# BRITISH CHEMICAL ABSTRACTS STANDARD PROMINTS CORP. (U.S.P. 1898.331 S.129., result in a daugonous rise of temperature. Addition of Appl., 16.6.28 U.K. 29.1.27). See 5.17 29.3 (0): molecure protocole she initial heating of the collected.

B.—APPLIED CHEMISTRY Centrificial reparator. W. C. Liveaux Liveaux: Entrop. Cost. (U.S.P. 199<del>1)</del> Appl. 204,275,—See D.P. 285,318 (J., 12

MARCH 29, 1929.

# I.-GENERAL; PLANT; MACHINERY.

Surface increase in mists from the "Schlick" spray. Anon. (Chem.-Ztg., 1929, 53, 33).—The average size of particle, the volume of liquid handled per minute, the number of single particles per litre of liquid handled, and the total surface of liquid in the mist are given for pressures of 2, 4, and 6 atm. With a pressure of 8 atm. the mist has a total particle surface 1700 times the surface of the liquid fed in. S. I. LEVY.

Removal of thin films of solution in centrifugals. H. V. WARTENBURG and H. PERTZEL [with F. SAVAAGE] (Chem. Fabr., 1929, 61-63).-The thickness of a film of liquid on a vertical flat surface, originally  $D_0$ , is shown to diminish in a time, t, according to D = $\sqrt{hD_0^2/(2D_0^2 \cdot \rho gt + h)/3\eta}$ , where h is the height and  $\eta$  the viscosity of the film. The formula is compared with actual determination of mother-liquor content in Glauber's salts during centrifuging, the variation being principally due to the effect of capillarity. A more exact comparison was obtained by the use of a number of brass tubes wetted with castor oil, olive oil, and paraffin oil of known viscosity. The discharged and residual oil were weighed and g in the formula was replaced by the centrifugal force. Whilst the results confirm theory to the extent that the film thickness is inversely proportional to the square root of the time of centrifuging, agreement is far from exact. For stationary films and slow-running centrifugals capillarity is important. C. IRWIN.

Correlation of freezing points and vapour pressures of aqueous solutions by Dühring's rule. C. C. MONRAD (Ind. Eng. Chem., 1929, 21, 139-142).-Since Dühring's rule holds good for aqueous solutions if two points on the vapour-pressure curve are known, the entire curve may be calculated and the f.p. and b.p. of the solution deduced. Temperature differences between the Dühring line of the solution and that of pure water are transferred to the pressure-temperature phase diagram of water. The accuracy of Dühring's rule for low vapour pressures was checked by reference to a large number of data in literature. This method, however, is not applicable to concentrations higher than that existing at the cryohydric point. In such cases the vapour pressures of saturated solutions must be known. If the vapour pressure of the solution at any other temperature is known, a system of parallel Dühring lines may be drawn. C. IRWIN.

Comparator for the colorimetric determination of the hydrogen-ion concentrations of coloured solutions. D. McCANDLISH and G. HAGUES (J. Inst. Brew., 1929, 35, 66-68) .- Reflected by means of two

mirrors from the same source of illumination, two beams of light of equal intensity emerge at right angles to each other after passing through the coloured solutions. To render the colour matching more delicate and accurate, the beams of light are then brought parallel and adjacent by means of a pair of right-angled prisms cemented together by Canada balsam, so that the balsam junction extends over only one half of the vertical contiguous surfaces, and there is an air gap between them over the other half. C. RANKEN.

# PATENTS.

Crushing machines. D. B. COOPER (B.P. 302,135, 22.2.28).-A bearing block for the rollers of sugar-cane mills and other purposes is described.

# B. M. VENABLES.

Catalytic apparatus. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,685,672, 25.9.28. Appl., 27.10.26). -The central cooling tubes are spaced closer than the peripheral elements or the velocity of the cooling gas or liquid in the cooling tubes is varied, e.g., by means of plugs, so as to secure maximum cooling in the central elements and relatively uniform temperature in the catalyst layer. With horizontal tubes, similar arrangements secure a maximum cooling effect in the upper catalyst laver, first in contact with the reaction gas. Where one or more reaction components are solid or liquid at ordinary temperatures, or where inert vapours may be present, the latent heat of vaporisation may be utilised for cooling purposes. R. BRIGHTMAN.

Methods and apparatus for catalysing gaseous reactions. H. WADE. From SILICA GEL CORP. (B.P. 304,251 and 304,269, 12.7.27).-A finely-divided catalyst consisting of adsorbent silica gel etc., together with activating material, if desired, is sprayed into a stream of gases before their entry into a reaction chamber, and the catalyst is subsequently removed from the reaction products, passed through a regenerator, and returned to the gas stream. The process is described with reference to (A) the cracking of oil vapour using silica gel activated with iron oxide etc., and (B) the production of sulphur trioxide and sulphuric acid, in which case the catalyst is activated with platinum, L. A. Coles. copper, iron, etc.

Gas-analysis apparatus. R. H. KRUEGER, Assr, to C. ENGALHARD, INC. (U.S.P. 1,698,887, 15.1.29. Appl. 27.8.27).-A resistance heating element is mounted in a thermal conductivity cell having walls adapted for dissipating heat, and adjustable means are provided for varying their heat-dissipating capacity.

J. S. G. THOMAS.

Apparatus for production of dispersions of solids in liquids. W. H. WHATMOUGH, Assr. to 230

STANDARD PRODUCTS CORP. (U.S.P. 1,698,354, 8.1.29. Appl., 16.8.28. U.K., 29.4.27).—See B.P. 293,510; B., 1928, 658.

Centrifugal separator. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,699,471, 15.1.29. Appl., 20.1.27).—See B.P. 283,918; B., 1928, 320.

Metal grinding balls (B.P. 277,044).—See X-Temperature-measuring device (U.S.P. 1,695,867). Gas-purifying plant (B.P. 278,710). Gas-purification electrode (G.P. 451,041).—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Maceration method in microscopical examination of coal. H. BODE (Berg-Technik, 1928, 21, 205; Fuel, 1929, 8, 86-90).-In this method the coal is prepared for microscopical examination by treatment with a solution of potassium chlorate in nitric acid (Schulze's solution) and subsequently with ammonia. Lignin and the coalification products of lignin and cellulose are thereby oxidised and removed. Cellulose itself, if present, and the bituminous substances of the coal are unattacked. From the amount and appearance of the residue, e.g., from the occurrence therein of fossil wood, spores, cuticle, etc., it is possible to determine the degree of coalification of the material, to distinguish vitrain, durain, and fusain in powdered coal, and to determine the nature of the plants which contributed to the formation of the coal. The method is preferable to that of the observation of etched polished surfaces in reflected light, which can lead to false conclusions. A. B. MANNING.

Spontaneous combustion of semi-coke from brown coal: its causes and prevention. P. ROSIN (Braunkohle, 1928, 27, 241-256, 282-292; Fuel, 1929, 8, 66-78).-The influence of various factors, such as moisture content, temperature of carbonisation, fineness of subdivision, the addition of catalysts, etc., on the spontaneous heating of a brown coal semi-coke has been studied. The apparatus consisted of an insulated metal cylinder capable of holding about 11 kg. of material, through which a current of oxygen could be passed, while observations were made of the temperature at several points within the material. Measurements have been made also of the adsorption of gases by the semicoke and of the heat developed on wetting it. The material used was taken directly from the retort and cooled in the absence of air. Self-heating did not occur uniformly within the material; local centres of heating were formed showing a more rapid rise of temperature than elsewhere. The first gradual rise of temperature, due to adsorption of oxygen, was followed at about 70° by a phase of rapid oxidation resulting in combustion. The adsorption of all gases by the semi-coke was accompanied by the evolution of heat, that evolved on the adsorption of carbon dioxide being sufficient to raise the temperature of the coke to 70°. This carbon dioxide is lost on subsequent exposure of the coke to the air, the heat thereby absorbed compensating to some extent for that evolved due to oxidation. Moist semi-coke "adsorbs" more oxygen than dry coke, due probably to increased oxidation rather than to true adsorption. The heat evolved when the semi-coke is wetted may also

result in a dangerous rise of temperature. Addition of moisture promotes the initial heating of the coke, and, although it retards spontaneous combustion, wetting alone is not a sure means of preventing such combustion. Spontaneous heating of the semi-coke can be prevented or much diminished by artificial ageing, treatment with carbon dioxide, or the addition of magnesium or ammonium chloride to the coal. The coke also loses all liability to self-heating with the removal of the inorganic constituents soluble in hydrochloric acid. Of the inorganic substances the addition of which promoted self-heating, potassium carbonate had the most marked action, but no correlation could be found between the potassium content of a coke and its liability to spontaneous combustion. The addition of marcassite, iron sulphide, or calcium sulphide had no marked influence on the spontaneous heating of the semi-coke.

**美国的集团**主义

A. B. MANNING.

Influence of inorganic materials in lignite carbonisation. A. W. GAUGER and D. J. SALLEY (Fuel, 1929, 8, 79-85).—The effect of the addition of 5% of various inorganic constituents on the character and yields of the products from the carbonisation of a North Dakota lignite has been studied. 150-225 g. of the material were briquetted and carbonised either at 510° in an aluminium retort as used by Fischer, or at 850° in a copper retort of similar construction. The addition of the hydrated salts of aluminium, magnesium, or strontium alters the structure of the char produced, yielding a hard pseudo-coke differing materially from the soft powdered char of the untreated coal, whereas addition of the anhydrous salts produces soft, fine chars. The addition of hydrated ferric chloride increases the yield of char at the expense of the tar. The addition of sodium carbonate decreases the yields of char and tar and increases the gas yield. Addition of silica has no A. B. MANNING. effect.

Briquetting of coal dust without the use of binding materials. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAŻY (Przemysl Chem., 1929, 13, 2-12, 40-49) .---A well-defined point of maximum plasticity is found for coking but not for non-coking coals; in no case is this point attained below 400° or above 480°. The resistance to erosion or mechanical shock of briquettes made of coking coal is small compared with that of ordinary briquettes; the greatest strength is obtained if briquetting is carried out at such a temperature that the surface of fracture has a vitreous appearance (about 450°). Non-coking coals give much the stronger briquettes, the optimum temperature in this case being 405-415°, the time of heating 30-40 min., and the pressure 400-600 kg./cm.<sup>2</sup>, or twice that used ordinarily. The best results are obtained when mixtures containing under 30% of coking coal are used. Inferior briquettes are yielded by coal dust the grains of which are <0.02mm. or >3 mm. in diam. Briquettes so prepared are slightly more resistant to erosion, but not to shock, than those made with the use of binding material; their ignition point is more than 100° lower than that of ordinary briquettes; they burn with the production of less smoke; and their calorific value is higher than that of the original coal, owing to their low water content. R. TRUSZKOWSKI.

Adsorption of pyridine vapour by coal from Upper Silesia and by its petrographic modifications. W. SWIENTOSLAWSKI and M. CHORAZY (Bull. Acad. Polonaise, 1928, A, 361-365).—Vitrain, durain, and fusain separated from the coal of Upper Silesia absorb in 26 days 70.4%, 54.5%, and 12.8%, respectively, of their dry weight of pyridine vapour. The greater is the coking power of the coals, the greater are the amounts of pyridine absorbed. W. E. DOWNEY.

Chemical composition of peat. II. Chemical composition of various peat profiles. S. A. WAKS-MAN and K. R. STEVENS (Soil Sci., 1928, 26, 239-251).---Using the methods previously described (B., 1928, 880), a study was made of peats from a number of different sources. The chief interest lies in the comparison of high- and low-moor peats, which, by reason of their different floral origin, differ distinctively in the products and manner of their decomposition and serve to illustrate modifications of the general scheme of humification of organic matter. H. J. G. HINES.

Humic acids. G. STADNIKOV and P. KORSCHEV (Kolloid-Z., 1929, 47, 136-141).-The course of the adsorption of barium hydroxide by humic acid has been followed by titration with hydrochloric acid. Under the experimental conditions, marked adsorption occurred during the first 4 or 5 hrs. and thereafter slowly, not reaching a final value after 120 hrs. The amount of barium hydroxide adsorbed does not depend on the concentration of the solution, thus resembling the result obtained by Kawamura (A., 1926, 1201) using stearic acid, but differing from that obtained by this author with artificial humic acid obtained from sugar. The results indicate a value of 147 for the equivalent weight of humic acid. Experiments have also been performed on the reactions of barium humate with the chlorides and nitrates of sodium and potassium. These reactions take place slowly, reaching equilibrium after about six days. During the reaction the concentration of the soluble humate varies proportionally with the concentration of the alkali salt in the solution ; the concentration of the barium salt in solution varies in a complicated way, which is expressed by a curve very similar to the ordinary adsorption curve. An explanation of these facts is attempted. E. S. HEDGES.

Methods of heating coke ovens. E. WEISE (Gas- u. Wasserfach, 1929, 72, 73-78).-The calculated temperatures obtainable by burning coke-oven gas, blast-furnace gas, and mixtures of the two with different degrees of preheating of gas and air and different excesses of air are tabulated, with and without consideration of dissociation within the gases at high temperatures. The total and useful heat contents per cub. m. of the heating gases, together with the available heat per cub. m. of the flue gases, are calculated ; also the cost per cub. m. of coke-oven gas under different combustion conditions is compared with that of a standard coal giving an equal thermal efficiency when burnt under standard conditions. It is estimated that, with cokeoven gas, mixed gas I (1 vol. of coke-oven gas : 1 vol. of blast-furnace gas), mixed gas II (1 vol. of coke-oven gas : 3 vols. of blast-furnace gas), and blast-furnace gas (net cal. values 4336, 2620, 1763, and 904 kg.-cal./cub.m.,

respectively), the relative volumes of gas required to yield a given quantity of available heat are as 1:1.76:2.26:3.98. W. T. K. BRAUNHOLTZ.

Automatic combustion control. T. A. PEEBLES (J. Inst. Fuel, 1929, 2, 131—140).—The difficulties of the efficient working of fuel-burning equipment, together with descriptions of automatically controlled plants using steam coal, pulverised coal, and blast-furnace gases, are given. C. B. MARSON.

Determination of the sulphur content of gases, especially of coke-oven gas. F. HEINRICH and F. PETZOLD [with E. SCHUTH] (Z. anal. Chem., 1929, 76, 120-134).—The gas is passed at the rate of 0.2litre/min. through a solution consisting of 180 c.c. of 0.5N-sodium hydroxide and 90 c.c. of 5% hydrogen peroxide solution distributed between two absorption flasks; the initial and final volume of the gas being known, the sulphur content may be determined by titration of the solution with 0.5N-sulphuric acid. The method is applicable to gases containing sulphur in any inorganic or organic compound with the exception of thiophen. H. F. GILLBE.

Hydrocarbons in Peruvian petroleum having b.p. below 150°. W. F. SEYER and A. F. REES (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 359–360; cf. B., 1926, 653).—The oil used ( $d^{20} 0.8340$ ) was from the Lobitos Oil Field, in Peru. The distillate boiling below 175° was collected and fractionated. The physical properties indicate the presence of the hydrocarbons  $C_6H_{14}, C_7H_{14}, C_8H_{16}$ , and  $C_9H_{18}$ . A. J. MEE.

Ultramicroscopy of asphalts and allied products. F. J. NELLENSTEYN and J. P. KUÏPERS (Kolloid-Z., 1929, 47, 155-160).-Former ultramicroscopical investigations on solutions of asphalt and of coal tar (B., 1928, 324) have been repeated and the results confirmed. The observation of other investigators, that solutions of artificial asphalts do not show the presence of ultramicrons, cannot be substantiated; the difference is merely that natural asphalts give a better ultramicroscopical effect at higher dilutions. Under the ultramicroscope a mixture of artificial asphalt with a finelydivided inorganic powder produces the same effect as natural asphalt. When mixed with soot, solutions of asphalt in nitrobenzene or aniline resemble those of coal tar, both microscopically and ultramicroscopically. E. S. HEDGES.

Characterisation and classification of gasolines. T. NOWOSIELSKI (Przemysl Chem., 1929, 13, 16–23, 66–78).—The average b.p. of gasolines is a much more instructive feature of a given sample than is its sp. gr., which gives little indication of the volatility. For practical purposes, the average b.p., the percentage distilled over at this temperature, and the b.p. range of a given gasoline suffice to characterise it. The average b.p. of a mixture of gasolines can be calculated with a mean error of  $1.5^{\circ}$  from those of the individual components, on the basis of the rule of mixtures.

R. TRUSZKOWSKI.

Fuels—alternative or supplementary to petrol for use in§internal-combustion engines for road vehicles. I. Liquid fuels. W. R. ORMANDY (J. Inst. Fuel, 1929, 2, 199-204).—The production and economic value of such liquid fuels from high- and lowtemperature distillations of coal and from lignite, oil shales, hydrogenation of lignites or bituminous coals, and treatment of water-gas by catalysts are discussed, together with the manufacture and advantages of alcohol. C. B. MARSON.

Voltol. K. WOLF (Petroleum, 1929, 25, 95–98).— Lubricating oils are produced by subjecting mineral oils, tar, fatty oils, or mixtures of mineral and fatty oils to a glowing electrical discharge, alternating current at 5000 volts being employed. The process is carried out at about 80° and in a vacuum of 60–70 mm., using aluminium electrodes. W. S. NORRIS.

Standardisation of oil testing. M. FRIEDEBACH (Petroleum, 1929, 25, 93—95).—The variation in flash point observed with the thermometer in different positions in the sample of oil is not greater than the experimental error (cf. Schlüter, B., 1928, 325). The thermometer is best placed towards the centre of the oil, but a precise mechanism for locating it is a needless complication. Rate of heating, location of the ignition flame so that local heating does not occur, and constancy in the length of the flame are more important factors. A simple flash-point apparatus is described.

W. S. NORRIS.

"Tar value " of lubricating oils. S. GASIOROWSKI and S. PILAT (Przemysl Chem., 1928, 12, 235-239; Chem. Zentr., 1928, ii, 613-614).

Humic acids. Pyrohymatomelanic acid. D. J. W. KREULEN (Chem. Weekblad, 1929, 26, 101-103).-See B., 1928, 555.

Paint resistant to flue gas. VAN WÜLLEN-SCHOLTEN. See XIII.

# DA PATENTS.

Vertical rotary retort for low-temperature carbonisation. G. POLYSIUS (G.P. 452,620, 25.9.24).— The retort comprises an outer drum and an inner concentric cylinder of perforated metal, the material to be carbonised occupying the annular space between the cylinders. The walls of the drums are made smooth in order to facilitate the discharge of the carbonised material. A. B. MANNING.

Production of liquid hydrocarbons by the carbonisation and hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,343, 17.10.27).—In processes in which carbonaceous materials are heated in the presence of gases or vapours for the production of low-boiling hydro-carbons, e.g., in the destructive hydrogenation of coal, tar, etc., increased yields are obtained by giving the gases in the reaction vessel a rapid oscillating movement or by repassing them repeatedly through the chamber, so that their rate of flow therein is increased. At the same time the partial pressures of the substances under treatment are kept as low as possible. A. B. MANNING.

Production of hydrocarbons from coal and water: A. UHLMANN (B.P. 283,177, 5.1.28. Ger., 6.1.27).—Finely-divided coal or other carbonaceous material is mixed to a pulp with water and is driven through a pipe in which it is subjected to the action of a

high-frequency field produced preferably by a pulsating continuous current. Locally produced high temperatures ("micro-carbon arcs") decompose the water and bring about hydrogenation of the coal with the production of hydrocarbons. A. B. MANNING,

Working-up the oil-bearing residues of destructive hydrogenation of carbonaceous materials or the products obtained by extraction of substances of the nature of coal. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,404, 5.11.27).—Oil-bearing residues are mixed with finely-divided carbonaceous material, e.g., coal dust, wood flour, etc., and are subjected to low-temperature carbonisation.

A. B. MANNING.

Separation of hydrocarbon oils from mixtures thereof with solid substances resulting from industrial processes in which carbonaceous materials are subjected to a heat-treatment. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,941, 20.6., 3.10., and 29.12.27) .- The mixture of hydrocarbon oils and solids, e.g., from low-temperature carbonisation of coal, destructive hydrogenation, or cracking operations, is stirred with an aqueous liquid containing a wetting or emulsifying agent, at raised temperature if desired, and the whole is centrifuged. With light oils, an aqueous layer is formed between the solids and the oil; heavy oils form the middle layer. Salt solutions, e.g., 40-50% aqueous calcium chloride, may be used, and in some cases addition of alkali is advantageous. Alternatively, the oils may be emulsified in water, the emulsion separated from the solids, and the oils recovered by destroying the emulsion. Oxidation products of paraffin wax are suitable emulsifying agents in alkaline solution. C. HOLLINS.

Working-up of vegetable waste. GES. F. LUPINEN-IND. M.B.H. (G.P. 452,810, 12.4.25).—Vegetable waste of high potash and phosphoric acid content is carbonised and briquetted in order to form a fuel suitable for use in a gas generator. The potassium salts and phosphates can then be recovered from the ash by extraction. A. B. MANNING.

Agglomerated fuel. Soc. FRANÇ. DES CHARBON-NAGES D'ALONG ET DONG-DANG (F.P. 629,394, 27.4.26).— Coal dust is impregnated with a solution obtained by treating vegetable tissue with alkalis or reducing agents under pressure at various temperatures, and the mixture is then briquetted and dried. A. B. MANNING.

Compound fuel. P. H. LÉVÈQUE, ASST. to SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (U.S.P. 1,693,790, 4.12.28. Appl., 4.10.27. Ger., 11.10.26. Renewed, 12.10.28).—Anthracite is mixed with coal which has been purified by the Trent or similar oil process. The mixture is compressed, the oil acting as a binder, and then distilled. F. G. CLARKE.

Treatment of gases arising in the distillation or carbonisation of coal and like carboniferous material. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & CO. (B.P. 304,333, 15.7.27).—The crude hot gases from the carbonisation of coal and similar processes are brought into contact with salt solutions which act as a cooling medium, and are of such composition that they will take up water from the gas but not ammonia. Their sp. gr. is sufficiently high that any insoluble constituents which are condensed will float on the surface. Solutions of ammonium salts, alone or with other salts, are suitable for the purpose. The diluted solution is reconcentrated by exposure to the atmosphere, the evaporative effect being increased by bringing the solution into direct or indirect contact with the hot gases. A. B. MANNING.

Treatment of coal-distillation gases. IMPERIAL CHEM. INDUSTRIES, LTD., K. GORDON, and J. HUGHES (B.P. 304,345, 17.10.27) .- The principal constituents of coke-oven and similar gases, from which the tar, ammonia, and benzol have been removed, are separated by washing the gas under pressures of 100-250 atm. (a) with strong ammonia liquor which removes carbon dioxide, hydrogen sulphide, etc., (b) with an ammoniacal solution of a cuprous salt for the removal of carbon monoxide and unsaturated hydrocarbons (principally ethylene), and (c) with kerosene for the absorption of methane (cf. B.P. 293,138; B., 1928, 670). The liquor produced in the first washing may be used for the preparation of ammonium sulphate by interaction with calcium sulphate. The gases released from the ammoniacal cuprous solution may be washed with silver nitrate solution in order to separate the ethylene from the carbon monoxide (cf. B.P. 291,186; B., 1928, 560), or these gases may be released separately from the solution by suitable adjustment of the pressure and temperature. The methane recovered from the kerosene washing may be used for the production of acetylene or acetic acid.

A. B. MANNING.

Removal of dust from low-temperature gases. KOHLENVEREDLUNG A.-G. (G.P. 452,349, 9.5.22).—The gases, which pass from the retort through a dust separator to the condensing system, are heated before entering the separator, the amount of heat given to them corresponding with the heat subsequently lost in the separator. A. B. MANNING.

Purification of gases from organically combined sulphur. F. FISCHER (B.P. 282,634, 25.10.27. Ger., 24.12.26).—The organic sulphur compounds in gases which also contain hydrogen are converted into hydrogen sulphide by passage of the gas over finely-divided silver, or other precious metal of the first periodic group, at 200—300°. The hydrogen sulphide is subsequently removed in the usual manner. A. B. MANNING.

Removal of sulphur compounds from gases. F. NEUWIRTH (Austr. P. 107,600, 30.6.26).—The gases are passed through one or more layers of lignite.

A. B. MANNING.

Water-gas producer. KÖRTING & AHRENS G.M.B.H. (G.P. 452,401, 22.4.25).—The producer is surrounded by a fire-tube boiler and is so arranged that the blow gases pass up through an annular space between the producer and the boiler and then down through the tubes of the boiler and a waste pipe to the air. A. B. MANNING.

Continuous production of water-gas. Soc. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (F.P. 629,397, 27.4.26).—Pulverised fuels are treated with steam in an apparatus which is heated externally to a temperature necessary for the production of water-gas.

A. B. MANNING.

Treatment of coal tar. K. W. NIELSEN (Dan. P. 35,094, 24.10.25).—The warmed tar is treated with air in the presence of nitric acid or oxides of nitrogen in order to remove the free carbon. A. B. MANNING.

Separation of diphenylene oxide from coal tar. RÜTGERSWERKE-A.-G., Assees. of R. MÜLLER (G.P. 451,536, 6.12.25).—The fractions containing diphenylene oxide are treated with a dehydrogenating, polymerising, or condensing agent, *e.g.*, oxygen, lead or manganese oxides, halogens, sulphur, aluminium chloride, etc., under suitable conditions of temperature and pressure, with or without the addition of catalysts, and the diphenylene oxide is then recovered from the product by distillation or crystallisation. A. B. MANNING.

Preparation of stable emulsions of coal dust in oil. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 453,465, 2.6.26. Addn. to G.P. 444,420; B., 1928, 81). —Instead of alkali, ammonia or organic bases may be used as stabilising agents. Only phenols or oils rich in creosote are added to oils which already contain organic bases. Pyridine and its derivatives, quinoline, aniline bases, and the crude mixtures of bases from tar can be used. W. J. BoyD.

Fractional distillation of pyroligneous acid. Soc. Anon. des Prod. Chim. de Clamecy (F.P. 628,785, 22.4.26).-Methyl alcohol, acetone, and methyl acetate are removed from the crude acid by distillation with benzine, carbon disulphide, or a halogenated hydrocarbon, and when the temperature reaches 69° and the low-boiling constituents have been removed the solvent is expelled by a current of steam. The residue is distilled, using a second column, so that water passes over at 98-100°, after which residual volatile impurities are expelled by treatment with steam at 70-97°. The lower aqueous layer separating in the receiver is withdrawn, and the upper oily layer is returned to the column. The residual concentrated acetic acid is freed from tar by extraction or distillation. L. A. COLES.

Pitch-coking process and product. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,694,800, 11.12.28. Appl., 10.11.19).—A mixture of pitch, bauxite, and coke breeze is subjected to coking treatment.

L. A. COLES. Manufacture of impregnating and adhesive compositions. J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 302,710, 17.9.27).—Oily or fatty pitches, e.g., stearine pitch, are heated with vegetable or animal oils, which may have been oxidised and/or polymerised, and sulphur or substances yielding it, e.g., thionyl chloride, material extracted from a tar and containing sulphur being added before, during, or after the sulphurising treatment. Pitch, asphalt, resins, fillers, etc. may also be added. L. A. COLES.

Operation of pressure stills [for cracking of hydrocarbon oils etc.]. J. E. BELL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,693,946, 4.12.28. Appl., 26.7.24).—Vapours from the still are refluxed in a zone separate from the vaporising zone in direct contact with fresh charging stock, and the reflux and unvaporised charging stock are introduced into the still stock that is to be immediately subjected to high temperature. The vapours pass through a series of condensing

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chambers, maintained at progressively lower temperatures by heat-interchange, in direct contact with fresh charging stock passing to the still, and a part of the condensate is refluxed from chambers of lower temperature to those of higher temperature. H. S. GARLICK.

Cracking of oil. R. T. POLLOCK, ASST. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,830, 23.10.28. Appl., 6.12.20. Renewed, 16.7.28).—Vapours from the cracking chamber are dephlegmated in presence of fresh charging oil and water or steam. The reflux is returned to the heating coil after separation of any water.

R. BRIGHTMAN.

Conversion of hydrocarbon oils. O. C. BREWSTER, Assr. to STANDARD OL CO. (U.S.P. 1,694,092, 4.12.28. Appl., 20.6.25).—Regulated air supply is used to maintain hydrocarbon oil, e.g., crude residue, d 0.933— 0.939, at conversion (distillation) temperature (380—  $405^{\circ}$ ) by internal oxidation under pressure, the feed oil being preheated similarly. Vapours from the still pass through a stripping column in countercurrent heatexchange with the feed oil, preheated to, e.g., 360—385°, the reflux and fresh oil passing back to the still, while the vapours pass to a condenser and receiver in communication with the uncondensed vapour from the feed preheater. R. BRIGHTMAN.

Converting [cracking] hydrocarbons into lowerboiling products. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,262, 4.12.28. Appl., 21.5.23).—Relatively clean oil, e.g., light gas oil, is vaporised in a heating coil at 480—540° and passed into a heat-insulated chamber in contact with heavy gas oil or other relatively dirty or coke-forming stock. The liquid and vapour products are then passed by a connexion having a pressure-release valve to a vapour separator, from which the tar is drawn off and the light and heavy vapours are passed to condensers.

## R. BRIGHTMAN.

Treating [cracking] hydrocarbons. A. SCHWARZ, Assr. to PETROLEUM SAND PRODUCTS CORP. (U.S.P. 1,691,085, 13.11.28. Appl., 1.3.24).—Heavy hydrocarbon oil is cracked as a vapour-oil emulsion by violent agitation, e.g., 400—600 r.p.m., at cracking temperature (cf. U.S.P. 1,458,443; B., 1923, 918 A).

#### R. BRIGHTMAN.

**Fractionation of hydrocarbons.** J. C. BLACK (U.S.P. 1,692,072, 20.11.28. Appl., 2.2.24).—Vapours from a continuous still are scrubbed with a "light intermediate" fraction in a condensing chamber, a, and passed into a chamber, b, from which the lighter vapours ascend through boiling-decks to the condenser, after heat-exchange with the feed to the still. Reflux from b is used to scrub the vapours in a, any surplus being stored as "light intermediate" or steam-distilled so that the vapours ascend through the boiling decks in b, while the reflux is stored as "heavy intermediate." The reflux from a is similarly treated, the light vapours ascending through the boiling decks in b.

R. BRIGHTMAN. [Steam] fractionation [of hydrocarbons]. I. B. FUNK, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,691,238, 13.11.28. Appl., 30.10.26).—In order to obtain close "cuts" when using fractionating columns

of the type having perforated plates with funnels, which lead the condensed liquid to the plate immediately below, horizontal partitions are placed in the upper part of the column to condense the steam. These partitions have apertures fitted with bubble caps, and the condensed liquids are led into traps outside the column. Vapour and oil are returned to the latter through separate tubes, and the water is removed from the bottom of the traps. F. G. CLARKE.

Treatment of liquids [petroleum oils]. L. CALD-WELL, Assr. to CELITE Co. (U.S.P. 1,691,266, 13.11.28. Appl., 27.1.26).—Petroleum oil is refined by treatment with the solid obtained by chlorinating the reaction product of silica (diatomaceous earth) and excess of calcium hydroxide. R. BRIGHTMAN.

Removal of petrolatum from oils. C. ZWEIG, Assr. to STANDARD OIL Co. (U.S.P. 1,694,090, 4.12.28. Appl., 21.6.26).—Oil containing petrolatum is diluted and agitated at a low temperature before settling to separate the wax. H. S. GARLICK.

**Production of thickened lubricating oil.** R. W. HENRY and E. D. STREETER, Assrs. to GULF REFINING Co. (U.S.P. 1,691,882 and 1,691,654, 13.11.28. Appl., [A, B] 24.3.26. Renewed, [B] 3.10.28).—Basic aluminium oleate, prepared, e.g., (A) by adding an aluminium salt to an emulsion of mineral oil and neutral aqueous salt solution, partly hydrolysing with alkali hydroxide, and adding an aluminium salt to reconvert neutral scap thus formed into aluminium trioleate, is used as thickener. (B) A basic aluminium salt solution, obtained by the addition of sodium hydroxide in presence of sodium citrate or other protective agent, is added to the neutral scap solution. R. BRIGHTMAN.

Reclaiming [lubricating oil] distillation residues. R. E. POWELL, ASST. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,694,280, 4.12.28. Appl., 25.2.25).—Still residues, e.g., from the distillation of lubricating stock containing naphthenic acids in presence of sodium hydroxide, are refluxed at 150—350° with 25% of gas oil and steam injection, and the residue is discharged hot on to the surface of a stream of water. After settling, the oil is separated from the aqueous naphthenate solution, continuously or in stages.

R. BRIGHTMAN.

Manufacture of motor fuel. L. KIRSCHBRAUN (U.S.P. 1,692,176, 20.11.28. Appl., 19.11.19. Renewed 12.5.28).—Fuel for internal-combustion engines is obtained by dispersing water in, *e.g.*, gasoline, with the aid of ammonia solution and phenol or naphthenic acid as emulsifier. R. BRIGHTMAN.

Fuel and fuel ingredients. J. W. ORELUP and O. I. LEE, Assrs. to BOYCE-ITE PRODUCTS, INC. (U.S.P. 1,692,784, 20.11.28. Appl., 23.1.25).—Addition to petrol of 0.1—3.0% of higher fatty acids or their esters, amides, halogenated derivatives, or metallic salts reduces the deposition of carbon in internal-combustion engines. As example, 0.1% of mixed esters, containing 60% of ethyl laurate, is mixed with an equal volume of o-nitrotoluene and added to the petrol.

R: BRIGHTMAN.

Ethyl alcohol substitute and its manufacture.

R. M. ISHAM, ASST. to DOHERTY RES. Co. (U.S.P. 1,692,662, 20.11.28. Appl., 6.5.26).—A mixture with an odour resembling that of ethyl alcohol is obtained from *iso*propyl alcohol by addition of 3 c.c. of acetaldehyde and 6 c.c. of butyl acetate per 5 gals.

R. BRIGHTMAN.

Manufacture of producer gas from coking coals. K. KOLLER (U.S.P. 1,697,920, 8.1.29. Appl., 18.7.25. Ger., 18.7.24).—See B.P. 254,392; B., 1926, 778.

Obtaining hydrogenation gas from hydrogenating carbon and hydrocarbons from gases containing methane and hydrogen. F. BERGIUS (U.S.P. 1,699,177, 15.1.29. Appl., 30.11.25. Ger., 18.12.24).—See B.P. 244,730; B., 1927, 66.

Separation of paraffinous constituents from fluid hydrocarbons. E. A. FORSBERG, Assr. to AKTIE-BOLAGET SEPARATOR-NOBEL (U.S.P. 1,699,286, 15.1.29. Appl., 28.6.27. Swed., 24.8.26).—See B.P. 276,658; B., 1929, 161.

Hydrogenation of tar oil. R. BERNHARD and W. DEMANN (U.S.P. 1,691,221, 13.11.28. Appl., 25.6.25. Ger., 30.6.24).—See B.P. 236,230; B., 1926, 973.

Coal washing apparatus. E. COPPÉE & CIE. (B.P. 288,252, 29.2.28. Belg., 5.4.27).

Gas burners. V. H. RICHARD (B.P. 304,938, 23.2.28).

Catalytic gas reactions (B.P. 304,251).—See I. Olefines and diolefines (B.P. 303,998). Sulphonic acids (B.P. 303,917). Hydrogenation of naphthalene (B.P. 304,403).—See III. Waterproofed materials (B.P. 303,935). Textile oils (B.P. 303,820).— See V. Bituminous emulsions (B.P. 301,544). Paving composition (B.P. 302,808).—See IX.

## III.—ORGANIC INTERMEDIATES.

Humic acids. STADNIKOV and KORSCHEV. Sulphur content of gases. HEINRICH and others.—See II. Nitrogen in urea-nitrate mixtures. LUCAS and HIRSCHBERGER.—See XVI. Absolute alcohol. VON KEUSSLER.—See XVIII.

#### PATENTS.

Manufacture of olefines and diolefines [butadiene]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,998, 14.12.27).—Paraffins, olefines, or alicyclic hydrocarbons are passed in vapour form at  $640-720^{\circ}$ over lustrous carbon (B., 1923, 1130 A; 1927, 802) deposited on a non-reducible oxide such as magnesia, lime, baryta, quartz, or metals such as chromium; preferably steam is used as a diluent. *cyclo*Hexane at 650° gives 90% of olefines, chiefly butadiene and ethylene; American petroleum or synthetic benzine at 700—720° yields 80% of olefines, of which one fifth is butadiene.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 303,772, 7.7.27. Addn. to B.P. 256,663; B., 1926, 897).—The vapours containing the acid (acetic) anhydride are mixed with vapour of an entraining liquid (benzene, carbon tetrachloride, petrol, mixtures of these, or of ether with light petroleum, b.p. 40—70°) at above 100° but below the b.p. of the anhydride. Water is thus carried away with the entraining liquid, and the anhydride is condensed. C. HOLLINS.

Manufacture of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 279,070, 5.10.27. Ger., 18.10.26).—A vaporous catalyst is used, preferably phosphoric acid entrained to the extent of 0.5 g. per kg. of acetic acid vapour at 600°. C. HOLLINS.

Production of acetone. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 280,184, 24.10.27. Ger., 4.11.26).—Acetylene and steam are passed at 470—500° over a heavymetal oxide (iron oxide) mixed with a promoter which increases the oxygen tension of the oxide to about the partial pressure of the oxygen in the reacting gases, so that no substantial reduction occurs. Examples are superficially rusted iron shavings, iron shavings coated with iron oxide and manganese oxide or with iron oxide and barium carbonate. C. HOLLINS.

Production of esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,411, 22.9.27).—Soaps, especially the fatty acid salts of tin, zinc, or magnesium, added in small proportion (0.1%) accelerate the esterification of higher fatty acids with polyhydric alcohols. C. HOLLINS.

Manufacture of substituted thioglycollic acids. I. G. FARBENIND. A.-G. (B.P. 287,178, 16.3.28. Ger., 18.3.27. Addn. to B.P. 281,290; B., 1928, 921).— The method of the prior patent is applied to the production of trihalogeno-derivatives of benzene and toluene. 1:2:3-Trichlorobenzene, m.p.  $53-54^{\circ}$ , yields with chlorosulphonic acid at 20—70° solely the 4-sulphonyl chloride, m.p.  $65-66^{\circ}$ , from which are obtained 2:3:4trichlorothiophenol, and 2:3:4-trichlorophenylthioglycollic acid, m.p.  $149^{\circ}$ . 2:3:4-Trichlorotoluene, m.p.  $41^{\circ}$ , similarly yields the 5-thioglycollic acid, m.p.  $157-161^{\circ}$ , and 1:2:4-trichlorobenzene, m.p.  $17^{\circ}$ , yields 2:3:5trichlorophenylglycollic acid. C. HOLLINS.

Preparation of the lactone of 14-oxy- [ $\omega$ -hydroxy-] tetradecane- $\alpha$ -carboxylic acid. Soc. Anon. M. NAEF & CIE., Assees. of M. NAEF & CIE. (B.P. 294,602, 26.6.28. Switz., 27.7.27).—The lactone of  $\omega$ -hydroxypentadecoic acid, m.p. 30°, b.p. 175°/11 mm.,  $d_4^n$  0.9383,  $n_5^n$  1.4633, is obtained by heating *cyclopentadecanone* with Caro's acid. It has a musk odour with a flavour of amber. The corresponding hydroxy-acid, m.p. 84°, is oxidised by chromic acid to tridecane- $\alpha\omega$ -dicarboxylic acid, m.p. 112°. C. HOLLINS.

[Catalyst for] catalytic preparation of oxygenated carbon compounds. E. I. DU PONT DE NEMOURS & Co., Assees. of W. A. LAZIER (B.P. 301,806, 13.6.27. U.S., 12.6.26. Cf. B.P. 272,555; B., 1929, 163).— A chromium mixed catalyst is prepared by heating a double chromate of a nitrogenous base and zinc, copper, cadmium, magnesium, manganese, silver, or iron. Catalysts from basic zinc ammonium chromate, zinc tetrapyridine dichromate, and manganese ammonium chromate are described. C. HOLLINS.

Hydrogenation of open chains containing nitrogen in unsaturated union. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 304,000, 20.12.27).— Unsaturated nitrogen linkings are reduced by treating the compound in aqueous solution with hydrogen in presence of nickel at 60—80°, an electrolyte being added which converts the reduction product (an amine) into a salt by double decomposition. Thus potassium cyanide is reduced to methylamine by hydrogen and nickel in presence of ferrous sulphate. Acetonitrile gives ethylamine; phenylacetonitrile,  $\beta$ -phenylethylamine; and azobenzene, cyclohexanol and cyclohexylamine.

C. HOLLINS.

Manufacture of aqueous "solutions" of organic compounds insoluble in water. I. G. FARBENIND. A.-G. (B.P. 273,757, 2.7.27. Ger., 2.7.26).—The sulphonic acids of saturated aliphatic hydrocarbons or saturated aliphatic acids, obtained as in B.P. 272,967 or 288,612 (B., 1928, 703, 865), are used for the solubilisation of water-insoluble organic compounds; *e.g.*, 20 pts. of methylcyclohexanol give a clear "solution" in 160 pts. of water to which have been added 70 pts. of a 50% aqueous solution of sulphonated lauric acid, and 8 pts. of *n*-butyl alcohol "dissolve" in 20 pts. of the same reagent neutralised with ammonia. Sulphonated paraffin oil is also effective. C. HOLLINS.

Production of sulphonic acids [wetting, emulsifying, and cleaning agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,917, 12.9.27).—Unsaturated aliphatic hydrocarbons or their halogen derivatives or mixtures containing these, *e.g.*, petroleum, brown coal tar oils, etc., are sulphonated by treatment with sulphuric acid, oleum, or chlorosulphonic acid in acetic anhydride. The products are acid-resisting wetting, emulsifying, and cleaning agents. C. HOLLINS.

Manufacture of substituted guanidines. SCHERING-KAHLBAUM A.-G. (B.P. 297,029, 13.8.28. Ger., 12.9.27). —Dialkylcyanamides (diethyl or dimethyl) react with amines mixed with their hydrochlorides to give trisubstituted guanidines. N-Phenyl-N'N'-diethyl- (picrate, m.p. 118°), NN-diethyl-N'-isoamyl- (picrate, m.p. 120°), NN-dimethyl-N'-isoamyl- (picrate, m.p. 104°), and NNdimethyl-N'-ethyl- (picrate, m.p. 152°) guanidines are described. C. HOLLINS.

Manufacture of disubstituted guanidines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,360, 19.10.27).—The reaction between cyanogen chloride or bromide with amines (aniline, ethylaniline, toluidines, benzylamine, methylamine, etc.) gives improved yields in presence of a salt of the amine ; *e.g.*, 5 pts. of aniline and 1 pt. of aniline hydrochloride or sulphate dispersed in carbon tetrachloride, water, etc. are treated with 1 pt. of cyanogen chloride at 0° and heated at 80—100° for 1 hr. C. HOLLINS.

Introduction of sulphocyanide [thiocyano-] groups into organic compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,813, 5.9.27. Cf. Söderbäck, B., 1920, 189 A).—Arylamines or phenols are treated with thiocyanogen or with a thiocyanate and halogen in an organic medium which is solvent both for the amine and the thiocyanate. The solvent may be protected from the halogen by saturating it with sodium chloride or bromide. *p*-Substituted compounds yield *o*-thiocyano-derivatives, convertible into benzthiazoles. Phenol and ammonium thiocyanate in methyl alcohol, treated with bromine in the same solvent at 15—20°, give p-thiocyanophenol, m.p.  $58^{\circ}$ . 4-Thiocyano- $\alpha$ naphthol, m.p. 118°, 5-chloro-3-thiocyano-o-toluidine, m.p. 102°, 3-thiocyano-p-phenetidine, m.p.  $85^{\circ}$ , 1thiocyano- $\beta$ -naphthylamine, 1-thiocyano-7-methoxy- $\beta$ naphthylamine, 2-amino-1-thiocyanoanthracene, m.p. above 300°, and 2 : 6-diamino-1 : 5-dithiocyanoanthracene are also described. C. HOLLINS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 296,071, 11.10.27, U.S., 26.8.27).—For a variety of catalytic oxidations (phthalic anhydride from naphthalene, anthraquinone from anthracene, benzaldehyde and benzoic acid from toluene, maleic acid from benzene, phenols, or furfuraldehyde, vanillic acid from eugenol, formaldehyde from methyl alcohol or methane, complete combustion of tars in coal-tar ammonia, etc.) a vanadium catalyst containing at least one multiple-component zeolite is used. An example is potassium vanadite, potassium silicate, and vanadyl sulphate, heated in burner gases at 400—500°, and, if desired, treated with dilute solutions of heavy-metal salts to effect baseexchange. Eight other examples are detailed.

C. HOLLINS. **Production of N-monoalkyl derivatives of aminophenols.** W. TRAUBE and E. HELLRIEGEL (B.P. 280,553, 9.11.27. Ger., 15.11.26).—*p*-Aminophenol is converted by means of aqueous potassium thiocyanate into the thiocarbamido-compound, which on desulphurisation with lead acetate and alkali yields p-cyanamidophenol, m.p. 265°. This may be methylated with the usual agents to form p-cyanomethylamidophenol [phydroxyphenylmethylcyanamide], m.p. 133—134°, which is readily hydrolysed by acids, water, or alkalis to N-methyl-p-aminophenol. C. HOLLINS.

Manufacture of N-[hydr]oxyethyl derivatives of 4-amino-1-[hydr]oxybenzene [p-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 290,997, 29.12.27. Ger., 23.5.27. Addn. to B.P. 280,873; B., 1929, 38). p-Aminophenol is treated in aqueous suspension with ethylene oxide in presence of calcium carbonate to give the N- $\beta$ -hydroxyethyl and NN-di-( $\beta$ -hydroxyethyl) derivatives. C. HOLLINS.

Manufacture of condensation products from m- or p-cresol and aliphatic ketones. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,684, 15.6.27. Ger., 29.6.26. Cf. Gaebel, A., 1902, i, 767).—m-Cresol condenses with acetone at 30—40° in presence of hydrogen chloride to give a compound, b.p. 187°/0.8 mm. (acetyl derivative, m.p. 122—123°), said to be a polymeride of 4-isopropenyl-m-cresol or of the cyclic anhydride of 4-j-hydroxyisopropyl-m-cresol. p-Cresol similarly yields a compound, b.p. 192°/0.8 mm. (acetyl derivative, m.p. 151—152°). C. HOLLINS.

Manufacture of alkylisopropenylphenols and alkylated coumarans. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,686, 15.6.27. Ger., 29.6.26).—The products from *m*- and *p*-cresols and acetone (B.P. 273,684, preceding) are depolymerised by distillation at 300°/100 mm., yielding 3 : 6- and 3 : 5dimethylcoumarans, b.p. 98°/11 mm. and 102°/11 mm., respectively, together with *iso*propenyl-*m*- and -*p*-cresols. C. HOLLINS. Manufacture of alkylisopropylphenols and their hydrogenation products. SCHERING-KAHLBAUM A.-G. (B.P. 279,855, 25.10.27. Ger., 26.10.26).—The isopropenyl-m- and p-cresols and dimethylcoumarans of B.P. 273,686 (preceding) are hydrogenated in presence of nickel until 2—8 hydrogen atoms are taken up.

C. HOLLINS.

Manufacture of thymol, its isomerides or homologues, and their hydrogenation products. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 276,010, 11.8.27. Ger., 16.8.26).—The direct condensation product from acetone and *m*-cresol (B.P. 273,684, preceding) takes up 2 atoms of hydrogen in presence of nickel and the corresponding aluminium phenoxide at 180—190° and 10—13 atm. to give thymol. The product from *p*-cresol yields 3-*iso*propenyl-*p*-cresol. The corresponding *cyclo*hexanols may be similarly prepared. C. HOLLINS.

Manufacture of thymol, its isomerides and their hydrogenation products. SCHERING-KAHLBAUM A.-G. (B.P. 280,956 and 280,924, [A] -21.11.27, [B] 16.11.27. Ger., [A] 22.11.26, [B] 16.11.26. Addns. to B.P. 276,010; preceding).—(A) The ethers obtained from *m*- or *p*-cresol and acetone are applied, in place of the normal condensation products, in the process of the prior patent. (B) The normal condensation products are hydrogenated at 280° instead of the usual 180°. C. HOLLINS.

Manufacture of substituted indoles. A. CARP-MAEL. From I. G. FARBENIND. A.-G. (B.P. 303,467, 4.10.27).—Monoacyl derivatives of arylamines containing an ortho-methyl group are converted into indoles by heating with sodamide at 205° in a diluent such as dimethylaniline, naphthalene, etc. Aceto-o-toluidide gives 80—84% of 2-methylindole; 2:5- and 2:7dimethyl- and 2-phenyl-indoles are similarly prepared. C. HOLLINS.

Hydrogenation of naphthalene. TECHNICAL RES. WORKS, LTD., and E. J. LUSH (B.P. 304,403, 4.11.27).— Pure tetrahydronaphthalene is obtained by passing naphthalene vapour and hydrogen over a nickel catalyst at a temperature and with an excess of hydrogen such that condensation of the naphthalene on the catalyst does not occur. B. FULLMAN.

Manufacture of new carboxylic acids of the fattyaromatic series. A. CARPMAEL. From I. G. FARBEN-IND. A.-G. (B.P. 303,389, 1.7.27).— $\alpha$ -Unsaturated carboxylic acids or their hydrogen halide additive compounds are condensed with aromatic hydrocarbons containing more than two fused benzene frings, in presence of an acid catalyst (hydrogen halides, aluminium chloride) and a fdiluent if desired. Anthracene with acrylic or  $\beta$ -chloropropionic acid yields a *compound* (I), m.p. 187—188°; with maleic acid a  $\beta$ -lactone (II), m.p. 255°; with cinnamic acid a *compound* (analogous with I), m.p. 232°. Similar *products* are obtained



from acrylic or  $\beta$ -chloropropionic acid and *ms*-dichloroanthracene (m.p. 237°), 1:5-dichloroanthracene (m.p. 218°), 2:7-dimethylanthracene (m.p. 205°),  $\alpha$ -anthrol, or phenanthrene (m.p. 132°). C. HOLLINS.

Manufacture of aminoanthraquinones. H. BERTHOLD, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,691,428, 13.11.28. Appl., 27.10.27. Ger., 2.12.25).- $\alpha$ -Nitroanthraquinones are reduced with hydrogenated quinolines in presence of acetic acid, pyridine, o-dichlorobenzene, quinaldine, or other diluent. The final mixture of quinaldine and excess unoxidised tetrahydroquinaldine may be catalytically hydrogenated and used again. 1:5-Dinitroanthraquinone affords 1:5-diaminoanthraquinone or 5-nitro-1-aminoanthraquinone; 1:5-dichloro-4-aminoanthraquinone and 8-nitro-1-amino-anthraquinone are similarly obtained. R. BRIGHTMAN.

Manufacture of benzanthraquinone derivatives and substitution products. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,263, 18.10.27).—4-o-*Carboxybenzoyl-1*: 8-naphthalic anhydride, m.p. 232°, obtained by oxidation of the acenaphthene derivative, is (unlike the latter) readily cyclised by means of sulphuric acid at 170—200° to give  $\alpha\beta$ -benzanthraquinone-4:5dicarboxylic anhydride (annexed formula), m.p. above



300°; the *imide*, m.p. above 300°. the N-methylimide, m.p. 280°, and the 8:11-dichloroanhydride are synthesised in a similar manner, the last-mentioned from 3': 6'-dichloro-2'carboxybenzoyl-1:8-naphthalic

anhydride, m.p. 274°. The condensation product of 4-o-carboxybenzoyl-1:8-naphthalic anhydride with o-phenylenediamine, viz., 4-o-carboxybenzoyl-1:8naphthoylenebenziminazole, may also be cyclised to the corresponding benzanthraquinone, which is a vat dye. C. HOLLINS.

Manufacture of dibenzanthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,538, 6.9.27).— Improved yields of dibenzanthrone are obtained in presence of aliphatic, non-sugar aldehydes or their bisulphite compounds or sulphoxylic acids, *e.g.*, paraformaldehyde, formaldehyde-bisulphite, sodium formaldehydesulphoxylate. C. HOLLINS.

Production of benzanthrone derivatives [isodibenzanthrones]. I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 303,454, 24.6. and 20.7.27).—Benzanthrone is converted smoothly into isodibenzanthrone by heating with alcoholic potassium hydroxide in a diluent (xylene, benzene, chlorobenzene, kerosene, or petrol) at 130°. Similar results are obtained with 9- and 10-methylbenzanthrones, 4:4'-dibenzanthronyl, benzanthrones from 1-chloroanthraquinone (previously separated from the product of m.p. 180— 181°) or 2-chloroanthraquinone (freed from 4-chlorobenzanthrone). [Stat. ref.] C. HOLLINS.

Rectification of acetic acid. G. F. LEGENDRE, Assr. to Soc. des Etabl. Barbet (U.S.P. 1,698,798, 15.1.29. Appl., 12.8.25. Fr., 14.8.24).—See B.P. 238,566; B., 1925, 899.

Manufacture of alkali salts of halogenated amides of aromatic sulphonic acids. H. GÜNZLER, Assr. to WINTHROP СНЕМ. Co., INC. (U.S.P. 1,699,888, 22.1.29. Appl., 12.10.25. Ger., 18.10.24).—See B.P. 241,579; B., 1926, 565.

Halides of aromatic oxamic acids. J. HALLER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,685,698, 25.9.28. Appl., 26.11.26. Ger., 25.11.25).—See B.P. 282,891; B., 1928, 151.

Treatment of coal-distillation gases (B.P. 304,345). Diphenylene oxide from coal tar (G.P. 451,536). Pyroligneous acid (F.P. 628,785).—See II. Vat dye intermediates (B.P. 303,203).—See IV. Protecting copper apparatus against corrosion by carboxylic acids (B.P. 284,685).—See X. Esters of alcohols and fatty acids (F.P. 615,953).—See XII. Separation of mixed substances (B.P. 272,968). Alkylaminoalkylamino-derivatives of aromatic compounds (B.P. 274,058 and 302,984—5).—See XX.

#### IV.—DYESTUFFS.

Red Congo acid and Congo-red lakes. WEISER and RADCLIFFE.—See XIII.

## PATENTS.

New basic and acid dyes of the Rhodamine type. IMPERIAL CHEM. INDUSTRIES, LTD., and L. J. ALLCHIN (B.P. 303,243, 7.11.27).—Fluorescein chloride is condensed with an unsulphonated aminophenol (e.g., *p*-aminophenol) to give basic dyes which may be converted by sulphonation into blue, acid rhodamines of good fastness properties. The basic dyes may also be synthesised from hydroxyaryl-*m*-aminophenols and phthalic anhydride. C. HOLLINS.

Manufacture of [sulphide] dyes. Soc. CHEM. IND. IN BASLE (B.P. 290,179, 7.5.28. Switz., 7.5.27).— Chromable azo dyes are converted into their chromium derivatives, which are then heated with sodium polysulphide to give sulphide dyes. Examples are 1:2:4aminonaphtholsulphonic acid  $\rightarrow \alpha$ -naphthol or 5:8dichloro- $\alpha$ -naphthol for greenish-blue dyes.

#### C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series], and intermediate products thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,203, 30.9.27 and 19.6.28) .- Arylsulphonamido-benzanthrones, -dibenzanthronyls, or -dibenzanthronyl sulphides are obtained by the action of arylsulphonyl chloride on corresponding amines, or of arylsulphonamides on suitable halogen derivatives, preferably in presence of acid-binding agents and/or catalysts. Those containing in position 3 or 4 the arylsulphonamido-group or another group eliminated during alkaline fusion are converted by alkaline fusion into vat dyes containing free aminogroups. Thus, 3-benzenesulphonamidobenzanthrone, m.p. 273°, gives by fusion with alcoholic potassium hydroxide a reddish-blue vat dye; 3:9-di(benzenesulphonamido)benzanthrone, m.p. 287°, gives a grey to black vat dye ; 3-bromo-9-benzenesulphonamidobenzanthrone, m.p. 300°, a similar dye; 2-benzenesulphonamidobenzanthrone, a greenish-grey after hydrolysis of the benzenesulphonyl residue ; di(benzenesul phonamido)-2 : 2'-dibenzanthronyl, grey to black ; the 3 : 3'-isomeride, olive-grey to black ;

6:6'-di(benzenesulphonamido)-3:3'-dibenzanthronyl sulphide, after hydrolysis, bluish-grey to black. C. HOLLINS:

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,184, 27.6.27).—8:9-Dimethyl-ms-benzdianthrone (I) is converted by alkaline condensing agents (baryta in boiling nitrobenzene, alcoholic potassium hydroxide at 170—175°, dry potassium hydroxide at 210° or in boiling aniline) into "allo-ms-naphthadianthrone" (II), m.p. 349—351°, an orange vat dye. 6:11-Dichloro-8:9-dimethyl-ms-benz-



dianthrone, prepared from 4-chloro-1-amino-2-methylanthraquinone, similarly yields a brown vat dye. These undergo further cyclisation by loss of hydrogen from positions 1 and 16 when stirred with manganese dioxide in sulphuric acid or heated with aluminium chloride, giving ms-anthradianthrone and its 5: 10-dichloro-derivative, both of which are yellow vat dyes, and are obtainable by alkaline condensation of ordinary ms-naphthadianthrones. allo-ms-Naphthadianthrone (II) gives on chlorination with sulphuryl chloride in nitrobenzene or with chlorine in trichlorobenzene a dichloro-derivative (orange); dibromo- (scarlet) and tetrabromo- (red) compounds are also prepared. Bromination of 5:10dichloro-allo-ms-naphthadianthrone yields a violet-red vat dye. Chloro- (orange) and dibromo- (orange) derivatives of ms-anthradianthrone may be obtained either by direct halogenation, or by cyclisation of halogenated allo-ms-naphthadianthrones. Dichlorodibromo-allo-msnaphthadianthrone, obtained by chlorination and bromination, is a brilliant red vat dye. C. HOLLINS.

Manufacture of new azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 301,175, 28.10.27).-Azo dyes giving yellowish-red to brown shades on wool, changed to blue, violet, or black shades on afterchroming, and reddish-brown shades when chromeprinted on cotton, are obtained by coupling diazo compounds with \$-naphthol-6-carboxylic acid. Reference is made to the dyes from aniline (yellowish-red), p-chloroaniline-o-sulphonic acid, p-nitro-o-aminophenol (brown), 2-nitro-6-aminophenol-4-sulphonic acid (violet), picramic acid, 1-amino-2-naphthol-4-sulphonic acid, 4-chloro-2aminophenol-6-sulphonic acid, p-nitro-o-aminosalicylic acid, o-aminophenol-p-sulphonic acid, 5-sulpho-3carboxy-2-hydroxybenzene-1-azo-4'-aminonaphthalene, 4-amino-1:2'-azonaphthalene-6:6'-disulphonic acid (dark violet), benzidine-mm'-disulphonic acid (reddish-brown), o-tolidine-m-disulphonic acid, m-dichlorobenzidinedisulphonic acid, 2-naphthylamine-1-sulphonic acid (red), o-toluidine-m-sulphonic acid, and acetyl-p-phenylenediaminesulphonic acid. The dyes obtained are superior in solubility and tinctorial power to corresponding dyes obtained from 2: 3-hydroxynaphthoic acid and afford yellower shades. The dyes from *p*-toluidine-*m*-sulphonic acid and aniline-*o*-sulphonic acids afford red barium and calcium lakes. R. BRIGHTMAN.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 275,258, 29.7.27. Ger., 29.7.26).—Diazo compounds are coupled with N-carbethoxyaminonaphthols, e.g., p-aminoacetmethylanilide  $\rightarrow$  N-carbethoxy-J-acid (level wool dye), and m-4-xylidine  $\rightarrow$  N-carbethoxy-Hacid (bluish-red wool dye). C. Hollins.

Manufacture of azo dyes. A. CARPMAEL. From I.G. FARBENIND. A.-G. (B.P. 302,965, 22.9.27).-An amidosulphonylanthranilic acid, NRH·SO2·Ar(NH2)·CO2H, is diazotised and coupled with a coupling component to give wool dyes suitable for chroming or pre-chroming and fast to washing, fulling, carbonising, and light. 5-o-Anisididosulphonylanthranilic acid [4-amino-3-carboxybenzenesulphon-o-anisidide] is prepared by condensing 2-chloro-5-chlorosulphonylbenzoic acid with o-anisidine and then with ammonia under pressure. It couples with  $\alpha$ -naphthol-5-sulphonic acid (yellowish-red on wool, becoming bordeaux-red by after-chroming), or with (reddish-brown; phenyl-y-acid black-brown Other examples are the 5-sulphonanilide chroming). with 1 -(2'-hydroxy-3'-carboxy-5'-sulphophenyl)-3methylpyrazolone (reddish-yellow; greener on chroming or coppering); the 5-sulphonamide with acetyl-H-acid, pre-coppered (wine-red), or with β-naphthylamine-7sulphonic acid, pre-chromed (red-violet pigment for varnishes); the 5-sulphon-p-toluidide with 3-naphthol-8sulphonic acid, pre-coppered (brownish-orange), or a-naphthol-4-sulphonic acid, pre-coppered (copper-red), or β-naphthol-6-sulphonic acid, pre-coppered (yellowishred-brown), or 1-p-sulphophenyl-3-methylpyrazolone, pre-coppered (greenish-yellow); the 5-sulphonanilide with β-naphthylamine-7-sulphonic acid (reddish-orange : bordeaux-red on chroming; pre-chromed, a violet pigment for varnishes). C. HOLLINS.

Manufacture of arylazodiarylamines [dyes for wool and acetate silk]. I. G. FARBENIND. A.-G. (B.P. 276,372, 22.8.27. Ger., 21.8.26).—An aminoazocompound is condensed with a halogenonitrobenzene containing reactive halogen and at least one sulphonic or carboxyl group. Examples are 4-aminoazobenzene with 1-chloro-2:6-dinitrobenzene-4-sulphonic acid (yellow on acetate silk), or 1-chloro-2-nitrobenzene-4-sulphonic acid (yellow on wool or acetate silk), or 2-chloro-3:5dinitrobenzoic acid (reddish-yellow on wool, goldenyellow on acetate silk). C. HOLLINS.

Manufacture of a black trisazo dye. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,424, 1.9.27).—p-Nitroaniline is diazotised and coupled acid with  $\gamma$ -acid; the product is reduced with sodium sulphide, rediazotised, and coupled alkaline with  $\gamma$ -acid; the disazo dye is finally diazotised and coupled with *m*-phenylenediamine. A further sulphonic group may be introduced into any of the components; *e.g.*, 2:8aminonaphthol-3:6-disulphonic acid may replace the second mol. of  $\gamma$ -acid. The resulting dyes give deep black shades on cotton or viscose silk, but leave acetate silk unstained. C. HOLLINS. Manufacture of nitrosoamine printing colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 303,942, 28.10.27).—Mixtures of nitrosoamine salts and coupling components (e.g., the Rapid Fast series of printing colours) are freed from sand, dust, iron particles, and other obnoxious impurities by dissolution in a neutral organic solvent, filtering, and removing the solvent. Suitable solvents are acetone, alcohol, methyl alcohol. C. HOLLINS.

Benzanthrone derivatives (B.P. 304,263 and 303,454).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER. R Tensile strength and fluidity of chemically modified cotton. D. A. CLIBBENS and B. P. RIDGE (J. Text. Inst., 1928, 19, 389-404 T).-Relations are established between the tensile strength and the fluidity in cuprammonium solution of samples of scoured, combed, Sakel yarns after treatment with hydrochloric and sulphuric acids, alkaline and neutral hypochlorite, alkaline hypobromite, and dichromate in the presence of sulphuric and oxalic acids. For a given fluidity, the same loss of strength is obtained irrespective of whether it is caused by the action of concentrated acids at the ordinary temperature or by dilute acids at the boil. Similarly, the relation between fluidity and tendering is the same whether the latter is caused by the action of hypobromite or of hypochlorite in neutral or alkaline solution, but for the same fluidity, acid attack corresponds with slightly greater loss of strength than does attack by these oxidising agents. Over the fluidity range 10-30 (corresponding roughly with 10-50% loss of strength) the curves for fluidity-% loss of strength are approximately straight lines for attack by both acids and hypochlorite solutions. Oxidation with dichromate in the presence of sulphuric or oxalic acid of concentrations insufficient to cause tendering in the absence of dichromate gives high fluidities accompanied by relatively slight loss of strength. For the yarns used, the percentage loss of strength of the single threads attacked with either acids or hypochlorite is equal to the loss of strength of the individual hairs composing the threads. When material modified by means of acids or alkaline hypochlorite is submitted to an alkaline boil, neither the strength nor fluidity of the product is materially affected, but for cotton modified by other oxidising agents both fluidity and loss of strength are increased. The most marked rise of fluidity on boiling is shown by neutral hypochlorite oxycelluloses, but the rise is slight for yarn oxidised by dichromate, although in this case the additional loss of strength is greatest. Cotton oxidised by dichromate differs from that oxidised by neutral hypochlorite in that (1) for a definite rise of fluidity it shows a much smaller loss of strength, and (2) alkali boiling produces a large additional loss of strength with a comparatively slight rise of fluidity. The fluidity of cotton attacked by dichromate indicates not the actual loss of strength, but a potential tendering which is realised only after the material is boiled with alkali. These facts vindicate the use of fluidity as a strength control, since material which would lose half its strength on laundering might be passed as satisfactory by a direct

tensile test, whilst being rejected as unsatisfactory by the fluidity measurement. Within the limits of experimental error, the same fluidity, after boiling, corresponds with the same loss of strength whether the modification is caused by hypochlorite in neutral or alkaline solution or dichromate in the presence of sulphuric or oxalic acid, whilst the fluidity-strength relationship for yarn tendered by acid is roughly the same as that for yarn submitted to oxidising attack and subsequently boiled with alkali. Although it cannot be maintained that a given rise of fluidity always corresponds with the same loss of strength if all possible causes of the chemical modification be taken into account as well as the possibility of subsequent hot alkali treatment, any chemical processes which are accompanied by a considerable rise of fluidity of the material reduce its strength, actually or after a mild alkali treatment, by an amount which constitutes serious practical damage. B. P. RIDGE.

Identification of rayon. W. D. GRIER (Ind. Eng. Chem., 1929, 21, 168-171).—The importance of microscopical examination is emphasised. Whilst the majority of textile fibres can be identified by longitudinal observation under the microscope, it is necessary to examine transverse sections in order to identify rayon filaments, since their longitudinal appearances are similar, and in some cases the results of chemical tests are inconclusive. The appearance of sections of the more important types of rayon in ordinary and polarised light is described, and diagrams and photomicrographs are given. The technique of section cutting and the apparatus required are described. B. P. RIDGE.

Characterisation of celluloses. T. LIESER (Cellulosechem., 1929, 10, 21-27).-Numberless natural and artificial celluloses are believed to exist each with different physical and chemical properties, and all natural cellulose fibres to contain many chemically different celluloses; a chemical method, based on viscose formation, for their differentiation is described. At a certain specific concentration of sodium hydroxide, which, at a given temperature, varies with the form of cellulose, a compound of the type 2C6H10O5, NaOH is formed, the formation of which is characterised by the possibility of undergoing the viscose reaction. Thus natural cotton cellulose forms this compound only with at least 16-18% sodium hydroxide solution, whilst cellulose-A (precipitated from concentrated hydrochloric acid solution) requires only a 7.5% solution. There exists a whole range of intermediate celluloses which form compounds, and, in consequence, undergo the viscose reaction, with increasingly lower concentrations of alkali. Thus, if the percentage of the sample undergoing the viscose reaction is plotted against the concentration of alkali a series of different curves are obtained with different types of celluloses. When natural cotton cellulose is treated with alkali of increasing concentration, above 2N the proportion undergoing the viscose reaction increases with increasing alkali concentration until it is complete at 5N. Lignocellulose gives the viscose reaction at a lower alkali concentration, whilst with artificial silk the proportion of viscose formed increases rapidly at concentrations of alkali above N. In the practical application of the method for the differentiation of celluloses and for the detection and

determination of cellulose-A (which is useless for the preparation of artificial silk), excess of alkali must be avoided, just sufficient being added to 1 g. of the cellulose to moisten all the fibres. After 1 hr. at 21°, 0.5 c.c. of carbon disulphide is added and viscose formation allowed to proceed in the thermostat. After dilution with water the insoluble residue is filtered through a hardened paper and washed until the filtrate gives no precipitate with mineral acids. The residue is then washed with 0.5N-hydrochloric acid, dried at 103°, and weighed. A number of typical results with various types of cellulose are given. J. W. BAKER.

Heartwood of the pine. III. Heating in sealed tubes. C. G. SCHWALBE and A. AF EKENSTAM (Cellulosechem., 1929, 10, 27-34; cf. B., 1929, 166).-The effect of drying, extraction with organic solvents and alkali, and previous impregnation with the various extracts, on the formation of cellulose has been studied in detail with standard sealed-tube heating in an attempt to elucidate the nature of the material present which inhibits the decomposition. The main results are as follows. The yields of cellulose obtained by heating spruce heart- and sap-wood, and Scottish pine sapwood with calcium bisulphite  $(1\% CaO, 4.05\% SO_2)$  are, respectively, 46.8, 46.0, and 48.2%, whilst under the same conditions the heartwood of Scottish pine is completely unattacked. Previous extraction of the latter with ether, benzene, or acetone before the bisulphite heating gives either no yield or only a poor-quality pulp, but pre-extraction with alcohol yields a good cellulose (cf. Hägglund, B., 1927, 294). If, however, the wood is previously air-dried (3 months) or, better, dried at 105° for 40 hrs. and then ether-extracted, bisulphite treatment then gives, respectively,  $57\cdot 8$  and  $64\cdot 5\%$  yields of good cellulose. The decomposition depends on the impregnatability of the wood with the bisulphite, and complete drying renders the wood more permeable to the ether, allowing the more complete removal of the ether-soluble portion. When the pine heartwood is previously treated for 10 days with 0.1N-sodium hydroxide and then washed with cold water for 3 days, the bisulphite heating yields  $66 \cdot 2\%$  of cellulose (ash  $1 \cdot 28\%$ ). Further washing for 10 hrs. with hot water gives a 65% yield (ash 0.979%), whilst a final washing with 0.5% acetic acid for 1 hr. and water for 2 hrs. gives a 63.4% yield (ash 0.491%). Although the substance which inhibits the bisulphite decomposition is readily removed from shavings with alkali, the use of the latter is not practicable with the larger pieces used technically. Similar pretreatment with calcium hydroxide solution yields specimens from which the calcium cannot be removed by washing and which contain about the same amount of calcium as is found after heating with calcium bisulphite. Subsequent heating with sulphurous acid (2.5-3.5% SO2) causes no decomposition. From a study of the effect of impregnating the pine and spruce sapwoods with the various organic and alkali extracts of the pine heartwood on the bisulphite decomposition it is concluded that the substance which inhibits the bisulphite decomposition is acidic, forms a soluble calcium salt, is soluble in alcohol, benzene, acetone, and ether, and insoluble in light petroleum. 'The calcium hydroxide extract of the pine heartwood differs from

the spruce extracts in that it becomes turbid on acidification, and on the basis of the above properties the isolation of this acidic substance in small quantity as a viscous brown liquid is described. J. W. BAKER.

Sulphonation of lignin from pine wood. I. E. HÄGGLUND and T. JOHNSON (Biochem. Z., 1928, 202, 439-452) .- Lignin isolated from pine wood by means of concentrated hydrochloric acid was treated with hot bisulphite solution, under conditions similar to those obtaining in the sulphite-cooking process. The solubility of the product was the greater the more protective the circumstances of isolation had been. Lignins as easily soluble as that in wood were obtainable. The presence of cellulose or of sugar did not affect the rate of dissolution. On sulphonation of the lignin insoluble salts of lignosulphonic acids are first formed : these are converted by dilute acids into soluble lignosulphonic acids. The rate of dissolution of lignin is directly related to the  $p_{\rm H}$  of the cooking liquid. The dissolved lignosulphonic acids behave like those of waste sulphite liquors. If sulphonation and dissolution succeed each other rapidly, sulphonic acids the naphthylamine compounds of which have the same sulphur content as those of the sulphonic acids of sulphite waste liquors are obtained; but if the reactions proceed slowly, as in the case of lignin treated with hydrochloric acid, the sulphur content of the lignin compounds precipitable by naphthylamine rises. The lignosulphonic acids which go into solution contain up to 15% of loosely bound sulphurous acid. In the sulphite waste liquors sulphur dioxide is loosely combined probably with the portion of the lignosulphonic acids which cannot be salted out. This portion has a copper number which is relatively high in comparison with that of the portion which can be salted out. Both solid lignosulphonic acids and those which dissolve acquire violet-coloured fluorescence when illuminated with a quartz lamp, provided that the lignin of the acids has been prepared under protective conditions. The molecules of lignosulphonic acids contain certain groupings which condition the fluorescence, and these are exceptionally sensitive towards acids. W. MCCARTNEY.

Hydrophilic properties of sthenosised cellulosic fibres. L. MEUNIER and R. GUYOT (Compt. rend., 1929, 188, 506-508; cf. B., 1927, 276).-Sthenosis, or the strengthening of viscose with a 5% aqueous, phosphate-buffered solution of formaldehyde  $(p_{\rm H} \ 1 \cdot 6 - 1 \cdot 2)$ , is best performed by immersion for 12 hrs. followed by centrifuging, heating at 60-90° for 2-48 hrs. according to the intensity of sthenosis required, and washing to remove acid. The strength and resistance to alkalis of viscose increases, but the powers of absorption and the humidity determined both by adsorption and desorption decrease proportionally with the intensity of sthenosis. The mechanism is a partial conversion of cellulose into hydrocellulose by the acid liquor, with an increase in the number of hydroxyl groups, and, during evaporation, the catalytic formation by the more concentrated acid of methylenic derivatives. If it is assumed that the cellulose crystallites are formed from a system of parallel chains of glucose residues (C6H10O5), then the changes in hydrophilic properties may be explained

by the reaction of different chains with 2 mols. of formaldehyde. J. GRANT.

Sulphite liquor. LAUBER.—See VII.

*Erratum.*—B., 1929, 201, col. 2, line 33, for "pine" read "spruce."

# 1891 vo PATENTS. next at they los but bus

Utilisation of palm leaves. J. AVRE (Austral. P. 1298, 6.4.26).—Woolly fibres obtained by mechanical treatment of the leaves of the zamia palm etc. are purified by treatment with sulphur dioxide and combed, and the residues are incinerated, the ash being used as a fertiliser. L. A. COLES.

Saturation of fibrous material. R. T. JOHNSTON, Assr. to FLINTKOTE Co. (U.S.P. 1,698,886, 15.1.29. Appl., 20.3.23).—Material after passing through hot saturant at atmospheric pressure is exposed to a vacuum out of the saturant. J. S. G. THOMAS.

Manufacture of a material for covering or wrapping perishable goods etc. Preservation of foods and other perishable goods. A. H. KINNER (B.P. 304,168 and 304,341, 15.10.27).—(A) Cotton or other fabric is passed vertically through a bath containing viscose, whence it is withdrawn through an adjustable slit at the bottom, so that the solution is forced into the threads and meshes of the fabric. The stretched material is passed through dilute sulphuric acid to coagulate the viscose and is then washed and dried. (B) Gas-tight bags or containers for enclosing food etc. and made of the treated material are described. F. R. ENNOS.

Gas-cell fabric. W. J. KELLY, ASST. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,691,764, 13.11.28. Appl., 23.6.25).—Balloon fabric is impregnated with a solution of cellulose xanthate containing acidified sodium sulphate or other material inhibiting bubble formation, and the cellulose is regenerated on the fabric and softened, preferably under slight tension. R. BRIGHTMAN.

Protection of materials such as wool, fur, etc. from the ravages of moth. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,092, 28.9.27).—Organoderivatives of phosphorus, arsenic, antimony, bismuth, or tin, *i.e.*, compounds in which these elements are linked directly to carbon, are used for moth-proofing purposes. Examples are tri-*p*-tolylphosphine, triphenylarsine, tribenzylarsine, tri(dimethylaminophenyl)arsine, triphenylarsine dihydroxide, triphenylstibine, tri-*o*tolylstibine, diphenylethylstibine, tri(bromophenyl)stibine, trimethylstibine sulphate, dibromide, and dichloride, triethylstibine, bismuth triphenyl, bismuth tri-*p*-tolyl, tin tetraphenyl, tin tetrabenzyl.

C. HOLLINS.

Waterproofing of textiles, paper, etc. M. E. P. FAVRESSE and J. C. PERES (F.P. 629,291, 18.2.27).— A suspension of flour in water is heated with sodium hydroxide at 120° until the starch is completely converted into dextrin. After cooling, a solution of casein and sodium carbonate or silicate is added and the articles to be waterproofed are soaked in this mixture, calendered hot, and treated with a solution of cellulose acetate in acetone and benzyl alcohol. A. R. POWELL. Hardening and waterproofing of porous or fibrous materials. H. A. AARONSON, and CHEM, WATERPROOFING CORP. (B.P. 303,935, 20.10.27).—Textile and paper materials are impregnated or sprayed with a suitable coal-tar pitch dissolved in a non-inflammable solvent such as trichloroethylene or tetrachloroethane, and the solvent is then removed by heating.

# A. J. HALL.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,685,168—9, 25.9.28. Appl., [A, B], 28.4.26).—Fur is treated with an aqueous solution of (A) an organic or (B) an inorganic acid containing a small amount of hydroxy-aliphatic acid, *e.g.*, glycollic or lactic acid. R. BRIGHTMAN.

Textile oils. H. WADE. From STANDARD OIL CO. (B.P. 303,820, 6.7.27).—Oils suitable for lubricating textile yarns, particularly artificial silk, for knitting or weaving consist of a refined distillate mineral oil of low viscosity, oleic acid, a suitable emulsifying agent, e.g., an alkali-metal soap, a sulphonated fatty oil compound, or an oil-soluble alkali-metal salt of a sulphonic acid derived from a sulphonated mineral oil, and an anti-oxidant (e.g.,  $\beta$ -naphthol or diphenylamine). A. J. HALL.

Application of highly sulphonated oils in the textile industry. H. FLESCH (B.P. 284,249, 4.8.27. Ger., 26.1.27).—Sulphonated oils (e.g., of dihydroxystearic acid) containing at least 6% of organically combined sulphuric acid, but differing from those described in B.P. 263,117 (B., 1928, 845), are particularly suitable as additions to liquors containing high concentrations of caustic alkalis or neutral salts for the purpose of assisting their penetration of textile materials.

A. J. HALL.

Cooking of sulphite pulp. G. H. TOMLINSON (U.S.P. 1,697,032, 1.1.29. Appl., 7.12.25. Renewed 14.5.28).—The hot liquor discharged from one cooking process is employed to heat a fresh charge of chips and to wash it into the digester, where it is mixed with a charge of cooking acid heated to approximately the same temperature by means of the hot gases obtained from a previous digestion. The cooking is then completed by means of steam. F. R. ENNOS.

Treatment of hydrated cellulose. I. G. FARBEN-IND. A.-G. (B.P. 278,684, 13.9.27. Ger., 5.10.26).— In order to diminish the tendency of hydrated cellulose to swell, it is immersed in or saturated with a neutral or feebly alkaline aqueous solution of a condensation product, *e.g.*, sulphur-o-cresol resin (cf. B.P. 186,107; B., 1922, 905 A), or the materials which can react to form such a product, with or without a catalyst. A further condensation is then brought about by addition of a substance such as formaldehyde with the formation of a product insoluble in water and dilute alkali.

Treatment of fabrics made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 297,712, and 299,042, [A] 15.6.28, [B] 6.10.28. U.S., [A] 15.7.27, [B] 20.10.27. Addns. to B.P. 293,858).—Mechanical treatments are claimed. F. R. ENNOS.

Conversion of nitrocelluloses which yield highly viscous solutions into nitrocelluloses which yield

solutions of low viscosity. I. G. FARBENIND. A.-G. (B.P. 289,387, 25.4.28. Ger., 25.4.27).—An unstable nitrocellulose is heated above 100° with water under pressure, with exclusion of acid and in the presence of an insoluble basic compound, *e.g.*, magnesia.

F. R. Ennos.

Acylation of carbohydrates. E. B. MIDDLETON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,685;220, 25.9.28. Appl., 3.6.25).—Carbohydrates are suspended in glacial acetic acid, or other medium, containing mineral acid as catalyst, and treated with keten. Thus, cellulose in presence of 1.5% of sulphuric acid is converted into chloroform-soluble cellulose acetate. R. BRIGHTMAN.

Manufacture of plastic compounds. G. E. ZELGER, Assr. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,685,443—4, 25.9.28. Appl., [A] 6.4.25, [B] 30.7.26.— (A) Phosphoric esters containing both aromatic and aliphatic radicals, e.g., diphenyl methyl phosphate, diphenyl ethyl phosphate, phenyl dimethyl phosphate, phenyl diethyl phosphate, or the corresponding tolyl derivatives, or (B) containing halogen-substituted aryl groups, e.g., di(chlorophenyl) methyl phosphate, phenyl chlorophenyl methyl phosphate, chlorophenyl dimethyl phosphate, dichlorophenyl dimethyl phosphate, chlorophenyl diethyl phosphate, are mixed with cellulose esters or ethers to give non-inflammable plastic compounds.

R. BRIGHTMAN.

Production of high-grade knifing compositions and mixed lacquers containing them. Production of knifing compositions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,615-6, 12.9.27).-(A) The compositions contain a cellulose ester or ether, inorganic fillers of a scaly, foliaceous, or fibrous nature, e.g., graphite, mica, asbestos powder, etc., and a finelydivided organic filler, e.g., wood, cork, lignite, coconut fibre, etc., with or without the addition of solvents, lacquers, resins, plasticising agents, colouring materials, etc., but excluding resinous or gummy products extracted from wood meal etc. by alkaline solutions. (B) Cellulose derivatives are incorporated with more than three times their weight of kieselguhr, powdered pumice, or precipitated chalk, other substances being added if desired. L. A. COLES.

Manufacture of waterproof straw pulpboard. E. B. WESTON and W. G. CLARK, ASSIS. to TERRE HAUTE PAPER Co. (U.S.P. 1,697,702, 1.1.29. Appl., 4.11.26).— After digesting straw with steam in the presence of alkali, it is thoroughly beaten to a pulp and washed to remove alkali. A rosin size and an alum mordant are then incorporated with the pulp, and the product is formed into sheet. F. R. ENNOS.

Cellulosic material and product obtained therefrom. H. DREYFUS and C. I. HANEY, ASSIS. to CELANESE CORP. OF AMERICA (U.S.P. 1,697,907, 8.1.29. Appl., 19.1.27. U.K., 8.10.26).—See B.P. 288,657; B., 1928, 444.

Manufacture of artificial silk and other filaments by applying electric current. K. HAGIWARA, Assr. to T. IWASAKI (U.S.P. 1,699,615, 22.1.29. Appl., 14.3.27. Japan, 3.8.26).—See B.P. 286,086; B., 1928, 329. CL. VI.-BLEACHING; DYEING; PRINTING; FINISHING. CL. VII.-ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS. 243

Machinery for softening or loosening artificial fibrous materials. I. G. FARBENIND. A.-G. (B.P. 286,302, 24.2.28. Ger., 4.3.27).

Manufacture of [twisted] yarns or threads, and apparatus therefor. BRIT. CELANESE, LTD., W. A. DICKIE, T. H. PLATTS, and L. LATHAM (B.P. 304,806, 28.9.27).

Manufacture of [fibrous] plastic material [for bottle caps etc.]. E. C. R. MARKS. From LAKEWEST CORP. (B.P. 302,767, 26.10.27).

Manufacture of [compound] paper. J. R. HAPPER (B.P. 304,863, 12.11.27).

Rubber goods with textile insertions (B.P. 304,157).—See XIV.

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

Suitability of various metals and alloys for dyeing machines. J. G. GRUNDY (J. Soc. Dyers & Col., 1929, 45, 39-42).-A number of metals were tested in order to ascertain their reaction with dyestuffs and with the reagents used in dyeing, dyes from all groups of organic colouring matters being examined and the results compared against an acid-resisting enamel as standard. The majority of acid colours were sensitive to cast and wrought iron, copper, Firth stainless steel, and phosphor bronze, but not sensitive to nickel, monel, Staybrite, Dyebrite, Silverite, and brass. Neolan colours were the least sensitive of those dyed from an acid dye-bath, owing, no doubt, to the fact that the colours were already metallic salts. Chrome colours, after-chromed, were very sensitive to iron, nickel, lead. and copper, but alloys of the Dyebrite type were satisfactory. Direct and basic colours appear to be only affected appreciably by wrought and cast iron. Sulphur colours were considerably affected by metals containing copper owing to the action of the sodium sulphide used in the dye-bath. Vat colours were not affected by any of the metals tested, whilst the azoic colours gave poor results with only cast and wrought iron.

#### L. G. LAWRIE.

Colouring of rubber. NAUNTON.-See XIV.

#### PATENTS.

Production and colouring of textile yarns, fabrics, and the like. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 303,958, 8.11.27).—Viscose silk yarn is immersed for about 1 hr. at ordinary temperature in a 3—6% solution of caustic soda, then freed from alkali by washing and souring, dried, and used together with untreated viscose silk in the manufacture of woven or knitted materials which are afterwards dyed with direct dyes; two-tone coloured materials are thereby obtained since the alkali-treated silk has a greater affinity than untreated silk for many direct dyes. Caustic potash may be used instead of caustic soda, but a less marked increase of affinity of the silk for direct dyes is obtained (cf. B.P. 295,488; B., 1927, 745). A. J. HALL.

Increasing the affinity of animal fibres for dyes. E. Du Bois, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,698,226, 8.1.29. Appl., 23.12.25. Ger., 6.1.25). --See B.P. 245,759; B., 1927, 475.

Apparatus for washing, bleaching, and dyeing artificial silk in hanks or spinning cakes. TF. KÜTTNER A.-G. (B.P. 288,990, 16.4.28. Ger., 16.4.27).

Collapsible spindle for dyeing and analogous treatment of wound yarns. J. and T. BRANDWOOD (B.P. 304,800, 28.7.27).

Wetting, etc. agents (B.P. 303,917).—See III. Printing colours (B.P. 303,942).—See IV. Colouring of rubber (B.P. 304,376 and G.P. 452,340). —See XIV.

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

Viscosity relationships in the system sulphuric acid-nitric acid-water. F. H. RHODES and H. B. HODGE, JUN. (Ind. Eng. Chem., 1929, 21, 142-150).-A large number of viscosity determinations were made on "mixed acid" at 0-75° with a view of identifying compounds which might be present. Capillary viscosimeters calibrated with sucrose solution were used. The binary system sulphuric acid-water shows in the viscosity curve maxima at 85% and 100% of acid and a minimum at 95% of acid, these points being less marked at the higher temperatures. In the system nitric acid-water less definite maxima occur at about 65% of nitric acid. Other variations in the curves are probably due to polymerising or depolymerising effects. In the system nitric acid-sulphuric acid maxima occur at 88% and 100% of sulphuric acid, with a minimum at 97% sulphuric acid. This is held to indicate the existence of a compound 10SO<sub>3</sub>, N<sub>2</sub>O<sub>5</sub>, 9H<sub>2</sub>O. Diagrams of the viscosities of the ternary system are given. They show the superimposed effects obtained with the various binary systems, and do not indicate the presence of any new compounds. C. IRWIN.

Manufacture of "converted" saltpetre. F. CHEMNITTUS (Chem.-Ztg., 1929, 53, 85-86).—Sodium nitrate of 96-98% and potassium chloride of 80% purity, the latter in slight excess, are mixed with a mother-liquor from a previous raw saltpetre crystallisation. The chloride should contain not more than 0.6% of perchlorate nor more than 0.5% of magnesium salts, which must be precipitated by addition of the equivalent amount of sodium carbonate. The reaction is controlled by determinations of the sp. gr. of the solution at different stages. A purified product containing only 0.3-0.5% of sodium chloride and 0.1% of sodium nitrate is obtained. F. R. ENNOS.

Preparation of sulphite liquor. H. LAUBER (Papier-Fabr., 1929, 27, 65—69).—The use of pyrites in the place of sulphur for the preparation of sulphite liquor is discussed. Sulphur losses in the roasting of the ores are dependent on the crystalline form and fineness of grinding of the material and on the presence of impurities (e.g., copper and zinc sulphides) which affect the fusion temperature. The efficiency of various types of pyrites burners is discussed, together with the technique of purification and absorption of the gases.

A. G. POLLARD.

Determination of potassium cyanate. J. LEBOUCQ (Ann. Falsif., 1928, 21, 595-600).—The method depends on the formation of s-dicarbonamidohydrazine (solubility in water 0.15 g./litre) by the action of semicarbazide hydrochloride on the cyanate. The product is weighed and the proportion of cyanate calculated. The method can be applied in the presence of cyanide, less than 10% of cyanate being detectable if care be taken to operate in an acid medium after fixing the hydrocyanic acid with dextrose. It may also be applied to the determination of cyanate formed by the action of copper sulphate on an alkaline cyanide. D. G. HEWER.

[Determination of free alkali in hypohalogenite solutions.] P. FUCHS (Z. anal. Chem., 1929, 76, 150).— Rupp and Lewy's method (B., 1928, 365) is not new. H. F. GILLBE.

Determination of nitrogen in cyanamide. S. REYNAERT (Natuurwetensch. Tijds., 1929, 11, 27–28).— It is unnecessary to continue heating in the Kjeldahl method until the mixture is decolorised; accurate results are obtained with 6 hrs.' heating only. Addition of potassium sulphate is also unnecessary. S. I. LEVX.

Action of water on calcium silicates. THORVALDSON and VIGFUSSON.—See IX. Radium and luminous paints. PARSONS.—See XIII. Agricultural lime. REYNAERT. Nitrogen in urea-nitrate mixtures. LUCAS and HIRSCHBERGER.—See XVI. Determination of potassium iodide. WEICHHERZ.—See XX. Adsorption of carbon dioxide by ferric hydroxide. TILLMANS and others.—See XXIII.

#### PATENTS.

Speeding of chemical reactions. H. B. KIPPER (U.S.P. 1,697,390, 1.1.29. Appl., 14.5.26).—The manufacture of hydrochloric acid and sodium sulphate from sodium chloride and sulphuric acid is hastened by grinding and beating the material in a heated vessel having rapidly moving metal parts, caking of the material on the vessel being prevented by means of slowly moving metal parts. W. G. CAREY.

Manufacture of hydrochloric acid. E. R. WOLCOTT, Assr. to TEXAS Co. (U.S.P. 1,691,452, 13.11.28. Appl., 1.10.22).—Sodium chloride and clay are intimately mixed with a small amount of binder, *e.g.*, asphalt, pitch, acid sludge, and distilled in steam below 820°.

R. BRIGHTMAN. Decolorisation of tartaric acid solutions. URBAIN CORP. (F.P. 622,649, 2.2.26).—The solution is treated with active carbon with or without an alkali or a colloidal precipitating agent, *e.g.*, an alkali alginate, and precipitation of the impurities is effected by subsequent addition of an electrolyte, *e.g.*, a solution of a copper, tin, or alkaline-earth salt. A. R. POWELL.

Production of alkali carbonates from alkali sulphates by the wet process. W. H. FRIEDRICH (G.P. 453,217, 24.2.26).—About 5% of an alkali hydroxide, calculated on the weight of the sulphate, is added as catalyst in the process described in G.P. 431,509 (B., 1926, 915). L. A. COLES.

Production of alkali cyanides. H. B. KIPPER (U.S.P. 1,697,529, 1.1.29. Appl., 1.3.26).—The materials are continuously and rapidly stirred and beaten in a closed vessel to maintain them in a finely-divided and disseminated condition while they are subjected to the action of highly heated reactive gases so that alkali cyanides are formed. The inner walls of the vessel are kept free by continuous and slow scraping.

W. G. CAREY.

Manufacture of potassium manganate. Soc. CHIM. DES USINES DU RHÔNE (B.P. 292,991, 19.3.28. Fr., 29.6.27).—Pure or dilute oxygen is blown through a mechanically stirred suspension of an oxide of manganese, or of compounds capable of liberating the oxides, in an excess of aqueous melted potash, containing 60—85% of potassium hydroxide, maintained at 160—200°. W. G. CAREY.

Decolorisation of alkaline-earth thiocyanate solutions. R. R. FULTON, Assr. to KOPPERS Co. (U.S.P. 1,691,345, 13.11.28. Appl., 31.12.26).—Derivatives of soluble fatty acids are added to the solutions, particularly in presence of cellulose. E.g., calcium thiocyanate solution is decolorised by being heated for 1 hr. at 120—130° with 0.4—2% of formaldehyde, or 3—5% of sodium formate. R. BRIGHTMAN.

Rendering calcium cyanamide free from dust. J. BRESLAUER and E. DE LUSERNA, ASSTS. to SOC. D'ETUDES CHIM. POUR L'IND. (U.S.P. 1,691,044, 13.11.28. Appl., 11.1.27. Switz., 22.1.26).—Calcium cyanamide is incorporated with a fusible potassium hydrogen salt, *e.g.*, potassium hydrogen sulphate, at 200°.

R. BRIGHTMAN. Drying of superphosphate. VER. F. CHEM. & MET. PROD. (F.P. 626,190, 13.12.26).—The material falls in a finely-divided condition from the decomposition chamber into a countercurrent of hot gases, preferably a mixture of air and combustion gases. L. A. COLES.

Production of basic copper carbonate from impure solutions of copper salts. A. M. BICKFORD & SONS, LTD., Assees. of J. HENDRY and P. A. BERRY (Austral. P. 2775, 13.7.26).—Bases other than copper are removed by selective precipitation before the basic copper carbonate is precipitated. L. A. COLES.

Apparatus for the production of silicon or titanium chloride or bromide. I. G. FARBENIND. A.-G., Assees. of M. SCHLIEMANN (G.P. 453,367, 6.8.26).— Apparatus of the usual type is constructed of metal and provided with efficient cooling arrangements.

L. A. Coles.

Production of titanium hydroxide. SILESIA VER. CHEM. FABR., Assees. of G. ALASCHEWSKI (G.P. 453,120, 25.11.25).—Solutions containing titanium salts are atomised and treated with a countercurrent of superheated steam and/or hot gases. L. A. COLES.

Simultaneous production of phosphorus or phosphoric acid and binding agents having latent hydraulic properties. I. G. FARBENIND. A.-G. (B.P. 297,416, 18.8.28. Ger., 21.9.27).—The process of B.P. 285,055 (B., 1928, 710) is modified by forming the charge of a mixture of crude calcium and aluminium phosphates in such proportions that a slag is produced similar in composition to that of the blast-furnace slag cement previously described, W. G. CAREY. Sodium sulphide pellets. R. Roos, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,699,377, 15.1.28. Appl., 13.11.24. Ger., 18.1.24).—See G.P. 426,052; B., 1926, 583.

Hydration of phosphoric anhydride. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,698,484, 29.1.29. Appl., 21.4.27. Fr., 21.4.26).—See B.P. 269,908; B., 1927, 748.

Catalytic gas reactions (B.P. 304,269).—See I. Vegetable waste products (G.P. 452,810).—See II. Catalysts (B.P. 301,806).—See III. Moth-proofing materials (B.P. 303,092).—See V. Recovery of metal compounds from ammoniacal liquors (B.P. 301,342).—See X. Determination of carbon dioxide (U.S.P. 1,695,031).—See XI.

## VIII.-GLASS; CERAMICS.

Relations between physical characteristics of glasses and their working properties on machines. G. GEHLHOFF (J. Soc. Glass Tech., 1928, 12, 145—157 P). —Data are collected to show the influence of viscosity, thermal expansion, conduction and radiation, devitrification, annealing rate, and resistance to thermal and mechanical shock on the capability of working various types of glass by machine. A list of temperatures is given at which typical lead and soda-potash glasses are sufficiently hot just to adhere to various mouldforming materials. A. COUSEN.

Refractory materials, with special regard to the glass industry. W. MIEHR (J. Soc. Glass Tech., 1928, 12, 158—176 P).—The selection and improvement of natural and artificial raw materials now used for making refractories for the glass industry, methods of producing these refractories, and the chemical and physical properties of the finished products are dealt with. Finally, an account of some experiences in the use of new kinds of refractory products in the works is appended. A. COUSEN.

Characteristics of refractory clays for the glass industry. G. GEHLHOFF, H. KALSING, K. LITZOW, and M. THOMAS (J. Soc. Glass Tech., 1928, 12, 280).— A correction (cf. B., 1928, 928). A. COUSEN.

Properties of glasses containing zinc oxide. S. ENGLISH, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1928, 12, 287-296).-Glasses of the molecular type 6SiO<sub>2</sub>, (2-x)Na<sub>2</sub>O, xZnO become increasingly quick-setting as the zinc oxide content increases. The values of d and  $n_D$  increase linearly from 2.4520 and 1.49975, respectively, for the first member of the series—SiO<sub>2</sub> 74.09%, Na<sub>2</sub>O 24.07%, ZnO 1.29%—to 2.5499 and 1.50829 for the last member -SiO<sub>2</sub> 71.94%, Na<sub>2</sub>O 14.70%, ZnO 11.39%. The average value of total dispersion is 0.00873 and of y is 57.8. For substitutions of sodium oxide by zinc oxide up to 6% there is no distinct increase in the annealing temperature, and up to 10% the effect is only small. Several change points occur in the rate of expansion of the samples between 0° and the lower critical temperature. Over the range 0-100° the thermal expansion decreases linearly with increase of zinc oxide content. A Cousen.

The glass-melting furnace as a heat machine. D. AUFHÄUSER (J. Soc. Glass Tech., 1928, 12, 297-306). —The furnace is considered from the point of view of a heat machine, heat evolution and exchange being velocity problems, whilst the intensity factors are temperature difference and area of exposed surface. A. COUSEN.

Ultra-violet light-transmitting glasses. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 306-320).—The transmission of eight commercial glasses of this type of 2 mm. thickness over the range 7000-2000 Å. has been determined. "Corex" glass transmitted practically as much light as quartz, having a transmission in the therapeutic region (2950-3150 Å.) of 89%, whilst the transmission of the remaining glasses were: "Sanalux" 73%, "Sun Ray" 62%, "Holvi" 61%, "Vita" 54%, "Helio" 52%, "Uviol" 46%, "Quartz-Lite" 4% (ordinary window glass 1%). Corex had the lowest iron oxide content, namely 0.012%, that of the remaining samples ranging from 0.021% to 0.054%. After a 12 weeks' exposure to sunlight, Corex became stained with a white film, but, on being cleaned, showed no change in transmission. The other glasses tested were more durable on exposure, but showed the following decreases in transmission : Vita 13%, Helio 11%, and Sanalux 7%. When artificially aged under the carbon arc the greatest change occurred in the first 2 hrs., the fall in transmission coinciding with a decrease in the proportion of ferrous oxide to total iron oxide in the glass. A. COUSEN.

Influence of ferric oxide content on the lighttransmission of soda-lime-silica glass, with special reference to the ultra-violet. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 324-332).--Soda-lime glasses of the approximate composition SiO<sub>2</sub> 75%, CaO 10%, Na<sub>2</sub>O 15%, and containing iron oxide in amounts varying from 0.005% to 1.0%, were studied over the range 7000-2000 Å. The intensity of transmission on either side of the yellowishgreen region decreased as the iron oxide content increased. By extrapolation, the limiting transmission for the glass free from iron was given as 2230 Å. All the glasses were melted in an oxidising atmosphere, and the ratio of ferrous oxide to total iron oxide averaged 6.9% as against 30% for commercial glasses transmitting ultra-violet light. A. COUSEN.

Change in optical transparency of certain ultraviolet transmitting glasses after exposure to Xrays and ultra-violet light. G. M. SHRUM, C. G. PATTEN, and H. D. SMITH (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 433-436).-Commercial ultra-violet glasses were examined and showed the effect of solarisation when exposed to ultra-violet light, and both solarisation and coloration when exposed to X-rays. If the glasses were kept at 100° for some time the coloration faded, and the effect of solarisation was reduced to zero when the glasses were gradually heated to 300°. A thermo-luminescence was observed for all samples, and it was found that the recovery from the solarisation effect coincided with the disappearance of the thermo-luminescence. It is possible that the solarisation is a photochemical oxidation, A. J. MEE.

Calculation of glass constants on the basis of recent investigation. E. ZSCHIMMER (J. Soc. Glass Tech., 1928, 12, 333—366).—A continuation of the systematic survey of recent determinations of the values of physical properties of glass (cf. B., 1928, 404), the properties dealt with being viscosity, annealing constants, density, thermal expansion, refractive index, and dispersion. A. COUSEN.

Action of fluorine in enamel smelts. H. G. WILCOX and F. C. WESTENDICK (J. Amer. Ceram. Soc., 1929, 12, 83—86).—By chemical analysis of the raw and smelted enamels it was shown that only part of the fluorine is lost during smelting. Longer smelting duration causes an increase and higher alumina content a decrease in fluorine lost. It is incorrect to assume that the fired and raw weights of either cryolite or fluorspar are the same. J. A. SUGDEN.

Some leadless borosilicate glazes containing nickel oxide. H. H. HOLSCHER (J. Amer. Ceram. Soc., 1929, 12, 111—117).—The literature pertaining to the use of nickel oxide is reviewed. The results of many trials, where comparison is possible, agree with previous investigators. It is concluded that nickel oxide has not proved useful in glossy, non-opaque glazes, and, in any case, is not recommended for use where a dark colour is desired, as all such colours are turbid. With suitable contents of lime (above 0.35%), barium oxide (not above 0.2%), and magnesia (barium oxide + magnesia not over 0.3%), nickel oxide can be made to produce attractive light tan, semi-matt, opaque glazes. J. A. SUGDEN.

Service of refractory blocks in a small experi-mental glass tank. W. L. PENDERGAST and H. INSLEY (J. Amer. Ceram. Soc., 1929, 12, 123-141, and Bur. Stand. J. Res., 1929, 2, 452-465) .- Trials have been made in a small, laboratory, continuous glass tank 1 ft.  $\times$  1 ft.  $\times$  4 ft. long. The sides of the tank were built up of six blocks (6 in. thick) under test. The batch used was one higher in soda than the average soda-lime bottle glass, and therefore more corrosive. Each run lasted about 30 days. Many types of fireclay and special refractories were tested. There appears to be little connexion between either chemical composition or porosity and resistance to glass attack. It is pointed out that homogeneity of a refractory must have a considerable effect on its resistance to attack, as shown by the many cases of pitting. Such corrosion may be brought about by the presence of coarse pores, difference in composition between grog and bond, or lack of adhesion between the particles in the refractory. In order to predict the resistance of a refractory to glass attack, means of measuring these properties will have to be devised. J. A. SUGDEN.

New apparatus for measuring the thermal expansion of refractory materials at 1600°. K. ENDELL (Feuerfest, 1929, 5, 3-4).—Prismatic or cylindrical test-pieces, 60—100 mm. long, plane at each end, are heated in an electric muffle furnace. Four heating elements ("silite" rods) are placed symmetrically about the test-piece, which rests on a refractory support. Two telescopes, with parallel optical axes the distance between which can be varied and accurately

measured, are focussed one on each end of the testpiece. The length (or distance between marks) of the test-piece before the test is accurately measured with callipers. The telescopes are fitted with adjustable ocular micrometers with glass scale. At a distance of 1 m. from the object, each division of the scale corresponds to 0.1 mm. Changes in length can thus be read off directly without moving the apparatus, and inaccuracy due to parallax is avoided. F. SALT.

Determination of the sp. gr. of refractory materials. E. KÜHN (Feuerfest, 1929, 5, 5).—A "volumoscope" is described by means of which more than 30 sp. gr. determinations can be made in 1 hr. The apparatus is filled with 200 g. of the material, a vacuum is produced, and by a simple operation of the hand the true sp. gr. can be read off. The material being tested comes into contact with air only. F. SALT.

Smooth-surface tile. V. S. SCHORY (J. Amer. Ceram. Soc., 1929, 12, 75—78).—Successful attempts to produce a white floor tile with a surface which is unglazed or non-vitrified but which is smooth enough not to hold the dirt are described. After trials with many bodies it was found that an oxidising fire was essential to the production of the desired surface. A consequent reduction in the amount of buff firing material had to be made in order to retain the ivory-white colour. The composition is given of a reliable body maturing at cone 7. The type of felspar used was found to affect the extent of the development of the smooth surface, but no explanation of this is given. J. A. SUGDEN.

Pyrophyllite, a new ceramic raw material. G. R. SHELTON (J. Amer. Ceram. Soc., 1929, 12, 79-82). -The physical and chemical properties of the mineral are given and a brief description of the deposits and their origin and distribution. Tests are being carried out on the possible substitution of the mineral for some of the usual ingredients of white-ware bodies. It has been found that when replacing ball clay an increase in the amount of pyrophyllite causes an increase in whiteness, density, and shrinkage, a marked decrease in plasticity, and a decrease in adsorption. Owing to its talc-like properties it is expected that pyrophyllite will reduce the abrasion of press dies. The use of the mineral as a refractory (cone 27) and in glazes is being examined. J. A. SUGDEN.

Scratch-hardness tests of ceramic materials. L. NAVIAS (J. Amer. Ceram. Soc., 1929, 12, 69-74).-Preliminary experiments are described. Three sets of standards were prepared: (1) Mohs' scale of minerals, (2) a set of steel samples, and (3) a set of Carboloy (tungsten carbide alloy) samples. The last two series were arranged according to the Brinell and Rockwell hardness tests. The three sets of standards were compared with one another according to their scratchhardness. Scratch tests were then carried out on stoneware, porcelains, glazes, glasses, and enamels. With one or two exceptions the order of hardness was the same with each of the three series of standards.

J. A. SUGDEN.

Absorption of two earthenware bodies and their resistance to crazing in the steam test. E. P.

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WRIGHT (J. Amer. Ceram. Soc., 1929, 12, 118—122).— An ivory and a white earthenware body matured to various absorptions were, when glazed, autoclaved to steam pressures of 100 and 150 lb./in.<sup>2</sup> The results show that bisque firing treatment as expressed by the absorption is of primary importance in resistance to crazing. Steam treatment at 100 lb. pressure will cause crazing above an absorption of 7—8% for the ivory body and above 8.5% for the white body. Treatment at 150 lb. pressure lowered the critical resisting absorption to 6% in each case. With both types of body there is a narrow range within which suitable glostfiring conditions may increase the resistance to crazing. The composition of bodies and glaze is not given.

# J. A. SUGDEN.

New method of direct firing glazed ceramic bodies. F. A. WHITAKER (J. Amer. Ceram. Soc., 1929, 12, 87—95).—The Carboradiant principle of oil firing has been applied to a rectangular sanitary-ware kiln. Improvement in control, increase in first-quality ware, and economy of fuel resulted. J. A. SUGDEN.

#### PATENTS.

Manufacture of reinforced glass. Soc. D'ETUDE DES VERRES ET GLACES DE SÖRETÉ (B.P. 293,053, 30.6.28. Fr., 1.7.27).—A sheet of transparent strengthening material, such as celluloid, is interposed between two sheets of glass the inner surfaces of which have been smoothed by grinding or by means of a concentrated varnish. The indices of refraction and the dispersion are the same for the two materials.

W. J. BOYD.

Manufacture of unsplinterable glass. J. H. ROBERTSON (B.P. 303,332, 25.6.28).—Two sheets of glass coated successively on one side with a solution prepared by treating casein with aqueous phenol solution, pouring off excess solvent, and dissolving the residue in strong ammonia, and with a solution of celluloid and phenol in, e.g., amyl acetate, the coatings being dried after each application, are united to an interposed sheet of celluloid by gentle squeezing in a bath of alcohol, followed by heating at 55° for 20 min. in a press; the composite sheet is subsequently heated at 55—60° for several hours. L. A. COLES.

White base for enamels and glazed. DEUTS. GASGLÜHLICHT AUER GES.M.B.H. (F.P. 625,260, 29.11.26. Ger., 10.12.25).—White coatings are obtained by using bases comprising mixtures of titanium oxide with cerium oxide, or of either of these with white oxides of multivalent elements, preferably those of the 3rd, 4th, and 5th groups of the periodic system, e.g., alumina, silica, stannic oxide, etc. L. A. COLES.

Production of cold glazes. KERAMENT- & KUNST-STEINWERKE C. H. JERSCHKE A.-G. (F.P. 625,203, 27.11.26).—The sand in the usual mixtures is replaced by material capable of absorbing water, preferably slowly quenched slag in grains of 1—2 mm. diam., in quantity not less than 25% of the mixture. In the production of coloured glazes, a thin layer of the coloured material may be applied over a colourless foundation layer. L. A. Coles.

Ceramic-glazing process. W. O. PROUTY, Assr. to AMER. ENCAUSTIC TILING CO., LTD. (U.S.P. 1,693,252, 27.11.28. Appl., 6.5.25).—Ceramic articles having a variegated colour are obtained by firing the bisque after the successive application of a glaze mixture of suitable colour, a glaze mixture of a different colour and containing insoluble material that is eliminated during the firing, and a final glaze mixture.

#### L. A. Coles.

Manufacture of chromite-containing refractory material. J. I. BROWN (B.P. 304,492, 10.2.28).—The usual clay, magnesia, or lime binder of the chromite material is replaced by a hydraulic cement  $(40\% \text{ Al}_2\text{O}_3)$ . The addition of 5—10% of such fused cement enables moulded blocks or bricks to be laid without previous burning. For rammed materials 10—20% of cement is added. J. A. SUGDEN.

Mould for high-temperature casting of refractory bodies. K. E. PEILER, Assr. to HARTFORD-EMPIRE Co. (U.S.P. 1,696,688, 25.12.28. Appl., 28.9.26).—A mixture of 80—85 pts. of fused silica, part of which is amorphous, ground to 20-mesh, 20—15 pts. of powdered lime, and a suitable quantity of water is dry-pressed and hardened by heat in the presence of steam. L. A. COLES.

Manufacture of waterproof polishing and abrasive paper etc. C. KLINGSPOR (B.P. 302,430, 5.10.27).— The binding material comprises animal glue previously soaked in water mixed at a temperature not above 75° with a liquid phenol-formaldehyde condensation product, with or without the addition of alcohol, glycerin, linseed oil, etc. After applying the abrasive powder, the binder is hardened by heating gradually to a temperature not exceeding 150°. L. A. COLES.

Manufacture of splinterless glass. G. B. RILEY (B.P. 304,630, 21.10.27).

Refractory materials (B.P. 302,087).—See X. Drycoating of lamp bulbs (U.S.P. 1,698,845).—See XI.

## IX.—BUILDING MATERIALS.

Setting of cement. III. H. GESSNER (Kolloid-Z., 1929, 47, 160-175; cf. B., 1929, 56).-Measurements of the hydration of cement show that definite amounts of water are taken up at different aqueous vapour pressures, the amount increasing with the time. The vapour-pressure isotherms resulting from the water-content curves show a definite break at a vapour pressure of 10.4 mm. in systems 3 days old; the break is shown less clearly by older systems. The conclusion is reached that in the initial stages of setting a definite hydrate is formed in the sheaths surrounding the clinker particles; during the subsequent course all the water bound by capillary forces becomes bound chemically. The velocity of diffusion of water in set cement is practically nil. Comparison of measurements of swelling with those of water content shows that the total system cement + water undergoes a contraction of about 7%. E. S. HEDGES.

Action of water on tricalcium silicate and  $\beta$ dicalcium silicate. T. THORVALDSON and V. A. VIGFUSSON (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 423-431).—The hydrolysis of fairly pure sampl s of tricalcium silicate and  $\beta$ -dicalcium silicate was studied in an attempt to discover if products of definite chemical composition are formed. The rates of hydrolysis of these two compounds were compared ; that of tricalcium silicate is much more rapid than that of  $\beta$ -dicalcium silicate. It is possible that the hydrolysis of the latter takes place in two steps. The hydrolysis curves for the two silicates were determined from the point of complete hydrolysis to a point where the solution was slightly supersaturated by the lime liberated. The product of hydrolysis highest in lime which can exist in equilibrium with a solution of calcium hydroxide below saturation has a lime : silica ratio of approx. 3:2. An inflexion in the hydrolysis curves occurs at a point where the lime : silica ratio is approx. 1:1; this indicates the existence of a hydrated monocalcium silicate. The products of hydrolysis of the two silicates are probably identical.

A. J. MEE.

## PATENTS.

Production of fused cement in rotating kilns. G. POLYSTUS (F.P. 625,600, 2.12.26. Ger., 23.12.25).— The hot gases leaving the fusion compartments are divided into two unequal portions, the greater being used for drying and calcining the charge, and the smaller for preheating the heating gases or the air for combustion. The calcining and drying compart-

ents project into the fusion chamber to prevent stoppage of the opening for withdrawing the smaller supply of gases. L. A. COLES.

Cements, paints, etc. [containing alkali silicates]. A. G. RODWELL, W. ROPER, and A. J. C. HART (B.P. 304,355, 19.10.27).—The products comprise sodium silicate and sodium nitrate solutions, together with mineral pigments. L. A. COLES.

**Production of porous building materials.** G. RICHTER (B.P. 305,415, 25.4.28).—To the concrete mixture is added a metal powder, *e.g.*, aluminium, which has been activated so as to decompose water readily, together with a non-hydrolysing aggregate, *e.g.*, flaky ashes. M. E. NOTTAGE.

Production of artificial stone. E. A. P. RENOUF (F.P. 629,450, 4.5.26).—The product comprises a hard nucleus (silica) and a softer matrix bound together by chemical reaction; *e.g.*, a mixture of alumina with pieces of quartz (5 mm. diam.) which have been coated with milk of lime and dried is heated at  $1150-1200.^{\circ}$  L. A. COLES.

Bituminous emulsions and their use in the coating of substances with bitumen. Colas Products, LTD., W. S. WHITING, and A. G. TERRY (B.P. 301,544, 2.9.27).—Mineral aggregates are coated with bitumen emulsions in the presence of a strongly electrolytic alkali salt, excluding ammonium salts, e.g., 0.5-2.0%of sodium chloride calculated on the weight of the emulsion, which may be added to the mineral aggregate or to a mixture of it with the emulsion. A filler capable of absorbing water, e.g., up to 4% of soft limestone, may be added to the aggregate. L. A. Coles.

Bituminous composition for paving. G. COBB (B.P. 302,808, 28.12.27).—A single-layer paving composition contains 31-5% of bitumen and crushed rock aggregate. L. A. COLES.

Imitating marble and other surfaces. E. C. R. MARKS. From Oxford VARNISH CORP. (B.P. 304,509, 12.3.28).

Manufacture of road-surfacing materials or compounds. J. Y. Johnson. From Amiesite Asphalt Co. of America (B.P. 304,226, 14.10.27).

Manufacture of cement. T. RIGBY (U.S.P. 1,699,451, 15.1.29. Appl., 20.7.25. U.K., 28.7.24).— See B.P. 243,410; B., 1926, 129.

Manufacture of concrete. M. LÉVY, Assr. to "PRODOR" FABR. DE PROD. ORGANIQUES SOC. ANON. (U.S.P. 1,699,705, 22.1.29. Appl., 2.11.22. Switz., 16.11.21).—See B.P. 201,650; B., 1923, 977 A.

Treatment of wood and like porous material-M. SMITH (U.S.P. 1,699,635, 22.1.29. Appl., 29.12.25. U.K., 17.8.25).—See B.P. 265,643; B., 1927, 328.

Waterproofed materials (B.P. 303,935),—See V-Binding agents having hydraulic properties (B.P. 297,416).—See VII. Cement with metallic lustre (B.P. 303,938).—See XIII.

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

Coalescence of non-metallic inclusions in steel. C. H. HERTY, JUN. (Proc. Eng. Soc. W. Penn., 1928, 44, 259-264).-Since the larger a non-metallic particle is, the faster it will rise to the surface of the steel bath, it is desirable that inclusions formed on deoxidation should coalesce as rapidly as possible in order to be eliminated from the steel before it is cast. If the inclusion has a m.p. higher than that of the bath, no coalescence can be expected, although the particles may coagulate in the steel to form clouds of particles, and, in general, the lower the m.p. of the impurity the greater is the rate of coalescence. Surface tension also affects coalescence to a minor degree. Manganese and double silicates have a m.p. which allows of ready coalescence, but additions of aluminium to killed or rimmed steel generally form inclusions having a higher m.p. than that of the steel, and particles of alumina can be observed in the metal. It is possible that the use of double deoxidisers, e.g., magnesium-aluminium alloys, might produce cleaner steel by reason of the formation of more fusible compounds. C. A. KING.

Troostite. A. E. CAMERON and I. F. MORRISON (Trans. Roy. Soc. Canada, 1928, [iii], 22, III, 289-291).-A series of heat-treatments on a medium-carbon (0.5%)C) and a high-carbon (0.8% C) steel were carried out. The specimens were subjected to drastic quenching operations (+ 1000°) followed by tempering and drawing operations at 100°, 200°, 300,° 400°, and 500°. The same area in each steel was microscopically examined after each drawing operation and the photographs are reproduced. It appears that globular troostite is not a decomposition product of martensite, but is a primary decomposition product of austenite. The troostite-like structures formed by tempering operations are in reality free-grained sorbite, and may be developed from either primary troostite or martensite. Martensite tends to sorbitise as soon as the slightest reheating is applied. The carbon content determines the granulation of troostite. In the 0.5% carbon steel troostite remains unchanged until tempered at 300°, whilst with 0.8% carbon steel the globular troostite has granulated appreciably at 200°. A. J. MEE.

Determination of molybdenum in steel in presence of tungsten and vanadium. I. KASSLER (Z. anal. Chem., 1929, 76, 113—120).—After dissolution of the steel in hydrochloric acid and oxidation with nitric acid, the solution is evaporated at 80—90° (but not boiled) in order to obtain the tungstic acid free from molybdic acid. Separation is effected not by filtration and washing, but by removal and filtration of an aliquot part of the diluted solution. If much molybdenum is present a small quantity may be retained by the tungstic acid, in which it may be determined colorimetrically. H. F. GILLEE.

Experimental demonstration of the refining of metals by oxidation. J. SILBERSTEIN (Ind. Eng. Chem., 1929, 21, 172).—In the refining of iron or copper by oxidation, losses of metal occur as oxide if the concentration of impurities is small. The effect is capable of demonstration on the laboratory scale with lead containing 0.2—0.5% Na. On melting and stirring, a white film of sodium oxide appears. If the film is removed as it is formed the colour becomes bluish, lead as well as sodium being oxidised. Some time is necessary before the sodium is completely oxidised. C. IRWIN.

Reduction of the alkaline-earth sulphates in metallurgical operations. G. CHARPY and L. JACQUÉ (Compt. rend., 1929, 188, 473–475).—The authors' experiments indicate that sulphates of calcium and barium and probably of strontium are equally effective for the sulphidation of castings, any differences previously observed being due to the physical rather than the chemical properties of the melt. The sulphur equivalents of the amounts used were 1-4% of the iron present. Reduction of the sulphate to the sulphide which may be due to carbon and/or metallic iron is perceptible at 600° and rapid at 900°. J. GRANT.

Determination of tin in rubber-coated [tinned copper] wires. A. LEJEUNE (Bull. Soc. chim. Belg., 1929, 38, 25—30).—The rubber is removed from 5 m. of the wire by rubbing with a cloth soaked in benzene, the wire is dried and cut into small pieces, and 3-4 g. of these are dissolved in 20 c.c. of nitric acid (d 1.36). The solution is evaporated to dryness, the residue extracted with 200 c.c. of boiling 1% nitric acid, and the insoluble metastannic acid is collected, washed with 1% nitric acid, ignited, and weighed. The copper retained by the precipitate is too small to affect the results, which are usually reported in g./m.<sup>2</sup> of surface. A. R. POWELL.

Metals and alloys for dyeing machines. GRUNDY. —See VI. Zinc foil for Leclanché cells. DROTSCH-MANN.—See XI. Protective layer in iron pipes. TILLMANS and others.—See XXIII.

# PATENTS.

Bonding of fine ores, burnt pyrites, etc. with liquid slags. M. PASCHKE and E. SCHIEGRIES (G.P. 453,469, 2.5.26).—The ore mixed with coke is introduced into molten slag from a blast furnace making ferromanganese to obtain a product containing 35-40%Fe and 5-10% Mn. A. R. POWELL.

Working of iron ores, slags, and residues containing lead, zinc, and silver. Soc. MIN. ET MÉTAL-LURG. DE PENARROYA (F.P. 620,994, 30.12.25).—The material is smelted in a blast furnace the hearth of which consists of a double layer of refractory firebricks. Between these layers and between the upper layer and the side walls outlets are provided for the removal of lead and silver. The furnace is operated with a hot blast so that the upper part of the shaft reaches a temperature of at least 450°, whereby the zinc is volatilised and may be recovered from the fume in settling chambers or by a bag plant. A. R. POWELL.

Detinning of tinned iron scrap. REYMERSHOLMS GAMLA INDUSTRI AKTIEBOLAG, Assees. of A. G. SUNDBERG (Swed. P. 59,566, 9.7.24).—The scrap is treated with a solution of copper salts containing alkali chlorides such as is obtained by the atmospheric oxidation of copper ores moistened with chloride solutions. The tin dissolves in the solution with the separation of an equivalent amount of copper. A. R. POWELL.

Utilisation of scrap metal. T. GOLDSCHMIDT A.-G. (B.P. 282,378, 5.12.27. Ger., 20.12.26).—The metal is formed into bundles which are heated by means of electrical eddy currents. The molten metal may be then worked up into cast iron or iron alloys by the adjustment of the carbon content and/or the addition of other substances. M. E. NOTTAGE.

Uniting metallically [welding] the seams of aluminium-plated sheet-iron. F. JORDAN (B.P. 304,170, 15.10.27).—The seams are pressed together and heated suddenly by electrical resistance, so that the aluminium coating at the joint is converted into oxide and the iron base melts and forms a tight weld. A. R. POWELL.

Alloy steels. TERNI (Soc. PER L'INDUSTRIA E L'ELECTRICITA) (F.P. 625,055, 24.11.26. Italy, 30.10.26). —Steels for use in the manufacture of turbines contain 0.05-1.5% C, 7-25% Cr, 0.05-20% Ni, 0.1-3% Si, and 0.1-2% Mn. A. R. POWELL.

Heat-treatment of [alloy] steel. F. KRUPP A.-G. (F.P. 626,672, 23.12.26. Ger., 1.4.26).—Nickel, chromium, silicon, vanadium, or tungsten steels are quenched from above 900°, to obtain a martensitic structure, and annealed at 600—650° to render them suitable for use in vessels handling gases and vapours at high temperatures and pressures. A. R. POWELL.

Manufacture of resistance-surface or abrasiveresisting alloys and castings, with special reference to a cast-metal grinding ball. L. J. BARTON (B.P. 277,004, 31.8.27).—The alloy contains  $98 \cdot 22 - 95 \cdot 54\%$ Fe,  $0 \cdot 8 - 1 \cdot 3\%$  C,  $0 \cdot 3 - 1 \cdot 0\%$  Si,  $0 \cdot 6 - 1 \cdot 8\%$  Mn, less than  $0 \cdot 18\%$  (S + P), and none or  $0 \cdot 5 - 3 \cdot 0\%$  Cr. It is made by melting steel scrap under a calcium silicate slag, adding ferromanganese until the slag becomes yellowish-green in colour to eliminate gases, deoxidising with ferrosilicon, and adding small amounts of aluminium as the metal is being poured into moulds.

entirential to retenotion of stating A. R. POWELL.

Production of metals or alloys together with refractory materials. T. R. HAGLUND (B.P. 302,087, 18.2.28).—Iron ores relatively rich in silica are smelted in an electric furnace to produce iron or ferrochromium, using magnesite and bauxite as fluxes in such proportions that the slag contains less than 15% SiO<sub>2</sub>, more than 15% MgO, more than 20% Al<sub>2</sub>O<sub>3</sub>, and up to 35%  $Cr_2O_3$ . The slag is cast into suitable shapes in moulds or is granulated, ground, and mixed with binding materials for the manufacture of refractory bricks.

A. R. POWELL.

Hardening of high-speed steel. W. STAUFFER (B.P. 289,082, 27.3.28. Switz., 23.4.27).—During heating, the articles to be hardened are packed in at least one non-caking carbide (e.g., silicon carbide) in a finelydivided form, such carbide having no appreciable carbonising action even at the temperature required (1380°) for hardening the steel. Graphite powder may be added to the carbide. M. E. NOTTAGE.

Hardening metal articles by nitrogenisation. P. F. M. AUBERT, A. J. P. and H. A. M. DUVAL (AUBERT & DUVAL FRÈRES) (B.P. 290,214, 5.3.28. Ger., 10.5.27; cf. B., 1923, 273 A).—The parts of the articles not to be treated are coated with a metal or metal alloy (e.g., of tin) and then covered with a layer of water-glass and aluminium powder, after which either the whole articles or the tinned parts are dipped in a bath of sodium or potassium nitrate heated to about 400°.

M. E. NOTTAGE.

Flotation process. C. SHAPLEY (U.S.P. 1,689,693, 30.10.28. Appl., 13.10.27).—Non-metallic minerals such as fluorspar, rutile, or corundum may be floated by immersing the ore in a 1% soap solution for a short period, draining off the excess solution, and agitating the ore in the usual flotation machines, using water free from any material which will decompose the soap.

A. R. POWELL.

Oil flotation process [for ores]. A. W. HAHN (U.S.P. 1,690,225, 6.11.28. Appl., 5.10.26).—Soluble lower salts or oxides of metals present in the ore pulp are oxidised by treatment with bleaching powder in presence of an alkali prior to adding a flotation agent.

R. BRIGHTMAN.

Roasting of sulphide minerals. J. B. READ and M. F. COOLBAUGH (U.S.P. 1,694,794, 11.12.28. Appl., 25.5.26).—Mineral sulphides and oxidising gases are passed in the same direction through a roasting furnace so that sulphates are formed and, as the charge approaches the end of the furnace, a predetermined weight of the original sulphide is added to convert a desired proportion of the sulphate into oxide. L. A. COLES.

Recovery of metals and metal compounds which are soluble in ammoniacal liquors. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,342, 24.8. and 7.11.27).—Oxidised copper ores are roasted at 600° in a current of chlorine or, after admixture with magnesium or ammonium chlorides, in a current of air. The product is leached with water, then with ammoniacal ammonium chloride solution to extract copper, silver, zinc, nickel, and cobalt. The filtered solution is heated with copper turnings to remove silver, then distilled to recover the ammonia and precipitate the carbonates or hydroxides of the other metals. The precipitate is converted into metal and the constituents are separated by electrolysis. A. R. POWELL.

Protection of copper apparatus against corrosion by carboxylic acids. Soc. CHIM. DES USINES DU RHÔNE (B.P. 284,685, 15.9.27. Ger., 4.2.27).—The ingress of oxygen into the apparatus during use is prevented by adding reducing agents, *e.g.*, sulphurous acid or its salts, covering the surface of the liquid with an inert oil, or passing a stream of inert gas through the apparatus. A. R. POWELL.

[Refining of] nickel and nickel alloys. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 304,371, 20.10.27).—The molten metal or alloy is treated with 0.25—0.5% of vanadium, niobium, or tantalum, preferably in the form of a 30% alloy with nickel, to remove the sulphur, then with 0.1% of magnesium to remove gases and oxides immediately before casting. A. R. POWELL.

Production of aluminium-silicon alloys. A. PHILLIPS, E. BARON, and METROPOLITAN-VICKERS ELEC-TRICAL Co., LTD. (B.P. 305,311, 24.11.27).—Alloys containing 5—15% Si may be prepared in the "modified" state by adding about 4% of anhydrous powdered carbonate or bicarbonate of an alkali metal to the molten alloy, heating to 1000°, and stirring.

M. E. NOTTAGE.

Treating impure lead and lead alloys. Treating mixtures containing alkali salts of certain metals [slags from lead refining]. Treating metal particles [lead alloys]. G. E. DALBEY and T. P. HANFORD, Assrs. to STANLEY CHEM. Co. (U.S.P. 1,693,639-1,693,642, 4.12.28. Appl., [A, C, D] 14.1.27, [B] 4.5.26).-(A) Antimonial lead containing tin is melted under a flux containing sodium antimonate, from which the antimony is displaced by the tin in the alloy. (B) Tin, arsenic, and antimony are removed from lead by melting it with litharge and adding gradually a concentrated solution of sodium hydroxide so that the material on the surface of the metal remains in a powdery condition. (c) Alkaline slags obtained in the above treatments are leached with a small quantity of water to remove most of the unchanged sodium hydroxide, then with more water to dissolve sodium stannate, zincate, and arsenate. The residue is heated with metallic tin and more water to decompose plumbates and plumbites, the resulting stannate solution being added to that obtained by leaching, and the insoluble sodium pyroantimonate is separated from the metallic lead by elutriation. The solution of sodium salts is treated with sodium sulphide to remove zinc and lead and then with carbon dioxide to precipitate stannic acid and leave a final solution of sodium carbonate and arsenate. (D) Finely-divided lead alloys are refined by digestion with concentrated sodium hydroxide solution in the presence of an oxidising agent to remove tin, arsenic, and antimony.

A. R. POWELL.

Activation of refractory metal filaments. J. W. MARDEN, ASST. to WESTINGHOUSE LAMP Co. (U.S.P. 1,698,850, 15.1.29. Appl., 31.3.23).—A refractory metal filament to which a mixture of potassium thorium fluoride and powdered aluminium is applied and retained by a suitable binding material is heated *in vacuo* so that the fluoride reacts with the aluminium and thorium is deposited on the filament. J. S. G. THOMAS.

Removal of vapours from annealing boxes in bright-annealing processes. T. STASSINET (G.P. 450,186, 13.12.25).—To prevent condensation of moisture or hydrocarbon vapours in the outlet tubes of annealing boxes through which a reducing gas or vapour is passed, these tubes are provided with an electric heating coil packed in insulating material surrounding the tube.

A. R. POWELL.

**Refining or purifying metals and alloys.** F. W. CORSALLI (B.P. 303,094, 23.6.27. Addn. to B.P. 297,759; B., 1928, 899).—Liquid metal in a collecting vessel is submitted to a swinging movement while being heated. Such a vessel may consist of a forehearth with a stepped wall over which the metal enters, the hearth being capable of a rocking motion; or the container may be bathshaped, pivoted on a central knife-edge, and operated by eccentric discs or crank rods. C. A. KING.

Electrodeposition of [iron-nickel] alloys. STAND-ARD TELEPHONES & CABLES LTD. From WESTERN ELECTRIC Co., INC. (B.P. 304,354, 19,10.27).—To obtain electrolytically an alloy of 79% Ni and 21% Fe an electrolyte containing 212 g. of nickel sulphate crystals, 22 g. of ferrous sulphate crystals, 18 g. of nickel chloride crystals,  $2 \cdot 5$  g. of ferrous chloride crystals, 25 g. of boric acid, and 180 g. of sodium sulphate crystals per litre is used with iron and nickel anodes, the surface areas of which are in the ratio of 15 : 85. The bath is operated at 50° with a current density at the anode of 4 amp./dm.<sup>2</sup>

A. R. POWELL. Electrolytic production of metals, especially magnesium. A. JESSUP (F.P. 626,497, 1.4.26).— The electrolysis is carried out in a cell divided into two compartments by a vertical wall terminating a short distance above the bottom of the cell, which is covered with a molten alloy of higher sp. gr. than the electrolyte. The electrolyte may be the same in both compartments or may consist of a single fused salt in one compartment and a mixture in the other. The cathode consists of a molten metal which floats on the surface of the electrolyte, and the anode of a carbon or graphite plate ; the alloy serves as the cathode in one compartment and the anode in the other. A. R. POWELL.

Chromium plating [bath]. Soc. NANTAISE ELECTRO-CHIM. ET MÉTALLURGIQUE (F.P. 625,313, 11.3.26).— The electrolyte contains more than 50% of chromium trioxide together with small quantities of chromium salts. The throwing power of this electrolyte is claimed to exceed considerably that of the solutions generally used. A. R. POWELL.

Coating metallic or non-metallic bodies with (A) rhodium, iridium, and ruthenium, (B) with osmium. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 304,396 and 304,580, 31.10.27). —The body to be coated, *e.g.*, a wire of tungsten, molybdenum, or nickel, is passed continuously between two electrical contacts in a chamber filled with a carbonyl chloride derivative of (A) rhodium, iridium, or ruthenium, or (B) osmium. As it passes between the contacts the

wire is heated by resistance to 500—600°, whereby a bright coating of the required metal is deposited on its surface. A. R. POWELL.

Uniting metallically the seams of aluminiumplated sheet iron. F. JORDAN (U.S.P. 1,699,434, 15.1.29. Appl., 12.11.27. Ger., 15.5.26).—See B.P. 304,170; B., 1929, 249.

Brass composition and its production. O. JUNKER (U.S.P. 1,699,665, 22.1.29. Appl., 23.7.25. Ger., 8.5.24).—See B.P. 243,998; B., 1926, 133.

[Spraying nozzle for introducing water into] cupola furnaces. A. J. STEPHENS. From VULCAN-FEUERUNG A.-G. (B.P. 304,887, 16.12.27).

Electric annealing furnace (B.P. 292,139).-See XI.

# XI.—ELECTROTECHNICS.

Zinc foil for the Leclanché cell. C. DROTSCHMANN (Chem.-Ztg., 1929, 53, 29-31, 50-52, 66-68).-The best conditions for, and effect of, amalgamation are discussed. With dry-cell mixtures containing 0.2-0.8% of mercurous chloride, the rate of amalgamation depends on this proportion, on the temperature, and on the time allowed, but is sufficiently rapid at 75° (3-10 min.). Amalgamated zinc electrodes in such mixtures, with no positive electrode, showed no corrosion after remaining sealed for 12 months. When the positive element is present, a dotting over of the zinc surface occurs rapidly and reaches a maximum, with amalgamated electrodes. in 48 hrs. Etching or washing of the zinc surface before amalgamation does not influence this change, which has no apparent effect on keeping the cell. Strips of copper, iron, lead, and tin soldered to the zinc foil caused no observable corrosion in 12 months in dry cells containing mercurous chloride. The presence of lead in the zinc foil is also not harmful, but foil containing arsenic suffered rapid corrosion. The presence of arsenic in the positive electrode mixture was found to affect the cell very badly. Hammering or deforming the surface of the zinc foil causes corrosion, but moderate local heating, such as is necessary for soldering, does not itself increase corrosion. The blackening of the zinc, observable in all cells partially or completely discharged, could not be explained by analysis of the black material, which contained only 1% Pb and 1% Hg. Chemical examination of the metal yields no information as to its utility for dry cells, and tests based on the rates of temperature rise when pieces of the same weight and surface are treated with dilute sulphuric acids in Dewar flasks are suggested. S. I. LEVY.

Effect of gases on the resistance of granular carbon contacts. P. S. OLMSTEAD (J. Physical Chem., 1929, 33, 69-80).—The resistance of granular carbon contacts, outgassed at 200° to a pressure of  $10^{-5}$  mm., has been measured by a special technique which is described, together with the effect of adsorbed nitrogen on the resistance. The contact resistance between carbon granules increases with the gas pressure over the range  $10^{-5}$  to 760 mm. The effect is reversible and varies exponentially with the pressure of the gas over this range. At constant pressure the increase in resistance diminishes with a rise in temperature. It

is concluded that the resistance of a granular carbon contact is partly determined by the amount of gas adsorbed at the surfaces of contact. At constant pressure, also, the contact resistance decreases with an increase in voltage, but at constant pressure and constant voltage the resistance decreases with a rise in temperature. These facts indicate that at least a part of the effect of applied voltage in reducing the resistance is due to the raised temperature of the contact. The possibility of an electrostatic effect, however, is not excluded. The effect of gas held in the pores of the carbon on contact resistance is also described and discussed. The falling resistance-voltage curve shows a sudden break at a higher voltage which depends on the kind of carbon used. This voltage is considered to mark the temperature at which further decomposition of the granules begins. At this point there is a linear relation between the resistance and the percentage of hydrogen found by analysis in the carbon.

L. S. THEOBALD.

Manufacture of magnesia insulating materials. F. E. M. BUSCHMANN (Chem.-Ztg., 1929, 53, 31—32).— The preparation of solid insulating blocks from basic magnesium carbonate and asbestos is described.

S. I. LEVY.

Comparator for determination of  $p_{\rm H}$  of coloured solutions. McCandlish and Hagues.—See I. Voltol. Wolf.—See II. Ultra-violet light-transmitting glasses. Starkie and TURNER.—See VIII.

#### PATENTS.

Electric furnace for annealing metals. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERT-WERKE GES.M.B.H. (B.P. 292,139, 7.5.28. Ger., 15.6.27). —Electric resistance heating elements, if desired mounted on frames, are arranged between the articles to be annealed, and similar heating elements are arranged on the inner walls of the furnace. J. S. G. THOMAS.

Magnesium primary cell. R. T. WOOD, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,696,873, 25.12.28. Appl., 5.8.25).—A magnesium electrode is placed in a neutral electrolyte containing a strong, soluble, oxidising agent which reduces corrosion of the magnesium on open circuit. J. S. G. THOMAS.

Mercury rectifier. C. KRÄMER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,694,328, 4.12.28. Appl., 24.4.26. Ger., 9.5.25).—An anode and a cathode are mounted in an evacuated vessel containing and interconnected with a receptacle for supporting hygroscopic material and having a porous wall so that water vapour may be admitted to, and mercury excluded from, the receptacle.

J. S. G. THOMAS. Oscillation generator. W. H. BURTIS (U.S.P. 1,692,074, 20.11.28. Appl., 21.4.27).—A piezo-electric crystal is coated with a silicate binder to retain a powdered metal, which forms an electrode.

F. G. CLARKE.

Dry-coating of lamp bulbs. D. S. GUSTIN, ASST. to WESTINGHOUSE LAMP Co. (U.S.P. 1,698,845, 15.1.29. Appl., 10.12.27).—An electric discharge is passed between electrodes which are respectively arranged near the external and internal surfaces of a hollow vessel containing the coating-material, the vessel being rotated during passage of the discharge. J. S. G. THOMAS.

Vacuum device and method of cleaning up residual gases therein. D. MACRAE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,699,112, 15.1.29. Appl., 9.1.23).—A galvanised iron electrode arranged in a bulb exhausted to a moderate degree is heated to cause evaporation of the zinc thereon and so effect a clean-up of the residual gases in the bulb.

J. S. G. THOMAS.

Electrode [for water-resistance steam generator]. H. W. MATHESON and J. C. SMITH (U.S.P. 1,693,794, 4.12.28. Appl., 23.8.24).—A hollow, inverted, frustoconical electrode forms an upwardly-enlarging steam conduit within a tubular discharge zone from which steam is directed into the conduit. J. S. G. THOMAS.

Electric gas-purifying plant. SIEMENS-SCHUCKERT-WERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 278,710, 4.10.27. Ger., 5.10.26).—The rectifier for producing high-tension continuous current consists of metallic bodies, *e.g.*, copper plates, coated on one side with a metallic oxide, *e.g.*, copper oxide, with interposed metal foil, such as lead foil, mounted alternately in series and in intimate contact with each other. [Stat. ref.] J. S. G. THOMAS.

Precipitation electrode for electrostatic gaspurifying apparatus. ELEKTRISCHE GASREINIGUNGS GES.M.B.H. (G.P. 451,041, 5.1.23).—The electrodes consist of metal rods shaped like the blades of a turbine and are so placed in the gas conduit that the gas stream impinges on them in a horizontal direction and after flowing over their entire surface leaves them in the same direction. A. R. POWELL.

Temperature-measuring device. F. S. STICKNEY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,867, 18.12.28. Appl., 6.4.27).—The cold junction of a thermocouple is connected to a Wheatstone bridge having side resistances respectively of high and low temperature coefficients of resistance. An electrical indicator is placed in the thermocouple circuit, and temperature changes in the cold junction are made sluggish by a heat-storage device. J. S. G. THOMAS.

Electrodes for indication and determination of the chemical composition of liquids. H. S. HAT-FIELD (B.P. 302,490, 5.12.27).—In apparatus for the determination of electrode potentials, two electrodes of relatively large area are placed on either side of a removably-mounted, thin, porous plate forming one wall of a chamber containing one liquid and making external contact with another liquid. The electrodes may be connected to actuate a low-resistance galvanometer etc. J. S. G. THOMAS.

Determining gaseous carbonic acid. H. SCHMICK, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,695,031, 11.12.28. Appl., 20.7.26. Ger., 1.9.25).—A quartz filter and a gas containing carbon dioxide are arranged between a source and a receiver of radiation, and means are provided for determining the action of heat rays at the receiver. J. S. G. THOMAS.

Dielectric substance and its manufacture. A. MOUILLEFARINE (U.S.P. 1,696,501, 25.12.28. Appl., 18.2.27. Fr., 18.2.26).—Wood is dyed to resemble ebonite by immersion for 24 hrs. in a liquid composed of Basic Black, acetic acid, and sodium chloride, heated to 100° for the first 4 hrs., and then cooled. The wood is further heat-treated and impregnated at 150—300° with an insulating mass of bituminous shale. It is then dried and varnished. J. S. G. THOMAS.

Mica plate [insulating] compositions. MICA INSULATOR Co., Assees. of J. M. COFFEY (B.P. 288,260, 13.3.28. U.S., 5.4.27).—Mica splittings are bound together by a reaction product of glycerol and phenol or its homologues. Thus the binder may be prepared by heating 10 pts. of phenol with 7 pts. of glycerin with a small amount of sulphuric acid at 160—190°, the reaction being complete when about 3½ pts. of water have been distilled. The acidity of the resultant rubbery mass is neutralised with sodium carbonate.

J. S. G. THOMAS.

Leading-in conductor. S. RUBEN (U.S.P. 1,692,998, 27.11.28. Appl., 8.9.27).—A layer of cuprous oxide is integrally formed with a metal body. J. S. G. THOMAS.

Primary battery. W. A. F. BLEECK (U.S.P. 1,699,580, 22.1.29. Appl., 4.11.25. Austral., 23.2.25).— See B.P. 248,011; B., 1927, 820.

Pasting of electric accumulator plates. C. A. VANDERVELL & Co., LTD., R. C. PATERSON, and P. J. BEDDOUS (B.P. 305,302, 17.11.27).

Electric discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 304,423, 23.11.27).

Cathodes for gas-discharge vessels. SIEMENS & HALSKE A.-G. (B.P. 279,497, 21.10.17. Ger., 23.10.26).

Insulation [tape] material for electric apparatus. H. D. SYMONS (B.P. 302,397, 17.9.27).

Hydrocarbons from coal and water (B.P. 283,177). —See II. Iron-nickel alloys (B.P. 304,354). Electrodeposition of magnesium (F.P. 626,497). Chromium plating (F.P. 625,313).—See X.

# XII.—FATS; OILS; WAXES.

Determination of water and fats in materials rich in fats. N. D. PRIANISCHNIKOV and S. M. TELNOV (Z. anal. Chem., 1929, 76, 161-166).-The method, especially suitable for the evaluation of oil-containing seeds etc., combines the volumetric determination of water by distillation with benzine and the removal of fats by benzine in one operation. The apparatus consists of a flask (100 c.c. capacity) with a bent neck the upper portion of which is vertical and enlarged to hold a glass extraction thimble containing about 5 g. of material. Solvent vapours, after passing the thimble, enter the usual arrangement of reflux condenser and graduated tube for the volumetric determination of water. The loss in weight of material is equal to the fat and water content. To obtain satisfactory separation of water the solvent (b.p. 95-120°) should contain 4-6% of isobutyl alcohol, when the observed volume of water should be multiplied by 0.945, or 4-6% of *n*-amyl alcohol, when the correction factor is 0.992. J. S. CARTER.

Examination and identification of fats and fat mixtures. II. Clouding point and setting curves. B. LUSTIG and G. BOTSTIBER (Biochem. Z., 1929, 204, 46-61; cf. B., 1929, 178.)—Every species of fat examined was shown to have its own clouding point and its own setting curve with definite maximum and minimum. By an examination of these curves fats may be identified and adulterations detected.

# J. H. BIRKINSHAW.

Fatty acids and component glycerides of some New Zealand butters. T. P. HILDITCH and (MISS) E. E. JONES (Analyst, 1929, 54, 75-95).-The composition of the mixed acids in 3 samples of New Zealand butter was determined by the following method Steamvolatile acids were removed by prolonged distillation in steam, recovered from the aqueous distillate by extraction with ether, and fractionally distilled. The nonvolatile acids were separated into solid and liquid acids by the lead salt method and converted into the methyl esters, which were quantitatively fractionated by distillation under reduced pressure. The approximate percentage composition of the acids was: butyric 3, hexoic 2, octoic 1, decoic 2, lauric 4, myristic 11, palm-itic 28, stearic 9, oleic 33-34, linoleic 4-5%. There was consistent evidence of the presence of a small percentage of acids less saturated than oleic acid. The butyric acid content as determined from the Kirschner value is about 20% in excess of the amount actually present. The percentage of fully saturated glycerides was determined by the permanganate method (B., 1928, 791), and the acids thus combined were fractionated via the methyl esters. The amount of saturated glycerides was about 30%; the fatty acids contained therein were the same as those in the whole fat and in proportions not greatly dissimilar. There was, however, a tendency for the lower acids to associate with the unsaturated acids and a slight compensating concentration of higher acids in the fully-saturated glycerides. These glycerides are probably of the complex, mixed type. The remainder (70%) of the butter fat consisted of mixed glycerides of saturated and unsaturated acids, the molecular proportions being 104 mols. of saturated acids to 100 mols. of unsaturated acids. The approximate composition of the butter fats might be thus formulated : mixed fully-saturated glycerides 30%, mixed mono-oleo-disaturated glycerides 36% and mixed dioleo-monosaturated glycerides 34%, all the saturated acids being comparatively evenly distributed throughout J. S. CARTER. the whole fat.

Deterioration of soap-nicotine preparations. II. C. C. MCDONNELL and J. J. T. GRAHAM (Ind. Eng. Chem., 1929, 21, 70-73; cf. McDonnell and Nealon, B., 1924, 803).—Commercial soap-nicotine preparations decrease in nicotine content on storage, the loss in the case of hard soaps being greater than with soft soaps. Excess of alkali or fat has no appreciable influence. When air is not excluded the loss of nicotine from soap preparations made from drying oils is due mainly to oxidation; in the case of those made from non-drying oils it is caused by volatilisation of the nicotine. Both hard and soft soaps, whether made from drying or non-drying oils, when packed so that they were completely protected from the air, suffered no loss of nicotine during 2 years' storage. F. R. Ennos.

Reactions of soya-bean oil. A. RICHARD (Ann. Falsif., 1928, 21, 579-582).—As little as 10% of soya-bean oil may be detected in admixture with arachis or olive oil by emulsifying 10 c.c. of the sample with 1 c.c. of nitric acid, and leaving the tube on a boiling water-bath for 15 min. Pure olive or arachis oil solidifies, as under these conditions the oleic acid appears to be transformed into its isomeride, and in 24 hrs. solidification is practically complete. Soya-bean oil shows no trace of solidification, and the viscous mass, instead of being yellow, is reddish-brown. After determining the presence of olive or arachis oil the proportion may be found from the iodine value.

## D. G. HEWER.

Coffee berry oil. L. von Noël (Pharm. Zentr., 1929, 70, 69-77; cf. Meyer and Eckert, B., 1911, 139).—By modifying the ordinary methods of separating the unsaponifiable constituents, the fatty acids of the oil were obtained white and without rotation; they consist of 29% of palmitic, 3% daturic, 14% of carnaubic, 2% of oleic, and 50% of linoleic acids, but decoic acid was not found. The oil is very rich in unsaponifiable constituents, which have a very high specific rotation; such rotation, however, is not due to the presence of sterols. The fatty acids are very little affected by roasting the coffee. S. I. LEVY.

## PATENTS.

Manufacture of esters of aliphatic, aromatic, and alicyclic alcohols and fatty acids by the action of alcohols on oils or fats or on glyceryl esters of fatty acids. Soc. ANON. ASSOC. PARISIENNE POUR L'IND. CHIM., Assees. of E. DESPARMET, R. WEIL, and F. SCHMITT (F.P. 615,953, 1.10.25).-The oil, or fat, or fatty acid is heated with the alcohol in the presence of a catalyst, such as a mineral acid or a salt, e.g., trisodium phosphate. E.g., 500 kg. of copra, 1000 kg. of butyl alcohol, and 10 kg. of hydrochloric acid (d. 1.16)are heated under reflux for 10 hrs., a further 10 kg. of hydrochloric acid are added, and the heating is repeated. Finally a third portion of acid is added and the same treatment repeated. The excess of butyl alcohol is distilled off, and the residual liquid, which separates into two layers, is transferred to a separating funnel. The lower layer contains the glycerol and the upper the butyl ester. The latter is removed, washed, neutralised, and purified by distillation in vacuo. A. R. POWELL.

Production of fatty bodies soluble in water. R. VIDAL (B.P. 289,001, 22.12.27. Fr., 19.4.27. Addn. to B.P. 280,193 and 285,473; B., 1929, 137).—Ricinoleic acid is used instead of oleic acid etc. as described previously, and the alkali hypochlorites or hypobromites are added repeatedly in small quantities. L. A. COLES.

Production of soap from sulphurised rosin. L. DE MOLTKE-HUITFELDT (F.P. 629,214, 17.2.27).— Rosin (500 g.) and sulphur (125 g.) are heated together for  $\frac{1}{2}$  hr. at 155—160°. The mass is finely powdered, 195 c.c. of sodium carbonate solution ( $d \ 1 \cdot 32$ ) diluted with 700 c.c. of water are added, and saponification is effected at 100°. A transparent, yellow, gelatinous soap

is obtained. Before treatment with sulphur the rosin may be dissolved in a medium such as turpentine or rosin oil. W. J. BoyD.

Manufacture of linoleum. G. E. HEVL, and HYCOLITE LIQUID WALLPAPER MANUF. Co., LTD. (B.P. 303,292, 7.2.28).—A mixture of 60—50% of finelydisintegrated, dry, oil-resistant paper or fibrous pulp (e.g., wood pulp) and 40—50% of a mineral lubricant such as china clay or plaster of Paris is substituted for part of the cork usually employed. The paper or pulp may be rendered non-absorbent of oil by soaking in varnish or a drying oil and drying prior to final disintegration. W. J. BOVD.

Treatment of tall oil. OEL- U. FETT-CHEMIE GES.M.B.H. (B.P. 281,637, 21.11.27. Ger., 4.12.26).— Tall oil is separated into fatty acid and resinic acid by distillation in a high vacuum (below 8 mm. of mercury), the process being interrupted when resinic acid begins to distil over. E. LEWKOWITSCH.

Impregnating compositions (B.P. 302,710). Lubricating oils (U.S.P. 1,691,882 and 1,691,654).— See II. Esters (B.P. 302,411).—See III.

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

Paint resistant to flue gas. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1929, 34, 1061-1063).—A preliminary account is given of investigations on the durability of paints designed for exposure to hot flue gases. Indications are obtained that, contrary to expectation, the introduction of tung oil does not improve the durability of such paints. Adhesion to red lead priming paint is, in general, very poor, no advantage being shown over tests carried out directly on bare metal. The relative efficiency of various pigments, *e.g.*, white lead, lamp black, graphite, is discussed. Types of failure are illustrated. S. S. WOOLF.

Radium, with special reference to luminous paint. A. T. PARSONS (J. Oil and Colour Chem. Assoc., 1929, 12, 3—25).—A general account of the chemical and radioactive properties and the manipulation of radium is given, and its applications to medical practice and chemical investigations are indicated. Radioactive luminous paints consist of a phosphorescent body, e.g., suitably prepared zinc sulphide etc., the luminescence of which is excited by radioactive material, e.g., radium, mesothorium, etc. The manufacture of luminous zinc sulphide is described. Work on the permanence of luminous paint and the recovery of radium therefrom is summarised. S. S. WOOLF.

Physical chemistry of colour lake formation. IV. Red Congo acid and Congo-red lakes. H. B. WEISER and R. S. RADCLIFFE (J. Physical Chem., 1928, 32, 1875—1885; cf. B., 1928, 131).—Aqueous solutions of Congo-red contain a red, colloidal anion, which is invisible in the ultramicroscope, but does not pass through a membrane permeable to ions in solution. Replacement of the sodium by hydrogen gives a blue colloidal acid according to the scheme 2Na<sup>+</sup> + R'' (red)  $+ 2H + 2Cl' = H_2R$  (blue)  $+ 2Na^+ + 2Cl'$ , and since the reaction is partially reversible, Congo-red is an unsuitable indicator in the presence of salts. The blue acid is slightly soluble in water yielding the red anion, the solubility increasing with a rise in temperature. In the blue sol the equilibria  $nH_2R$  (blue)  $= nH_2R$  (solution) + 2nH + nR'' (red) exist. Mordants such as alumina adsorb the blue colloidal acid forming lakes. The red lake is an adsorption complex of the hydrous oxide and red acid or alkali salt, and not a metallic salt (cf. Bayliss, A., 1911, ii, 886). The existence of the red Congo-acid in the solid state is still open to question. L. S. THEOBALD.

Determination of turpentine vapour in the air. W. D. BOGATSKI and W. A. BIBER (Z. anal. Chem., 1929, 76, 103-108).-The rose colour produced by turpentine in a hydrogen chloride solution containing 1% of vanillin requires the presence of at least 0.7 mg. of turpentine; on heating to boiling, however, the colour changes to a dull greenish-blue, visible with 0.5 mg. of turpentine. If a hydrogen chloride solution of d 1.19be employed, spontaneous heating occurs on addition of an alcoholic solution of turpentine, and the colour changes from rose to a bright greenish-blue, which reaches its maximum intensity after about 30 min. Under these conditions 0.14 mg. of turpentine may be detected, whilst if the mixture be heated the colour change is visible with 0.02 mg. Benzene, benzine, acraldehyde, carbon monoxide, xylene, and phenol give negative reactions, aniline produces a yellowish colour, whereas pyrogallol and resorcinol produce only the rose colour. A positive reaction is given by methyl ethyl ketone, camphor, pulegone, pinene, and dipentene.

H. F. GILLBE. Method of observing the drying times [of varnish films]. H. WOLFF and W. TOELDTE (Farben-Ztg., 1929, 34, 1060—1061).—The spreading of a drop of a solution of a red dye in boiled linseed oil on a drying varnish film reaches a minimum at the "dust-dry" stage, spreading occurring in the film prior to this stage and on the surface subsequently. By plotting "spreading area" against time, the progressive drying of films may be recorded. A permanent record may be obtained by "printing off" the test plates on photographic paper. The "sand method" is recommended for the observation of drying after the "dust-dry" stage. S. S. WOOLF.

Light-coloured condensation resin. H. A. GARDNER, C. A. KNAUSS, and A. W. VAN HEUCKEROTH (Ind. Eng. Chem., 1929, 21, 57).—Equimolecular proportions of phthalic anhydride, triethylene glycol, and tartaric acid on heating at 185—200° for 3 hrs. yield a pale amber resin in the form of a viscous solution, soluble in acetone, alcohol, and chloroform, insoluble in toluol, and compatible with nitrocellulose solutions. If heating is continued for 15—18 hrs. a dark amber plastic mass is produced which is incompatible with nitrocellulose solutions, but compatible with cellulose acetate solutions. These substances are apparently true liquid resins. Exposure tests on lacquers containing equal parts of the dark resin and cellulose acetate gave fairly satisfactory results. F. R. ENNOS.

#### PATENTS.

Paints and cements having a metallic lustre. C. NITTINGER (B.P. 303,938, 24.10.27).—A paint or cement consisting of ground coal slag, zinc powder prepared by grinding electrodeposited zinc, and a suitable vehicle is claimed. S. S. Woolf.

Red lead paint. C. D. FAHSEL (U.S.P. 1,697,388, 1.1.29. Appl., 10.11.21. Renewed, 5.9.28).—Red lead is mixed with boiled linseed oil, a light oil varnish, and a paraffin oil. S. S. WOOLF.

Paints, particularly for the surface-covering of thin rubber sheet material. P. SCHIDROWITZ and D. J. BURKE (B.P. 304,334, 16.7.27).—An improved paint suitable for application to thin flexible material, such as artificial leather, is produced by dissolving a gum resin, e.g., wax-free shellac or a dammar resin, in a low-viscosity solution of a cellulose ester in a highboiling solvent, e.g., ethyl lactate or benzoate, and intimately mixing this solution with a fine or colloidal suspension of the pigment in a non-drying oil or liquid plasticiser such as tricresyl phosphate. The paste is then thinned with a diluent comprising a liquid hydrocarbon, e.g., toluene or xylene, and an alcohol, e.g., amyl alcohol. Such paint avoids, largely or entirely, the common tendency to cracking and peeling. D. F. Twiss.

Paint and varnish remover containing an alkalimetal benzoate as thickening agent. B. N. Lougovoy, Assr. to CHADELOID CHEM. Co. (U.S.P. 1,691,771, 13.11.28. Appl., 25.2.26).—An alcoholic solution of sodium benzoate is mixed with acetone or other ketone. Benzene, toluene, or other hydrocarbons, but not chlorinated hydrocarbons, and, *e.g.*, ceresin or paraffin wax, may also be added. R. BRIGHTMAN.

Pigment for heat- and acid-resistant paints. H. HEMPEL and E. MURNSEER (F.P. 628,543, 4.2.27).— Iron or steel is dissolved in a mixture of hydrochloric and nitric acids, excess of acid is removed, and the paste formed after 8—10 days is dried slowly, ground, and dispersed in water. The water is evaporated and the treatment is repeated. The mass on being dried and calcined yields a brown-red product.

S. S. WOOLF.

Lacquers or enamels. Insulating enamels, varnishes, or like coatings. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, and A. T. WARD (B.P. 303,915 and 303,936, [A] 5.9.27, [B] 22.10.27).-(A) The adhesion of cellulose lacquers to smooth surfaces is improved by the incorporation of about 5% of a "glyptal" resin (in the partly condensed or soluble form) which has preferably been previously plasticised with oleic acid. (B) The flexibility of the usual insulating varnish system is improved by the use of a primer containing a "glyptal" resin which has been plasticised with oleic acid, tricresyl phosphate, etc., and which is cured by heat treatment in situ. One or two coats of the primer are applied and stoved for 1 min. at about 310°, subsequent coatings of oil varnish being stoved for 1 min. at 350°. A suitable primer consists of "glyptal" plasticised with 20% of oleic acid, furfuraldehyde, glycol diacetate, butyl phthalate, and coal-tar naphtha. S. S. WOOLF.

Manufacture of varnish. E. E. WARE, Assr. to ACME WHITE LEAD & COLOR WORKS (U.S.P. 1,697,213, 1.1.29. Appl., 23.6.24).—A liquid varnish-making ingredient passing continuously through a series of containers is subjected to predetermined temperature and pressure, and a resin ingredient is introduced at such a rate that uniformity in the proportion of ingredients and in their heat treatment is maintained.

S. S. WOOLF.

Plastic resinous material and its manufacture. H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,692,524, 20.11.28. Appl., 12.1.24).—Formalin is ground with 0.25—1.0 mol. of magnesium oxide and the mixture is heated, e.g., at 70°, with 1 mol. of m- and p-cresol or of cresylic acid containing not more than about 10% of o-cresol. The product is dried, incorporated with filler, and moulded at 160°. If the cresylic acid contains larger amounts of o-cresol, 1% of aluminium palmitate may be added to prevent sticking during R. BRIGHTMAN. moulding.

Resinous composition containing sulphur and its manufacture. C. ELLIS (U.S.P. 1,690,160, 6.11.28. Appl., 19.12.22).-A phenol is heated to above 160° with at least 1.6 pts. of sulphur in presence of an alkaline catalyst ; e.g., potassium carbonate with phenol gives a resin fusible at 130-140°. R. BRIGHTMAN.

New synthetic resins from aromatic hydrocarbons. P. KRISHNAMURTHY (B.P. 298,939, 6.2.28. India, 17.10.27) .- Aromatic hydrocarbons with sidechains (e.g., toluene) are chlorinated in the latter, and the purified product is treated in a solvent (carbon disulphide) with anhydrous zirconium tetrachloride or ferric chloride. B. FULLMAN.

Manufacture of condensation products of urea with formaldehyde. H. BARTHÉLEMY, Assr. to Soc. IND. DES MATIÈRES PLASTIQUES (U.S.P. 1,691,427, 13.11.28. Appl., 16.9.26. Fr., 24.9.25) .- The syrupy solution of the initial condensation product of urea and formaldehyde is treated with an aliphatic anhydride, e.g., acetic anhydride, preferably in presence of an alcohol.

R. BRIGHTMAN.

Purification of phenol-formaldehyde resins. F. SEEBACH, Assr. to BAKELITE GES.M.B.H. (U.S.P. 1,697,885, 8.1.29. Appl., 12.2.26. Ger., 6.3.25).-See B.P. 248,726; B., 1927, 532.

Azo dyes (B.P. 302,965) .--- See IV. Nitrocellulose of low viscosity (B.P. 289,387). Knifing compositions and lacquers (B.P. 302,615-6).-See V. Paints (B.P. 304,355).—See IX. Soap from sulphurised rosin (F.P. 629,214).-See XII.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Diffusion of water through rubber. E. E. SCHUMACHER and L. FERGUSON (Ind. Eng. Chem., 1929, 21, 158-162) .- Measurements by manometric and gravimetric methods indicate that the rate of diffusion of water through a rubber membrane is inversely proportional to the square of the thickness, and decreases greatly with increase in hardness of the rubber. Saturation with water increases the permeability, probably due in part to the increased vapour tension of water within the rubber and in part to the decreased hardness. The increase in permeability with temperature is also possibly associated with decreased hardness. No simple relation exists between the rate of diffusion and minor

alterations in composition of the rubber. A simple formula is mathematically derived applicable to the calculation of the rate of diffusion of water at various humidities through rubber samples of various dimensions D. F. Twiss. and composition.

Colouring of rubber. W. J. S. NAUNTON (J. Soc. Dyers & Col., 1929, 45, 31-35; cf. B., 1928, 720) .--Rubber can be coloured in the uncured state as latex or sheet or in the cured state as cold-cured, soft heatcured, or hard rubber (ebonite). Rubber colours can be classified as (a) soluble in rubber, (b) slightly soluble, and (c) insoluble in rubber, e.g., inorganic or organic pigments, or lakes. Latex can be coloured with almost any dyestuff which carries a negative charge, and hence does not cause coagulation. Vat dyes can be mixed with the latex and caused to oxidise in an insoluble form by agitation with air. Crêpe or uncured sheet rubber is best coloured by boiling it in an aqueous solution of a basic dye. Cold-cured rubber can also be dyed in a similar manner to crêpe, but usually a pigment is worked. into the dry rubber mix. The successful colouring of soft rubber depends on the use of accelerators, as almost all organic dyestuffs are suitable if the rubber is cured in the presence of a super-accelerator. Ebonite, which possesses a natural brownish-black colour, can be coloured by means of a balanced mixture of white pigment and colour. L. G. LAWRIE.

Practical method for obtaining dry air for humidity control in a rubber laboratory. F. S. CONOVER (Ind. Eng. Chem., 1929, 21, 162-164).-It is believed that zero humidity is more convenient and more satisfactory as a standard condition for the storage of rubber samples, before mixing, vulcanisation, and testing, respectively, than the relative degree of humidity recommended by the Physical Testing Committee of the American Chemical Society (cf. B., 1929, 104). A description is given of storage cabinets ventilated by a current of air dried by passage through a tower of silica gel. D. F. Twiss.

#### PATENTS.

Factice. [Rubber substitute.] Soc. ANON. LE TANK (F.P. 629,401, 28.4.26) .- Before treatment with sulphur chloride the oils are mixed with talc, calcined magnesia, or a mixture of magnesite with zinc oxide or manganese dioxide. Oils which react feebly with sulphur chloride are treated with magnesium linoleate, aluminium chloride, and zinc oxide. If mineral oils are used, a preliminary chlorination is effected by means of chlorine or hydrogen chloride in the presence of a catalyst. In this way uniformly vulcanised oils are obtained which can be used for the production of resilient tyres. D. F. Twiss.

Preparation of aqueous emulsions or dispersions of rubber, gutta-percha, balata, or similar materials. J. M. A. TOUCHON (F.P. 628,093, 25.1.27). -A concentrated solution of rubber in a volatile solvent is mixed in a colloid mill with water in which emulsifying or dispersing agents such as soap, saponin, casein, dextrin, glue, or naphthenates may be present. The rubber solution may be replaced by finely-divided rubber to which dispersing agents may be added. D. F. Twiss.

Production of rubber and other goods with textile insertions attached thereto or embedded therein. ANODE RUBBER CO. (ENGLAND), LTD. From ANODE RUBBER CO., LTD. (B.P. 304,157, 10.8.27).— Fibrous materials and fabrics to be impregnated or coated with organic materials, such as rubber, by agglomeration from dispersions, *e.g.*, by electrophoretic migration, are wetted with the dispersions or similar dispersions prior to the process of impregnation or coating. Such preliminary treatment ensures a high degree of adhesion. D. F. TWISS.

Vulcanisation of plates or articles of rubber. PHIL. PENIN GUMMI-WAAREN-FABR. A.-G., Assees. of E. WLCECK (G.P. 452,466, 7.5.25).—Rubber mixtures containing pigments or other ingredients of an inorganic or organic character are vulcanised by successive immersion, repeated, if necessary, in a 4—10% aqueous solution of sulphur dioxide and in saturated aqueous hydrogen sulphide. D. F. TWISS.

Colouring of rubber. IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 304,376, 21.10.27).—A pigment comprising a vat dye, such as duranthrene-blue, or dimethoxydibenzanthrone, and a substratum, such as clay or light magnesium carbonate, substantially insoluble in water, is prepared by mixing this substratum with the dye in the presence of water (and of a protective colloid, if desired) or of a medium (e.g., benzene) possessing a definite small solvent action for the dye; as a further alternative the dye may be added to the substratum in the form of an alkaline solution of the leuco-compound and then oxidised. The pigment preparations so obtained are then incorporated into rubber, increased tinctorial value being obtained.

D. F. Twiss.

Colouring of rubber with lipoid-soluble dyes. CONTINENTAL-CAOUTCHOUG U. GUTTA-PERCHA CO. (G.P. 452,340, 21.3.25).—Lipoid-soluble dyes or colour bases are dissolved in a molten mixture of rosin and free fatty acid or esters of such acids; the solidified mixture is introduced into rubber in the customary manner and generally dissolves completely, thereby enabling the production of transparent rubber articles.

#### D. F. Twiss.

Moulding of ebonite composition. H. GRAY, Assr. to B. F. GOODRICH CO. (U.S.P. 1,691,347, 13.11.28. Appl., 14.6.23).—A composition comprising 100 pts. of rubber, 40 pts. of sulphur, and 2 pts. of organic accelerator is mixed with 35—65 pts. (per 100 pts. of composition) of clay, asbestos, whiting, gas black, or other inert pigment, and vulcanised, *e.g.*, first in a mould, for 2 min. at 175°, and finally with open heat, at 145°, giving ebonite of high softening point. R. BRIGHTMAN.

Controlling the vulcanisation of rubber and similar materials. S. M. CADWELL, Assr. to NAUGA-TUCK CHEM. Co. (U.S.P. 1,698,712—5, 15.1.29. Appl., [A—C] 1.11.27, [D] 25.6.23. Renewed [D] 12.7.28).— See B.P. 218,247; B., 1925, 772.

Separation of mixed substances (B.P. 272,968).— See XX. XV.-LEATHER; GLUE.

Tanning value of "Takaout" galls from Tamarix articulata. F. and M. H. DE BALSAC and A. DEFORGE (J. Soc. Leather Trades' Chem., 1928, 12, 559-564).—Samples of "Takaout" galls, derived from Tamarix articulata, Wahl, and grown in various parts of Morocco and Algeria, contained water  $11 \cdot 7$ —13·1, tannin  $41 \cdot 6$ —56·3, soluble non-tans  $11 \cdot 4$ —17·1, insoluble matter  $21 \cdot 5$ — $28 \cdot 2\%$ ; the ratio tans : non-tans was  $2 \cdot 4$ —4·9. They contain a pyrogallol tannin which penetrates rapidly yielding a soft, full, creamy-white leather. Very little tannin is present in other parts of the tree. The galls are used by the native tanners. D. WOODROFFE.

Effect of heat on wetted, vegetable-tanned leathers. I. W. J. CHATERS (J. Soc. Leather Trades' Chem., 1928, 12, 544-558).-Strips of the leather were suspended in water in a specially devised apparatus, and the shrinkage of the leather was observed as the water was heated. Temperature-shrinkage curves when plotted showed a pronounced shrinkage at 70°, due to gelatinisation of the leather substance. The shrunk leather dried out hard and brittle. The shrinkage was least along the lines of tightness of the leather and most in a direction at right angles to them. The middle layer of a piece of sole leather was scarcely affected, and the flesh part shrank less than the grain. The shrinkage temperature was unaffected by dyeing the leather, but was lowered D. WOODROFFE. by stripping it with borax.

Determination of fat in leather. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1928, 12, 569—572; cf. B., 1926, 926).—Water is not retained by oils if they are heated at 105° for 3 hrs., but this may involve slight decomposition. A greater residue is obtained by evaporating off the solvent after extracting air-dry leather with light petroleum and drying the residue in a water-oven than by drying it at 105° or by extracting leather dried at 105° and drying the extract in either a water-oven or at 105°. The higher figure is attributed to the extraction of moisture from the leather and its retention by the fat extracted if the latter is heated in a water-oven only. The leather should be dried at 105° prior to the fat extraction to avoid this. D. WOODROFFE.

Significance of mechanical wood-joint tests for the selection of woodworking glues. T. R. TRUAX, F. L. BROWNE, and D. BROUSE (Ind. Eng. Chem., 1929, 21, 74-79).-Wood-joint tests are not considered so suitable for grading animal glues for woodworking as viscosity and jelly-strength tests. Different gluing conditions are necessary with different kinds of glue, and no deductions can safely be drawn from tests made under any one set. When a glue is applied under the most suitable conditions, the failure of the joint depends on the strength of the wood rather than on the grade of glue. Wood-joint tests are used to some extent for casein and vegetable glues, as a more refined technique for evaluating them has not yet been developed and as these glues yield stout joints under a comparatively wide range of 

Nature of adhesion between glue and wood. F. L. BROWNE and D. BROUSE (Ind. Eng. Chem., 1929,

British Chemical Abstracts-B.

21, 80-84).—The mechanical adhesion theory of th strength of glued wood joints is criticised, and experiments are described which suggest that specific adhesion is essential for satisfactory gluing of wood.

## F. R. ENNOS.

Determination of tannins in drugs. LINDE and TEUFER.—See XX.

#### PATENTS.

Manufacture of synthetic tanning agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,454, 9.1. and 9.7.28).—Hydroxymethyl derivatives obtained by the action of formaldehyde on phenols or their carboxylic acids are condensed with resorcinol below  $50^{\circ}$  in presence of a condensing agent to yield tanning agents; one or several hydroxymethyl groups may be present in the starting material. In the examples phenol, salicylic acid, and *p*-cresotic acid are used.

C. HOLLINS.

Adhesives for use in shoe-making. BRIT. UNITED SHOE MACHINERY CO., LTD., F. RICKS, and R. A. LINE-HAM (B.P. 304,823, 27.10.27).

## XVI.—AGRICULTURE.

Scottish soil types with special reference to North-East Scotland. G. NEWLANDS (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 187—192).—The general characteristics of the profiles of Scottish soils are typical of the podsol type. Further subdivision of this type is possible on the basis of organic and moisture contents, texture, and chemical composition. A. G. POLLARD.

Substances removed by the drainage from a Scottish soil. J. HENDRICK and H. D. WELSH (Proc. 1st Internat. Cong. Soil. Sci., 1927, 4, 163-171) .--Results of lysimeter experiments with cropped and manured soils are recorded and discussed. The small loss of nitrate from cropped soils even when highly fertilised was noteworthy. The amount of sodium in the drainage water was nearly as great as that of calcium, although the respective proportions of these two bases in the total replaceable bases of the soil were 4.5 and 85%. No appreciable amounts of phosphates were found in the drainage even where phosphatic fertilisers were used. The leached sulphate was increased by liming, presumably as a result of stimulated bacterial decomposition of the soil organic matter. In the chalk-free soil examined considerable amounts of silica appeared in the drainage. A. G. POLLARD.

Mineral composition of the soil as a factor in soil classification. J. HENDRICK and G. NEWLANDS (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 183—186).—An outline of the process for separating the minerals in the fine-sand fraction of soils by means of bromoform is described. The proportions of the various minerals present serve as a means of classification of soils according to origin. Characteristics of the parent rock are retained in cultivated soils. A. G. POLLARD.

Changes in soil reaction effected by long-continued manuring. B. THOMAS and F. J. ELLIOTT (J.S.C.I., 1929, 48, 51-54 T).—The Tree Field plots at the Northumberland County Experiment Station,

Cockle Park, form the subject of this investigation. Changes in soil reaction and base status produced by the long-continued application of lime, basic slag, superphosphate of lime, dissolved bones, and dung are examined. It is shown that dung from sheep fed with cake and also superphosphate produce no significant effect on  $p_{\rm H}$  or exchangeable bases, dissolved bones produce only a slight increase in the bases and no effect on  $p_{\rm H}$ , basic slag gives a slight increase in  $p_{\rm H}$  and a large increase in exchangeable bases, and lime produces a large increase in  $p_{\rm H}$  and a very large increase in exchangeable bases.

Influence of the replaceable bases on the soil solution formation in mineralised soils. F. MEN-CHIKOWSKY and S. RAVIKOVITCH (Soil Sci., 1929, 27, 49-68).-The distribution of replaceable bases varies with the depth of soil examined. Water extracts of soils remove amounts of bases corresponding to the proportions present in the absorbing complex, the relationship being the more definite in soils having the greater absorbing capacities. The amounts of bicarbonate and silicate ions in water extracts of soils increased with the water : soil ratio adopted. It is considered that water brings about the hydrolysis of the aluminosilicate nucleus of the colloidal complex, and that the reaction is independent of adsorbed sodium ions. The relative proportions of cations in water extracts of soils do not alter with the proportion used. The amount of ions of an individual base present in a water extract is the outcome of a definite partial decomposition pressure of compounds of adsorbed cation with the aluminosilicate complex. In highly mineral soils the nature of the soil solution reflects to a greater or smaller degree the composition of the adsorbed bases. A. G. POLLARD.

Nature of the nitrogenous compounds in fungous tissue and their decomposition in soil. A. F. HECK (Soil Sci., 1929, 27, 1-47).-Analyses of a number of fungi show the carbon contents to range between 40 and 44%, with nitrogen 1.5-7%. The carbon: nitrogen ratio of the substrate determines both the amount of mycelium produced thereon and its nitrogen content, which may vary considerably. As the available nitrogen of the substrate is decreased the nitrogen content of the mycelium falls to 2-3%. Further reduction in nitrogen supplies when the carbon : nitrogen ratio is of the order 10 or 12:1 lowers the amount of mycelium produced, without markedly affecting its nitrogen content. In dry fungous tissue 40-70% of the total nitrogen is water-soluble, and of this 80-90% is dialysable. Of the total nitrogen 80-85% is soluble in 0.05N-alcoholic soda; 40-65% of the water-soluble nitrogen consists of free amino-acids. No urea was found. After decomposition in soil for 26 days 40-60% of the carbon is liberated as carbon dioxide and 30-40% of the nitrogen appears as nitrate. In the absence of other sources of energy living fungous tissue liberates its own nitrogen by autolysis. Nitrification proceeds quite as rapidly as is the case with other materials of similar nitrogen contents. The rate of nitrification depends on the nature of the source of energy. Where the latter is of simple composition decomposition of fungous tissue is mainly bacterial and much nitrate is produced. Where cellulosic matter is the source of energy, decomposition of fungous tissue is mainly effected by fungi and the yield of nitrate is small. A. G. POLLARD.

Conditions of nitrification [in soil]. J. HENDRICK (Proc. 1st Internat. Cong. Soil Sci., 1927, 4, 175-182).-Analysis of drainage water from variously manured soils indicates that nitrification can take place freely and for long periods in acid, chalk-free soil in spite of increasing acidity resulting from continued use of ammonium sulphate. Ammonium sulphate is almost completely nitrified when used alone or in conjunction with other fertilisers, even when relatively large applications are made. Mineral fertilisers stimulate the activity of soil bacteria responsible for the decomposition of organic matter from which ammonia and ultimately nitrate is produced. The conception that the nitrogen of fertilisers may be utilised by soil organisms and stored in an insoluble form within their tissues is discredited. No evidence is obtained that nitrogen in a gaseous form is lost from soil when excessive amounts of nitrogenous fertilisers are used. A. G. POLLARD.

Tolerance limit of seedlings for aluminium and iron and the antagonism of calcium. J. R. SKEEN (Soil Sci., 1929, 27, 69—80).—Comparison of the growth of *Lupinus albus* and *Phaseolus vulgaris nanus* in the presence of iron and aluminium shows the former to be about three times as resistant as the latter to these toxic ions. The iron ion is 5—7 times as toxic as the hydrogen ion. The antagonism of calcium for iron and aluminium in this respect is shown, and limiting values for soil solution concentrations are discussed and demonstrated in pot experiments. Both iron and aluminium ions tend to become more toxic at 29°. Hydrogen-ion concentration of soils is of little significance as an ecological factor. A. G. POLLARD.

Deleterious action of smoke gases on vegetation. K. NOACK [with O. WEHNER and H. GRIESSMEYER] (Z. angew. Chem., 1929, 42, 123-126).-The effect of nitrous gases on vegetation as shown by the change in the absorptive power for carbon dioxide is similar to that of sulphur dioxide (cf. B., 1926, 458); in the concentrations found in smoke gases hydrochloric acid is less harmful and ammonia has scarcely any action. A certain minimum concentration of the noxious gas is required to diminish the absorptive power for carbon dioxide, but for concentrations below the minimum there is at first an increase in the amount of carbon dioxide absorbed, which after longer exposure of the plant to light falls again to the normal. The affected plant shows an increased ratio of soluble to insoluble iron compounds; by soaking in a very dilute solution of an iron salt, e.g., ferrous ammonium citrate, its absorptive power is considerably increased, but in no case is it restored to normal. The phenomenon appears to depend on a change in the condition of the iron of the chloroplasts, with a consequent reduction in its catalytic activity. F. R. ENNOS.

Comparison of the Robinson, International, and Bouyoucos methods of mechanical analysis of non-organic soils, and the analysis of such soil

with and without preliminary treatment with hydrogen peroxide. J. H. DENNETT (Malayan Agric. J., 1928, 16, 374-377).-The International and Robinson's methods are interchangeable and values are obtainable by interpolation of the summation curves of the latter. Clay determinations after 24 hrs.' sedimentation with a sampling depth of 30 cm. by the International method are as accurate as and more convenient than the usual sampling after 8 hrs. at a depth of 10 cm. Hydrogen peroxide treatment is unnecessary in non-organic soils. Bouyoucos' hydrometer method is of value in determining general limiting values of soils for routine purposes, but the total sand is preferably determined by direct sedimentation rather than by difference. A. G. POLLARD.

Colorimetric determination of phosphorus in acid soil extracts. W. N. C. BELGRAVE (Malayan Agric. J., 1928, 16, 361-371).-Atkins' modification (B., 1924, 483) of Denigès' method fails in acid soils where the proportion of iron to phosphate is great (cf. Greenstreet, B., 1928, 682). Reduction of the ferric salts followed by adjustment of the  $p_{\rm H}$  of the solution leads to a satisfactory determination. The extract from 1 g. of soil, from which silica and nitric acid have been substantially removed by gentle ignition, is dissolved in concentrated hydrochloric acid and heated with 2-3 g. of granulated zinc. When reduction is complete (spot test with thiocyanate), but while hydrogen is still freely evolved, the liquid is filtered through cotton-wool and made up to 100 c.c. To 40 c.c. are added 3 drops of thymol-blue solution and 5M-ammonium acetate solution until the pink colour begins to fade. 1 c.c. of Atkins' reagent-A and 3 drops of reagent-B are added and the original method is proceeded with. An equal amount of thymol-blue is added to the standard solution to maintain similar tints. 0.1 mg. of phosphorus pentoxide may be determined in 50 c.c. of solution in the presence of 1 mg. of silica without decrease in the colour A. G. POLLARD. intensity.

Improved method of fusion for soils. J. H. DENNETT (Malayan Agric. J., 1928, 16, 372—373).—In certain soils silica and sesquioxides fail to respond to fusion with "fusion mixture." A quantity of potash is prepared by fusion in a nickel crucible till all frothing ceases. A portion is removed by means of a nickel ladle holding 5 c.c. and placed in a small nickel crucible with 0.5 g. of soil. A homogeneous fusion is obtained by heating for 15 min. in a blowpipe flame. The water extract of the melt contains little or no nickel, and is satisfactory for the determination of iron, aluminium, and silica. A. G. POLLARD.

Examination of agricultural lime. S. REYNAERT (Natuurwetensch. Tijds., 1929, 11, 25—27).—The importance of composition in connexion with price is indicated. S. I. LEVY.

Determination of total nitrogen in urea-nitrate mixtures. R. LUCAS and W. HIRSCHBERGER (Z. angew. Chem., 1929, 42, 99—100).—About 10 g. of material are quickly weighed and dissolved in 1 litre of water. To 50 c.c. of solution are added 5 g. of powdered iron and 50 c.c. of 1:3 sulphuric acid, a funnel leng placed in the neck of the flask. When reaction has ceased, the flask is heated until the contents become turbid through separation of ferrous sulphate. After cooling, 15 c.c. of concentrated sulphuric acid are added and the concentration is continued until white fumes are freely evolved. The contents are transferred to a distillation flask, decomposed with excess of sodium hydroxide, and ammonia is absorbed in 40 c.c. of 0.5N-sulphuric acid. The back-titration is effected with 0.5N-sodium hydroxide, using nitrophenol as indicator. Adherence to the quantities indicated is essential. J. S. CARTER.

Apparatus for incinerating plants for microscopical examination of the ash. O. WERNER (Mikrochem., 1929, 7, 110—115).—The material to be ashed is placed between two perforated aluminium discs which fit into a thin cylindrical framework so as to leave a space between the discs. The framework is carried on an asbestoscovered iron gauze stretched on a rectangular framework over Teclu burners. The ash is mounted for microscopical examination in fused phenol between two cover glasses joined around their circumferences with paraffin wax. For permanent mounting Canada balsam is used in place of phenol. A. R. POWELL.

Soap-nicotine preparations. McDonnell and GRAHAM.—See XII.

#### PATENTS.

Manufacture of a [mixed] fertiliser. I. G. FARB-ENIND. A.-G. (B.P. 294,654, 26.7.28. Ger., 29.7.27).— Ammonium nitrate is caused to react with a mixture of sodium and potassium chlorides in aqueous solution, the mixture of salts obtained being separated from the liquor. W. G. CAREY.

Material for the destruction of plant pests. TEKNISKA FABR. JOFUR, N. I. BRUZELIUS, Assees. of E. G. A. WIKSTRÖM (Swed. P. 59,458, 2.3.22).—The material comprises a mixture of tall oil with liquids capable of dissolving fats and waxes, *e.g.*, trichloroethylene, tetrachloroethane, benzol, etc., preferably with the addition of alkaloids, *e.g.*, nicotine.

#### L. A. Coles.

Utilisation of palm leaves (Austral. P. 1298).— See V. Superphosphate (F.P. 626,190).—See VII. Sewage sludge (B.P. 305,346). Insecticides and fungicides (B.P. 303,932).—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

Reaction between amino-acids and dextrose. J. A. AMBLER (Ind. Eng. Chem., 1929, 21, 47-50).— This reaction was studied under varying conditions of concentration and heating. If the volatile products, e.g., carbon dioxide and aldehydes, are removed during the reaction the loss of reducing sugar on boiling the solution is relatively high, amounting in certain cases to as much as 10 mols. per mol. of amino-acid; the reaction becomes the more complete the longer the heating, progressive dehydroxylation of the dextrose molecule taking place. When the volatile products are not removed the amount of dextrose destroyed is smaller, probably owing to side reactions of the aldehydes with

the amino-acids. Carbon dioxide is produced only with the more concentrated solutions, and then in equimolecular proportions with the aldehyde. The yield of melanoidin is also the greater in the more concentrated solutions. These highly coloured substances may be a contributing factor to the discoloration of syrup during the manufacture of sugar products, and to the formation of froth in the fermentation of syrups etc. owing to their effect on the surface tension of liquids. F. R. ENNOS.

Comparative precipitation method for the qualitative identification of each of the common gums. W. WEINBERGER and M. B. JACOBS (J. Amer. Pharm. Assoc., 1929, 18, 34—36).—A method is established for the differentiation of the following gums : acacia, tragacanth, Irish moss, agar-agar, Indian gum, and quince seed, by the characteristic precipitate which is formed when each gum is precipitated from aqueous solution by alcohol. The texture, appearance, adherent quality, and manner of settling are different for each gum and are tabulated. E. H. SHARPLES.

# PATENTS.

Better utilisation of the lime in the precipitation of calcium saccharate. C. STEFFEN, SEN., and C. STEFFEN, JUN. (G.P. 452,728, 20.7.24).—Cooling of the sugar solution is continued until lime begins to settle as a deposit, and then with continued cooling the liquid is kept in circulation until the greater part of the lime has settled. The deposited lime sludge is introduced into a cold sugar solution and fresh lime is then added. W. J. BOYD.

Purification of crystalloid [sugar] solutions. K. KOMERS and K. CURER (B.P. 283,564, 14.10.27. Austr., 14.1.27).—The active adsorption surfaces of vegetable cell conglomerates, such as beet slices or comminuted sugar cane, which have been chemically treated after lixiviation, are further increased by the addition of finely-divided substances such as active carbon, cellulose, metal powder, metallic oxides or salts, and/or by the formation of finely-divided precipitates in the suspension of such conglomerates, e.g., by addition of iron sulphate to an alkaline conglomerate suspension. W. J. BOYD.

Manufacture of starch. CORN PRODUCTS REFINING Co. (B.P. 286,289, 16.1.28. U.S., 3.3.27).—An improved cyclic system of starch manufacture is described in which the water is used repeatedly and at the same time the building up of the sulphur dioxide content of the wet starch to an undesirable extent is avoided.

W. J. BOYD.

Composition of gum chicle, rubber, and an aqueous colloid. A. BIDDLE, ASST. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,691,460, 13.11.28. Appl., 22.5.26).—Adhesives are obtained, *e.g.*, by dissolving gum chicle in linseed oil or other organic solvent, and adding casein or other hydrophilic colloid and dispersed rubber or rubber latex. R. BRIGHTMAN.

Crushing machines (B.P. 302,135).—See I. Spirit and yeast from molasses (B.P. 283,969).—See XVIII. Separation of mixed substances (B.P. 272,968).— See XX.

#### XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. IV. Influence of hydrogen-ion concentration in fermentation. II. G. HAGUES (J. Inst. Brew., 1929, 35, 51-60; cf. B., 1928, 620) .- The mechanism of cell reproduction of Saccharomyces cerevisiæ and S. ellipsoideus is almost identical and depends on the activities of two groups of proteolytic enzymes or of two single enzymes the optimum activities of which occur at  $p_{\rm H}$  4.0 and 8.5, respectively. With S. Pastorianus I and S. Pastorianus III (S. validus), a third enzyme, which may be a phosphatase, with an optimum activity at  $p_{\rm H}$ 6.5 appears to be present in addition to the same groups which stimulate the reproduction of the previous two yeasts. The reproductive capacity of a white torula is probably due to the activity of only one enzyme the optimum of which is at  $p_{\rm H} 5.0$ . When S. Pastorianus I and S. cerevisiæ are grown in symbiosis, the presence of the one species is deleterious to the reproductive power of the other. C. RANKEN.

First chemical phases of hexose decomposition in alcoholic fermentation. A. J. KLUYVER and H. P. STRUYK (Woch. Brau., 1929, 46, 51-55; cf. A., 1928, 398) .- Figures are put forward showing that a high maximum rate of evolution of carbon dioxide during the phosphorylation period and a low ratio of hexosemonophosphate to -diphosphate are brought about in cell-free fermentations by increasing the concentration of the yeast macerate. According to the theories of Harden and Henley and of Meyerhof, the molecular ratio of the increase of carbon dioxide, resulting from the addition of phosphate, to the hexosediphosphate formed should be 2:1. The authors obtain in some cases much greater values (up to 11.4) for this ratio, which is only approached as a minimum with high concentrations of yeast macerate. They consider these observations to support their views that Robison's hexosemonophosphate results from the stabilisation of an active hexosemonophosphate, and that hexosediphosphate is formed by the combination of 2 mols. of glyceraldehyde monophosphate. F. E. DAY.

Manufacture of absolute alcohol by the spiritbenzol distillation under pressure. O. von KEUSSLER (Z. Spiritusind., 1929, 52, 36-38).-Young's method of the manufacture of absolute alcohol by distilling spirits with benzol or similar substances which form a mixture with minimum b.p. has been extended and improved by Merck, who carries out the distillation under a pressure of 10 atm. When the pressure is increased from 0 to 10 atm., corresponding to a rise in temperature of 65-144°, the amount of water removed from the alcohol by the benzol, after allowing for the intersolubilities of the liquids, is 2.6 times greater. In practice, benzine (b.p. 90-110°) is admixed with the benzol, which then acts as a solvent between the aqueous spirit and the benzine. If the benzine is added in correct amount it prevents the formation of layers and gives a more homogeneous mixture. The danger of separation is greater at lower temperatures, and at 65° the benzol can contain only 20-40% of benzene, whereas at 144° the content may safely amount to

60-65%. The high benzine content of the added liquid gives improved dehydration, facilitating the rapid separation of the distillate into two layers and reducing to a minimum the addition of water to the distillate, which is so necessary when no pressure is used. Since the heat of vaporisation decreases with the rise of temperature and pressure, about 20% less heat is required for every kg. of vaporised liquid when the pressure is raised to 10 atm. The high pressure does not diminish the temperature difference between the top and bottom of the high-pressure column, the difference being 5° at 10 atm. and 4° with no pressure. Under actual working conditions, the yield is increased  $2-2\frac{1}{2}$  times at the high pressure, although theoretically it should be 10 times greater. C. RANKEN.

Neutralisation of brewing liquor. D. MCCAND-LISH and G. HAGUES (J. Inst. Brew., 1929, 35, 61-66).-The brewing liquor is neutralised by sulphuric acid, thorough mixing being ensured by dripping the acid into the water as the latter passes into the mash-liquor tank. Owing to the danger of tank corrosion, if the  $p_{\rm H}$ value of the water is reduced below 7.0 the rate of admixture is so regulated that the  $p_{\rm H}$  of the treated water is approximately maintained at that value. The low  $p_{\rm H}$  value of the brewing water favours the production of a correspondingly low  $p_{\rm H}$  of the final beer, which gives the beer a bacterial stability provided infection is kept at a minimum. In addition, there is rendered possible a low initial value of the  $p_{\rm H}$  of the wort in which the yeast is grown, which secures a better reproduction and aggregation of the yeast and gives rise to a clearer racking C. RANKEN. beer.

Wine refining. M. RÜDIGER and E. MAYR (Kolloid-Z., 1929, 47, 141-155).-The effectiveness of the refining process is a property not of the kind of gelatin employed, but of the nature of the turbidity of the wine. From an analysis of the wine no conclusions can be reached as to the nature of the refining. The following conditions have been found most suitable for the process of refining: (a) the swelling-water should be used for dissolving the gelatin, especially with those poor in glutin; (b) the concentration of gelatin should depend on its glutin content, *i.e.*, not above 0.25% for gelatin rich in glutin, about 1% for medium glutin content, whilst concentrated solutions may be used with gelatin poor in glutin; (c) better results are obtained by allowing the gelatin to age for 1 to 2 days before use; (d) within limits, rise of temperature favours the process; (e) the most favourable method of mixing is either a continuous addition of the gelatin solution to the wine in proportional amounts, or a rapid admixture of the entire volumes. The tannin content of wine decreases with the amount of gelatin added. A part of the refining solution remains in the wine, the nitrogen content of which increases when large amounts of gelatin are used. The viscosity of the wine is less and the surface tension greater with increasing amounts of gelatin addition. The acidity of the gelatin solution and of the wine are important factors, increasing acidity favouring the process within limits. Experiments on the charge of the colloidal particles in turbid wine during the course

of refining indicate that the process is essentially one of electrical adsorption. E. S. HEDGES.

Comparator for determination of  $p_{\rm H}$  of coloured solutions. McCandlish and Hagues.—See I.

#### PATENTS.

Manufacture of yeast. K. A. JACOBSEN (B.P. 291,770, 8.6.28. Denm., 8.6.27).—The continuous supply of nutrient solution to the fermentation vat is periodically varied in such a manner that an increased quantity is added in time to prevent the occurrence of the yeastripening periods. In this way the yeast is continuously kept in a budding condition and at a constant amount. The fermented wort containing the unripened yeast is led into a separate container, in which the yeast ripens under aeration. C. RANKEN.

Manufacture of spirit and yeast by working-up molasses. A./S. DANSK GAERINGSIND. (B.P. 283,969, 20.1.28. Austr., 21.1.27).—Difficulties in the purification of the waste waters are obviated by avoiding during the process, which follows known methods, the use of primary and auxiliary materials containing inorganic sulphur compounds and particularly  $SO_4$  ions. Thus, hydrochloric acid replaces sulphuric acid for adjusting the  $p_{\rm H}$  value of the mash, and yeast nutrients are added in the form of the chloride, carbonate, and phosphate of ammonium in place of the sulphate. C. RANKEN.

Material for denaturing alcohol. H. M. CHABROL (F.P. 626,495, 1.4.26).—Shale oil, b.p. 75—86°, and phenolphthalein are used. L. A. Coles.

Production of glycerin. K. and N. LÜDECKE (U.S.P. 1,698,800, 15.1.29. Appl., 15.7.26. Ger., 21.6.26).—See B.P. 278,086; B., 1927, 921.

Decolorising tartaric acid solutions (F.P. 622,649). —See VII.

#### XIX.-FOODS.

Peptisation of wheat flour proteins by inorganic salt solutions. R. A. GORTNER, W. F. HOFFMAN, and W. B. SINCLAIR (Cereal Chem., 1929, 6, 1—17).—The amount of protein extracted from any given wheat flour by various salt solutions of equivalent ionic concentration is markedly different, whilst the peptisation of a variety of flours by a single salt solution is equally variable. The salt-soluble protein does not represent a mixture of albumin and globulin, nor yet the nongluten proteins, some salts extracting only a part of the non-gluten proteins. These differences appear to be determined by the ease of peptisation of the protein of the particular flour, and the specific properties of the anions and cations of the salt solution used.

# F. R. ENNOS.

Relation of hydrogen-ion concentration and buffer value to the baking quality of flour. I. E. A. FISHER and P. HALTON (Cereal Chem., 1929, 6, 18–33). —The baking quality of two flours as revealed by the gas production and volume of the loaf produced was practically unaffected by the addition of sufficient tartaric acid to diminish the  $p_{\rm H}$  value by 0.7-0.8; in one case the fermentation was slightly hastened. A more marked effect was shown on the dough toughness and loaf flavour, but this was only pronounced with large additions of acid. It is concluded that hydrogen-ion concentration is a factor of little importance in bread making, and contributes little or nothing to loaf quality. F. R. ENNOS.

Effect of dry skim milk on the fermentation and hydrogen-ion concentration of doughs. J. L. ST. JOHN and C. H. BAILEY (Cereal Chem., 1929, 6, 51—59).—Addition of dry skim milk to the baking mixture increases the total production of carbon dioxide and of the carbon dioxide lost from the dough, whilst the rate of increase in the volume of the dough is practically unaffected. The buffer action of dry skim milk is appreciable, the acidity of the doughs containing this ingredient being lower at all stages of the fermentation. F. R. ENNOS.

Relation of quantity of sodium sulphate to time of digestion in protein determination. C. G. HARREL and J. H. LANNING (Cereal Chem., 1929, 6, 72—78).— The weight of sodium sulphate used in the Gunning method is a vital factor in protein determination owing to its effect on the b.p. of the digestion mixture. For a given source of heat, the time required for complete digestion can be shortened by increasing the ratio of sodium sulphate to acid and vice versa. A larger quantity of the salt is necessary with copper as catalyst than with mercury. F. R. ENNOS.

Determination of thallium [in wheat] and its toxicity. C. STICH (Pharm. Ztg., 1929, 74, 231; cf. A., 1929, 164).—Wheat containing thallium is ignited with sulphuric acid, and the thallium is determined colorimetrically. A mouse fed with 5 grains (1 g.) of wheat containing  $1\cdot 8-1\cdot 9\%$  Tl (as sulphate) died in 12-15 hrs.; rats fed with 20 grains died in 2-3 days.

S. I. LEVY.

Flour colour tests. D. W. KENT-JONES and C. W. HERD (Cereal Chem., 1929, 6, 33-50).—A critical survey of the authors' method (cf. B., 1927, 762) in consequence of certain criticisms by Hooft and de Leeuw (cf. B., 1928, 909). F. R. ENNOS.

Method of measuring colour in bread. E. GREWE, W. K. MARSHALL, and C. G. HARREL (Cereal Chem., 1929, 6, 60-68).—An application of the Munsell method to the measurement of the colour of crust and crumb of bread. F. R. ENNOS.

Determination of chlorides in milk. G. RODILLON (Ann. Falsif., 1928, 21, 576—578).—Raquet and Kerleveo's criticisms (B., 1928, 208) of the author's method are refuted. D. G. HEWER.

Determination of chlorine and sodium in sheep's milk. L. BARTHE and DUFILHO (Ann. Falsif., 1928, 21, 578—579; cf. B., 1928, 385).—Analysis of a series of sheep's milks showed that the chlorine and sodium present per litre was: colostrum period 0.994, 0.191; 1 month 1.207, 0.212; 2 months 1.065, 0.193; and 4 months 1.136, 0.338 g., respectively. The chlorine is present in the same proportion as in cows' milk, and the sodium, varying round 200 mg. per litre at first, then

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increases to the amount in cows' milk. Sheep's milk, in respect of chlorine and sodium, approaches most nearly to cows' milk, and ruminants' milk appears to be distinct from other mammalian milks in the high proportion of sodium. D. G. HEWER.

Rapid methods for determination of bacteria in milk. A. J. J. VANDE VELDE (Natuurwetensch. Tijds., 1929, 11, 9—14).—The methods based on titration of the acid, measurement of the enzyme-reducing power, and direct counting, respectively, are described. The results of a large number of tests by the third method with different culture media are tabulated, and Skar's method is recommended as most rapid and accurate.

S. I. LEVY.

Sampling apples in the orchard for the determination of arsenical spray residue. J. W. BARNES (Ind. Eng. Chem., 1929, 21, 172—174).—Determinations were made of the residual arsenic in a large number of apples sprayed with lead arsenate, the results being expressed as grains of arsenic per apple, per pound of fruit, and per unit area of apple surface. A statistical study shows that in order to obtain a result with a probable error of 5% in the value for the mean arsenical residue per pound of fruit it is necessary to analyse a sample of about 50 apples picked at random.

F. R. ENNOS.

Nutritive value of extracted cattle foods. G. B. VAN KAMPEN (Chem. Weekblad, 1929, 26, 98—101).— The disease appearing in cattle fed on soya-bean cake obtained by extraction of the beans with trichloroethylene is found to be due to the removal by this solvent of part of the phosphatide content of the bean. The amount removed corresponds to the reduction in milk yield of cows fed with 4 kg, per day of the cake. The phosphorus-containing organic material removed by trichloroethylene, after a thorough extraction with light petroleum, is found to contain, besides some calcium and a little magnesium, considerable proportions of silica. S. I. Levy.

Auxiliary tables useful in the determination of nitrogen in cattle foods. G. JORGENSEN (Ann. Falsif., 1928, 21, 601-604).—For 1 g. sample of food the number of c.c. (ranging from  $5 \cdot 7$  to  $60 \cdot 5$ ) of  $0 \cdot 1N$ -acid used in the Kjeldahl titration are tabulated and arranged in consecutive horizontal lines. By direct reading from other horizontal or vertical columns corresponding values may be obtained from which the percentage of nitrogen and of protein in the sample may be deduced.

# D. G. HEWER.

Fatty acids and glycerides of New Zealand butters. HILDITCH and JONES. Coffee berry oil. VON NOËL.—See XII.

#### PATENTS.

Manufacture of a vegetable alimentary product. J. PERINO (B.P. 279,487, 20.10.27. Ger., 22.10.26).— Leguminous vegetable material is treated with a solution of alkali phosphates at a temperature below 60°, under reduced pressure, in the absence of free oxygen, and under ultra-violet irradiation. Insoluble alkali hydroxides are added to the mixture and the liquor is then expressed from the residue. Vitamin-containing herb- or leaflike or bulbous vegetables are treated with a solution of neutral alkali monohydrogen phosphates under the same conditions, and the resulting liquor is expressed from the residue. The two liquors may be desiccated together or separately. In the treatment of the second class of vegetables ultra-violet irradiation may be omitted. W. J. BOYD.

Vegetable waste products (G.P. 452,810).—See II. Wrapping of perishable goods (B.P. 304,168 and 304,341).—See V.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Recognition and differentiation of acetone, methyl alcohol, and isopropyl alcohol present together in pharmaceutical preparations. W. MEYER (Pharm. Ztg., 1929, 74, 230—231).—The liquid is subjected to a careful fractional distillation; methyl alcohol and acetone are concentrated in the first fraction, and isopropyl alcohol is found in the second. The latter is divided into two halves, one of which is tested directly, the other, after chromic acid treatment, for acetone; the two results are compared to decide if isopropyl alcohol is present. The second fraction contains methyl alcohol, detected by the usual tests for formaldehyde after oxidation, only if considerable proportions are present in the sample. S. I. LEVY.

**p-Dimethylamidobenzaldehyde as a reagent for** organic medicinal substances. H. W. VAN URK (Pharm. Weekblad, 1929, 66, 101–108).—A table is given of the colour reactions with the principal medicinal substances. Amino-compounds give yellow to orange colorations, hydroxyl compounds yellow, blue, green, and violet, the colour usually deepening with the number of hydroxyl groups. Pyrrole and indole compounds give colorations. The carboxyl group generally inhibits. Several cases are described in detail. A second table gives results obtained with other aldehydes.

S. I. LEVY.

Examination of therapeutic preparations. K. NEUMAYER (Arch. Pharm., 1929, 267, 27-30).—A lecture dealing mainly with the microscopical examination of hormone preparations etc. S. COFFEY.

Ammonium acetylsalicylate. "Ammonaspirin." N. E. WOLDMAN (J. Amer. Pharm. Assoc., 1929, 18, 14—17).—The preparation of salicylic acid, and of acetylsalicylic acid and its ammonium salt is described. Ammonium acetylsalicylate, difficultly soluble prismatic needles, decomp. above 70°, has the same physiological action as a mixture of aspirin and free ammonia in the same proportions.

E. H. SHARPLES.

Determination of potassium iodide in tincture of iodine according to D.A.B. VI. J. WEICHHERZ (Arch. Pharm., 1929, 267, 36–42).—The permanganate method described in D.A.B. VI. affords inaccurate results on account of the formation of manganese dioxide and iodic acid. Satisfactory results are obtained by the addition of small amounts of manganese sulphate and several c.c. of carbon tetrachloride, the latter serving the double purpose of preventing to a large extent the formation of iodic acid and also loss of iodine in the stream of carbon dioxide resulting from the oxidation of oxalic acid. S. COFFEY.

Determination of tannins in drugs. O. LINDE and H. TEUFER (Pharm. Zentr., 1929, 70, 21-26, 53-60).--The determinations were made by the following methods : (a) precipitation by means of stannous chloride, the washed and dried precipitate being weighed before and after ignition; (b) precipitation by means of copper acetate; (c) extraction by means of hide powder, the difference in weight of organic content of the aqueous solution before and after extraction being observed, and an allowance made for the organic matter yielded to the solution by the powder; and (d) permanganate titration before and after extraction of the tannin from the aqueous solution by means of hide powder. The precautions adopted in each case are described. The values obtained show poor agreement, those obtained by method (c) being highest, and by method (d) generally lowest. Method (c) is regarded as most accurate, and best suited for ordinary laboratory examination. Where alkaloids are also present it is necessary to decompose the alkaloid tannates by means of alkali.

S. I. LEVY.

Odour and constitution among the mustard oils [thiocarbimides]. VI. Natural mustard oils. G. M. Dyson (Perf. Ess. Oil Rec., 1929, 20, 42-44).— The synthesis and properties of allylthiocarbimide and of some other naturally occurring thiocarbimides are described. Specifications of 8 national pharmacopœias for mustard oils are tabulated (cf. B., 1929, 188).

E. H. SHARPLES.

Determination of essential oils by bromometric and thiocyanogen methods. I. H. P. KAUFMANN (Arch. Pharm., 1929, 267, 1-27).—The following iodine values of a number of essential oils, which furnish unsatisfactory results with the Hübl and Wijs methods, have been determined bromometrically by using a solution of bromine in methyl alcohol saturated with sodium bromide: geraniol (329·3), linalöol (329·3), terpineol (164·6), anethole (171·3), thymol (338·1), citral (336·6), vanillin (166·9), cinnamaldehyde (192·1), carvone (338.1), geranyl acetate (258.7), linalyl acetate (258.7), whilst the thiocyanogen method afforded the following values: citronellol  $(162 \cdot 5)$ , geraniol  $(164 \cdot 6)$ , linalöol  $(164 \cdot 6)$ , terpineol  $(164 \cdot 6)$ , anethole  $(171 \cdot 3)$ , thymol (169), citral (166.8), citronellal (164.6), geranyl acetate (129.4), linalyl acetate (129.4). The bromometric method cannot be applied to citronellol, cinnamyl alcohol, citronellal, bromostyrene, or coumarin, and the thiocyanogen method fails with geranyl formate and safrole, although in some of these cases normal values may be obtained by carefully adjusting the time and conditions of the reaction. Thiocyanogen does not combine with cinnamaldehyde, coumarin, or vanillin. S. COFFEY.

## PATENTS.

New antirachitic preparations and their manufacture. W., K., L., W., and F. MERCK (E. MERCK)

(B.P. 285,083, 9.2.28. Ger., 12.2.27).—The unsaponifiable constituents of yeast fat or of the corresponding extracts from ergot etc. are esterified and irradiated with ultra-violet light, or irradiated and then esterified. Water-soluble products are obtained when esterification is accompanied by the introduction of residues carrying salt-forming groups. B. FULLMAN.

Separation of mixed substances. CHEM. FABR. U. SERUMINST. "BRAM." G.M.B.H. (B.P. 272,968, 21.6.27. Ger., 21.6.26).—Separation is carried out at an optimum  $p_{\rm H}$  predetermined for each particular mixture by experiment. For example, the  $\psi$ -globulins containing diphtheria antitoxins are freed from the euglobulins which cause anaphylaxia and serum disease by precipitation at  $p_{\rm H}$  6·4; this can be produced by removal of ammonium sulphate etc. by electro-osmosis. Purification of sugar solutions and glycerol lyes, and precipitation of caoutchouc from latex are other examples mentioned. [Stat. ref.] C. HOLLINS.

Manufacture of alkylaminoalkylamino-derivatives of aromatic compounds. I. G. FARBENIND. A.-G. (B.P. 274,058 and 302,984-5, 23.6.27. Ger., [A] 8.7.26. Addns. to B.P. 267,169; B., 1927, 379).-Aminophenols or aromatic diamines in which the nitrogen or one nitrogen atom carries an alkyl substituent containing an alkylated amino-group, such, for example, as the intermediates required for the production of the polyaminoquinolines etc. of B.P. 267,169, are valuable against blood-parasites. The compounds may contain an N-alkyl side-chain (A) united directly or (B) through nitrogen, oxygen, or sulphur atoms to the alkylated amino-group, or (c) may contain two or more alkylated aminoalkylamino-groups. The following are described : (A) N-β-diethylaminoethyl-m-aminophenol, b.p. 171°/1.5 mm.; its N-methyl derivative, b.p. 151°/0.5 mm.; N-δ-diethylamino-β-amyl-m-aminophenol,b.p. 171°/1 mm.; N-methyl-N-B-diethylaminoethyl-p-aminophenol, b.p. 164°/ 3 mm.; N-ethyl-N-γ-dimethylamino-β-butyl-m-aminophenol, b.p. 175°/2 mm.; N-ethyl-N-B-diethylaminoethyl-2-amino-p-cresol, b.p. 176°/2 mm.; p-amino-N-methyl-Nβ-diethylaminoethylaniline, b.p. 161-163°/3 mm. (by reduction of the *p*-nitroso-compound); (B) compounds of the type  $NHR \cdot C_6H_3(OMe) \cdot OPr^8(1:3:4)$  in which R is β-(β'-diethylaminoethylthiol)ethyl (b.p. 225-227°/5 mm.), B-(B'-diethylaminoethoxy)ethyl (b.p. 186-188°/1.5 mm.), and β-(ethyl-β'-diethylaminoethylamino)ethyl, i.e.,  $CH_2 \cdot CH_2 \cdot NEt \cdot CH_2 \cdot CH_2 \cdot NEt_2 (m.p. 68-69^\circ, b.p. 145^\circ/9$ mm.); (c) N-methyl-NN'-di-(β-diethylaminoethyl)-pphenylenediamine, b.p. 180°/1 mm.; 3:4-diethoxy-NN-di-(β-diethylaminoethyl)aniline, b.p. 203-204°/1·5 mm.; 3:4dimethoxy-NN'-di-(B-di-ethylaminoethyl) - 0 - phenylenediamine, b.p. 203°/2 mm.; 2:6-diisopropoxy-NN-di-(β-diethylaminoethyl)aniline, b.p. 188-190°/2.5 mm.; 3-dimethylamino - 6 - (bis-B-diethylaminoethyl)aminothioanisole, SMe C<sub>6</sub>H<sub>3</sub>(NMe<sub>2</sub>) · N(CH<sub>2</sub> · CH<sub>2</sub> · NEt<sub>2</sub>)<sub>2</sub>, b.p. 198-204°/2 The following new intermediates are also mm. described : (A)  $\gamma$ -dimethylamino- $\beta$ -chlorobutane ; (B) 3nitro-6-isopropoxyanisole, m.p. 54-55°, and the corresponding amine, m.p. 68-69°, b.p. 145°/9 mm.; N-ethyl-N-( $\beta$ -diethylaminoethyl)- $\beta$ -aminoethyl alcohol, NEt2 · CH2 · CH2 · NEt · CH2 · CH2 · OH, b.p. 111-113°/15

CL XXI .- PHOTOGRAPHIC MATERIALS. CL. XXII .- EXPLOSIVES; MATCHES.

mm. (from  $\beta$ -diethylaminodiethylamine and ethylene oxide), and the corresponding *chloride* (by action of thionyl chloride on the alcohol); (c) 2:6-*diisopropoxyaniline*, m.p. 63°, b.p. 123°/3 mm. (by reduction of the *nitro*-compound); 4-*amino*-3-*methylthioldimethylaniline*, b.p. 135°/3 mm. (by methylation of the thiophenol). C. HOLLINS.

Manufacture of new derivatives of aromatic amino-[hydr]oxy- and polyamino-compounds. I. G. FARBENIND. A.-G. and A. CARPMAEL (B.P. 303,093, 23.6.27. Addn. to B.P. 267,169; B., 1927, 379; cf. B.P. 274,058, preceding).—Compounds valuable against blood parasites are obtained by introducing alkylated amino-groups into aromatic polyamino- or aminohydroxyderivatives, the alkylated amino-group forming part of an aliphatic side-chain or of a hydroaromatic or heterocyclic residue. The following compounds, derived from 1-chloro-2: 4-dinitrobenzene, are described: 2: 4-diamino-N-methyl-N-(B-diethylaminoethyl)aniline, b.p. 163°/ 3 mm. (hydrochloride, m.p. 205°); 3-methoxy-4-isopropoxy-N-(2'-dimethylaminocyclohexyl)aniline, b.p. 173-175°/ 2 mm.; 3-methoxy-4-isopropoxy-N-(3-hydroxy-y-1-piperidylpropyl)aniline, m.p. 92-94°, b.p. 225-230°/5 mm.

C. HOLLINS.

Solution of ammonium salt of hydroxyacetylaminophenylarsenic acid. E. FOURNEAU (U.S.P. 1,697,910, 8.1.29. Appl., 30.9.27. Fr., 19.1.26).—See B.P. 264,797; B., 1927, 573.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Dyes in photographic reductions and oxidations. A. STEIGMANN (Z. wiss. Phot. 1929, 26, 299—304).— An attempt is made to explain the effect of certain dyes on the sensitivity of a photographic plate by supposing that the dye activates the halogen first split off from the silver halide, and possibly also atmospheric oxygen, and that these then bleach the silver nuclei. Alternatively, the dye may be considered to take up hydrogen from the gelatin, and according as its affinity for this active hydrogen is greater or less than that of the silver halide, it will act as a desensitiser or sensitiser, respectively. The action of a sensitiser may also depend in part on the dye protecting the silver nuclei from dissolution by the acid fixing bath. R. CUTHILL,

Oiling of plates for ultra-violet photography. A. C. G. BEACH (Nature, 1929, 123, 166).—The plates are treated with a 0.5% solution of vaseline in light petroleum; they may be developed without previous cleaning. A. A. ELDRIDGE.

Determination of ripening silver. H. KIESER (Z. wiss. Phot., 1929, 26, 305—310).—Schmidt's experiments on the determination of ripening silver in a photographic emulsion (A., 1928, 1340) are criticised. The values found are almost certainly much too high, probably because the grains of the emulsion contain inclusions of silver nitrate, so that the ratio of silver to halogen seems abnormally large. Treatment of the plate with chromic acid before exposure will destroy the ripening nuclei on the surface of the grains, and as these would normally act as exposure nuclei, the sensitivity on chemical and primary physical development will appear to be reduced. From the fact, however, that on exposing and developing again the sensitivity proves to be normal, it must be concluded that ripening nuclei are present within the grains as well as on the surface. R. CUTHILL.

# Spontaneous form. STATENTS, protective layer

Manufacture of photographic films. J. CHAMBER-LAIN and H. PERIAM (B.P. 304,632, 20. and 26.10.27).— To improve the adherence of the emulsion to the celluloid film base the latter is coated with a substratum consisting of a concentrated solution of ethyl cellulose in benzol admixed with an aqueous solution of gelatin containing a small amount of sulphonated castor oil. If the film is required for colour photography, pigment particles or dyed transparent particles, usually of the three primary colours, may be dispersed in the substratum before coating. J. W. GLASSETT.

Method of colour photography. J. F. SEVENTAHL (U.S.P. 1,697,194, 1.1.29. Appl., 31.1.23).—Separated registered images are produced in a single layer of sensitive emulsion, and, after development and fixing, the colloid is treated with a substance which retards the action of the subsequently applied bleaching agent. J. W. GLASSETT.

**Production of colour cinematograph films.** A. and O. PILNY (B.P. 296,422, 11.2.28. Switz., 1.9.27). —A film negative is first prepared which contains two different colour record images either side by side or one above the other. For printing from this negative the two images are projected simultaneously, but are separated by an opaque screen placed perpendicular to the film at the dividing line of the images. By reflection from a series of right-angled prisms the projected images are made to register on opposite sides of a doubly-coated positive film, which is moved in the same direction and at the same speed as the negative film. After development each side of the positive film is dyed its respective colour. J. W. GLASSET.

Method and apparatus for developing exposed photographic surfaces [by atomisation]. A. E. WHITE. From AMER. PHOTOTURE Co. (B.P. 304,636, 22.7.27).

[Producing the effect of relief or depth in] photography. L. M. DIETERICH (B.P. 304,703, 25.7.27).

#### XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. Influence of the temperature of the gas surrounding the powder particles on velocity of combustion. H. MURAOUR (Z. physikal. Chem., 1928, 139, 163—168).— A recapitulation of work already published (B., 1928, 141, 770).

## PATENTS.

Explosive. A. C. SCOTT, Assr. to MEXCO, LTD. (U.S.P. 1,700,085, 22.1.29. Appl., 21.11.25. U.K., 29.11.24).—See B.P. 248,089; B., 1926, 422. : Coating [with wax the metal case of] ammunition cartridges. J. D. PEDERSEN (B.P. 304,948, 20.3.28).

Matches [in disc form]. F. KÄPPLER, and BRIT. BOOKLET MATCHES, LTD. (B.P. 304,883, 13.12.27).

# XXIII.-SANITATION; WATER PURIFICATION.

Spontaneous formation of a protective layer in iron pipes by cold water. I. Adsorption of carbon dioxide by ferric hydroxide. J. TILLMANS, P. HIRSCH, and K. SCHILLING (Gas- u. Wasserfach, 1929, 72, 49-53, 78-82).-Ferric hydroxide adsorbs carbon dioxide from water containing free carbon dioxide and sodium, ammonium, or calcium bicarbonate. The hydroxide may be precipitated in the solution or may be added to it, and is more active if precipitated in the cold than from a boiling solution. The quantity of carbon dioxide adsorbed increases with the quantities of iron, and of free and bicarbonate carbon dioxide present, and appears to be definitely related to the concentration of free and combined carbon dioxide. With water containing much carbon dioxide and very little bicarbonate, and vice versa, adsorption is practically absent. The production of a protective layer of rust and calcium carbonate in iron water-mains depends primarily on the presence in the water of oxygen, which leads to the formation of ferric hydroxide. This adsorbs bicarbonate ions from the water, liberating an equivalent quantity of hydroxyl ions which unite with the free carbon dioxide present to re-form the original quantity of bicarbonate ions. The net result is a disappearance of free carbon dioxide and, in waters in which this is not originally present in excess, the precipitation of calcium carbon-W. T. K. BRAUNHOLTZ. ate.

Determination of manganese in water. J. DE GRAAFF (Chem. Weekblad, 1929, 26, 103-104).-The sample is boiled with nitric acid, treated with a small excess of silver nitrate, and boiled with ammonium persulphate until clear; the permanganate formed is determined colorimetrically without filtering. If the sample is kept for some time before the determination, manganese compounds may settle out on the walls of the S. I. LEVY. containing vessel.

#### PATENTS.

Treatment of sewage sludge. G. H. HADFIELD (B.P. 305,346, 29.12.27).—The drying of sewage sludge for manurial purposes by natural means may be facilitated by thoroughly mixing partly dried press-cake with a proportion of fine, dry boiler ash and powdered quicklime or Portland cement. The mixing may be carried out in a pan with a perforated bottom, the final product being friable instead of sticky.

C. JEPSON.

Purification of sewage and analogous liquids. F. A. DALLYN (B.P. 305,717, 11.11.27).-A mixture of the flocs precipitated from compounds of iron and aluminium is agitated with sewage under strictly aerobic conditions; the iron floc absorbs molecular oxygen, and by releasing it in the atomic state oxidises the organic matter present while the aluminium floc coagulates and precipitates emulsified and unoxidised matter by being brought into intimate contact therewith. After

a short period of settlement, the purified effluent is discharged and the settled sludge, after re-aeration, is introduced into a fresh supply of sewage, and, by initiating the coalescence of the iron and aluminium compounds usually present, continues the process with a minimum of expense. The reaction is non-biological, and any undesirable increase in the oxygen demand, due to fermentation changes, may be controlled by partial sterilisation without deleteriously affecting the process. C. JEPSON.

Treatment of garbage. E. H. BRUNE (U.S.P. 1,700,608, 29.1.29. Appl., 25.9.26).-A mixture of 1 pt. of iron sulphate, 18 pts. of water, and 100 pts. of calcium oxide is added to garbage to deodorise it and increase its food value for animals. H. ROYAL-DAWSON.

Disinfecting agent. K. DAIMLER, F. JUST, G. BALLE, and S. FUCHS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,691,228, 13.11.28. Appl., 31.7.26. Ger., 3.8.25).-Aromatic sulphonic acids containing at least one side chain of three or more carbon atoms, e.g., sodium butylnaphthalenesulphonate, dibutylnaphthalenesulphonate, benzylbutylsulphonate, or pyridine cyclohexyltetrahydronaphthalenesulphonate, are used.

R. BRIGHTMAN.

Manufacture of disinfecting, bactericidal, insecticidal, fungicidal, and vermin-destroying preparations. I. G. FARBENIND. A.-G., W. BONRATH, and W. SCHEPSS (B.P. 303,932, 17.10.27. Addn. to B.P. 232,249; B., 1925, 940).-Instead of the arsenic or arsenious salts of basic dyes, mixtures of salts of the dyes with arsenic or arsenious acid or their salts are used. C. HOLLINS.

Automatic and continuous activation of water by means of radium emanation on an industrial scale. SPA-MONOPOLE, COMP. FERMIÈRE DES EAUX ET DES BAINS DE SPA (B.P. 298,915, 15.10.28. Belg., 15.10.27).-The emanation from a slightly acid aqueous solution of a radium salt is transported by means of an inert gas to a scrubber in which it is removed by a spray of water which by operating at varying rates becomes radioactivated to any desired degree up to saturation and may be disposed of as required. The gas leaving the scrubber is freed from carbon dioxide and again bubbled through the radium salt solution. All parts of the apparatus are of glass sealed together to ensure airtightness. The direction of flow in the circuit is maintained by the introduction of suitable mercury seals, and the circulation is induced by the rise and fall of a mercury piston actuated intermittently by a suction pump so as to prevent a too copious evaporation of the radium solution by a continuous stream of gas. An accumulation of explosive gas is prevented by the introduction of a heated tube containing copper oxide in the circuit at a point preceding the scrubber.

記述 Apparatus for charging furnaces for burning refuse or like material. WOODALL-DUCKHAM (1920), LTD., and F. A. HORNBY (B.P. 305,451, 5.5.28).

Gas masks, respirators, etc. J. A. SADD (B.P. 305,080, 29.9.27).

Protective gas-masks. E. DRÄGER (B.P. 289,810, 30.4.28. Ger., 2.5.27).

C. JEPSON.