a heavy soil, injury did not appear until growth had proceeded for several weeks, but in sand cultures, the harmful influence of straw or a water extract of straw was evident soon after germination. Seedlings also showed injury at a very early stage in water culture, even under sterile conditions or when the solutions were changed very frequently. It is generally accepted that abundance of highly carbonaceous material stimulates development of micro-organisms which compete with the plants for the available nitrogen, but this can hardly account for the effects upon quite young seedlings. It is suggested that a second factor is involved—the presence of toxic compounds in the straw. Analyses indicated the presence of salicylic acid, dihydroxystearic acid, and vanillin, all known to be toxic to plants. C. T. GIMINGHAM.

Is the potato an acid-sentitive plant? M. TRENEL (Die Kartoffel, 1926, [3], 28—30; Chem. Zentr., 1926, I., 2042).—Potatoes show vigorous growth in soils of p_H 4·0—6·0. Injury appears below p_H 4·0 and in soils of alkaline reaction.

C. T. GIMINGHAM.

Influence of [seed treatment with] magnesium chloride and mercury-containing materials on plant yield. D. MEYER (Deuts. Landw. Presse, 1924, 51, 461—462; Chem. Zentr., 1926, I., 2044).—The so-called seed-stimulating solutions are discussed in connexion with "seed-pickling" solutions. 2·5% magnesium chloride solution acts unfavourably on the germination of oats, but not of barley. "Uspulun" and "Germisan" have little effect on germination. Pot and field experiments with oats, wheat, and barley gave no increased yields due to treatment of the seed. C. T. GIMINGHAM.

Adherence to foliage of sulphur in fungicidal dusts and sprays. R. W. THATCHER and L. R. STREETER (New York Agr. Exp. Stat., 1925, Tech. Bull. 116, 18 pp.).—Determinations are recorded of the amount of sulphur remaining on apple foliage after treatment with sulphur dusts and "lime-sulphur" sprays. During the first week after treatment, 89—94% of the sulphur applied as dust was lost from the leaves; in the same time, the loss of sulphur derived from "lime-sulphur" spray was from 45 to 75%. Addition of calcium caseinate to the spray fluid slightly reduced this loss. After this rapid mechanical loss during the first days, the decrease in the amount of sulphur adhering proceeded slowly and fairly regularly. The larger loss of sulphur from the dust application is attributed to the large size of the particles as compared with those derived from the decomposition of the polysulphides of "lime-sulphur." C. T. GIMINGHAM.

Concentration of phosphorite ore. I. VERCHOVSKI (Trans. Inst. Fertilisers [Russ.], 1924, Lief. 22, 75 pp.; Chem. Zentr., 1926, I., 2040).—By heating phosphorite ore to 500—600° before grinding, the cohesion between the phosphorite concretions and the matrix is, to a great extent, broken down and the mass becomes more friable. A mechanical

concentration of the phosphorus-containing part of the mineral can thus be obtained.
C. T. Gimingham.

Determination of perchlorate in Chile saltpetre. Leimbach. Hahn.—See VII.

Patents.

Fertiliser containing urea. Comp. de l'Azote et des Fertilisants S.A. (F.P. 600,016, 23.6.25. Conv., 26.6.24).—A mixture of calcium cyanamide with a solution of a salt, *e.g.*, potassium sulphate, containing an acid radical capable of precipitating calcium, but in quantity insufficient to precipitate the whole of it, is heated under atmospheric or increased pressure, preferably after removal of the precipitated calcium salt. The solution, which contains urea and potassium hydroxide, is saturated with carbon dioxide and evaporated to dryness, or is used in the treatment of further quantities of calcium cyanamide. L. A. Coles.

Storing mixed fertilisers containing ammonium nitrate. J. Heppes (G.P. 423,806, 24.12.24).—Caking of the fertilisers is prevented by storing them at a temperature below 5°.
L. A. Coles.

Preservation of organic nitrogen compounds in liquid manure. L. Kuchler and J. Bodler (G.P. 424,667, 23.1.23).—Fresh farm or stable manure, in the form of liquid or paste, is subjected to the action of an alternating current, and when completely sterilised is stored in tightly closed pits.
L. A. Coles.

XVII.—SUGARS; STARCHES; GUMS.

See A., April, 386, **Occurrence of crystallised lævulose** (von Lippmann). 387, **Molecular weight of soluble starch** (Pictet). 441, **Pectins of the sugar-beet** (Ehrlich and von Sommerfeld).

Patents.

Decolorising and defecating product (U.S.P. 1,575,561).—See II.

Treating bagasse (U.S.P. 1,574,254).—See V.

Lactose from whey (E.P. 248,998).—See XIX.

Glutamic acid (E.P. 248,453).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Valuation of hops. W. Windisch, P. Kolbach, and W. Banholzer (Woch. Brau., 1926, 43, 79—82).—In the valuation of hops by expert examination and handling, the visual estimate of the amount of lupulin present is supposed to give a rough measure of the richness of the hops in bitter principles. Working on 14 samples of hops from different districts, the authors have compared the visual estimates of lupulin-content with the results of analytical determinations of the bitter acids and resins by the most recent methods, and found no correspondence whatever. J. H. Lane.

Wort boiling under pressure. P. Petit and J. Raux (Fond. Brass. et Malterie, 1925, Bull. 4, 62; Woch. Brau., 1926, 43, 29—30).—The effect of excess pressures up to 1 atm. during wort boiling was studied on a wort made from malt with 12·5% of maize and rice, by a two-mash process. The wort was treated with 100—150 g. of hops per hl., two-thirds being added at the commencement of boiling and the remainder $\frac{1}{2}$ hr. before the end. Compared with boiling under atmospheric pressure the higher pressures were found to permit a better utilisation of hops and to produce an earlier and better break or flocculation of proteins. On the other hand they intensified the colour of the wort and to a smaller extent that of the fermented beer, and tended to produce an unpleasantly bitter flavour. Better results were obtained by boiling for a short time at relatively high excess pressures, such as 1 atm., than for a longer time at lower ones; thus the intensification of colour was much less in $\frac{1}{4}$ hr. under 1 atm. than in 1 hr. at 0·1—0·5 atm. above the normal. J. H. Lane.

Influence of sugar on determination of ammonia in grape must. J. Ventre and E. Bouffard (Compt. rend., 1926, 182, 784—786).—In presence of lævulose or, to a less extent, of dextrose (but not sucrose), ammonia cannot be completely removed from aqueous solution by the usual distillation process. To determine ammonia in must therefore, it is necessary to distil to dryness with magnesia in a vacuum. R. Cuthill.

See also A., April, 431, **Enzymic breakdown and synthesis of carbohydrates** (von Euler and Myrbäck). 432, **Activity and iron content of highly active catalase preparations** (Hennichs). 433, **Yeast maltase. Separation of maltase and invertase** (Willstätter and Bamann). 434, **Effect of hydrogen cyanide on alcoholic fermentation** (Warburg); **Effect of hydrogen sulphide on chemical processes of the cell** (Negelein); **Alcoholic fermentation in presence of hydrogen sulphide and hydrogen cyanide** (Neuberg and Perlmann); **Growth of yeast in wort** (Ludwig). 435, **Lactic acid fermentation** (Virtanen, Karström, and Bäck); **Fermentation of α-ketoglutaric acid by *Bact. xylinum*** (Iwatsuru).

Patents.

Preparing yeast for use in food products. C. Hoffman and C. N. Frey, Assrs. to Fleischmann Co. (U.S.P. 1,575,762, 9.3.26. Appl., 15.9.23).—A product containing ammonium sulphate and devitalised yeast in which the protoplasmic material in the cells has been coagulated, is obtained by treating yeast with saturated ammonium sulphate solution at 85°, washing out at least a portion of the ammonium sulphate, filtering, and drying the residue in such a manner that the cell walls are not ruptured.
L. A. Coles.

Fixation of solar or ultra-violet energy (E.P. 226,534).—See VII.

Glutamic acid (E.P. 248,453).—See XX.

XIX.—FOODS.

Reagent for detection of peroxydase in milk. P. Borinski (Z. angew. Chem., 1926, 39, 281—283).—0·85 g. of guaiacum resin is dissolved in 85 c.c. of 70% alcohol, and 10 c.c. of phenol and 5 c.c. of 3% hydrogen peroxide are added. The reagent may be prepared in 1 hr., can be used immediately, and is stable for at least a week. Milk no longer gives a blue colour with this reagent after heating for 30 min. at 70°, 3 min. at 75°, or 1 min. at 85°; the colour is given if 10% of unheated milk is added. A. Geake.

Action of streptococci on milk. C. Gorini (Compt. rend., 1926, 182, 946—947).—Streptococci may be divided into two classes, the one class (saprophytic), occurring in bovine excreta, clot milk which has been thoroughly sterilised until of yellow tint, giving rise to a large amount of lactic acid and yielding a clot which is not digested readily; the other class (parasitic), found on udders and in bovine and human infections, will only clot milk which has been mildly sterilised, do not produce much lactic acid, and the clot formed is readily digested by acids. L. F. Hewitt.

Composition of fenugreek seeds and their admixture with wheat used for flour-milling. E. Fleurent (Compt. rend., 1926, 182, 944—946).—Fenugreek imparts a disagreeable bitter flavour to flour when mixed with it, even to the extent of 0·01%, and the bread made from the flour is unsaleable. This effect is due to the fact that fenugreek seeds contain about 9·5% of a brown oil, smelling of fennel, d^{18} 0·97, iodine value 110—114, extracted by ether, and a resin (13·5%) with a bitter taste, extracted by alcohol. L. F. Hewitt.

Determination of the age of bread. J. P. Peper (Chem. Weekblad, 1926, 23, 163—168).—The ageing of bread is accompanied by a shrinkage in volume of the starch grains and loss of water; these phenomena are inhibited by presence of acetaldehyde. The method devised by Katz consists in kneading a weighed quantity of the crumb with water through silk gauze, making up to a known volume in a measuring cylinder, adding toluene and acetaldehyde, and noting the height of the column of sediment after 24 hrs. This height is compared with readings obtained from breads of known age. It is found possible in this way to determine whether a bread is quite fresh or more than 12 hrs. old. S. I. Levy.

Tin-plate for containers for food products (preserves etc.). H. Serger (Chem.-Ztg., 1926, 50, 201—202).—The most frequent source of trouble in tin-plate to be used for food containers is local porosity due to the presence of particles of slag, dirt, etc., accidentally introduced into the iron before or during the rolling process. These particles absorb acid during the pickling, and bubbles result when the sheet is tinned, which ultimately form porous spots on the plate. Exposed iron, which may readily be detected by the potassium ferricyanide–gelatin method, is due to incomplete removal of oxide scale from the sheet during the pickling process, with the result that tin is not deposited on these spots. The chemical composition of the iron is of considerable importance in obtaining a satisfactory tin-plate. B. W. Clarke.

[Decomposition of] pectins. A. Mehlitz (Konserven-Ind., 1926, 13, [2], 1—3; Chem. Zentr., 1926, I., 2154; cf. B., 1925, 687).—If apple-pectin and pectin-sugar solutions are heated for 10 hrs., 16% and 19·4% of their pectin-contents respectively are destroyed, mostly in the first few hours. In the latter case the decomposition is increased and decreased by the presence of sugar and acid (produced in the initial stages) respectively, the high final value being due to the higher boiling point of the solution. When the duration of boiling is less than 2 hrs. the decomposition of pectin is appreciably less in presence of sugar. J. Grant.

Relative nutritive value of various proteins contained in Japanese food articles. U. Suzuki, Y. Matsuyama, and N. Hashimoto (Sci. Papers Inst. Phys. Chem. Res., 1925, 4, 1—48).—By feeding experiments on rats, the relative nutritive values of the proteins of various kinds of fish, rice, wheat, soya beans, kaoliang, Indian corn (maize), barley, oats, rye, peanut, cotton seed, banana, and potato have been studied. A table is given showing the different effects of the various proteins on the growth of rats. H. J. Channon.

Preventing bumping in the determination of crude fibre [in feeding stuffs etc.]. W. Lepper (Chem.-Ztg., 1926, 50, 211).—In the determination of crude fibre in material containing large amounts of mineral constituents, the preliminary boiling with sulphuric acid presents no difficulties, but when the insoluble material from this operation is collected on asbestos filters and the fibre and asbestos subsequently boiled with potassium hydroxide there is a marked tendency for the liquid to bump. This may be avoided by just covering the bottom of the beaker with glass beads 5 mm. in diameter. These may subsequently be separated by decantation or by screening on a sieve with 3 mm. holes. A. R. Powell.

See also A., April, 428, **Maintenance values for proteins of milk, meat, bread and milk, and soya-bean curd** (Rose and McLeod). 436, **Biological assay of fat-soluble vitamins** (Chick); **Maintenance of standardised breed of rats for work on fat-soluble vitamins** (Smith and Chick). 437, **Vitamin-B of lemon rind** (Willimott); **Use of colloidal ferric hydroxide for adsorbing vitamins-B and -D** (Zajdel and Funk); **Water-soluble vitamin content of velvet bean** (Salmon and Miller); **Action of *n*-butyl nitrite on activated cholesterol and vitamin-D** (Bills); **Effect of irradiation on antirachitic properties of milk** (Steenbock and others); **Antirachitic value of fresh spinach** (Chick and Roscoe); **Antirachitic value of winter spinach** (Boas).

441, **Alcohol-soluble protein from polished rice** (Hoffman). 444, **Determination of calcium in milk** (Corley and Denis); **Determination of protein nitrogen** (Voit); **Determination of pectin** (Emmett and Carré).

Patents.

Obtaining milk sugar [lactose] from whey. A. G. Bloxam. From Elektro-Osmose A.-G. (Graf Schwerin Ges.) (E.P. 248,998, 17.7.25).—Whey is neutralised, *e.g.*, with sodium carbonate, boiled for a short time, and then, while cooling, is gradually acidified, *e.g.*, by addition of strong hydrochloric or sulphuric acid. After filtering off precipitated albumins, the solution is decolorised and further quantities of the albumins are removed by stirring it with about 1—2% of an adsorbent, such as clay or kaolin, and again filtering. Electrolytes and other diffusible impurities are subsequently removed by electro-osmotic treatment under such conditions that losses of lactose by diffusion and inversion are reduced to a minimum. For example, the liquid may be subjected to the action of a continuous electric current in a narrow compartment between anode and cathode diaphragms constructed respectively of animal membranes, such as skin, bladder, or gut, and woven vegetable material, such as cotton cloth, the temperature being kept below 40°. Lactose of 96—97% purity is obtained on evaporation of the solution *in vacuo* or by spraying, and pure lactose can be obtained from it by crystallisation. L. A. Coles.

Desiccating apparatus [for milk and buttermilk]. N. P. Collis, Assr. to Collis Co. (U.S.P. 1,575,414, 2.3.26. Appl., 14.1.22).—A desiccating apparatus for milk and buttermilk comprises a steam-heated rotating drum beneath which is a pan containing the liquid to be desiccated. The buttermilk is pumped from the pan and sprayed through slots on to the drum where it is desiccated and from which the dried material is removed in the course of a rotation. Any excess of liquid is returned from the spray pipe to the pan, the contents of which it serves to agitate. A second pump and spray system are provided for use with sweet milk and as this dries more rapidly than buttermilk it is delivered to the drum nearer the scraping point. Gearing is provided by which either pump can be coupled with the driving engine. T. S. Wheeler.

Making concentrated sour milk. A. Neuhauser (U.S.P. 1,576,351, 9.3.26. Appl., 19.1.24).—Skim milk is concentrated to about one-fourth of its bulk, and the product is soured at about 32—38° to an acidity of over 4%, and is then homogenised. L. A. Coles.

Self-preserving stock food. W. P. M. Grelck (E.P. 249,746, 9.9.25).—See U.S.P. 1,554,913; B., 1925, 939.

Recovery of solid constituents of liquids (E.P. 248,081).—See I.

Preparing yeast for use in food products (U.S.P. 1,575,762).—See XVIII.

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

Iodoeosin as indicator in alkaloidal determinations. D. B. Dott (Pharm. J., 1926, **116**, 357).—As an indicator for the titration of strychnine, iodoeosin is inferior to cochineal, hæmatoxylin, or methyl-orange. E. H. Sharples.

Alleged deterioration of Indian opium on keeping. D. B. Dott (Pharm. J., 1926, **116**, 356).—Examination at intervals over a period of two years, of a sample of Indian opium which had been dried at 95—99° and preserved in a stoppered bottle in the dark, demonstrated that the morphine content decreased by only 0·24%. E. H. Sharples.

Benzylmorphine tartrate. D. B. Dott (Pharm. J., 1926, **116**, 356).—The solubilities in water of the neutral tartrate (containing 1 mol. H_2O) and of the sulphate of benzylmorphine, are 1 in 3 and 1 in 170 pts. respectively. E. H. Sharples.

Determination of small amounts of alcohol and ether vapours in the presence of each other. E. von Somogyi (Z. angew. Chem., 1926, 39, 280—281).—Alcohol vapour is absorbed in a mixture of 3 pts. of water and 1 pt. of concentrated sulphuric acid by vol., and ether vapour in a mixture of equal volumes of concentrated sulphuric acid and *N*-potassium dichromate. Ether retained by the first absorbent is drawn into the dichromate by sucking air through the apparatus for 3 hrs., and the dichromate reduced measures the ether content. The alcohol is also determined by oxidation with dichromate. A. Geake.

Adsorption from solution and the valuation of adsorbents used in therapy. T. Sabalitschka and W. Erdmann (Pharm. Ztg., 1926, 71, 374—376, 390—392).—Experiments on the adsorption of barium chloride, ferric chloride, basic aluminium acetate, potassium hydroxide, hydrochloric acid, oxalic acid, acetic acid, sulphosalicylic acid, nicotine, methylene-blue, tannin, and iodine by kieselguhr, boneblack, barium sulphate, lime-, beech-, and animal-charcoals, and charcoal sponge ("Schwammkohle") indicate that the order of the adsorptive powers of the adsorbents for the above compounds is not uniform. Animal charcoal, charcoal sponge, and boneblack generally possessed the highest, and barium sulphate the lowest adsorptive powers. Boneblack had the highest adsorptive power for bases, but was inferior to lime- and animal-charcoals and charcoal sponge for the adsorption of acids. The present knowledge of the application of adsorbents in therapy is discussed and it is concluded that generalisations regarding the behaviour of adsorbents in the animal body deduced from experiments *in vitro*, are only valid within very narrow limits owing to the predominating effect of exterior conditions on the adsorptive power. E. H. Sharples.

See also, A., April, 408, **Existence of *iso*pulegone in nature** (Grignard and Savard); **Terpene and sesquiterpene of Mitsuba-zeri** (Hirano). 409, **Purification of digitonin** (Windaus). 417, **Conversion of berberine into β-homochelidonine (α-allocryptopine)** (Haworth and Perkin, jun.). 430, **Relation between chemical constitution and taste of sweet substances** (Täufel). 440, ***Chlorocodon whiteii*: its constituents and their harmacological actions** (Dilling). 444, **Micro-determination of lactic acid and lactates** (Hansen)

Patents.

Manufacture of acridine derivatives [bactericides]. Brit. Dyestuffs Corp., W. H. Perkin, and A. W. Burger (E.P. 248,182, 12.3.25).—2:8-Diamino-3:7-dimethoxy- (or -ethoxy-)acridine derivatives or the corresponding 3 : 7-2 : 8-compounds prepared by standard methods, and the acridinium compounds derived from them by acetylation, alkylation, and hydrolysis, have bactericidal properties, the acridinum compounds being especially suitable for therapeutic use, owing to their greater solubility. 2:8-Diamino-3:7-diethoxyacridine, m.p. 281°, is prepared by heating a mixture of 42·5 pts. of 2:4-diaminophenetole, 35 pts. of oxalic acid, 40 pts. of zinc chloride, and 45 pts. of glycerin, to 200° in 1½ hrs., and maintaining it at that temperature for 2 hrs. When cool, the product is ground and extracted for several hours with cold, dilute ammonia, and, after filtration, the acridine derivative is extracted from the residue with boiling alcohol, and recovered by evaporation or by precipitation with dilute ammonia; it is purified by crystallisation from pyridine and water. 2:8-Dimethoxy-3:7-diaminoacridine: a mixture of 1 pt. of sodium nitrite with 34·2 pts. of 3:3′-diacetamido-4:4′-dimethoxydiphenylmethane, m.p. 159—160°, prepared by the acetylation of 3:3′-diamino-4:4′-dimethoxydiphenylmethane (cf. F.P. 322,985; B., 1903, 414), is added to a mixture of 68 pts. of sulphuric acid, *d* 1·7, and 21·6 pts. of nitric acid, *d* 1·49, at 0—5°. After stirring for 2 hrs., during which the temperature rises to 15—20°, the mixture is poured on to 2000 pts. of ice and water, and the dinitro-compound, m.p. 260—261°, is recovered by filtration, and hydrolysed by heating with strong sulphuric acid on the water bath to 6:6′-dinitro-3:3′-diamino-4:4′-dimethoxydiphenylmethane, m.p. 226—228°. A solution of 17·4 pts. of the product in 207 pts. of hydrochloric acid, *d* 1·185, is treated with 21·5 pts. of zinc dust, heated under a reflux condenser for 24 hrs., diluted with an equal volume of water, boiled, and oxidised with ferric chloride; the acridine compound is then salted out and crystallised from water.

L. A. Coles.

Manufacture of glutamic acid and salts thereof. K. Ikeda (E.P. 248,453, 3.12.24).—Glutamic acid is recovered from vinasses or other beet sugar residues in the form of its calcium salt, $C_5H_7O_4NCa,H_2O$, either by digestion of the liquor with lime or, preferably, with an acid hydrolytic agent, followed by neutralisation with lime. Separation of the sparingly soluble calcium salt is facilitated by addition of alcohol or a soluble calcium salt and ammonia. Alternatively, glutamic acid may be separated from the acid hydrolysis product in the form of its hydrochloride, which is converted into the calcium salt by treatment with lime. From the calcium salt, by addition of mineral acid, glutamic acid may be obtained, or, by treatment in solution with an alkali salt of an acid which forms insoluble calcium salts, soluble alkali glutamates can be prepared. The soluble calcium salt, $Ca(C_5H_8O_4N)_2$, can be obtained by treating the sparingly soluble salt with carbon dioxide in presence of water.

E. H. Sharples.

Manufacture of monoacyl derivatives of aminoarylarseno-compounds. G. Newbery, and May and Baker, Ltd. (E.P. 248,523, 6.1.25).—Monoacylamidoarylarseno-compounds are prepared by combining equimolecular proportions of an arylarsenic compound and an acylamidoarylarsenic compound. Thus, an arylarsenious oxide or an aryldichloroarsine is condensed with an acylamidoarylarsine, or an acylamidoarylarsenious oxide or an acylamidoaryldichlorarsine with an arylarsine. For example, 3-acetamido-3′-amino-4 : 4′-dihydroxyarsenobenzene (light yellow powder) is prepared by condensing 3-acetamido-4-hydroxyphenylarsenious oxide with 3-amino-4-hydroxyphenylarsine hydrochloride in 10% sodium hydroxide solution. The compounds are also prepared by reduction of an arylarsinic acid and an acylamidoarylarsinic acid in admixture. For example, 3-benzamido-3′-amino-4 : 4′-dihydroxyarsenobenzene is obtained by reducing a mixture in equimolecular proportions of 3′-amino-4-hydroxyphenylarsinic acid and 3-benzamido-4-hydroxyphenylarsinic acid in dilute sodium carbonate solution with sodium hyposulphite and magnesium chloride. E. H. Sharples.

Manufacture of pure urea [carbamide]. J. Y. Johnson. From Badische Anilin & Soda-Fabr. (E.P. 249,041, 5.11.25).—Pure carbamide is prepared by treating concentrated solutions of technical carbamide at moderately elevated temperatures and in presence of at least 0·5—1% of ammonia with an oxidising agent leaving no soluble residues, preferably with air, oxygen, or manganese dioxide, and separating from the precipitated impurities. Compounds of the heavy metals are precipitated practically entirely in a form in which they can be easily separated. After separation, a clear colourless solution is obtained from which, by crystallisation or complete evaporation, pure, white carbamide is produced. E. H. Sharples.

Producing chloro-derivatives of methane. C. B. Carter and A. E. Coxe, Assrs. to S. Karpen & Bros. (U.S.P. 1,572,513, 9.2.26. Appl., 9.8.23).—A mixture of 100 vols. of chlorine, 100 vols. of methyl chloride, and 200 vols. of methane is passed into a chamber at about 450°, when chlorination of the methane with formation of 80% of methylene chloride, 15% of chloroform, and 5% of carbon tetrachloride rapidly occurs, all the chlorine being consumed. The issuing vapours are washed in a tower with water to remove hydrochloric acid and then compressed to 10—15 atm. to condense methylene chloride, chloroform, and carbon tetrachloride. The condensate is distilled and any methane and methyl chloride evolved are returned to the uncondensed gases which, after expansion, are mixed with 100 vols. of chlorine and 46 vols. of methane and passed again to the reaction chamber. The process is continuous and all danger of explosion is obviated. T. S. Wheeler.

Extraction process [for concentration of acetic acid]. H. Suida (F.P. 594,925, 10.3.25).—In a continuous process for concentrating aqueous solutions of substances volatile in steam, *e.g.*, acetic acid, the dilute solution is heated under pressure in a still and the vapours are led into the base of an extraction column down which trickles a boiling solvent not readily miscible with water. Acetic acid vapours are absorbed by the solvent, which is drawn off at the base of the column. The solution then passes to a second still which is heated at a slightly lower pressure by hot vapours from the top of the preceding column, and the operations are repeated in a succession of stills and columns, the pressure in the final still being atmospheric. R. B. Clarke.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Intensification by dye-toning. A. and L. Lumière and A. Seyewetz (Brit. J. Phot., 1925, **73**, 147—148; Bull. Soc. Franç. Phot., 1926, iii., **13**, 5—8).—The silver image is mordanted in a bath containing copper sulphate 40 g., tripotassium citrate 60 g., glacial acetic acid 30 g., ammonium thiocyanate (dissolved separately) 20 g., water 1 litre. After washing, the image is dyed in a mixture of 1% solutions of Methylene Blue (287 c.c.), Rhodamine S (333 c.c.), and Phosphine M, patent extra concentrated (380 c.c.), with addition of 1% acetic acid. This bath gives a neutral coloured image, and an intensification greater than that obtained by other means. The intensified image can be reduced by a solution of potassium permanganate 1 g., sulphuric acid 5 c.c., water 1 litre, or by hyposulphite which, however, alters the colour. The colour of the image can be altered by various modifications to obtain different results in printing. W. Clark.

Patents.

Dye composition [for treating photographic emulsions]. E. J. Wall and D. F. Comstock, Assrs. to Kalmus, Comstock and Wescott (U.S.P. 1,573,595, 16.2.26. Appl., 6.7.17. Renewed 12.9.23).—A composition for treating photographic emulsions contains a protective emulsoid colloid for preventing the coagulation of the suspended dye. For example, with an isocyanine dye a fraction of 1% of gelatin may be used. E. S. Kreis.

Treating photographic emulsion, and colour-sensitising composition. E. J. Wall and D. F. Comstock, Assrs. to Kalmus, Comstock and Wescott (U.S.P. 1,573,596, 16.2.26. Appl., 11.7.17. Renewed, 12.9.23).—The sensitising composition contains an oxidising agent to prevent reduction of the emulsion by the protecting agent for the colloidal dye, and an alkali to prevent bleaching of the dyestuff. Hydrogen peroxide is a suitable oxidising agent. E. S. Kreis.

Photographic medium. M. C. Beebe and A. Murray, Assrs. to Wadsworth Watch Case Co. (U.S.P. 1,574,359, 23.2.26. Appl., 18.11.22; cf. E.P. 203,285; B., 1924, 493).—The medium consists of a hydrophobic colloid with lead triethyl iodide as sensitiser. W. Clark.

Photographic sensitiser. M. C. Beebe and A. Murray, Assrs. to Wadsworth Watch Case Co. (U.S.P. 1,575,143, 2.3.26. Appl., 18.11.22).—The sensitiser consists of a hydrophobic protective colloid, containing a dispersed halogen-liberating compound. W. Clark.

Light-sensitive layers. B. Stange (G.P. 421,138, 18.10.21).—The layer contains silver halide particles stained in three primary colours. It consists of organic particles, such as bacteria (yeast cells, acetic bacteria), dyed in the appropriate colours, and treated first with a silver salt solution and then with a solution of a halide. W. Clark.

Toning sulphide-toned silver images on development and printing-out papers. Leonar-Werke Arndt & Löwengard (G.P. 422,295, 5.8.24, and 422,296, 18.9.24. Addns. to 413,360; B., 1925, 787).—Thiocarbamide, thiosinamine, alkali thiocyanate, sodium thiosulphate, or mixtures of these are added to simple or slightly complex gold or platinum salts, with or without the addition of lead salts, and either in the solid state or in solution. The images may be toned with selenium as well as with gold. W. Clark.

Selenium toning bath for developing and printing-out papers containing silver. Leonar-Werke Arndt & Löwengard (G.P. 422,297, 11.11.24. Addn. to 419,428).—The selenium is dissolved in hyposulphite solution in presence of alkaline reacting substances other than sodium carbonate, *e.g.*, sodium sulphite, ammonia, organic ammonium bases, caustic soda, or caustic potash. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Pressure wave sent out by an explosive. I. W. Payman and H. Robinson (Safety in Mines Res. Bd., Paper No. 18, 1926, 60 pp.).—As part of an investigation of the effect of the detonation of mining explosives on the atmosphere surrounding a borehole, the waves sent through air by different types of explosion have been photographed by the "Schlieren" (striæ) method devised by Töpler

(Ann. Physik, 1867, **131**, 33). The apparatus and technique used in the present investigation are fully described. The waves were set up by the "explosion" produced when a celluloid disc was caused to burst under gaseous compression, by the pressure and shock waves from a gaseous detonating mixture, and by the explosion of a low-tension detonator. A series of 48 photographs of atmospheric shock waves are reproduced, and a non-mathematical explanation is given of their initiation and propagation. A method is also described for obtaining an accurate record of the rate at which the shock wave sent out by an explosive is propagated through the atmosphere, the length and duration of the flame produced, and the relative positions of shock wave and flame at any instant. An appendix contains a summary of other methods which have been proposed for investigating these waves.

S. Binning.

Patent.

Explosives. A. C. Scott, and Mexco, Ltd. (E.P. 248,089, 29.11.24 and 19.8.25).—The particles of an explosive mixture containing a combustible substance and a compound containing readily available oxygen are coated with a synthetic resin, *e.g.*, a phenol–aldehyde condensation product. By using a liquid or plastic resin the explosive will be rendered plastic. A gel of a phenol–formaldehyde resin and a nitro-compound may be used as a coating. The phenol–aldehyde condensation product may form the combustible constituent of the mixture. Explosives made in this way are waterproof and may be grained.

S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Control of the aluminium sulphate process for the purification of drinking water. F. Egger (Chem.-Ztg., 1926, **50**, 167).—The determination of the sulphate content of the water before and after the clarification process gives a more accurate indication of the efficiency of the precipitation than the determination of the aluminium, which gives unsatisfactory results. The process may be represented

$$Al_2(SO_4)_3+3Ca(HCO_3)_2=3CaSO_4+2Al(OH)_3+6CO_2$$

and owing to the corrosive nature of the carbon dioxide, the amount of this left in the water must be carefully controlled by milk of lime, the acidity of the filtered water as indicated by phenolphthalein being a satisfactory guide to the efficiency of the removal. The determination of the hydrogen-ion concentration is the most accurate method of control. The determination of organic impurities and bacteria enables the filtration process itself to be controlled.

B. W. Clarke.

Effect of temperature on rate of deoxygenation of diluted sewage. R. E. Greenfield and A. L. Elder (Ind. Eng. Chem., 1926, **18**, 291—294).—The rate of deoxygenation of sewage samples diluted with aerated distilled water and incubated at temperatures of 20° and 14° is fairly adequately represented by the formulæ connecting rate, time, and concentration developed by Theriault, Streeter, and Phelps (cf. B., 1925, 565), and others; but this is not the case at lower temperatures (2—6°), chiefly on account of the occurrence at these temperatures of a lag phase during the first few days of incubation when little or no oxygen is absorbed. The lag phase, the duration of which decreases with increase in sewage concentration, is attributable to the slow development at low temperatures of the bacteria required to carry on the deoxygenation. Biochemical oxygen demand curves for diluted sewage give no indication of two stages of deoxygenation such as those obtained by Theriault for Ohio River water. Such curves were obtained, however, from samples of Illinois River water. The two-stage phenomenon seems to be one encountered in river water but not always in diluted sewage. It is suggested that the two stages, rather than representing carbonaceous oxidation and nitrification (cf. Adeney, B., 1896, 463), indicate first, the decomposition of the dead organic matter contained in the river water, and second, the decomposition of dead plankton which at the time of collection of the sample are living organisms.

W. T. Lockett.

Treatment of packing-house sewage. W. C. Moor and W. P. Wayne (Ind. Eng. Chem., 1926, **18**, 239—242).—In 1916 an experimental activated sludge plant was constructed at Fort Worth, Texas, for the treatment of packing-house waste. With an aeration period of 10 hrs. clear and stable effluents were obtained, but the cost of aeration was excessive. The present system consists of treatment by screens and Dorr clarifiers, the resulting partially purified waste being then passed to the city sewers. About 2½ million gallons of stock-yard waste are treated per day. Screening removes 24% of the suspended solids and 27% of the grease content of the waste; the Dorr clarifiers, which are provided with grease skimmers, remove a further 50% of the suspended solids and 49% of the grease. By the entire process (screens and clarifiers) a total of 98% of the suspended solids (as determined by settling for 2 hrs.) is removed. The city disposal plant comprises screens, grit chambers, Imhoff tanks, sprinkling filters, Dorr clarifiers for the retention of humus, and drying beds for sludge.

W. T. Lockett.

Patent.

Processes involving exchange reactions, particularly softening of water by zeolite-like bodies. E. B. Higgins (E.P. 248,414, 1.10.24).—Instead of passing the water through a bed of the softening material, the softening material is suspended in the water, by giving the water an upward flow. Various forms of apparatus are described. For example, by causing the water to flow upwards in a vessel like an inverted cone, the softening material in one zone will rise with the water, in another it will just keep in suspension, and in the uppermost zone it will settle through the water. Regeneration and washing of the material are effected in a similar manner. By this method larger quantities of water can be treated in a shorter time than by the old method, owing to the more intimate contact achieved and greater surface of softening material exposed.

E. S. Kreis.