

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 30, 1927.

I.—GENERAL; PLANT; MACHINERY.

Heat transmission by radiation from non-luminous gases. H. C. HOTTEL (Ind. Eng. Chem., 1927, 19, 888—894).—Coefficients of heat transmission based on the assumption of convection as the controlling mechanism cannot be satisfactory except for the apparatus in which they are determined, as radiation is neglected. When analysed the infra-red radiations from gases occur in bands, three each for carbon dioxide and water vapour. The calculation of heat quantities transmitted from gas mixtures containing these two gases is discussed and equations and curves are developed which enable them to be calculated with approximate accuracy from the gas and surface temperatures, gas partial pressures, shape factors, and the "black-body coefficient." The case of a flue etc. with change of temperature along the length is also considered.

C. IRWIN.

Critical analysis of equations for the design of fractionating columns. L. H. SHIRK and R. E. MONTONNA (Ind. Eng. Chem., 1927, 19, 907—911).—The distillation of a mixture of ethyl alcohol and water in a column of nine plates of the bubbling hood type was studied. The relation of the actual number of plates used to the theoretical number as calculated by various methods to give similar results, termed the "column efficiency," was calculated. This gives a criterion by which the methods of calculation can be checked. The graphical stepwise method of McCabe and Thirle was found most satisfactory. Methods assuming continuous rather than stepwise change were markedly erroneous for such a column. The composition of the alcohol-water mixtures was determined by the immersion refractometer. Dilute mixtures (below 0.5% of alcohol) were determined by concentration by distillation and comparison with a prepared dilute mixture.

C. IRWIN.

Suction of acid fumes from fume-cupboards. K. VESELY (Chem. Listy, 1927, 21, 359—360).—Suction is produced by forcing air through an injector into a broader tube, thereby causing a partial vacuum which is in connexion with the fume-chamber. Corrosion of ventilating mechanism is thus avoided.

R. TRUSZKOWSKI.

Industrial photo-colorimeter T.C.-B., independent of the eye. R. TOUSSAINT (Bull. Soc. d'Encour., 1927, 126, 421—429).—The colour to be measured is successively submitted to light passing through various coloured filters, and the intensity of the reflected light in each case is recorded by means of a photo-electric

circuit. The curve obtained by plotting intensity against wave-length is characteristic of the colour. The relative brilliance of colours may also be assessed by measuring their true reflection of white light, allowance being made for difference in colour of the reflecting surface.

S. S. WOOLF.

Rapid counter-current mixer. KALINOWSKY-STIER (Chem.-Ztg., 1927, 51, 630—631).—A description of the Erich mixer, the cylindrical body of which rotates in the opposite direction to an eccentrically placed vertical shaft carrying inclined and staggered mixing arms.

S. I. LEVY.

Sources of error in glass volumetric apparatus. H. V. E. M. RENN (J. Amer. Ceram. Soc., 1926, 9, 850—859).

PATENTS.

Apparatus for the quantitative analysis of substances with the aid of Röntgen or cathode rays. F. DESSAUER, Assee. of METALLBANK & METALLURGISCHE Ges. A.-G. (E.P. 252,207, 17.5.26. Conv., 15.5.25).—Apparatus for the quantitative analysis of substances by means of Röntgen or cathode rays dependent upon the observation of characteristic discontinuities in the ionisation-voltage curve in a chamber containing the substance, comprises an ionisation chamber in which the substance is arranged in the path of the rays, and a meter for registering the ionisation by reflecting a beam of light on to a drum coupled to a regulator for varying the voltage applied to the tube emitting the Röntgen or cathode rays.

J. S. G. THOMAS.

Separation of solid materials of different specific gravities. G. RAW (E.P. 274,578, 30.4.26 and 26.2.27).—An even bed of the material, in such a state of subdivision that it offers resistance to air, is disposed on a pervious table with upstanding sides, and a static condition of air pressure, greater beneath the table than above it, is maintained so as to produce a mechanically fluid state and to separate the materials by density action. Stratification is increased by synchronising sharply defined pulsations of air and vibration of the table. The table may be inclined and scrapers may be provided to assist the discharge of material of lesser density.

W. G. CAREY.

Roller mills for grinding and mixing. G. DÖRSTLING (E.P. 274,784, 18.5.27).—Uniform pressure is applied to all the rollers of a roller mill by a single weighted lever system operated by a hand lever which acts directly on one of the rollers, the movement being transmitted to the other rollers by carriers. The rollers can be separated from one another to a variable extent

when mixing rather than grinding chocolate, pigments, etc., such separation being shown on an index.

W. G. CAREY.

Grinding bodies for ball, drum, and tubular mills. O. KORDT (E.P. 274,786, 26.5.27. Addn. to E.P. 261,264).—Grinding bodies for mills consist of globular steel bodies having not more than five flattened surfaces to intensify the beating, rocking, and rolling motions, and preferably any two of these surfaces are parallel to each other.

W. G. CAREY.

Centrifugal apparatus for treating gases with liquids. L. and F. H. E. THEISEN (E. THEISEN) (E.P. 270,340, 30.4.27. Conv., 3.5.26. Addn. to E.P. 265,120).—The annular rotary plates are swaged or stamped to produce thereon annular series of protuberances each in the form of a truncated cone or provided with opposed walls which are segments of truncated cones.

H. HOLMES.

Drying agent for gases. I. G. FARBENIND. A.-G. (E.P. 272,173, 25.3.27. Conv., 7.6.26).—A solution of phosphorus pentoxide in 100% phosphoric acid is used for drying gases even when they are hot, and is employed, e.g., for drying air for the combustion of phosphorus or gas mixtures containing phosphorus in the manufacture of phosphorus pentoxide. Phosphoric acid is obtained as a by-product, by using the drying mixture until all the phosphorus pentoxide is used up.

W. G. CAREY.

Heat-exchange apparatus. R. L. MUNDAY (U.S.P. 1,639,051, 16.8.27. Appl., 21.12.26. Conv., 5.12.25).—See E.P. 255,711; B., 1926, 729.

Separation of the elements of air or of other gaseous mixtures by liquefaction and rectification. J. LE ROUGE, Assr. to Soc. L'AIR LIQUIDE (Soc. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE) (U.S.P. 1,638,005, 2.8.27. Appl., 21.7.22. Conv., 12.8.21).—See E.P. 184,454; B., 1922, 859 A.

Apparatus for heating, cooling, or drying materials. V. CANO (Re-issue 16,699, 9.8.27, of U.S.P. 1,592,078, 13.7.26).—See B., 1926, 808.

Furnaces (E.P. 257,261).—See X.

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

Siberian boghead coals. G. STADNIKOV (Brennstoff-Chem., 1927, 8, 244—245).—Three samples of Siberian boghead coals contained roughly 1.3—4.0% of water, 5.9—14.8% of ash, and 70.4—81.0% of volatile matter (on the dry coal), 0.7—3.1% of sulphur and 0.6—1.1% of nitrogen (on the dry and ash-free coal). Their gross calorific value was 7584—8325 kg.-cal. On low-temperature carbonisation they yielded 39.1—48.8% of tar, with semi-coke containing 12.8—39.2% of ash and 11.9—14.9% of volatile matter. Analyses of the low-temperature tars are mainly remarkable for the low content of phenols (1.3—4.0%) and bases (0.2—0.9%) in the distillate up to 350°.

W. T. K. BRAUNHOLTZ.

So-called "algæ" of boghead coal. HELLMERS and POTONIE (Z. angew. Chem., 1927, 40, 895—897).—The products obtained from boghead coal by maceration with Schulze's mixture, and named "algæ" by Bertrand

and others, show no membranous structure. Aggregates of similar nature can be prepared by suitably introducing molten paraffin into aqueous gelatin solution, and are due to balling together of small drops. The phenomenon could be explained by a coagulation of bituminous constituents of the coal.

A. COUSEN.

Oxidation of different coals at different temperatures. II. D. J. W. KREULEN (Brennstoff-Chem., 1927, 8, 241—244).—When a flaming gas coal, a gas coal, a fat coal, or a lean coal is heated for several hours in air at 125°, 150°, 175°, or 225°, the resulting oxidation causes an increase in the fixed moisture content and a parallel increase in the humic acid content, the effect being greatest for the flaming gas coal and least for the lean coal, and being greater with increased temperature. The coking properties are reduced, 1 hr.'s oxidation at 175° being sufficient completely to destroy them. Even at 100—105° the coking properties are impaired, but heating at 175° in an atmosphere of carbon dioxide does not affect them. By prolonged heating of a flaming gas coal in air at 175°, and repeated extraction of the humic acid formed, 95% of the coal is converted into humic acid. W. T. K. BRAUNHOLTZ.

Effect of weathering on the softening and solidification points of coal. T. E. LAYNG and A. W. COFFMAN (Ind. Eng. Chem., 1927, 19, 924—925).—Nitrogen was passed through a heated mass of 20—60-mesh coal and the back pressure due to softening measured. Tests with coal naturally weathered or oxidised by heating at 110° for various lengths of time showed that weathering raises the temperature of initial plasticity, decreases the maximum pressure developed, and decreases the temperature of coke formation. The curves obtained may therefore be used to indicate the extent of weathering. Coal cannot be preheated above 150° in air without injuring its coking properties, but in some cases higher temperatures in nitrogen were not harmful.

C. IRWIN.

Influence of ash content on the calculated calorific value of ash-free coal. II. R. STUMPER (Brennstoff-Chem., 1927, 8, 261—262; cf. B., 1927, 130).—The calorific value of the ash-free coal in mixtures of coal and calcium sulphate, calculated from the calorific value found by the bomb method and from the actual content of non-combustible matter, is constant until the gypsum content exceeds 20%, above which it rapidly falls. Calcium sulphate loses combined water and also suffers dissociation at 800°. The ash content experimentally determined is therefore less than the actual content, and the calorific value of the ash-free coal, calculated from the experimental ash content, steadily decreases with increasing content of gypsum.

W. T. K. BRAUNHOLTZ.

Utilisation of marine-animal and fish oils in motors. G. LUMET and H. MARCELET (Compt. rend., 1927, 185, 418—420).—Marine-animal and fish oils have, in general, been found satisfactory as fuels in Diesel or semi-Diesel engines. The behaviour of the engine is more flexible than with gasoline, and the power developed is approximately the same. High viscosity at low temperatures presented the only difficulty.

C. W. GIBBY.

Determination of corrosive sulphur in motor benzol. R. KATTWINKEL (Brennstoff-Chem., 1927, 8, 259—260).—The corrosive ("active") sulphur is the difference between the total sulphur in the benzol before and after treatment with a metal such as copper or mercury. The total sulphur is determined by Schenk's method (cf. B., 1914, 130), a fresh sample of the benzol being then heated for 1 hr. under a reflux condenser with finely-divided copper bronze to remove the corrosive sulphur. An alternative method is to decompose the metallic sulphide with acid and determine the hydrogen sulphide liberated. W. T. K. BRAUNHOLTZ.

Illuminating gas from lignite by processes employing internal heating by inert gases. F. SEIDENSCHNUR (Gas- u. Wasserfach, 1927, 70, 728—736).—Low-temperature carbonising processes employing internal heating by gases have the advantages of more efficient and uniform heating and less decomposition of tar and semi-coke. The Limberg and Lurgi processes are not suitable for supplying illuminating gas from lignite, since the gas produced is used up for heating purposes in the processes themselves. A more promising method is carefully to dry screened or briquetted lignite by heating it in air of gradually diminishing humidity, to distil the dried product by internal gas heating at 420—440°, and finally to degasify the semi-coke at red heat. The gas produced at 440° (83 litres/kg. of dry lignite) has, after removal of benzene, a gross calorific value of 1660 kg.-cal./m.³, contains 73.5% CO₂, and may be utilised for drying the raw lignite. The corresponding semi-coke, containing 16.1% of ash and 23.4% of volatile matter, yields, when heated to redness, a gas (266 litres/kg.) containing 12.8% CO₂, and a porous and easily combustible coke with 4% of volatile matter and a nett calorific value of 6340 kg.-cal. The carbon dioxide-free gas contains 26.0% CO, 51.5% H₂, 20.8% CH₄, 0.8% N₂, and has d 0.418 and gross calorific value 4480 kg.-cal., thus resembling ordinary coal gas. The yield of low-temperature tar in the above process is 100—110% of that found by the Fischer assay, the loss due to decomposition being only 1.5%. By cracking, this tar could be converted into benzene and a gas of high calorific value. Large-scale tests of the above process have not yet given results equal to those above quoted, obtained in laboratory experiments.

W. T. K. BRAUNHOLTZ.

Low-temperature distillation of long-flame coals. A. LÉAUTÉ (Compt. rend., 1927, 185, 465—467).—The influence of the binding material upon the distillation of long-flame coal has been studied. Porosity of the residue can be avoided by using as a binding material anthracene oil, which has a minimum content of tar. The residue is then hard and compact, similar to anthracite, and can be used as such. A quantity of oil is obtained about equal to that of the binding material used, and 2—15% of gas. C. W. GIBBY.

Evaluation of water-gas oil. T. MITSUHASHI (J. Fuel Soc. Japan, 1927, 6, 2—3).—The oil is fed into the top of a vertical porcelain tube, 3 cm. in diam. and 60 cm. long, filled with broken firebrick and heated in an electric furnace. The apparatus is first filled with carbon dioxide, and again swept out with carbon dioxide

at the end of the experiment, the gases being passed through potassium hydroxide on their way to a gasometer. The yield and quality of the gas produced at any given temperature are determined.

W. T. K. BRAUNHOLTZ.

Eislingen shale oil. K. NEUBRONNER (Petroleum, 1927, 23, 789—796).—The shale was carbonised in a rotary cylindrical retort, yielding 6—6.4% of crude oil and 2.9%, or 35 litres, of gas per kg. The net calorific value of the shale was 1380. The crude oil had d 0.900—0.920, open flash pt. 20°, wax content 0.45—0.50%, sulphur content 2.3—3.5%, and contained about 0.75% of aromatic hydrocarbons. The distillation test gave 30—34% at 200° and 63—71% at 300°. When the crude oil was distilled in an atmosphere of nitrogen, and the fraction up to 150° repeatedly redistilled, 10.6% of the crude oil distilled over up to 125°, and 6.4% from 125° to 150°. The fractions up to 74° remained colourless, whilst those up to 125°, though colourless at first, became dark and gave a reddish-violet deposit on keeping. The benzene fraction 60—180° had d 0.790—0.795, sulphur content 3.5—4.0%, and polymerised readily with deposition of asphaltic substances. In refining the 150° fraction the diolefines were removed by acid and soda treatment. Refining with about 5% of sulphuric acid and 1—1½% of 15% soda solution gave a spirit with passable smell. Better results were not obtained with sodium plumbite solution. The refined benzene had d 0.780—0.790, n_D^{20} 1.4500—1.4540, mercury acetate value 300—340, sulphur content 2.55—2.65%. A commercially feasible method of desulphurising the benzene was not found. H. MOORE.

"Pinking" in internal-combustion engines. G. B. MAXWELL (J. Inst. Petrol. Tech., 1927, 13, 224—243).—The researches and opinions of other workers on pinking, detonation, and flame phenomena are reviewed. The distinction between pre-ignition and pinking and the effect of various anti-knock and knock-inducing compounds are discussed, as also the effect of carbon disulphide on the lag period, and of the shape of the combustion chamber and engine details on pinking. H. MOORE.

Effect of metallic vapours on the ignition of substances. A. EGERTON and S. F. GATES (J. Inst. Petrol. Tech., 1927, 13, 244—255).—The temperature of ignition of petrol in a Moore apparatus was raised by 160° by the addition of 0.25% of lead tetraethyl, and by 145° when the vapour of that compound was admitted to the crucible. Lead oxide, produced as a fine dust by sparking between lead electrodes, only caused a slight rise in ignition temperature, but argon passed over a low-tension arc between lead terminals and directly introduced into the crucible caused a rise of 60°, or 70° with a similar arrangement using nitrogen. The rise for a given quantity of lead was of the same order as when lead tetraethyl was added to the petrol. A table is given of elements effective or ineffective when tested by this method. The effect of the metal was found to be similar to that of its organo-derivative. The metal itself in a state of incipient oxidation is the effective agent. Among the rises of temperature noted with a concentration of 0.1% of the substance tested are selenium

diethyl 140°, iron carbonyl 130°, bismuth triethyl 120°, lead ethyl 90°, nickel carbonyl 40°, tellurium diethyl 55°, and bismuth triphenyl 42°. The effects of lead tetraethyl on the ignition temperatures of different combustibles are as follows. Normal alcohols are little affected; aldehydes, very greatly raised; methylcyclohexane, more affected than cyclohexane; benzene, effect only slight; and isohexane, less affected than *n*-hexane. Anti-knock compounds diminish the rise in temperature by hindering oxidation of the fuel. H. MOORE.

Significance of igniting temperatures. A. EGERTON and S. F. GATES (J. Inst. Petrol. Tech., 1927, 13, 256—272).—The ignition point is defined as that at which the rate of gain of heat by a volume element of vapour balances the rate of loss of heat, and the temperatures given in ignition testers similar to that of Moore are termed "igniting points." The results of different investigators on igniting points are reviewed and shown to be tolerably concordant. The authors' experiments were conducted with an iron pot 1½ in. diam. and 2¼ in. high, heated air being introduced at the rate of 330 c.c. per min. and temperatures measured by a thermo-couple near the bottom of the pot, the surface of which was polished. The size of the drop did not greatly affect the igniting temperature, nor did the rate of air supply, so long as this was adequate. Oxidation of the surface of the pot caused a rise in igniting temperatures. Differences of material of the surface had considerable influence on the temperature, especially in the case of acetone. An increase of surface by the introduction of iron plates or glass wool into the pot gave higher igniting temperatures, flameless combustion occurring before ignition. The introduction of a halide lowered the temperature. The introduction of ions from radium bromide did not influence the temperature. Aldehydes ignite at lower temperatures than the corresponding alcohols and hydrocarbons. The igniting temperatures are related to the knocking tendencies of fuels in engines. H. MOORE.

Effect of certain organic compounds on the igniting and "knocking" characteristics of petrol. A. EGERTON and S. F. GATES (J. Inst. Petrol. Tech., 1927, 13, 273—280).—The effects of various purely organic compounds on the knocking tendencies of petrol were studied in connexion with their influence on the igniting temperature. *m*-Xylidine is the most effective anti-knock of the arylamines, but is 30 times less effective than lead tetraethyl. Aldehydes, acids, nitro-compounds, nitrates, etc. promote knocking; *m*-compounds are rather more effective than *o*- and *p*-compounds, and diethylamine is less effective than diphenylamine. Quinoline, pyridine, and carbylamine are ineffective in comparison with aniline. Ethyl iodide, benzoquinone, cresol, phenol, and diphenyl ether are effective, but less so than aniline. The comparative effects of iron carbonyl, lead ethyl, nickel carbonyl, aniline, *m*-xylidine, and toluene are respectively 600, 400, 160, 11, 12, and 1. Alcohol and benzene act as diluents, raising the H.U.C.R. according to the quantity added; 50% of carbon disulphide raises the H.U.C.R. 6%, in agreement with its influence on igniting temperatures. Ether destroys the influence of lead tetraethyl, in accordance with its influence on igniting temperature. Aniline raises the igniting tem-

perature of acetaldehyde by 110°. The addition of hydrocarbons of high mol. wt. depresses the H.U.C.R. disproportionately. No substance effective on the engine did not affect the igniting point, but the reverse does not always hold. The molecules of lead tetraethyl and aniline are effective on the igniting point in the proportion of 70:1. A table showing the effect on igniting point of various organic compounds is given. H. MOORE.

Theories of anti-knock action. A. EGERTON and S. F. GATES (J. Inst. Petrol. Tech., 1927, 13, 281—299).—Anti-knock compounds are negative catalysts which remove the substances which autocatalyse combustion, generally peroxides. Such metallic compounds form comparatively stable peroxides which destroy fuel peroxides and are then regenerated. The organic anti-knock compounds behave in a similar manner, but themselves undergo combustion. The anti-knock effect implies the slowing down of the initial stage of combustion, is chemically the same as the effect on igniting temperatures, and suppresses detonation, even when the fuel and the anti-knock compound are both in the gaseous state. The active constituent of an organo-metallic anti-knock compound is the metal atom, and this must be oxidisable. Rise of igniting temperature and anti-knock effect are attended by diminution of the rate of oxidation of the fuel. The effective organic compounds are amines, quinones, phenols, etc. with high b.p. and igniting temperatures. Anti-knock compounds affect the combustion of aldehydes, but hardly that of alcohols. The chemistry and physics of the action of the compounds are discussed. H. MOORE.

Anti-knock dopes. R. A. WEERMAN (J. Inst. Petrol. Tech., 1927, 13, 300—307).—The spontaneous ignition temperatures of a number of organic and organo-metallic compounds were tested in an apparatus consisting of an iron pot roughly polished inside, at the bottom of which air is passed at the rate of 330 c.c. per min., a drop (0.013 c.c.) of the liquid to be tested being allowed to fall through a hole in the cover of the pot while the temperature is rising at intervals of 10° and 5°. The effect of the addition of various small percentages of different compounds to a fuel was tested, and it was found that the more notable anti-knock compounds raised the spontaneous ignition temperature by an amount of the order of 105° to 170°. Compounds which raised this temperature had an anti-knock effect in the engine and *vice versa*, though not in exact proportion. An anti-knock compound must be volatile and capable of reduction during the compression stroke in the engine. Silicon tetraethyl lowers the spontaneous ignition temperature and increases knocking. On condensing the products of combustion from the apparatus it was found that the addition of 2 g. per litre of lead tetraethyl to petrol caused less water to be formed and a reduction of the acid value of the oil layer, the consumption of oxygen being also reduced. Importance is to be attached to the reactions which occur during the compression stroke in an engine. H. MOORE.

Fuller's earth and its uses in the petroleum industry. A. RAUCH (J. Inst. Petrol. Tech., 1927, 13, 325—330).—Fuller's earth has an important action in neutralising acid oils, thus saving the expenses of soda

washes and the risk of emulsion formation. The earth itself has generally an acid reaction, and acid earths have the better bleaching properties, though not in proportion to their activity. The deodorising action of fuller's earth in vapour-stage refining is attributed to the polymerisation and adsorption of unsaturated and sulphur compounds. On opening the filter presses in which fuller's earth has been separated from edible oils, fires sometimes occur owing to rapid oxidation in contact with air.

H. MOORE.

Action of sulphuric acid on unsaturated and aromatic hydrocarbons. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1927, 13, 311–320).—The assumption that shaking with 98% sulphuric acid completely removes aromatic and unsaturated hydrocarbons is erroneous, a large proportion of the olefines being separated as saturated hydrocarbons, probably open-chain paraffins. Alkyl hydrogen sulphates only are formed in small quantities from some unsaturated compounds, and a large proportion of aromatic sulphonic acids disappears when olefines are present. The barium carbonate method carried out in the cold gave very accurate results in determining aromatic compounds. In mixtures of a turpentine fraction and aromatic-free petrol and the same petrol with added aromatic compounds the loss to sulphuric acid was about equal to the turpentine plus aromatics, but when amylene was substituted for turpentine the results only corresponded to about one half of the unsaturated compounds. The conductivity of the final liquor was adopted as criterion of the sulphonic acids present, and gave very accurate figures for the aromatic compounds present in a mixture. In the absence of olefines aromatic compounds may be determined by the amount of sulphonic acids formed on shaking with 98% sulphuric acid, but in the presence of olefines aromatic compounds are not entirely removed, partly condensing with the hydrocarbons formed from the olefines. The sulphuric acid wash from amylene contains very little amyl hydrogen sulphate.

H. MOORE.

Carbon ratio. M. STUART (J. Inst. Petrol. Tech., 1927, 13, 308–310).—It is pointed out that the carbon ratio, often used as a guide to the character of oils found in the vicinity of coal, or as an indication of the degree of probability of finding petroleum oil in a given district, is often erroneously described as the carbon/hydrogen ratio, as if it were the ratio of these elements found by ultimate analysis. It is actually the percentage ratio of fixed carbon to the total of fixed carbon and volatile matter found by proximate analysis in a coal. This ratio in certain coals may be 50, whilst the same coals contain more than 90% of carbon. H. MOORE.

Economy of low-boiling distillate of fuel gasoline. Y. TANAKA (J. Fuel Soc. Japan, 1927, 6, 39–40).—The volatility of a gasoline poor in the lower-boiling fractions, e.g., one from which a low-boiling fraction has been removed for solvent or other uses, can be increased by the addition of alcohol, which forms minimum-boiling azeotropic mixtures with the gasoline hydrocarbons. From a study of such mixtures the following formulæ have been established: $W_H = (160 - T_H)/1.15$ for paraffins and naphthenes, $W_H = (140 - T_H)/0.89$ for

aromatic hydrocarbons, and $(T_H - T_{AZ}) (81 - T_{AZ}) = K$, where T_H is the b.p. of the hydrocarbon and W_H its percentage by weight in the azeotropic mixture, of which T_{AZ} is the b.p. K is about 236 for paraffins and naphthenes, and about 150 for aromatic hydrocarbons.

A. B. MANNING.

Preparation of benzene by polymerisation of acetylene. S. IKI and M. OGURA (J. Soc. Chem. Ind. Japan, 1927, 30, 461–469).—Acetylene was passed through a hard glass U-tube containing activated carbon of many kinds. There is little difference in catalytic activity between activated charcoal, wood charcoal, coke, and coalite. A special relationship is not found between the decolorising action and the catalytic action of the carbons. The coarse granular catalysts are suitable for the purpose; those in a fine state are apt to cause local heating in the reaction tube. The optimum temperature is 650–660°, and gas velocity 10 litres per hr.; the yield of the oil is then 60–70% of the acetylene used, and it contains 50–60% of light oil distilling below 150°, composed mainly of benzene. Under constant conditions the results of the experiments are almost similar for all catalysts. K. KASHIMA.

Crystallisation of paraffin wax. F. H. RHODES, C. W. MASON, and W. R. SUTTON (Ind. Eng. Chem., 1927, 19, 935–938).—Paraffin wax was fractionally melted in a thermostat, the mol. wt. of each fraction determined, and its behaviour on solidification studied microscopically. All the fractions behaved in a similar way. Small plates were first formed, but with fairly rapid cooling needles then developed. With very slow cooling plates only were formed. The two forms are therefore of identical composition, and are not even allotropic modifications, as they may co-exist at constant temperature. The needles, as is shown by examination under crossed Nicols, are really plates tightly rolled up. The plates may be hemimorphic, and the rolling caused by differences in surface tension. C. IRWIN.

Structure of the asphalt micelle. F. J. NELLENSTEYN (Chem. Weekblad, 1927, 24, 414–421).—The disperse phase consists of carbon particles which adsorb and are surrounded by heavy protective hydrocarbons and their dissociation products, forming micelles in the medium. Asphalt shows analogies with such highly-protected organosols as sodium chloride in benzene.

S. I. LEVY.

Production of oil from bituminous coal. Work of the Fuel Research Station. C. H. LANDER (J. Inst. Petrol. Tech., 1927, 13, 151–168).

Refining dry-cleaners' solvent. FLOWERS and others.—See V.

Ammonium sulphate. TERRES and SCHMIDT.—See VII.

Determination of carbon monoxide. KAST and SCHMIDT.—See VII.

Refractories for oil-gas manufacture. KNOLL-MAN.—See VIII.

PATENTS.

Gas-producer and combined furnace. T. R. WOLLASTON (E.P. 274,286, 3.8.26).—The furnace described in E.P. 243,092 is modified to provide ready

access to the ports and burners supplied with secondary air, means for closing these ports and burners independently, as by detachable firebrick plugs, and means for controlling the actual and relative amounts of primary and secondary air supplied. The dampers or valves controlling the primary and secondary air supplies are positioned from a common lever with provision for independent adjustment of the effective leverages. When applied to a boiler furnace, water may be circulated between the producer jacket and the boiler, or water preheated in the jacket may be pumped into the boiler. Hot water from the jacket may be atomised at the primary air inlet.

H. HOLMES.

Coking retorts. COLLIN & Co., and J. SCHAEFER (E.P. 266,286, 23.9.26. Conv., 20.2.26).—The coke discharged from any one of a row of retorts is conveyed selectively to any one of several cooling chambers in front of the retorts across a platform movable transversely to the retorts and arranged on a level with the discharge openings of the retorts and the charging openings of the chambers. The platform may be a rotary disc provided with a stripper, adjustable rotarily for directing the coke into the selected chamber, or it may be provided with a stripper or a base, such as an endless conveyor band, movable parallel to the axes of the retorts. A container wall arranged around the platform may be provided with closable openings opposite the retorts and the chambers and, if desired, with quenching sprayers.

H. HOLMES.

Coke ovens. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co. G.M.B.H. (E.P. 268,832, 4.4.27. Conv., 3.4.26).—Each inspection opening in the top of the oven over a vertical heating flue is provided with a heat-insulating block and a plugged covering block, both removable. The heat-insulating block is seated on an annular shoulder, and is apertured in line with the plugged aperture in the cover block. A distance tube, *e.g.*, of brick or iron covered with asbestos, may be provided between the blocks.

H. HOLMES.

Regenerative coke ovens. SIMON-CARVES, LTD., and J. H. BROWN (E.P. 274,653, 6.8.26).—The vertical heating flues of a regenerative coke oven are divided into four sections, two up- and two down-flow flues, each pair of down-flow flues being connected to a regenerator placed between adjacent oven walls. The horizontal flue connecting the vertical flues is divided into two halves, which are each tapered from the centre to each end, and thus allow the vertical flues to extend to a higher level.

H. D. GREENWOOD.

Production of active carbon. K. S. C. BONE, and WILSON BROTHERS BOBBIN Co., LTD. (E.P. 274,538, 19.3.26).—Finely divided wood, impregnated with calcium acetate, is fed by means of helically-grooved internal conveyors through one or two retorts, in which it is gradually heated to 1000°. As a result of the progressive heating creosote oil is produced instead of the usual wood tar distillates, and this together with the decomposition products of calcium acetate are withdrawn below 450°.

H. D. GREENWOOD.

Dissociation of carbonaceous gases or vapours. H. E. POTTS. From CANADA CARBIDE Co., LTD. (E.P. 274,573, 24.4.26).—In a continuous process for the

manufacture of carbon black by the dissociation of hydrocarbon gases in a special vertical retort, the composition of the gas is such that the heat of reaction is sufficient to maintain the required dissociation temperature. The gases are introduced into the retort without preheating in order to prevent polymerisation, and suddenly heated to dissociation temperature. The liberated hydrogen is burnt from the base of the retort, the carbon being collected in flues with baffles and continuously removed by a screw conveyor.

H. D. GREENWOOD.

Motor fuel. C. E. H. NORTH and A. H. HUDSON (U.S.P. 1,637,007, 26.7.27. Appl., 5.8.25).—Detonation is suppressed by adding 0.05—1.5% by vol. of carbon tetrachloride to motor fuel.

C. O. HARVEY.

Gaseous fuel [for welding etc.]. J. HARRIS, ASSE. to J. R. ROSE (U.S.P. 1,637,187, 26.7.27. Appl., 28.5.23).—A mixture of acetylene with 5—20% by vol. of benzine vapour is used.

C. O. HARVEY.

Recovery of benzene from coal gas. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co. G.M.B.H. (E.P. 265,984, 11.2.27. Conv., 12.2.26).—The loss of benzene in the exhaust gas of the vacuum pump during the distillation of wash oil is reduced by (1) adding the exhaust gas to the coal gas before washing, (2) washing the exhaust gas in a separate washer, (3) extracting the benzene from the gas by an absorbing agent such as activated carbon or silica gel.

H. D. GREENWOOD.

Low-temperature distillation or coking of fuels. J. PLASSMANN (E.P. 258,261, 7.9.26. Conv., 10.9.25).—The close packing of fuel, prior to distillation, is effected in a vertical retort by the jiggling movements (without rotation) of a tapered plate suspended within the retort or by the horizontal movement of a vertical tapered plate arranged at one side of the retort. The shape of the shaking device may be such that it exerts a pressing action.

H. D. GREENWOOD.

Treatment for transport and/or utilisation of bituminous and like materials. N. TESTRUP, T. BOBERG, and TECHNO-CHEMICAL LABORATORIES, LTD. (E.P. 274,540, 20.3.26).—Insoluble matter is removed from Trinidad asphalt by treating the molten water-free asphalt with about an equal weight of a heated volatile solvent in a vertical tubular vessel. The solution overflows into a cylindrical settling tank with conical bottom from which the settled impurities are withdrawn, whilst the solvent is recovered by heating the mixture in a closed steam coil evaporator and finally with superheated steam. The hot purified asphalt can be transported in heated tank steamers or sufficient solvent can be left in the residue to render it fluid at ordinary temperatures.

H. D. GREENWOOD.

Production of hydrocarbons and derivatives thereof from mineral oil or other bitumens and products obtained therefrom. I. G. FARBEIND, A.-G. (E.P. 249,501 and 274,401, 8.3.26. Conv., [A] 19.3.25. Addns. to E.P. 247,583; B., 1927, 595).—The process described in the main patent can be applied to (A) crude mineral oils, shale oils, fractions or residues of such materials, ozokerite, mineral pitch, etc., (B) conversion products of mineral oils and bitumens, *e.g.*, cracked products, acid sludge, etc., in order to convert them

into valuable oils consisting principally of colourless, saturated benzines. A. B. MANNING.

Production of hydrocarbons [from coal etc.]. I. G. FARBENIND. A.-G. (E.P. 249,156 and 274,404, 13.3.26. Conv., 14.3.25; cf. E.P. 247,582—4; B., 1927, 595).—(A) Coal or similar carbonaceous material is treated with hydrogen under pressures of at least 50 atm. and at high temperatures (e.g., under 200 atm. at 450°) with or without the aid of contact masses, and the tar-like products so obtained are further treated with hydrogen in the presence of catalysts under similar conditions of temperature and pressure. 70% or more of the carbon contained in the solid fuels is thereby converted into valuable hydrocarbons. (B) The tar vapours are subjected to the second stage of the treatment without condensing them intermediately. A. B. MANNING.

Preparation of mixtures of carbon monoxide and hydrogen from hydrocarbons. L. CASALE (E.P. 274,610, 2.6.26).—Gaseous hydrocarbons mixed with excess of steam are completely decomposed to hydrogen and carbon monoxide when heated above 1000° at reduced pressure. The heat absorbed in the reaction is supplied by admitting the required proportion of oxygen or air into the mixture. If air or air enriched with oxygen is used, the mixture of hydrogen and nitrogen required in the synthesis of ammonia is obtained. H. D. GREENWOOD.

[Cracking] treatment of petroleum. J. W. LEWIS, JUN., ASSR. to ATLANTIC REFINING Co. (U.S.P. 1,636,520, 19.7.27. Appl., 4.4.25).—The oil (in liquid phase) is heated and cracked while passing through a tube containing a helical spiral, the cross-section of the spiral stream of oil being but a small fraction of that of the tube. C. O. HARVEY.

Process and apparatus for cracking hydrocarbons. C. ARNOLD. FROM STANDARD DEVELOPMENT Co. (E.P. 274,763, 14.3.27).—Reduced crude oils are cracked so as to produce not more than 0.25% of sediment in the tar by heating the oil in a coil, and then passing it to an enlarged insulated vessel, where cracking is completed under pressure. The cracked product is run at reduced pressure to a tubular still, fitted with a fractionating column, where it is further heated to vaporise at least 30% of the contained gas oil. The tars obtained from the cracking of the gas oil and from the reduced crude oil are mixed for fuel oil. H. D. GREENWOOD.

Process and apparatus for the conversion of heavy hydrocarbon oils into lighter oils. V. W. NORTHRUP (E.P. 275,120, 21.3.27).—Increased yields of gasoline are obtained by mixing the vapours from heavy hydrocarbon oils with hydrogen or a gas containing hydrogen (natural gas) and subjecting the mixture to an alternating current field of 2000–8000 volts to effect ionisation. Thence the vapours pass through a catalysing tube at temperatures of about 385–538°, a suitable catalyst being nickel containing 5% Fe and 5% Al. After passage through a dephlegmating column the vapours are partly condensed in a tubular cooling chamber maintained at 93–204°, the condensate being returned for retreatment and the uncondensed vapours passing to the condenser. In order to obtain gasoline

of satisfactory boiling range etc., a battery of catalysing tubes maintained at different temperatures is used in conjunction with each still. C. O. HARVEY.

Apparatus for the solidification of liquid hydrocarbons. H. NEVEU, ASSR. to Y. DE PANTAGUA (U.S.P. 1,636,644, 19.7.27. Appl., 27.9.24. Cf. U.S.P. 1,525,409; B., 1925, 276).—The apparatus comprises a vessel fitted with mixing arms provided with conduits and attached to two vertical shafts, the one shaft serving for the admission of the liquid hydrocarbon and an emulsifier and the other shaft for the admission of the hardening agent. C. O. HARVEY.

Distilling [oils] with aluminium chloride. A. McD. McAFEE, ASSR. to GULF REFINING Co. (U.S.P. 1,636,144, 19.7.27. Appl., 20.7.25).—In a cracking distillation process employing aluminium chloride the oil is subjected to a preliminary purifying distillation with exhausted aluminium chloride sludge. C. O. HARVEY.

Distillation of crude oils. F. TINKER (E.P. 274,959, 28.4.26).—A heat-interchange arrangement wherein the recovery of outgoing heat from various parts of the system is facilitated by delivering hot liquids or vapours or liquid-vapour mixtures into a mixing chamber, where they attain a common temperature prior to their entering a chamber for separating liquid and vapour. C. O. HARVEY.

Decolorising, clarifying, and purifying petroleum oils. H. L. KAUFFMAN and I. A. CLARK, ASSRS. to H. L. KAUFFMAN (U.S.P. 1,636,938, 26.7.27. Appl., 11.11.24. Renewed 25.2.27).—Untreated, finely-divided clays, containing enough water to render them sufficiently fluid for pumping through pipes, are mixed with the oil, the mixture being heated at a temperature not above the b.p. of the oil, and then steamed down to a temperature over 100°. The vapours removed with the spent steam are condensed. After further cooling, the oil is separated from the filtering medium. C. O. HARVEY.

Purification of oils. F. W. WEBER (U.S.P. 1,636,946, 26.7.27. Appl., 18.3.26).—After refining with sulphuric acid and separation of the acid sludge, the acid-oil mixture is treated with a colloidal solution containing basic chlorides of iron and rare-earth metals, whereby ferric hydroxide is precipitated. The further addition of a practically saturated solution of sodium silicate produces a voluminous heavy precipitate of silicon and rare-earth metal hydroxides, and, after settling and separating operations are completed, the oil is distilled. C. O. HARVEY.

Retort oven for low-temperature carbonisation. P. C. ZUYDERHOUDT (U.S.P. 1,636,975, 26.7.27. Appl., 27.9.24. Conv., 22.10.23).—See E.P. 229,880; B., 1925, 345.

III.—TAR AND TAR PRODUCTS.

Naphthalene formation in coal tar. Y. KOSAKA and Y. OSHIMA (J. Fuel Soc. Japan, 1927, 6, 4–8).—Phenol and its homologues, when passed over coke at 850°, are decomposed to varying extents with formation of benzene, naphthalene, anthracene, and a gas comprising mainly hydrogen, carbon monoxide, and methane. Under the same conditions benzene and toluene yield

naphthalene, anthracene, and a gas comprising mainly hydrogen and methane. Methane is formed by demethylation of homologues of benzene and phenol, whilst carbon monoxide results from the decomposition of or action of carbon on phenols. It is suggested that naphthalene in coal tar is derived partly from the decomposition of alkylnaphthalenes and of naphthols and their derivatives, but mainly from the decomposition of phenols formed during the early stages of coal carbonisation.

W. T. K. BRAUNHOLTZ.

Determination of the softening point of pitch and asphalt by Kraemer and Sarnow's method. H. MALLISON (Z. angew. Chem., 1927, 40, 927—928).—The softening point of pitch or asphalt as determined by Kraemer and Sarnow's method depends on the rate of heating. Values 2—3° lower are obtained for the softening point when the increment of temperature is 1° per min. than when it is 2° per min. Klinger's modification of this method (B., 1914, 130) is recommended, the initial temperature of the water-bath being 20—25° below the expected softening point of the pitch.

R. BRIGHTMAN.

Sulphur in motor benzol. KATTWINKEL.—See II.

Action of sulphuric acid on hydrocarbons. ORMANDY and CRAVEN.—See II.

PATENT.

Benzene from gas (E.P. 265,984).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Hydro- ψ -thiocyanic acid as a dye for cotton. B. MELIS (Annali Chim. Appl., 1927, 17, 346—351).—This acid (cf. A., 1927, 346) when dissolved in sodium hydroxide or sulphide dyes cotton a lemon-yellow colour. The dye is fast to light, washing, boiling, rubbing, hot ironing, the action of sulphur dioxide, ammonium acetate, 10% sulphuric acid, or 30% acetic acid. Alkali causes a slight deepening of colour, boiling sodium hydrogen sulphate solution a slight heightening; the dye is not, however, transferred to admixed undyed cotton. Washing with soap and sodium carbonate causes a slight modification, chlorine a slight bleaching, and mercerisation a modification of colour with transference to undyed cotton. The mechanism of the dyeing is discussed; it is attributed to the presence of SH- and S-S-groupings.

E. W. WIGNALL.

PATENTS.

Manufacture of compounds of aromatic *p*-diamines with sulphur dioxide. I. G. FARBENIND. A.-G. (E.P. 269,583, 14.4.27. Conv., 17.4.26).—Aromatic *p*-diamines on treatment with sulphur dioxide in the absence of water (e.g., by saturating their benzene solution with the gas) are converted into water-soluble compounds remaining unchanged in air, which are used as hair dyes, photographic developers, etc.

B. FULLMAN.

Reduction of aromatic nitro-compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 274,562, 21.4.26).—In the reduction of aromatic nitro-compounds, finely-divided iron oxide of pigmentary value may be obtained by carrying out the reduction with iron and an acid-reacting concentrated solution of an easily

soluble salt of a metal more electro-positive than iron, the acid being either an added non-reducing mineral acid or derived from the hydrolysis of the salt (of a non-reducing mineral acid). Easily soluble chlorides (e.g., those of the alkali or alkaline-earth metals, magnesium, and zinc) are especially suitable, as well as ferrous chloride. A sludge of black ferrosiferrous oxide or yellow to brown hydrated ferric oxide remains, which on sedimentation etc. may either be used as a pigment immediately or converted into a red shade by calcination.

B. FULLMAN.

Manufacture of cyclic hydrocarbons and derivatives thereof. I. G. FARBENIND. A.-G., Assees. of FARBW. FORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,270, 19.4.26. Conv., 24.4.25).—Aromatic ketones having both a methyl or methylene group and a free hydrogen atom *ortho* to the carbonyl group condense internally to form anthracene derivatives when their vapours are passed over heated porous contact substances (silica gel, alumina gel, active charcoal, etc.); e.g., phenyl *m*-xylyl ketone (active carbon at 380—400°) gives β -methylantracene, whilst *o*-tolyl α -naphthyl ketone (aluminium oxide at 420°) gives naphthanthracene, m.p. 167° [Elbs (Ber., 1919, 52, 2211) gives 141°]. Similarly, mixed aliphatic aromatic ketones yield derivatives of 5-membered rings; e.g., *m*-xylyl methyl ketone (active charcoal) gives methylinene.

B. FULLMAN.

Manufacture of colour lakes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 274,627, 28.6.26).—Diphenylamine derivatives, made by condensing a halogenated aromatic nitro-compound with a primary or secondary arylamine which may contain carboxylic or sulphonic groups, are mixed with the usual lake components or, if acidic, are converted into insoluble barium or calcium salts. Thus sodium 2:4-dinitrodiphenylamine-2'-carboxylate and sodium carbonate are dissolved in water, barium sulphate and a solution of aluminium sulphate are added, and an orange lake is precipitated by addition of barium chloride solution. 2:4-Dinitrodiphenylamine-4'-sulphonic acid gives a greenish-yellow lake.

C. HOLLINS.

Manufacture of [chrome] dyes of the triaryl-methane series. I. G. FARBENIND. A.-G. (E.P. 263,879, 3.1.26. Conv., 2.1.26).—The carbinols obtained by condensing substituted benzaldehydes with 2 mols. of *o*-cresotic acid are converted into soluble compounds by heating with sodium sulphite solution. The filtered solutions on evaporation give colourless products, which regenerate the carbinols on treatment with acid, and dye wool blue to violet and brown shades by after-chroming. Products derived from 4-*p*-tolylaminobenzaldehyde (violet), 4-(6'-chloro-*o*-tolylamino)benzaldehyde (violet), *p*-chlorobenzaldehyde (greenish-blue), 2:6-dichlorobenzaldehyde (reddish-brown), 2:6-dichloro-3-hydroxybenzaldehyde (brown), 2:4:6-tribromo-3-hydroxybenzaldehyde (brown), and hydroxynaphthaldehydedisulphonic acid (E.P. 19,498 of 1897; brownish-violet) are described.

C. HOLLINS.

Manufacture of chloranil and bromanil. L. B. HOLLIDAY & Co., LTD., and C. SHAW (E.P. 274,700, 26.11.26).—Chloranil results when *p*-nitrosophenol is chlorinated in 30% hydrochloric acid, the temperature

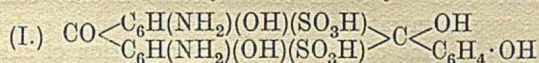
being raised slowly to 90°. Bromination of *p*-nitrosophenol in glacial acetic acid gives bromanil.

C. HOLLINS.

Manufacture of derivatives of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 250,968, 17.4.26. Conv., 17.4.25).—4 : 8-Diaminoanthrarufin-2 : 6-disulphonic acid, when heated with rather more than 2 mols. of 40% aqueous formaldehyde at 95° for 1½ hrs., gives the NN'-dimethyl derivative, precipitated by addition of concentrated hydrochloric acid. It dyes wool blue from an acid bath. One or both of the sulphonic groups may be removed, e.g., by reduction with sodium hyposulphite, or by hydrolysis with 90% sulphuric acid and boric acid, especially in presence of a little stannous chloride. The monosulphonic acid (aniline salt prepared) gives a light greenish-blue on wool. Similar series of compounds are obtained from the 3 : 7-disulphonic acid and from 4 : 5-diaminochrysazin-2 : 7-disulphonic acid.

C. HOLLINS.

Manufacture of anthraquinone derivatives. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 274,211, 19.4.26).—The boric esters of 4 : 8-diaminoanthrarufin-2 : 6-disulphonic acid and its *N*-methyl derivatives condense in concentrated sulphuric acid below 20° with phenols or their ethers to give compounds, which when heated in dilute mineral acid or alkali (or in the cold with caustic alkali) are converted into 2-*p*-hydroxyaryl derivatives, with loss of the 2-sulphonic group. The remaining sulphonic group may be removed by known methods, e.g., by reduction with sodium hyposulphite. 4 : 8-Diaminoanthrarufin-2 : 6-disulphonic acid is heated with sulphuric and boric acids to form the boric ester, and condensed with phenol at 10–20° to give the compound (I), which may be isolated by addition of acetic



acid, or by liming out and evaporation in a vacuum. This product is converted, if desired without isolation, into 4 : 8-diamino-2-*p*-hydroxyphenylanthrarufin-6-sulphonic acid (pyridine salt described), which is a blue acid wool dye. Removal of the sulphonic group gives a compound which dyes chrome-mordanted wool in blue shades. Similar series of products are obtained from 4 : 8-dimethyldiaminoanthrarufin-2 : 6-disulphonic acid, and by using other phenols or their ethers, e.g., anisole, resorcinol, quinol, naphthols, diphenyl ether, etc. C. HOLLINS.

Manufacture of vat dyes of the 1-thionaphthen-2'-indoleindigo series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 274,527, 17.2.26).—Thioindoxyls containing in 4 : 6-positions halogen or alkyl groups, and hydrogen, alkyl, or halogen in position 5, are condensed with α -anils or α -halides of isatins, particularly of 5 : 7-dihalogeno-isatins (or the condensation product may be halogenated). 4 : 6-Dichlorothioindoxyl, m.p. 140–143°, 4 : 5 : 6-trichlorothioindoxyl, m.p. 150–153°, 4-chloro-6-methylthioindoxyl, m.p. 106–108°, 6-chloro-4-methylthioindoxyl, m.p. 120–123°, 4 : 6-dichloro-3-methylthioindoxyl, m.p. 135°, and 4 : 6-dichloro-5-methylthioindoxyl, m.p. 110°, are prepared, respectively, from 2 : 4-dichloro- and 2 : 3 : 4-trichloro-anilines, 5-chloro-*o*-toluidine, 3-chloro-*p*-toluidine, and 4 : 6- and

2 : 6-dichloro-*m*-toluidines, by the usual methods. The condensation of these thioindoxyls with 5 : 7-dichloro-, 5 : 7-dibromo-, and 5 : 6 : 7-trichloro-isatin α -chlorides or α -anils in chlorobenzene or xylene at 60–90° gives reddish-violet to bluish-violet vat dyes. 4 : 5 : 6-Trichloro-*o*-toluidine, m.p. 94–95° (acetyl derivative, m.p. 199–200°), is prepared from 3 : 4-dichlorotoluene by nitration, further chlorination, and reduction; 4 : 5 : 6-trichloro-*m*-toluidine from 4 : 5 : 6-trichlorotoluene, m.p. 41°, by nitration, separation of *m*-nitro-compound by fractional distillation, and reduction; 4 : 6-dichloro-3-aminoethylbenzene from *p*-chloroethylbenzene by chlorination, nitration, and reduction. C. HOLLINS.

Manufacture of azo dyes [pigments and ice-colours]. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 255,072, 7.7.26. Conv., 7.7.25).—Compounds of the type Ar·SO₂·NR·Ar'·NH₂(o), in which Ar and Ar' are aromatic residues and R is alkyl or aralkyl, are diazotised and coupled in substance or on the fibre with an arylamide of 2 : 3-hydroxynaphthoic or acetoacetic acid. Benzenesulphon-*o*-aminomethylanilide gives with 2 : 3-hydroxynaphthoic *o*-toluidide or 5-chloro-*o*-toluidide a red, with bisacetoacetylolidine a greenish-yellow dye. C. HOLLINS.

Manufacture of alkylated or cycloalkylated arylsulphonic acids. I. G. FARBENIND. A.-G. (E.P. 268,375, 25.3.27. Conv., 27.3.26. Addn. to 250,241; B., 1927, 470).—Anhydrous aromatic sulphonic acids are condensed with aliphatic or alicyclic alcohols and chlorosulphonic acid. The products show variation of the position of the sulphonic groups as compared with those obtained by the similar condensation of aromatic hydrocarbons. E.g., naphthalene is sulphonated with chlorosulphonic acid, and the reaction mixture treated simultaneously with butyl alcohol and chlorosulphonic acid, and then with chlorosulphonic acid. The sodium salt of the product is a wetting and emulsifying agent.

B. FULLMAN.

Manufacture of *N*-monoalkyl-*p*-aminophenol. H. E. POTTS. From CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER A.-G. (E.P. 274,175, 10.4.26).—Acylated *ON*-alkyl-*p*-aminophenols (e.g., *N*-methylaceto-*p*-phenetide) are converted into *N*-monoalkyl-*p*-aminophenols by hydrolysis with sulphuric acid; the amount of the latter may be varied continuously from 6 equivalents of 50% acid (heating for 3 hrs. at 139°) to 3 equivalents of 70% (4½ hrs. at 146°). The acid is neutralised with chalk, and on evaporation the pure alkylaminophenol crystallises. B. FULLMAN.

Manufacture of anthraquinone derivatives. W. H. PERKIN, A. W. FYFE, and M. MENDOZA, ASSTS. to BRITISH DYESTUFFS CORP., LTD. (U.S.P. 1,636,485, 19.7.27. Appl., 5.4.26. Conv., 8.7.25).—See E.P. 252,922; B., 1926, 658.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Hygroscopic properties of cotton wool and its charred product. S. V. GORBATSCHEV and E. N. VINOGRADOVA (Biochem. Z., 1927, 186, 413–418).—The adsorption of water vapour by cotton wool and its

charred product ("granulose") has been measured at 37° in an atmosphere saturated with water vapour. The kinetics of the adsorption deviate considerably in each case from the adsorption of gases, for which Iljin has given a theoretical formula (A., 1924, ii, 92). The adsorption isotherms for cotton wool and "granulose" show that the latter possesses a distinctly greater adsorptive power at higher humidities, and is therefore to be preferred for bandages. Used clinically, "granulose" has given encouraging results. A. WORMALL.

Kapok. R. O. BISHOP and G. L. TEIK (Malayan Agric. J., 1927, 15, 97—103).—Samples of kapok from various sources do not differ materially in their chemical and physical properties, nor is it possible to effect a satisfactory differentiation by buoyancy tests. Extraction of kapok with fat solvents has no appreciable effect on buoyancy. The mean fibre lengths of Serdang kapok, prime Samarang, and average Samarang kapok are 6.5, 7.5, and 10.0 mm. respectively. D. J. NORMAN.

Determination of copper in rot-proof canvas. BONNARD and LEBLANC (Ann. Chim. Analyt., 1927, [ii], 9, 233—235).—Owing to the presence of chromium in rot-proof canvas the copper cannot be completely dissolved by acid treatment. The sample is therefore incinerated in a platinum dish and the residue fused with 5 g. of potassium hydrogen sulphate. The product is leached with hot water containing a little hydrochloric acid, and the copper in the solution is separated by a current of hydrogen sulphide. A. R. POWELL.

Chemical processes for re-refining and decolorising dry-cleaners' solvent by continuous automatic methods. A. E. FLOWERS, F. H. MCBERTY, and M. A. DIETRICH (Ind. Eng. Chem., 1927, 19, 868—873).—100 kg. of goods cleaned yield 1.6 kg. of solid dirt and 1.8 kg. of dark-coloured, soluble impurity. As little as 0.1% of moisture may cause dirty solvent to mark goods subsequently treated. The odour which petroleum solvents communicate to clothes etc. is due mainly to unsaturated hydrocarbons. The centrifugal clarifier is suitable for the continuous removal of solid impurities. Distillation in steam for the removal of colour and odour is relatively costly and unsatisfactory. For this purpose a continuous process using sulphuric acid has been developed which is much superior to the employment of alkali, fuller's earth, or activated carbon. The washing apparatus is fitted with a number of discs upon which the solvent mixed with 3% of acid falls. In a second apparatus neutralisation with dilute ammonia is performed. This does not cause emulsification. The treated solvent is returned to the clarifier to remove moisture. The process is more suitable for "Stoddard-solvent" (a spirit with restricted distillation range) than ordinary petrol as the end-point of the latter is unduly raised. C. IRWIN.

Measurement of the "wetness factor" of mechanical wood pulp by the Schopper-Riegler apparatus. W. BRECHT and E. SCHAUN (Papier-Fabr. 1927, 25, Fest-u. Ausland-heft, 45—60).—The Schopper-Riegler apparatus has attained very considerable importance for the control of the grinding operation, since in the case of ordinary "news" paper manufacture under rigorously standardised conditions, all the technically

important qualities of the paper can be governed by the factor established by this test. An investigation of the sources and limits of error in the Schopper-Riegler test has led to the recommendation that the standard test should be carried out on 1000 c.c. of a suspension containing 0.30% of absolutely dry fibre at a standard temperature of 20°. The error due to variations of temperature is a fall of 0.462 "wetness" or "slowness" unit per 1° rise in temperature. Variations in the volume of the suspension with constant concentration induce a rise in the "slowness" factor with fall in volume, but not in direct proportion. The principal source of error is a variation in consistency, i.e., dry fibre-substance present in the 1000 c.c. of suspension. This error is large if concentrations below 0.3% are selected for the test; on the other hand, if higher concentrations are adopted, the error is reduced but the sensitiveness of the test is much impaired. Errors in concentration may be minimised by adopting standard manipulation in preparing the suspension. It is recommended that 250 c.c. of the sample of fluid pulp be collected on a filter in a Buchner funnel and pressed to a cake of standard dry-substance by means of a brass plate and weight calculated to give a uniform pressure of 0.445 kg. per sq. cm. for 3 min. The calculated quantity of this cake necessary to give 3 g. of dry fibre is shaken with the complementary volume of water for the test-suspension. Lastly, a slight modification of the test-apparatus for eliminating the personal factor in releasing the flow for the test is recommended, or alternatively the adoption of the improved pattern of instrument devised by the Canadian manufacturers. J. F. BRIGGS.

Digestion of wood with liquors of low sulphite content. E. HÄGGLUND (Papier-Fabr., 1927, 25, Fest-u. Ausland-heft, 60—63).—In certain cases liquors containing low concentrations of sulphite are used in order to shorten the time of digestion, the proportion of sulphite being an important factor in the speed of the digestion. This is because the acidity with low concentration of sulphite is greater than with high sulphite concentration. In the first stage of the digestion, while the sulphite is combining with the lignin, there is not much difference between the two types of liquor, since this phase is not dependent on the hydrogen-ion concentration, and large quantities of lignosulphonic and sulphuric acids are not present in the liquor. In the second phase, however, the difference is very marked, but shows itself more in the larger quantity of direct cupric-reducing sugars than in the degree of digestion of the wood. The yield of cellulose is only markedly affected in the last phase of the digestion, and a liquor of low sulphite content gives in 15 hrs. the same yield of cellulose as the less acid liquor in 17 hrs. Owing to the greater hydrolytic action of the less basic liquor, the proportion of direct reducing sugars is consistently higher, even at equivalent yields of cellulose, and the quality of the cellulose is lower both in tensile strength and particularly in the folding test; the cellulose produced has a brittle fibre. J. F. BRIGGS.

Digestion of plant materials with nitric acid. P. KRAIS, K. BILTZ, and O. RENNER (Papier-Fabr., 1927, 25, Fest-u. Ausland-heft, 43—44).—Several charges

on an industrial scale up to 900 kg. of wood waste, straw, and reeds have been put through with favourable results. The consumption of nitric acid has been rather higher than in the laboratory, but improvements in the recovery of gaseous products are in hand and the institution of rotary digesters of larger dimensions. The celluloses produced on the large scale have copper values of 2.4 to 2.7 and the α -cellulose content ranges between 78.2 and 86.3% according to the raw material. It is true that small quantities of hydrocyanic acid vapours are produced, but greater personal inconvenience is probably caused by the escape of nitric oxide, which at the same time acts as a warning. The yield and quality of the cellulose produced by the nitric acid process are comparable with those of sulphite pulp. J. F. BRIGGS.

Decomposition of cellulose. HEUKELEKIAN.—See XXIII.

PATENTS.

Treating plant material for the removal of incrusting materials from the fibre. F. K. FISH, JUN. (E.P. 255,029, 4.6.26. Conv., 7.7.25).—Fibrous material is fed into a digester which already contains a proportion of liquor and is provided with a device whereby liquor is continuously withdrawn from the bottom of the digester and sprayed on top of the charge. When completely charged the digester is sealed and evacuated. A further quantity of liquor is then admitted and digestion completed under pressure. During each of these operations circulation and spraying of the liquor are maintained. The cooking liquor may be acid, neutral, or alkaline, but should contain the volatile plant extractives recovered from previous operations.

D. J. NORMAN.

Wadding. PAPETERIES DE LA ROBERTSAU (E.P. 265,125, 10.6.26. Conv., 29.1.26).—Non-hydrophile wadding is obtained by sizing the cellulose pulp in the hollander with, *e.g.*, 3% of resin soap using aluminium sulphate as precipitant, or with gelatin with tannic acid as precipitant.

D. J. NORMAN.

Extracting fats from textiles. A. BEIL and W. A. MEYER, Assrs. to A. RECHBERG-G.M.B.H., and G. BRAUN-G.M.B.H. (U.S.P. 1,636,351, 19.7.27. Appl., 11.3.26. Conv., 18.3.25).—The material is treated with liquid methylene chloride.

D. J. NORMAN.

Desizing process. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 274,795, 18.1.26).—The desizing of fibrous material of animal, vegetable, or artificial origin is facilitated by the addition of wetting agents to the desizing liquor.

D. J. NORMAN.

Manufacture of artificial silk etc. W. P. DREAPER (E.P. 273,354, 11.1.26).—During the replacement of one centrifugal box by another in the centrifugal spinning process, the thread is diverted before reaching the funnel guider and collected in an untwisted condition on a reel or bobbin. The funnel is preferably of the type described in E.P. 171,719 (B., 1922, 52 A). The untwisted filaments thus obtained may conveniently be converted into staple fibre.

D. J. NORMAN.

Manufacture of threads, filaments, etc. from viscose. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (E.P. 273,506, 19.7.26).—Hollow viscose

filaments, which consist chiefly of narrow tubes practically free from spaced bubbles, are obtained by spinning a viscose solution containing 1–3% of sodium carbonate into a bath containing 9–11% of sulphuric acid, 10–14% of sodium sulphate, 8–14% of magnesium sulphate, and 0–8% of zinc sulphate, the proportions being so arranged that the bath contains a total of 25–28% of metal sulphates.

D. J. NORMAN.

Manufacture of artificial materials from viscose.

L. LILIENTHAL (E.P. [A] 274,521 and [B] 274,690, 11.1.26).

—(A) Viscose silk of high strength, *e.g.*, 2–5 and 1.5–2.5 g. per denier in the dry and wet state respectively, is obtained by spinning, preferably stretch-spinning, viscose solution into sulphuric acid of 55–85% strength. Unmatured or relatively slightly matured viscose made from alkali-cellulose that has matured only for a short time, *e.g.*, 3–36 hrs. is, in general, more satisfactory than fully ripened viscose, being less affected by the strong acid; in certain circumstances, however, it is advantageous to use a more fully ripened viscose in the preparation of which more than 40% of carbon disulphide (on the weight of cellulose) has been added. The viscosity of the spinning solution should be at least twice that of glycerin. The length of travel of the thread both in and outside the bath may vary within wide limits depending on the concentration of the acid, the ripeness of the viscose, etc. Means should be provided for diluting the acid on the thread before or during the reeling operation. Any suitable additions may be made to the spinning bath or to the viscose solution. (B) The whole or a part of the sulphuric acid in the spinning bath described above can be replaced by another strong mineral acid, *e.g.*, hydrochloric acid of 25–40% strength, nitric or arsenic acid of 60–90%, or phosphoric acid (*d* 1.5–1.86).

D. J. NORMAN.

Production of foils, skins, bands, etc. from cellulose. WOLFF & Co., E. CZAPEK, and R. WEINGAND (E.P. 257,924, 27.8.26. Conv., 1.9.25).—Solutions of suitable inorganic acids or salts or mixtures thereof in organic solvents are used as coagulating agents for aqueous cellulose solutions in the manufacture of articles which have a larger cross-section than that of artificial silk. Alcohols of low b.p., particularly methyl alcohol, may be used as organic solvents, and suitable coagulating baths consist of methyl alcohol in admixture with (a) 40% of sulphuric acid, (b) 10% of magnesium chloride, or (c) 10% of magnesium chloride and 10% of free hydrochloric acid. Methyl alcohol alone may be used to effect a preliminary coagulation of the cellulose solution if desired.

D. J. NORMAN.

Treating artificial silk produced in centrifuges.

VEREIN. GLANZSTOFF-FABR. A.-G. (E.P. 265,996, 12.2.27. Conv., 13.2.26).—In the manufacture of artificial silk, particularly viscose silk, by the centrifugal process a more regular product is obtained if the freshly spun cake of silk is washed and, as far as possible, dehydrated centrifugally before removing it from the spinning pot. The silk is then reeled off and the resulting hanks are dried, preferably on a stretching carriage. To prevent damage to the cake during washing, water in a fine state of division is used.

D. J. NORMAN.

Treating cellulose acetate. E. S. FARROW, JUN., ASSR. to EASTMAN KODAK CO. (U.S.P. 1,634,986, 5.7.27. Appl., 12.10.25).—Acetone-soluble cellulose acetate is produced by treating chloroform-soluble cellulose acetate, dissolved in acetic acid, with an aqueous solution of phosphoric acid. D. J. NORMAN.

Production of fibre half-stuff. I. G. FARBENIND. A.-G. (E.P. 264,803, 9.12.26. Conv., 25.1.26).—Pulp of good quality can be obtained from flax straw, corn straw, jute, hemp, etc. by digestion under pressure with a liquor containing sodium bisulphite and sodium sulphite in the ratio 3—3.5 : 1 with addition of neutral salts, preferably those which undergo hydrolysis at elevated temperatures. A certain proportion of the alkali bisulphite and sulphite may be replaced by the corresponding alkaline-earth salts if the ratio of bisulphite to sulphite remains unaltered. D. J. NORMAN.

Producing white pulp of high strength. G. A. RICHTER, ASSR. to BROWN CO. (U.S.P. 1,635,637, 12.7.27. Appl., 11.7.25).—Raw cellulosic material is digested in an acid sulphite liquor in which the free sulphur dioxide is not in excess of the combined sulphur dioxide. The resulting pulp is washed, treated with a solution containing chlorine, again washed, and finally digested with a solution containing alkali. D. J. NORMAN.

Process of making paper from straw. C. D. WOOD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,634,603, 5.7.27. Appl., 12.1.26).—The alkali-soluble components obtained by digesting fibrous material with soluble sulphides are incorporated with the pulp during its conversion into paper. D. J. NORMAN.

Treatment of [wool-scouring] liquids containing oil or fatty substances and the like. J. AVERY, ASSR. to AUSTRALIAN LANOLINE PROPRIETARY LTD. (U.S.P. 1,638,977, 16.8.27. Appl., 27.9.23. Conv., 23.10.22).—See E.P. 205,833; B., 1925, 281.

Dry distillation of alkalisied waste liquor from soda cellulose manufacture. E. L. RINMAN (U.S.P. 1,638,061, 9.8.27. Appl., 4.6.24. Conv., 6.6.23).—See E.P. 217,235; B., 1925, 496.

Treating fibres etc. (E.P. 273,830, 272,165, 263,102, 274,940, 273,406, and 274,584).—See VI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Is digallic acid identical with tannin as a mordant for basic dyes? P. P. VIKTOROV (Z. angew. Chem., 1927, 40, 922—925).—Synthetic *m*-digallic acid obtained by Fischer's method possesses the characteristic properties of the natural tannins, being precipitated by tartar emetic and affording precipitates with basic dyes which dissolve in excess of the acid. Digallic acid is adsorbed by cotton, the adsorption increasing with the time of immersion in the bath. With tartar emetic and basic dyes it affords dyeings on cotton which in the case of Victoria Blue B and Night Blue are deeper, purer, and bluer than the shades obtained under corresponding conditions with tannin. Methylene Blue and Malachite Green, however, afford deeper and greener shades when dyed with tannin than with digallic acid. In mordanting with tannin, the tannin is decomposed and only the digallic acid plays an active part in the formation

of the antimony mordant compound, $\text{OH} \cdot \text{Sb}(\text{C}_{14}\text{H}_9\text{O}_9)_2$. In both mordant and dyeing processes the active constituent of natural tannin is that which appears in synthetic pentadigalloyl- β -glucose containing digallic acid. Doubt is thrown on the pentagalloyl- β -glucose structure assigned to Turkish tannin by Fischer, since gallic acid is only very slightly adsorbed by cotton, and in a 0.25% acetic acid bath. It is possible, however, that if Turkish tannin possesses a pentagalloylglucose structure its mordant properties are to be attributed to the 2.7—3.8% of ellagic acid also present.

R. BRIGHTMAN.

PATENTS.

Treatment of cotton fabrics containing artificial silk. CALICO PRINTERS' ASSOC., LTD., L. A. LANTZ, and C. M. KEYWORTH (E.P. 273,830, 12.4.26).—Cotton in admixture with cellulose acetate silk can be mercerised with ordinary mercerising solutions under ordinary conditions of time, temperature, etc. without injury to the cellulose acetate if the fabric is impregnated, prior to mercerisation, with a liquid which neither mixes nor reacts with alkali solution of mercerising strength, but prevents or retards hydrolysis of the cellulose acetate. Suitable impregnating agents include benzene and its homologues, hydrogenated naphthalenes, petroleum distillates, solvent naphtha, and essential oils. D. J. NORMAN.

Chemically varying vegetable or artificial fibres. HEBERLEIN & CO. A.-G. (E.P. 272,165, 29.11.26. Conv., 5.6.26).—By the use of suitable resists the process described in E.P. 255,453 and 261,792 (B., 1927, 103, 473) may be used for the production of two-colour effects on fabrics; e.g., bleached cotton fabric is printed with a mixture of caustic soda and dextrin and is then drawn through a 3% solution of phosphorus oxychloride in xylene. Those parts of the fabric that have been printed with alkali show modified dyeing properties, whilst the remainder is substantially unaffected. D. J. NORMAN.

Preparation for sizing textile fabrics. A. F. GALVIN (E.P. 254,720, 30.6.26. Conv., 4.7.25).—A sizing preparation which neither damages nor discolours textile fabrics is obtained by adding linseed oil previously boiled with a cobalt acetate, oleate, or resinate drier to a refined drying oil or light drying oil varnish, the total quantity of drier being less than 4% on the weight of drying oil present. Other constituents may be added to produce various effects. A suitable solution contains petrol 2 litres, drying oil produced as above 1 litre, tung oil 0.5 litre, soap 0.1 litre, grease 30 g., Japan wax 30 g. D. J. NORMAN.

Manufacture [sizing] of artificial silk and other textile fibre. NEUTRASOL PRODUCTS CORP., ASSEES. OF E. POHL (E.P. 255,909, 27.7.26. Conv., 27.7.25).—A coating material for artificial silk etc. is obtained by partially or completely saponifying a mixture of beeswax and other waxes and fatty acids, e.g., 3 pts. of beeswax, 3.5 pts. of Japan wax, 3.5 pts. of stearic acid, and 1 pt. of oleic acid saponified with 1 pt. of caustic soda dissolved in 20 pts. of water. D. J. NORMAN.

Improving vegetable textiles. A. MEYER-SANS-ŒUF G.M.B.H. (E.P. 263,102, 4.11.26. Conv., 21.12.25).—Textile materials in loose or fabric form show improved

resistance to ultra-violet light, water, and dilute solutions if they are subjected to the successive action of a tanning solution containing a wetting agent, a mordanting solution, and an oiling solution containing an aldehydic or ketonic substance to control the rate of oxidation of the oil during the final drying operation; *e.g.*, cotton yarn is impregnated for 0.5 hr. at 90° in a bath containing 5% of catechu and 5% of Tetracarnit (a mixture of Turkey-red oil, pyridine, and tetrachloroethane), centrifuged, and transferred for 0.5 hr. to a 5% solution of lead acetate at 50°. The oiling bath consists of a 5% solution of linseed oil in tetrahydronaphthalene with addition of 2% of paraldehyde; after impregnation for 10 min. at the ordinary temperature, the material is centrifuged and allowed to dry.

D. J. NORMAN.

Treatment of cellulosic materials. W. KERSHAW, F. L. BARRETT, C. S. PARKER, and BLEACHERS' ASSOC., LTD. (E.P. 274,940, 23.4.26).—Cotton fabrics are treated with sulphuric acid of parchmentising strength containing dissolved cellulose, so that, when the acid is washed out, cellulose is precipitated within and upon the fabric, giving a stiff material, which resists deformation by stretching even when wet, and, after suitable conditioning, is capable of taking embossed patterns which are not destroyed by washing or boiling. A solution of 5 pts. of pure air-dry cotton cellulose in 95 pts. of sulphuric acid (*d* 1.575–1.675) at 0° is suitable for parchmentising; it is fairly stable at low temperatures, but should not be kept many hours. D. J. NORMAN.

Manufacture [finishing] of fabrics. BRITISH CELANESE, LTD., W. A. DICKIE, and H. HALKYARD (E.P. 273,406, 1.4.26).—Embossed effects, which are not affected by ordinary laundering operations and cause no substantial weakening of the fabric, are produced on fabrics composed of or containing cellulose acetate or other thermoplastic cellulose derivatives by impregnating the fabric with a volatile liquid which causes the cellulose derivative to swell, but not to dissolve, and exposing the treated fabric to heat, *e.g.*, 110–120°, and pressure. Suitable liquids are water, aqueous solutions of, *e.g.*, ammonium thiocyanate, alcohol, etc., solutions of acetone, cyclohexanone, ethyl lactate, etc. in volatile organic solvents which have no action on cellulose acetate, *e.g.*, benzene; plasticising agents and lubricants may be added if desired. D. J. NORMAN.

Treatment of yarns or fabrics consisting of or containing artificial silk. C. S. PARKER, W. KERSHAW, F. L. BARRETT, and BLEACHERS' ASSOC., LTD. (E.P. 274,584, 29.4.26).—Crêpe-like effects are obtained on fabrics consisting of or containing cellulose esters by treating the fabric for 3–180 sec. at 0–55° with dilute nitric acid (*d* 1.15–1.25). D. J. NORMAN.

Apparatus for bleaching, dyeing, washing, and drying yarns or fabrics. W. WINTER and H. JORDAN (E.P. 275,363, 31.5.26).

Arylsulphonic acids (E.P. 268,375). **Compounds of *p*-diamines and sulphur dioxide** (E.P. 269,583).—See IV.

Resinous condensation products (E.P. 274,155).—See XIII.

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

Repairing sulphuric acid chamber bottom during operation. W. C. KENDRICK and M. E. SOUDER (Ind. Eng. Chem., 1927, 19, 954–955).—A leak which had developed over the full width of an acid chamber bottom was repaired without stopping the chamber as follows:—A sectional wooden tunnel, covered on the outside with composition and pitch, was prepared. The chamber was put under suction, a hole cut in it, and the tunnel inserted, the sections being jointed together with acid-proof cement. The acid inside the tunnel, which had no bottom, was taken up with dry earth and the paste pushed through loose boards to form a dam outside. The repair was then carried out, the whole operation taking 25 hrs. C. IRWIN.

Ammonium sulphate. Manufacture of household ammonia. G. WALMSLEY (Gas J., 1927, 189, 391–392).—A considerable increase in the revenue arising from the disposal of gas-works' ammonium sulphate appears possible by the conversion of part at least into ammonia solution (10–12%) for domestic use. The process consists in distilling a concentrated solution of ammonium sulphate with lime, using a small steam-heated still of special design. Working up one ton of sulphate can be made to yield a profit of approximately £50. A. B. MANNING.

Physico-chemical principles of the recovery of ammonium sulphate from ammoniacal gases and sulphuric acid. E. TERRES and W. SCHMIDT (Gas- u. Wasserfach, 1927, 70, 725–728, 762–766, 808–813).—The solubility of ammonium sulphate in water containing various proportions of sulphuric acid was determined at temperatures between 0° and 100°. With an acid concentration of 0–22%, the solid phase is normal ammonium sulphate, the solubility decreasing with increasing acidity at temperatures up to 40° and increasing slightly with increasing acidity at higher temperatures. With an acidity of about 22–35%, the solid phase is the double salt ammonium sulphate-bisulphate, the solubility of which increases with increasing acidity, whilst at still higher acid concentrations it is ammonium bisulphate alone, the solubility of which decreases to a minimum at an acidity of about 58%. The solubility of ammonium sulphate in water containing free ammonia decreases regularly and rapidly with increasing ammonia content, falling to zero (by extrapolation) at a free ammonia concentration of about 30% at 0°. Vapour pressure curves are also established for the above solutions and temperatures. The vapour pressure of ammoniacal solutions falls rapidly and regularly with decreasing alkalinity. With increasing acidity the vapour pressure continues to decrease, though less rapidly, the effect being more marked the higher the temperature, and breaks in the curves indicate the change in the solid phase at about 35% acidity. The bearing of these physico-chemical data on the practical manipulation of the direct and semi-direct ammonia recovery processes and of the Burkheiser ammonium sulphate-bisulphate process is discussed.

W. T. K. BRAUNHOLTZ.

Manufacture of bismuth salts. F. CHEMNITZ (Pharm. Zentr., 1927, 68, 513–518).—The preparation of the normal and basic nitrate, oxide, hydroxide, sulphate, benzoates, subcarbonate, citrates, subgallate, subsalicylate, tartrate, lactate, oxyiodide, iodogallate, tannate, albuminate, phenoxide, and β -naphthoxide is described.

Composition of technical calcium hypochlorite and comparison of its behaviour on heating with that of bleaching powder. H. DITZ and R. MAY (Z. Elektrochem., 1927, 33, 265–272).—Technical calcium hypochlorite (Griesheim) has been analysed and the results are compared with those obtained by Kast and Metz (cf. B., 1927, 297). The much higher value for free lime given by the latter results from their assumption that it can be regarded as the difference between the total lime and that combined as hypochlorite, no allowance being made for that present as chloride, chlorate, or carbonate. The same error is involved in their values for free lime in the bleaching powder samples examined. The results of Kast and Metz on the tendency of calcium hypochlorite and bleaching powder to decompose on heating, and on the action of carbon dioxide on bleaching powder are compared with previous results of Hoffmann and Ritter (A., 1914, ii, 612). Further experiments have been made on the behaviour of technical calcium hypochlorite when heated alone, or with addition of calcium chloride, in a current of dry air free from carbon dioxide. On the basis of the results a critical examination is made of the conclusions derived by Kast and Metz from experiments on the action of heat on technical calcium hypochlorite and bleaching powder, and on the action of carbon dioxide on bleaching powder.

H. J. T. ELLINGHAM.

Colorimetric determination of carbon monoxide by means of an ammoniacal silver solution. H. KAST and A. SCHMIDT (Gas- u. Wasserfach, 1927, 70, 821–822).—Whereas Wein's method of determining carbon monoxide by means of palladium chloride cannot be applied to mixtures containing ethylene or acetylene, Thiele's method, using an ammoniacal silver solution, is unaffected by the presence in the mixture of up to 2% of ethylene, or of acetylene provided its concentration does not exceed one tenth of the carbon monoxide content. Methane and hydrogen do not interfere with the method, which can therefore be used for determining carbon monoxide in technical gases, e.g., hydrogen for the synthesis of ammonia. W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of sulphuric acid. C. W. FIELDING (E.P. 274,918—9, 29.1.26).—(A) Gases containing sulphur dioxide and free oxygen, with or without oxides of nitrogen and water vapour, are brought into intimate contact with nitrosylsulphuric acid or with sulphuric acid sufficiently concentrated to absorb oxides of nitrogen in a vessel consisting of a series of tubes or columns provided with mixing devices. The nitrated acid is continuously circulated and cooled, so that it acts as a true surface catalyst and oxidation proceeds in the liquid phase. (B) Intimate contact is made between the nitrosylsulphuric acid and gases containing sulphur dioxide and free oxygen by forcing the gases through

the acid contained in tubes, in which are supported perforated septa above and below the surface of the liquid, or the gases are forced to pass through fine films of the acid supported on perforated or slotted sieve plates so that a foam is produced. W. G. CAREY.

Producing active silicic acid. RING GES. CHEM. UNTERNEHMUNGEN M.B.H. (E.P. 254,726, 30.6.26. Conv., 1.7.25).—Silicon fluoride is treated with small quantities of water or steam, alone or mixed with gas or other vapour, the resulting silicic acid being then washed and moulded under pressure, and the adherent moisture removed by suction without depriving the product of its hygroscopic water content. H. ROYAL-DAWSON.

Recovery of phosphorus and hydrogenated compounds thereof in the form of phosphoric acid. URBAIN CORP. (E.P. 269,908, 20.4.27. Conv., 21.4.26).—Gases generated by the combustion of phosphorus are passed into a Gaillard tower where they meet and concentrate a phosphoric acid solution which has already undergone a first concentration in a heat exchanger; the gases then proceed to a coke container in which the phosphoric anhydride is hydrated. The heat generated by the combustion of the phosphorus is used to concentrate the solution of phosphoric acid, the water evaporated being used again for hydrating. The mass of water in circulation is maintained constant.

W. G. CAREY.

Treating superphosphates. A. GAILLARD (E.P. 269,921, 22.4.27. Conv., 22.4.26).—After the first drying process the superphosphate is stored for some time in a silo to allow any unfinished chemical changes within the mass to conclude. The material is then passed through the dryer a second time. A. R. POWELL.

Decomposition of salts of complex hydrofluoric acids. A. F. MEYERHOFER (E.P. 249,860, 23.3.26. Conv., 24.3.25).—The salts are injected in powdered form by means of gas into a heated chamber, the charge being kept in continuous motion during the operation, or the powdered charge is dispersed by injecting heated neutral or active gas or steam. Substances may be introduced for reacting with the decomposing salts or for producing gases for reacting therewith, and waste gases from the decomposition chamber may be used for heating or for producing compounds required for the decomposing process.

W. G. CAREY.

Oxidation of ferrous sulphate in solution. A. ELLIOTT (U.S.P. 1,636,296, 19.7.27. Appl., 31.3.26).—Air is passed through hot ferrous sulphate solution under pressure and at a temperature below 100°.

W. G. CAREY.

Manufacture of pure lead carbonate from crude lead sulphate. R. DALOZE (E.P. 273,660, 19.3.27. Conv., 3.7.26).—Crude lead sulphate, e.g., the slime from lead chambers, is treated with a solution of lead acetate and excess of calcium acetate, and filtered. The calcium sulphate is separated and milk of lime is added to the solution, forming basic lead acetate, which is decomposed with carbon dioxide, precipitating the lead carbonate, and leaving lead and calcium acetates in solution to be used again.

H. ROYAL-DAWSON.

Manufacture of titanium oxide. J. BLUMENFELD, Assee. of FABR. DE PROD. CHIM. DE THANN ET DE

MULHOUSE (E.P. 253,550, 11.6.26. Conv., 11.6.25).—Ilmenite ore is treated with sulphuric acid, the iron compounds are reduced to the ferrous state, and the solution is cooled to about 0° to crystallise out the ferrous sulphate. The mother-liquor is then subjected to hydrolysis and concentration for treating further quantities of ore.
H. ROYAL-DAWSON.

Process of mining insoluble boron compounds. H. BLUMENBERG, JUN. (U.S.P. 1,636,455—6, 19.7.27. Appl., 23.3.27).—(A) Hot calcium chloride solution is introduced into the calcium-boron deposit, thus disintegrating and dissolving a portion; the resulting magma is removed and cooled, whereby the dissolved compounds are precipitated. These are removed, leaving the calcium chloride solution with only a small content of calcium-boron compounds. (B) Hot ammonium chloride solution may be used in the same manner.

W. G. CAREY.

Conversion of oxides into anhydrous fused chlorides. I. G. FARBENIND. A.-G., and K. STAIB (E.P. 275,116, 5.3.27).—Finely-divided oxides, *e.g.*, oxides of calcium, strontium, magnesium, zinc, are introduced into a heated reaction chamber with the aid of and suspended in a mixture of chlorine and carbon monoxide, or carbon is mixed with the oxide and chlorine alone is used to introduce the oxide.

W. G. CAREY.

Production of alkali-metal sulphhydrates. E. E. NAEF, ASSR. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,636,106, 19.7.27. Appl., 26.1.24. Conv., 31.1.23).—See E.P. 214,358; B., 1924, 511.

Production of cobaltous acetate. H. SCHATZ, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,637,281, 26.7.27. Appl., 28.10.26. Conv., 27.11.25).—See E.P. 262,075; B., 1927, 601.

Dissolving a mixture of hafnium and zirconium phosphates and separating hafnium and zirconium. A. E. VAN ARKEL and J. H. DE BOER, ASSRS. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,636,493, 19.7.27. Appl., 4.1.26. Conv., 6.6.24).—See E.P. 235,217; B., 1925, 713.

Production of activated nitrogen and of oxides of nitrogen therefrom. W. A. BONE (U.S.P. 1,639,584, 16.8.27. Appl., 19.2.24. Conv., 28.2.23).—See E.P. 217,277; B., 1924, 712.

Drying agent (E.P. 272,173).—See I.

Carbon monoxide and hydrogen (E.P. 274,610).—See II.

VIII.—GLASS; CERAMICS.

Determination of the constitution of glasses. K. TABATA (J. Amer. Ceram. Soc., 1926, 9, 823—849).—The method used is based on experiments on the causes of devitrification of glasses. Devitrification is arbitrarily defined in five degrees, which are illustrated by photographs. The degree of devitrification varies directly with the amount of silica dissolved in the glass. The experiments were conducted on alkali silicates and on alkali lead silicates. The constitution of the former is $x\text{Na}_2\text{O}, 2\text{SiO}_2 + y\text{K}_2\text{O}, 2\text{SiO}_2 + z\text{SiO}_2$, and of ordinary flint glasses is $x(\text{R}_2\text{O}, 2\text{SiO}_2, \text{PbO}, 2\text{SiO}_2) + y(\text{R}_2\text{O}, 2\text{SiO}_2) +$

$z(\text{PbO}, 2\text{SiO}_2) + w\text{SiO}_2$. The dissolving powers of $\text{Na}_2\text{O}, 2\text{SiO}_2$ and of $\text{K}_2\text{O}, 2\text{SiO}_2$ for silica vary considerably, owing to the difference in the atomic structure of both atoms of sodium and potassium. In accordance with the constitution of glasses thus determined, glass-melting pots should be rich in either alumina or silica according to the solvent power of the glass for silica. F. SALT.

Resistance to abrasion of ceramic glazes: its control and methods of determination. A. C. HARRISON (J. Amer. Ceram. Soc., 1927, 10, 77—89).—The resistance to abrasion of ten brands of hotel ware was tested by allowing silica sand to fall through a funnel orifice 0.4 in. in length and 0.24 in. in width from a height of 40 in. on to the glazed surface, and measuring the degree of abrasion by means of a glarimeter. The resistance of the glazes was proportional to the percentage relative humidity of the atmosphere. A high degree of accuracy was obtained by this method when the humidity was maintained constant. A simpler method, suitable for works control, is described, in which abraded samples are referred to arbitrary standards. The effect on the resistance to abrasion of varying the silica and alumina content, the glaze thickness, and the firing treatment was studied on a number of experimental glazes. Insufficient data were obtained to indicate definitely the effect of varying the silica and alumina contents, but the resistance was inversely proportional to the thickness, and was markedly increased by a higher glaze fire. The type of body used was of secondary importance, the glaze composition and the temperature of the glaze fire being the chief factors. F. SALT.

Chromium red glaze. C. F. BINNS and E. CRAIG (J. Amer. Ceram. Soc., 1927, 10, 73—74).—The red colour of basic lead chromate has been successfully utilised in producing a red in- or under-glaze colour from a batch mixing of 200.60 pts. of red lead, 14.40 pts. of potassium dichromate, 6.12 pts. of soda ash, 25.80 pts. of clay, and 54.00 pts. of flint. The whole glaze was fritted, ground, and fired at cone 08. F. SALT.

Gas-fired enamelling furnaces for cast iron. H. L. READ (J. Amer. Ceram. Soc., 1926, 9, 797—804).—The relative efficiencies of gas-fired and electrically-heated enamel kilns are compared. Details of the construction are given, together with operating data, of a kiln of the surface-combustion type. F. SALT.

Defects in enamel due to cast iron. A. MALINOVSKY (J. Amer. Ceram. Soc., 1926, 9, 805—813).—To determine the cause of pinholing and blistering of enamel on cast iron, records were kept of changes in materials, methods, etc. Samples of castings were examined microscopically, and photomicrographs indicate that pinholing and blistering are due almost entirely to the condition of the carbon in the iron, and not merely to the presence of carbon or of other ingredients.

F. SALT.

Coloured [ceramic] bodies. C. F. BINNS and H. E. WARDNER (J. Amer. Ceram. Soc., 1927, 10, 75—76).—Experiments with different mixtures of raw materials led to the preparation of blue, green, red, brown, and yellow bodies of uniform shrinkage and porosity, which could be worked together successfully, and a suitable colourless, transparent glaze maturing at cone 04 was

evolved. The blue and green body mixtures were also used successfully as engobes on a body made up of the other three. F. SALT.

Impact and static transverse strength [modulus of rupture] of wet-process electrical porcelain. L. NAVIAS (J. Amer. Ceram. Soc., 1927, 10, 90—97; cf. B., 1926, 823).—Tests were made with a Charpy (pendulum) impact machine, adapted for testing porcelain specimens varying from $\frac{1}{2}$ to $1\frac{1}{8}$ in. in diameter, the support for the test pieces being variable. Results are given for cylindrical specimens of wet-process porcelain 6 in. long, tested across a 4-in. span. Specimens of $\frac{3}{4}$, 1, and $1\frac{1}{8}$ in. diameter gave an average value of 1.53 ft.-lb./sq. in., with an average deviation of 2.4%. Similar specimens were tested for modulus of rupture across a 4-in. and a 5-in. span; an average value of 11,000 lb./sq. in. was obtained in each case. F. SALT.

Gas-expansion porosimeters [for ceramic products]. A. E. MACGEE (J. Amer. Ceram. Soc., 1926, 9, 814—822).—Three types of porosimeters based on the principle of gas expansion are described, which are modifications of the original Washburn-Bunting type (cf. B., 1922, 253 A). A direct-reading porosimeter enables both the porosity and the apparent sp. gr. to be read directly from burettes; another, of the indirect-reading type gives accurate results with test pieces varying widely in shape and size; and, finally, an instrument which operates without the use of a vacuum pump, by means of which the porosity of full-sized bricks can be determined.

Laboratory apparatus for the refining of Pacific Northwest kaolin. R. J. CLARK (J. Amer. Ceram. Soc., 1927, 10, 98—108).—The various methods of purifying clay are briefly reviewed. Samples of kaolin from Washington State were washed in a laboratory plant consisting of a feeding hopper, a rotary blunger, a sand-settling tank, a cone classifier, and two large settling tanks. A small quantity of alkali was added during blunging. The minimum feeding rate of the hopper was 240 lb. of crushed kaolin per hour. From 30 to 38% of the crude kaolin consisted of china clay, the impurities being quartz, muscovite, partially decomposed felspar, and a small amount of ferruginous matter. F. SALT.

Refractories for oil-gas manufacture. H. J. KNOLLMAN (J. Amer. Ceram. Soc., 1926, 9, 860—887).—In generators of the two-shell type used for the manufacture of gas from crude oil, the refractories have to withstand alternating oxidising and reducing conditions, the latter strongly predominating. Other destructive factors are the deposition of carbon in the bricks, abrasion by hot gases, effect of long flames, abrupt changes of temperature, action of salt water, sulphur, and supersaturated steam. Bricks in the lower part of the generators must be hard-fired and highly refractory; those in the upper part must have a dense surface to resist the action of steam, hot gases, etc. In addition to fireclay bricks, other suitable materials are magnesite, silica, carborundum, fused magnesia, alumina, and spinel. The thermal properties of these materials are discussed. F. SALT.

Properties of refractories in the metallurgy of zinc. I. Raw materials and body mixtures now in use. E. S. WHEELER and A. H. KUECHLER (J. Amer. Ceram. Soc., 1927, 10, 109—131).—The results of a preliminary investigation of the properties of the raw materials used in making zinc retorts are reported. Confirmation of the following points was obtained: the properties of the clays varied but slightly; the use of old retort material as grog in making new retorts must be carried out with care; it must be free from slag and made from clay of good quality; a portion of the grog should consist of calcined flint clay of good quality; the presence of zinc lowers considerably the deformation points of fireclays and body mixtures. F. SALT.

Pickling iron for enamelling purposes. STALEY.—See X.

PATENTS.

Furnace for melting and refining glass etc. J. S. SHAW (U.S.P. 1,636,151, 19.7.27. Appl., 16.6.24. Conv., 23.8.23).—See E.P. 224,622; B., 1925, 100.

Drawing of pieces of silica glass or any other vitreous material. QUARTZ & SILICE (E.P. 270,692, 29.4.27. Conv., 4.5.26).

IX.—BUILDING MATERIALS.

Preliminary petrographic study of Portland cement. J. L. GILLSON and E. C. WARREN (J. Amer. Ceram. Soc., 1926, 9, 783—786).—A petrographic study was made of five common brands of Portland cement clinker. In each case more than 95% of the material consisted of three crystalline constituents, two of which greatly predominated. The optical properties of these three constituents were determined and were found not to agree with any of the compounds of the $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ system produced artificially by Rankin and Wright. One of the three crystalline compounds, which is biaxial, optically positive, and has indices of refraction of 1.718, 1.720, and 1.733, was also found in the set concrete, proving it to be inert and unaffected by water, a fact which was confirmed by X-ray examination. F. SALT.

Wood preservation. Production of acid by wood-rotting fungi. L. P. CURTIN (Ind. Eng. Chem., 1927, 19, 878—881).—The difficulty in finding a satisfactory inorganic wood preservative lies in the fact that toxicity implies solubility and that soluble salts cannot be very permanent. If, however, a sparingly soluble salt can be dissolved by some secretion of the fungus, the dilemma may be avoided. Tests made in nutrient jelly with *Fomes annosus* and other fungi and also moulds, with suitable indicators, showed a development of acid. Sodium alizarinsulphonate was the most suitable indicator. The result was confirmed with wood which had been neutralised and sterilised. Calcium and strontium carbonates precipitated in agar-syrup gels were dissolved by acids produced in this way. The acid solution is of approximately p_{H} 5. C. IRWIN.

PATENTS.

Manufacture of artificial stone. M. HARNISCH (E.P. 271,108, 13.5.27. Conv., 14.5.26).—Residue or waste of asbestos material, e.g., serpentine, is treated

with dilute sulphuric acid (d 1.02—1.04), and kept for some time; it is then mixed with a binder, *e.g.*, sodium silicate and calcium chloride, or tar, forming a plastic mass, which is moulded to shape and dried in the air.

H. ROYAL-DAWSON.

Production of heat-insulating material. H. and J. SCHEIDEMANDEL (E.P. 253,919, 17.6.26. Conv., 18.6.25).—A hot solution of aluminium sulphate or zinc sulphate is stirred into a hot aqueous suspension of calcium or barium hydroxide, to which asbestos, magnesia, or kieselguhr may be added. The viscous mixture is poured into moulds to harden, and then dried.

H. ROYAL-DAWSON.

Production of a cold glaze upon building material, such as concrete, sandstone, clay, brickwork, etc. K. FRIEDRICH (U.S.P. 1,636,860, 26.7.27. Appl., 8.10.21).—See E.P. 190,267; B., 1923, 146 A.

Wood preservation. A. M. HOWALD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,638,440, 9.8.27. Appl., 23.1.24).—See E.P. 228,119; B., 1925, 284.

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

Proposed theory of the hardening and tempering of steels. O. E. HARDER and R. L. DOWDELL (Trans. Amer. Soc. Steel Treat., 1927, 12, 51—68).—The mechanism of recrystallisation from γ - to α -iron on cooling is discussed. When the rate is slower than that required to produce maximum hardness, there is a slight coalescence of the carbide, the particles of which are not stable enough to resist acid reagents and the specimen etches dark, giving troostitic microstructure. Martensite needles follow the octahedral planes in austenite, which are those of greatest atomic concentration and interplanar distance, and therefore those of easy slip. The austenite previous to recrystallisation has been stressed sufficiently to cause slip. After quenching in oil, the stresses are insufficient to produce slip and more austenite is retained. The hardness of martensite may be due to intra-atomic forces, the extremely fine α grain-size, the saturation of the α lattice with carbon atoms, the distortion of the lattice caused by stress, and the interference to slip caused by the martensite needles. The evolution of heat in a quenched steel may be due to the shrinkage of the expanded space lattice.

T. H. BURNHAM.

Effect of silicon, nickel, chromium, and tungsten on the hardening of tool steel. W. HAUFFE (Stahl u. Eisen, 1927, 47, 1365—1373).—The number of annealings at temperatures between 780° and 1030° followed by quenchings in water at 0° before hardness cracks begin to appear in a hypereutectoidal steel (1.2% C) to which varying amounts of silicon, manganese, nickel, chromium, and tungsten have been added, has been determined, and the results are reproduced in graphical form, together with the corresponding volume changes. With increasing silicon content, the tendency to fracture on repeated hardening is increased at the higher temperatures, but high-silicon steels may be annealed up to 930° and quenched in oil without showing hardness cracks. Manganese decreases the hardening range of hypereutectoidal steels very considerably, so that, by hardening

at the usual temperatures, there is a tendency towards the production of cracks after quenching. Hypereutectoidal steels with a sufficiently high manganese and silicon content behave like plain carbon steels when hardened at 780°, and better than the plain steel when hardened at 830°. Up to 1% Cr decreases the tendency of high carbon steels to crack during hardening; with 1—2% Cr these steels become extremely hard without, however, losing their toughness, and hardness cracks may be avoided by quenching from 930° in oil. Nickel has scarcely any action on the hardening properties of hypereutectoidal steel, and the action of tungsten is uncertain owing to the separation of carbide during annealing.

A. R. POWELL.

Effect of heat treatment on the combined carbon in grey cast iron. E. L. ROTH (Trans. Amer. Soc. Steel Treat., 1927, 12, 27—40).—Samples from two grey cast irons $\frac{3}{8}$ in. thick, containing 3.52 and 3.25% of total carbon, respectively, when heated at temperatures from 625° to 925° for $\frac{1}{2}$ —3 hrs., showed that the combined carbon reached a maximum at 760°. At this temperature very little increase in precipitated graphitic carbon was obtained by heating longer than $\frac{1}{2}$ hr., but the same amount of graphitisation was obtained by heating for longer periods at lower temperatures. Silicon did not have a marked effect on graphitisation. Microscopic examination showed that the formation of graphitic carbon was not propagated from the edges to the centre. Little change occurred in the amount of total carbon. It is considered that, on heating, the Fe_3C in the matrix begins to decompose into iron and graphite, and up to 760° the iron can only dissolve graphite to a limited extent, but is capable of dissolving the graphite above this temperature, so that a minimum amount of Fe_3C is found at this point.

T. H. BURNHAM.

Theory of pickling of sheet iron and steel for enamelling purposes. H. F. STALEY (J. Amer. Ceram. Soc., 1926, 9, 787—796).—Efficient pickling depends on rapid dissolution, with copious evolution of hydrogen, of a thin layer of iron immediately beneath the scale. The rate of pickling depends upon the amount of acid present, the proportion thereof which is ionised, and the mobility of the ions. The most effective concentrations of acids lie between 15% and 20%. The viscosity of acid solutions is decreased, and the particles are rendered more mobile by heating. Hydrochloric acid is more effective than sulphuric; it acts more rapidly, with dissolution of less iron. The addition of small amounts of sodium chloride has a marked effect upon the pickling action of sulphuric acid. The chief object of adding auxiliary substances is to reduce the amount of acid fumes from pickling vats, and thus permit the use of highly efficient concentrations of acid. Hydrogen absorbed by metals may be removed by immersing the metals in boiling water for a few minutes.

F. SALT.

Analysis of iron and steel. Determination of silicon, phosphorus, sulphur, and manganese. M. MARQUEYROL and L. TOQUET (Ann. Chim. analyt., 1927, [ii], 9, 225—233).—10 g. of the sample are dissolved in 80 c.c. of nitric acid (d 1.33), to which are added, in small quantities at a time, 5 g. of potassium chlorate. The solution is evaporated to dryness and the nitrates

are converted into chlorides by evaporation with hydrochloric acid. The silica is removed by filtration and determined as usual; the filtrate is diluted to 500 c.c. in a graduated flask, and 250 c.c. are used for the determination of sulphur, 150 c.c. for manganese, and the remainder (measured accurately) for phosphorus. The last portion is heated to 70–80°, treated with 60 c.c. of molybdate mixture, and just neutralised with ammonia. The yellow precipitate is collected, washed, and dissolved in ammonia, the solution treated with 10 c.c. of 35% ammonium nitrate, 2 c.c. of nitric acid, and 5 c.c. of molybdate mixture, and the resultant yellow precipitate collected, washed, dried, weighed, and calculated to phosphorus. For the determination of sulphur, 20 c.c. of 12% barium chloride solution are added to the solution, which is then boiled, set aside for 12 hrs., and filtered, the precipitate being washed with 10% hydrochloric acid containing 1% of barium chloride, then with water, dried, ignited, and weighed. Manganese is determined by converting the chloride solution back into nitrate by repeated evaporation with nitric acid, precipitating the manganese as dioxide by boiling with 2 g. of potassium chlorate, and dissolving the precipitate in an excess of standard hydrogen peroxide.

A. R. POWELL.

Iron-carbon-vanadium alloy for Brinell balls. G. W. QUICK and L. JORDAN (Trans. Amer. Soc. Steel Treat., 1927, 12, 3–26).—10 mm. balls of iron-carbon-vanadium alloy containing 2.93% C and 13.5% V, quenched from 850° in water, tempered at 100°, and work-hardened were used in testing hardened steels in comparison with ordinary Brinell balls and worked-hardened and etched Hultgren balls. The flattening of the alloy balls was least and the ordinary balls showed the greatest deformation. For testing steels up to 700 Brinell hardness, however, the alloy balls offered no appreciable advantage over Hultgren balls. When testing hard steels, balls deform both elastically and plastically, the former type of deformation playing an important part. For very hard materials, therefore, a ball with greater resistance to elastic deformation such as corundum or diamond is necessary.

T. H. BURNHAM.

Aluminium-silicon alloy "alpax," and its applications. L. GUILLET, DE FLEURY, and S. DE LAVAUD (Compt. rend., 1927, 185, 327–329).—Alpax is a light alloy of silicon and aluminium in eutectic proportions (13% Si), which is used for making moulded articles on account of its small shrinkage after quenching. The microscope shows a fine structure of silicon grains in aluminium, but annealing even at low temperatures produces coalescence and impairs its mechanical properties. Alpax may be refined before pouring by means of the alkali fluorides and chlorides, and its mechanical properties then depend on the temperature of pouring, the rate of cooling, and the amount of sodium present.

J. GRANT.

Influence of sulphur on copper. P. SIEBE (Z. Metallk., 1927, 19, 311–315).—The presence of sulphur in copper leads to the formation of sulphur dioxide during melting and casting, whereby the castings rise in the mould and are extremely porous. If the sulphur is present as cuprous sulphide and oxidising conditions

are carefully avoided during melting, sulphur has a much less deleterious effect on the mechanical properties of the metal than has oxygen owing to the greater softness and plasticity of cuprous sulphide compared with cuprous oxide. Up to 0.12% S has very little effect on the mechanical properties of copper: larger proportions of sulphur increase the hardness and tensile strength very slightly but appreciably reduce the resistance to impact and the ductility. The rate of change of the values for the various mechanical properties is, however, much less for increasing amounts of copper sulphide than for the corresponding quantities of cuprous oxide.

A. R. POWELL.

Heat treatment and structure of $\alpha + \beta$ -brass. R. HINZMANN (Z. Metallk., 1927, 19, 297–303).—The effect on the structure of an $\alpha + \beta$ -brass (50% Cu, 2% Pb, 48% Zn) of extrusion at 720° followed by annealing at various temperatures has been investigated. As the extension was effected just within the β range, and during passage through the press the metal cooled below this range into the field of duplex structure, the microstructure varied from one end to the other of the pressed rod. The first portion passing through the press completely as β showed on cooling evenly distributed needle-like crystals of β in a ground-mass of α , the middle portion passing through the press at about the decomposition point of β showed some of the β needles to have been broken up or elongated in a longitudinal direction, and the end portion of the rod which had been extruded below the decomposition point of β had a granular structure composed of evenly distributed fragments of β needles in a ground-mass of α . The mechanical properties of the end portion of the rod were therefore less satisfactory than those of the first portion, and this defect could not be completely eliminated by annealing again at 750–800° as the middle portion had a higher recrystallisation temperature and formed coarser crystals than the end portion. Characteristic photomicrographs of the different structures obtained are given.

A. R. POWELL.

Separation of vanadium from tungsten. S. G. CLARKE (Analyst, 1927, 52, 466–467).—In the presence of large and moderate amounts of tungsten good results for vanadium may be obtained by adding to the solution 10 c.c. of hydrofluoric acid, neutralising with ammonia, adding 20 c.c. of hydrochloric acid, diluting to about 300 c.c., and precipitating with cupferron (1 g. in about 20 c.c. of water). The precipitate is filtered off after 15 min. on a pulp filter, washed with dilute cupferron solution containing a few drops of 1:3 sulphuric acid, and burnt off in a platinum dish. If the vanadium pentoxide can be kept unfused it may be dissolved in concentrated sulphuric acid, diluted to 200 c.c., and after adding 20 c.c. of saturated sulphur dioxide solution, boiled for 20 min. and titrated with 0.1N-permanganate at 80°. If the oxide is fused it is brought into solution with fusion mixture. Impure tungstic oxide, as separated from steel, may be dissolved in sodium hydroxide or ammonia and boiled, and the remaining undissolved ferric hydroxide collected on the filter, washed, dissolved in hydrochloric acid, and added to the main steel solution. The vanadium is then recovered as above.

D. G. HEWER.

Electrolytic deposition of chromium. F. SALZER (Z. Elektrochem., 1927, 33, 309).—Contrary to the statement by Liebreich (cf. A., 1927, 322), the author's chromium plating process is used commercially.

H. J. T. ELLINGHAM.

Economic and social development of the American iron and steel industry. T. W. ROBINSON (Iron and Steel Inst., Sept., 1927. Advance copy. 19 pp.).

Refractories in metallurgy of zinc. WHEELER and KUECHLER.—See VIII.

PATENTS.

Protecting iron or steel melts in furnaces or converters from oxidation and adsorption of gases. B. ZWIEBEL (E.P. 275,002, 28.6.26).—A reducing atmosphere is formed over the surface of the slag of an iron or steel melt by introducing a heavy hydrocarbon, e.g., coal tar, in either a solid or atomised condition, into the furnace at an appropriate time towards the end of the process. Adaptations suitable for Siemens-Marten furnaces and converters are described. C. A. KING.

Making malleable iron castings. H. A. SCHWARTZ, ASSR. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,636,657, 19.7.27. Appl., 7.12.26).—In making malleable castings of low silicon content, a compound which tends to increase graphitisation is added to the iron, and the metal is cast with minimum separation of carbon. The casting is afterwards annealed to cause precipitation of finely-divided carbon. C. A. KING.

Manufacture of moulded castings of non-rusting chromium-nickel steel. F. KRUPP A.-G. (E.P. 266,305, 22.1.27. Conv., 18.2.26).—For making small castings of a nickel-chromium steel fragments of the steel are charged into a hopper above the mould, covered with a deoxidising flux, and melted down by means of an oxy-hydrogen flame in which a large excess of hydrogen is used. As soon as the metal is melted the mould is rotated rapidly so as to force the molten metal into all the contours of the mould. A. R. POWELL.

Purifying iron scale. A. LUCAS (E.P. 275,099, 14.2.27).—For the production of iron oxide suitable for use in aluminothermic mixtures, mill scale is crushed between rollers which are not under sufficient pressure to crush impurities harder than the scale. The crushed scale is then screened or treated with an air blast. C. A. KING.

Selective flotation of minerals from crude ores. O. WISER (U.S.P. 1,636,974, 26.7.27. Appl., 21.6.26).—Ore pulp containing lead, zinc, and iron sulphides is treated with sulphur dioxide (0.05—1.50 lb. of sulphur per ton of ore), and lead sulphide is floated preferentially from an approximately neutral pulp. C. A. KING.

Treatment of ores, wastes, oxides, metals, and other substances. M. FOURMENT (E.P. 265,548, 3.12.26. Conv., 2.2.26).—Metallic values are obtained by volatilisation by means of high-frequency currents induced by a winding not containing iron. Secondary crucibles may be used for fractional condensing purposes. A convenient apparatus consists of a deep cylindrical crucible into the bottom of which the raw material is fed by a central vertical tube. In the annular space formed landing stages may be provided with run-off

pipes for the separation of volatilised components. The degree of fractional distillation is regulated by separately winding the different vertical stages of the column or crucible. C. A. KING.

Sintering refractory materials [e.g., zirconium]. N. R. DAVIS, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (E.P. 274,283, 26.7.26).—The powdered refractory metal, e.g., zirconium, is pressed into a rod and heated *in vacuo* in a wire-wound resistance furnace at 350° to drive off occluded gases at a temperature below that at which they combine with the metal. The temperature is then raised to 800—1000° and heating continued until all volatile matter is expelled. After cooling in the vacuum the partly sintered mass is transferred to another furnace in which it is heated to the sintering temperature by passing a current through it or by induction. The final heating may be conducted in a vacuum or in an atmosphere of an inert gas either at the ordinary or at a reduced pressure. A. R. POWELL.

Metallurgical furnaces. CARBORUNDUM Co., LTD., ASSEES. of C. E. HAWKE (E.P. 257,261, 16.8.26. Conv., 20.8.25).—The refractory lining is composed of a material, such as silicon carbide, many times as heat-conductive as fireclay, and is made of a thickness such that heat is conducted through it at a rate which ensures the formation upon it of a congealed protective layer of the fused furnace contents. The lining may be built up of blocks shaped to provide passages for the circulation of a cooling medium (air). To conserve the heat conducted through the lining the circulated air is subsequently supplied to the tuyères or otherwise utilised. H. HOLMES.

Furnace for treating refractory ores containing precious metals. G. W. B. EVANS, ASSR. to EVANS ORE REDUCTION Co. (U.S.P. 1,636,467, 19.7.27. Appl., 13.8.26).—Ores are treated in an atmosphere of hydrogen in a rotating cylindrical chamber which is enclosed in an outer chamber also filled with hydrogen. C. A. KING.

Ore furnace. F. M. SIMONDS, ASSR. to A. F. HYDE (U.S.P. 1,637,163, 26.7.27. Appl., 19.2.25).—An ore-treating furnace consists of a number of superimposed distributing chambers having perforated walls. A second perforated ore-confining wall surrounds the distributing chambers to form a space for descending ore. Different qualities of gas may be supplied to the different chambers of the furnace. C. A. KING.

Metallic composition. H. BOVING, ASSR. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,636,763, 26.7.27. Appl., 26.5.20).—When making an alloy containing metals having a low volatilisation temperature, the finely-divided components are mixed and melted under sufficient pressure of an inert gas to inhibit volatilisation of the volatile components. C. A. KING.

Fluxes for soldering. J. BLACKFORD (E.P. 274,693, 16.11.26).—A flux for use with iron, brass, etc. consists of anhydrous zinc chloride 1 pt., finely ground ammonium chloride 8 pts., and sodium fluoride 1½ pts. H. ROYAL-DAWSON.

Welding copper and aluminium. J. V. CAPICOTTO, ASSR. to DUBILIER CONDENSER CORP. (U.S.P. 1,636,656, 19.7.27. Appl., 10.6.24).—Aluminium is brought into

contact with copper coated with solder and the junction spot-welded. C. A. KING.

Steel of high-temperature stability. W. R. SHIMER, Assr. to BETHLEHEM STEEL CO. (Re-issue 16,683, 19.7.27, of U.S.P. 1,624,075, 12.4.27).—See B., 1927, 415.

Furnace for heating metal sheets etc. BRITISH FURNACES, LTD., and E. W. SMITH (E.P. 275,513, 23.3.27).

Fuel [for welding etc.] (U.S.P. 1,637,187).—See II.

Coating metals (E.P. 274,333 and 274,355).—See XIII.

XI.—ELECTROTECHNICS.

Strength of electrical porcelain. NAVIAS.—See VIII.

Chromium. SALZER.—See X.

PATENTS.

Oxide cathodes. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABRIEKEN (E.P. 274,981, 6.5.26).—A metal wire, *e.g.*, of platinum or platinum alloy, preferably coated with an alkaline-earth oxide, is wound helically on a core of highly refractory metal, preferably a poor conductor of heat, *e.g.*, tungsten or molybdenum.

J. S. G. THOMAS.

Electrical heating-body for high temperatures especially for ceramic, metallurgical, and chemical processes. F. WECKERLE, Assr. to STUDIENGES. FÜR WIRTSCHAFT U. IND.M.B.H. (U.S.P. 1,637,167, 26.7.27. Appl., 24.8.25. Conv., 28.8.24).—See E.P. 239,222; B., 1925, 927.

[Cooling device for use when] coating articles with metal in vacuum by electrode dispersion. W. A. F. PFANHAUSER (E.P. 275,523, 14.4.27).

Apparatus for analysis (E.P. 252,207).—See I.

Conversion of heavy hydrocarbon oils into lighter oils (E.P. 275,120).—See II.

XII.—FATS; OILS; WAXES.

Trichloroethylene as a solvent in the determination of fats. J. GROSSFELD (Chem.-Ztg., 1927, 51, 617—618).—The advantages of trichloroethylene as compared with ether are enumerated, and a formula is given for determining the quantity of fat in a simple mixture by direct extraction with the solvent and weighing the residue left after evaporation of the solvent from an aliquot proportion of the solution. S. I. LEVY.

Experiments on olive oil at Ghaba. M. ROUSSEAU (Bull. Mat. Grasses, 1927, 223—231).—During 1925—1926 experiments were conducted by the Department of Agriculture of Tunis to test the effect of decantation by centrifuges on the quality and preservation of olive oil. Samples of oil separated from the pulp by the older decantation method of ladling, by the Hignette, Sharples, and Alfa-Laval supercentrifuges respectively, were examined both before and after filtration in respect of acidity, taste, and power of keeping. The results prove that the centrifugal separation of the oil does not provoke any change in the quality of the resultant products, and that the keeping quality and taste of the oil are not less inferior to those of oil produced by the older methods

of separation. The results for oil separated by decantation by ladling, by the Hignette centrifugal filter, and by the Sharples and Alfa-Laval supercentrifuges respectively were: acidity 0.5, 0.5, 0.45, 0.45%; water, 1.7, 1.6, 0.36, 0.40%; mineral matter and other impurities, 0.32, 0.18, 0.04, 0.04%. H. M. LANGTON.

Action of heat and blowing on linseed and perilla oils and glycerides derived from them. J. S. LONG, W. S. EGGE, and P. C. WETTERAU (Ind. Eng. Chem., 1927, 19, 903—906).—The synthesis of linolenic monoglyceride from linseed and perilla oils is described, the products being fairly pure and showing association in benzene. Very little difference exists between the glyceride obtained from linseed oil and that from perilla oil. On heating at 225° linolenic monoglyceride apparently changes to an isomeride which does not form a solid hexabromide. At 250°, however, this isomeric change is masked by changes producing rapid increase in mol. wt. and decrease in iodine value. The glyceride rapidly decomposes at 293°. The reactions produced by blowing and by heating oils are discussed and contrasted. S. S. WOOLF.

Effect of foreign oleaginous seeds, when crushed with flax seed, on the drying and bodying properties of linseed oil. W. H. EASTMAN and W. L. TAYLOR (Ind. Eng. Chem., 1927, 19, 896—897).—Raw linseed oil pressed from flax seed containing 6% of dockage (rich in small oleaginous seeds) was compared with oil pressed from the same seed after removal of all the foreign seeds by means of suitable cleaning machinery. Differences in the constants, particularly the iodine value and n_D^{25} , are noticed in the raw oils, and are maintained or accentuated when the oils are boiled, blown, heat-bodied, bleached, refrigerated, etc. S. S. WOOLF.

Chaulmoogra-group oils. II. Refining and isolation of hydnocarpic acid. G. A. PERKINS, A. O. CRUZ, and M. O. REYES (Ind. Eng. Chem., 1927, 19, 939—942).—*Hydnocarpus wightiana* oil is preferable to chaulmoogra oil both as to cost and quality, and in a refined state is very suitable for use in the treatment of leprosy. Details of the refining of the oil, chiefly to remove free fatty acids, are given. The preparation of hydnocarpic acid in unprecedented yield (150 g. of esters per kg. of oil) and quality from *H. wightiana* oil is described, nearly pure ethyl hydnocarpate being obtained by fractionating the ethyl esters of the oil. Characteristics of some minor oils of this group are also tabulated. S. S. WOOLF.

Determination of some physical constants of marine animal oils. H. MARCELET (Compt. rend., 1927, 185, 455—457).—The heats of combustion, ignition temperatures, densities, fluidities, and viscosities of twenty-nine marine animal and fish oils have been determined. C. W. GIBBY.

Detection of whale oil. M. TSUJIMOTO (Chem. Umschau, 1927, 34, 217).—A reply to Davidsohn's criticism (B., 1927, 303) of the author's suggested method of detection of whale oil (B., 1926, 986), pointing out probable causes of the discrepancies. E. HOLMES.

Origin of the yellow colour of beeswax. G. F. JAUBERT (Compt. rend., 1927, 185, 405—406). In view

of the predilection of bees for mignonette, yellow-weed (*R. luteola*), heather, parsley, etc., it is probable that the 1:3-dihydroxyflavone, m.p. 285°, previously observed in beeswax (B., 1927, 707) is accompanied by traces of other derivatives of this dye (cf. Rivière and Bailhache, Ann. Sci. agron., 1920, 38, 82). R. BRIGHTMAN.

Marine-animal and fish oils for motors. LUMET and MARCELET.—See II.

Action of cathode rays on drying oils. LONG and MOORE.—See XIII.

Oil in soya beans. COLE and others.—See XVI.

Cacao butter substitutes. KNAPP and others.—See XIX.

PATENTS.

Hydrolysis or saponification of glycerides or other organic esters. V. R. KOKATNUR (E.P. 251,290, 23.4.26).—Glycerides or other esters are treated with a substantially anhydrous saponifying agent in the presence of an organic liquid diluent, *e.g.*, kerosene, that is chemically inert to the materials present, and is insoluble in and a non-solvent for the resulting alcoholic component, the latter and the diluent being subsequently recovered by distillation at the combined partial pressures of the two. S. S. WOOLF.

Production of emulsifying agents. J. H. DISNEY and J. C. KERNOT (E.P. 274,142, 15.1.26).—An emulsifying agent for use with bituminous substances, oils, etc. consists of glue or gelatin (which has been purified by washing, treating repeatedly with alkali, rewashing, and heating repeatedly with dilute acid), mixed with saponifiable material, *e.g.*, resin, tallow, vegetable or animal oil, at a temperature of 40–50°. A suitable resinous material is the liquid resin obtained in the manufacture of sulphite wood pulp, a preferred mixture being 1 pt. of treated glue, 1 pt. of water, and 2 pts. of liquid resin. S. S. WOOLF.

Decomposing emulsions in the splitting of fats and oils, and recovering the splitting agent. G. PETROFF (E.P. 274,741, 1.2.27).—The emulsions formed in the splitting of fats and oils by various sulpho-acids are broken by the addition of 0.1–0.5% of calcium sulphate. The sulpho-acids may be regenerated from their calcium salts or double salts with calcium sulphate formed in the process, or the salts themselves may be used as splitting agents. S. S. WOOLF.

Production of margarine (E.P. 274,949).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Absorption of ultra-violet light by paint vehicles. G. F. A. STRUTZ (Ind. Eng. Chem., 1927, 19, 897–901).—The ultra-violet transmission of thin dry films of various oils, varnishes, and lacquers, as well as the change in absorption caused by exposure of the films to sunlight and to the mercury arc, were measured, using a quartz spectrograph. A number of continuous spectrograms of the tungsten spark under water, taken through wet and dry, raw and treated, linseed oil films are also given. Raw linseed oil is quite transparent to ultra-violet light; heat-bodied oils and heavy-bodied air-blown oils are more opaque. Varnishes are quite opaque, but clear

nitrocellulose is transparent, opacity being afforded by adding plasticisers and resins. The effect of ultra-violet light on the various films is discussed, with special reference to weathering tests. S. S. WOOLF.

Action of cathode rays on drying oils. J. S. LONG and C. N. MOORE (Ind. Eng. Chem., 1927, 19, 901–903).—The changes in n_D , iodine value, mol. wt., and hexabromide value when thin oil films were exposed to high-voltage cathode rays for various periods are tabulated for linseed, perilla, and tung oils that had been subjected to preliminary treatment, *e.g.*, heating, blowing, boiling with driers. Mol. wt. increases and iodine value decreases usually on exposure to the rays, whilst n_D increases for linseed and perilla oils but decreases for tung oil, pronounced bleaching occurring in all cases. The improvement in drying time observed is attributed to isomeric change induced by the cathode rays. S. S. WOOLF.

Zinc compounds as driers. F. WILBORN (Farben-Ztg., 1927, 32, 2654–2655).—The statements by Rhodes and others (cf. B., 1923, 1233 A; 1926, 201) that zinc compounds do not increase the rate of absorption of oxygen by linseed oil are not confirmed. The use of zinc driers in conjunction with lead or cobalt driers is recommended, accelerated drying action and improved mechanical properties of the film resulting, but manganese-zinc combinations were not satisfactory. S. S. WOOLF.

Mechanical properties of varnish films etc. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1927, 32, 2708–2710).—The conclusions to be drawn from measurements of tensile strength, elasticity, etc. of detached films are discussed, the importance of changes in these properties with age being stressed. The preparation of films by the application of the varnish etc. to be examined to coloured gelatin foil which is dissolved away with warm water when the coating is dry, is preferred to the other available methods, and details are given. The method of expressing results is described and some typical curves are reproduced, illustrating the loosening of structure undergone by a film on exposure to ultra-violet light, and the irregular extension of a nitrocellulose lacquer. S. S. WOOLF.

Degree of saturation of the resin acids. III. B. M. MARGOSCHES, K. FUCHS, and W. RUZICZKA (Chem. Umschau, 1927, 34, 215–217; cf. B., 1927, 228, 371).—Mainly a theoretical discussion of the mechanism of the production of halogen acids during the determination, by various methods, of the iodine values of resin acids and allied substances. It is considered that the production of acids in the Hübl and Wijs methods is a secondary reaction resulting in the incipient re-formation of the double linking, but probably a primary reaction in the Margosches-Hinner-Friedmann rapid method (*loc. cit.*). Further evidence, based on the determination of "Plus-säure" values, is adduced to demonstrate the analogy between pinene and abietic acid. E. HOLMES.

Linseed oil. EASTMAN and TAYLOR. **Linseed and perilla oils.** LONG and others.—See XII.

PATENTS.

Bituminous paints and like coating and impregnating compositions. COLAS PRODUCTS, LTD., and

F. LEVY (E.P. 274,955, 28.4.26).—Paints or impregnating compositions containing the reversed bitumen-in-water emulsions or jellies described in E.P. 251,323 (B., 1926, 595) are claimed. Either oil or water paints may be incorporated with the emulsion, reversal of which may take place before or after the incorporation.

S. S. WOOLF.

Coating metal and like articles. K. LOOS, M. LEHNIG, C. HENNING, and R. DASSDORF (E.P. 274,333, 15.11.26, and 274,355, 11.1.27).—(A) A smooth, chemically resistant coating is given by a mixture of artificial resin, water-glass, clay or metallic oxides, and silicic acid compounds, the other components being added to the artificial resin at any stage before the final hardening. (B) The final hardening is carried out in the absence of air, *e.g.*, in a bath of oil of high b.p.

S. S. WOOLF.

Synthetic resins of the phenol-aldehyde type. A. A. DRUMMOND (E.P. 274,581, 26.4.26).—Phenol-aldehyde resins, made by any known method and whether initially in solution or not, are treated with the vapour of an organic solvent of lower b.p. than water, and preferably one with which the resin is miscible, *e.g.*, alcohol, until no further free phenol is removed. The solution of purified resin may be used as such or may be heated *in vacuo* at a temperature below 80° to remove the solvent, which is subsequently recovered.

S. S. WOOLF.

Manufacture of moulded phenolic compositions. E. C. R. MARKS. From BAKELITE CORP. (E.P. 275,011, 12.7.26).—"Cured" moulded phenolic compositions are subjected to further heating for 48–80 hrs. at 125–135°, the dielectric strength of the product at 100° being increased by the treatment to at least 50% of that at 20°.

S. S. WOOLF.

Manufacture of condensation products of carbamide or its derivatives and formaldehyde. F. POLLAK (E.P. 248,729, 17.2.26. Conv., 3.3.25).—Hydrophobic resins are obtained when organic compounds capable of forming resinous condensation products with formaldehyde in an acid medium, *e.g.*, thiocarbamide, phenol, are added to the initial condensation product of carbamide etc. and formaldehyde (*i.e.*, before the "gelatinisation phase" of the first stage commences) and the mixture is further heated in the presence of free hydrogen ions until, on cooling, the resin is precipitated. The mixture may be neutralised or rendered slightly alkaline before cooling since resins produced in this manner retain their hydrophobic qualities in both acid and alkaline solutions.

S. S. WOOLF.

Manufacture of condensation products from carbamide or a derivative thereof and formaldehyde. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 253,094, 21.5.26. Conv., 5.6.25).—The products are condensed in the presence of active carbon, which is removed at any desired stage of the condensation. An alcohol, ketone, ether, or ester, or mixture of any of these may be present as a retarding agent or diluent, particularly in the conversion of a product at a lower stage of condensation into one at a higher stage in the presence of acid. Effective control of the various stages of the process may be obtained by carrying out the condensation at ordinary temperatures.

S. S. WOOLF.

Production of resinous condensation products from amines of the aromatic series. [Resists for batik dyeing.] A. L. MOND. From I. G. FARBENIND. A.-G. (E.P. 274,155, 15.3.26).—By condensing an arylamine with formaldehyde in aqueous alcohol in presence of an arylamine salt (up to 10% of the total amine used) the yield of resin is increased and the softening point of the product is raised by an amount depending on the proportion of salt present. Technical formaldehyde contains formic acid, and mineral acid or formic acid may be added to make up the desired quantity. The amount of amine salt necessary is less at b.p. than at lower temperatures. The softening point of the resin is further raised by using 25–50% excess of aldehyde. Volatile matter may be removed in a vacuum. The resins, being stable to alkali, are suitable as resists in batik dyeing, and may be removed from textiles by means of organic solvents, weak acids, or hot water; resins softening below 60° are preferred for this purpose.

C. HOLLINS.

Manufacture of a cement suitable for linoleum etc. A. B. CRAVEN, C. S. BEDFORD, and YORKSHIRE DYEWARE & CHEM. CO., LTD. (E.P. 274,300, 30.8.26).—From 20 to 60% of rosin with or without the addition of other suitable resins is incorporated with waste or unused vulcanised rubber and the mixture heated at 110–135°, a definite change taking place in the rubber permitting the formation of a completely homogeneous plastic cement which may be blended with cork, wood, pigments, etc. in the manufacture of linoleum. The rubber may be softened by the use of organic solvents, *e.g.*, xylene, and such other softening agents as "sharp oil," "dead oils," tricresyl phosphate, vegetable oils, etc.

S. S. WOOLF.

Manufacture of a cement suitable for linoleum etc. A. B. CRAVEN, C. S. BEDFORD, and YORKSHIRE DYEWARE & CHEMICAL CO., LTD. (E.P. 274,722, 3.1.27. Addn. to E.P. 274,300; cf. preceding abstract).—The semi-fluid resinous material obtained from gutta-percha or from balata by extraction with light petroleum etc. or the solid resin obtained from the semi-fluid by oxidation at 100° is incorporated with vulcanised rubber in accordance with the original patent.

S. S. WOOLF.

Preparation of pigments and paints. J. BLUMENFELD, ASST. to BLUMANN RARE EARTHS CHEMICAL CO. (U.S.P. 1,639,423, 16.8.27. Appl., 22.4.26. Conv., 22.4.25).—See E.P. 256,302; B., 1926, 889.

Production of coats of lacquers, lacquer and basic oil paints, etc. A. PAHL (E.P. 275,477, 7.1.27).

Colour lakes (E.P. 274,627). **Azo dyes** (E.P. 255,072). **Reduction of nitro-compounds** (E.P. 274,562).—See IV.

Emulsifying agents (E.P. 274,142).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Modifications [in rubber technology] resulting from applications of the antioxygenic theory. J. DUGUÉ (Compt. rend., 1927, 185, 389–391).—In view of the impracticability of collecting the rubber latex in the absence of air the immediate addition of anti-oxygens is suggested, to supplement the natural anti-oxygens present in event of deficiency or partial destruc-

tion. The plasticity and adhesivity of fresh latex, containing 20–25% of moisture, and the loss of plasticity occurring when the product acquires the elasticity of rubber indicate that the polymerides formed in the ageing of rubber are more or less rigid and brittle and not elastic. On this view the cold oxidation or "ageing" of rubber is exactly analogous to vulcanisation, and treatment which effectively prevents autoxidation should also prevent vulcanisation. In vulcanisation, however, a third process of depolymerisation can also take place, with a restoration of elasticity. In vulcanisation under pressure, however, the oxygen present is unable to escape and in the cold catalyses the polymerisation of the rubber. Proposals are made to utilise the plasticity and adhesivity of the freshly-prepared latex for moulding etc. R. BRIGHTMAN.

PATENTS.

Producing aldehyde amine condensation products [vulcanisation accelerators]. GRASSELLI CHEM. CO., Assees. of I. WILLIAMS and W. B. BURNETT (E.P. 263,853, 8.3.26. Conv., 13.3.25).—One mol. of primary or secondary amine reacts with a number of mols. of higher aliphatic aldehyde, water being eliminated, with the formation of highly unsaturated products (viscous liquids or non-crystalline solids), the properties of which change continuously with changing proportions of amine and aldehyde. They are used as vulcanisation accelerators, especially when formed from primary amines and aldehydes in which the α and β carbon atoms are joined by a single linking. As the proportion of aldehyde in the product increases, the "curing" power increases to a maximum and then decreases. The 22 examples given include the preparation of the 2:1, 3:1, 5:1, 7:1, 10:1, and 15:1 heptaldehyde-*n*-butylamine compounds. B. FULLMAN.

Manufacture of liquefied crude rubber. H. P. BUTLER (E.P. 274,742, 2.2.27).—Crude rubber is dissolved in benzene etc., carbon tetrachloride is added to render the mixture non-inflammable, and the temperature is raised to 100° for 2–3 hrs. The mixture may be used alone or with the addition of resins, oils, thinners, pyroxylin, etc. as surfacing compositions.

S. S. WOOLF.

Process and apparatus for regenerating vulcanised rubber. SOC. ITAL. PIRELLI (E.P. 260,620, 29.10.26. Conv., 31.10.25).—Combined and uncombined sulphur are removed from ground vulcanised rubber scrap by heating with a solution of caustic soda or other alkali, of medium concentration and in amount slightly in excess of that corresponding to the total sulphur present at ordinary pressures. S. S. WOOLF.

Regeneration of vulcanised rubber. C. O. CARLSON (E.P. 274,387, 15.3.27).—Scrap or waste vulcanised rubber is dissolved in tar oil at about 150°, and the dissolved rubber is precipitated by suitable agents, *e.g.*, alcohol, after removal of mineral matter by centrifuging, if desirable. The tar oil and precipitating liquid are subsequently recovered.

S. S. WOOLF.

Finishing rubber articles. L. MELLERSH-JACKSON. From L. CANDEE & Co. (E.P. 274,357, 15.1.27).—The tackiness of the finish produced on rubber articles by coating them with a vulcanisable varnish (usually

consisting of a drying oil, sulphur, and a resin) is removed by a superficial treatment with a halogen or a compound comprising a halogen and a member of the sulphur group, excess of halogen etc. being neutralised.

S. S. WOOLF.

XV.—LEATHER; GLUE.

Effect of hydron concentration of tan liquors on absorption of tannin by hide. J. G. PARKER and J. A. GILMAN (J. Soc. Leather Trades' Chem., 1927, 11, 213–220).—Pieces of fresh ox hide were limed, unhaired, fleshed, delimed in boric acid solution, washed, and tanned in liquors of p_H 5.0, 4.0, 3.0, 2.5, and 2.0 respectively. The tan liquor was prepared from valonia, myrobalans, mimosa bark, and chestnut extract. The pieces were completely penetrated after 21 days in the liquors of p_H 5.0, 4.0, and 3.0, after 28 days at p_H 2.5, and after 36 days at p_H 2.0. The pieces tanned at p_H 2.0 and 2.5 contained more absorbed matter after 21 days than the other pieces which were completely penetrated so that decreased rate of penetration does not necessarily mean a decreased degree of absorption. The leather tanned at p_H 2.0 and 2.5 was of bad colour and harsh. The leather tanned at p_H 3.0 appeared to have the properties most desirable in sole leather, *viz.*, firmness without brittleness, good substance and colour, and a fairly high tannin content. With a high acidity there is less tendency to bloom deposition. Above p_H 3.0 there was a heavy deposit of bloom on the leather and in the liquors. The greatest yield of leather is obtained at p_H 3.0.

D. WOODROFFE.

Action of sulphuric acid on leather. D. WOODROFFE and F. H. HANCOCK (J. Soc. Leather Trades' Chem., 1927, 11, 225–227).—In tests on pieces of sumach-tanned skiver and East Indian tanned sheepskin treated with solutions of sulphuric acid of different strengths, the acid content of the leather was found to increase and the tensile strength and stretch to diminish with decreased p_H value of the acid solutions. The weakening effect of solutions of $p_H > 2.0$ was appreciable but not serious. The presence of 1% of free sulphuric acid in the East Indian tanned sheepskin did not cause serious damage, although it did in the skiver.

D. WOODROFFE.

Batch B.14 hide powder. D. BURTON (J. Soc. Leather Trades' Chem., 1927, 11, 227–233).—Serious differences have been obtained in tannin analyses using B.14 powder as compared with B.13. Treatment with buffer solution of p_H 5.0 caused it to give fair agreement, but the non-tans of quebracho and blended extracts were lower. By treating it with a buffer solution of p_H 5.6 it gave higher non-tans with these extracts than B.13. Probably p_H 5.6 is too high. D. WOODROFFE.

Determination of total sulphur dioxide set free by acid from a bleaching extract. (Report of Committee of Society of Leather Trades' Chemists.) D. BURTON (J. Soc. Leather Trades' Chem., 1927, 11, 221).—The committee recommends the following provisional method for the determination of the total sulphur dioxide set free by acid from a bleaching extract. About 1.2 g. of the bleaching extract are dissolved in 100 c.c. of distilled water, and boiled with about 3 g. of phosphoric

acid in an atmosphere of carbon dioxide so that 50 c.c. distil over into 25 c.c. of 0.1*N*-iodine. The excess iodine is titrated with 0.1*N*-sodium thiosulphate solution. The Burton and Charlton apparatus (cf. B., 1927, 20), or one of which sketch is given, should be used.

D. WOODROFFE.

Titration method for evaluating enzyme bates. J. SCHNEIDER, JUN., and A. ULČEK (Collegium, 1927, 342–349).—The bate (5 g.) is extracted with 1 litre of water containing 5 g. of ammonium sulphate and 2 g. of ammonium chloride at 18°. 30 c.c. of a standard casein solution and 20 c.c. of water are measured into each of seven flasks placed in a thermostat at 40°, and 50 c.c. of water into each of seven other flasks. Different amounts of bate infusion are now pipetted into the first set of flasks and corresponding amounts into the other set containing water, and serving as blank tests. After 1 hr. the unchanged casein is precipitated with 100 c.c. of a solution of Glauber salt, 45 c.c. of formaldehyde are added to each filtrate, which is then titrated with 0.1*N*-sodium hydroxide until a violet colour appears. The differences in the numbers obtained by titrating the filtrates and blanks, respectively, are plotted against the amount of bate used and compared with the results for a standard bate.

D. WOODROFFE.

Digallic acid and tannin as mordants. VIKTOROV.—See VI.

PATENTS.

Treatment of hides and skins preliminary to tanning. R. BOTSON (E.P. 272,195, 4.8.27. Conv., 2.6.26).—Hides and skins are delimed, before tanning, in weak acid baths, such as 1 pt. of acetic acid in 2000 pts. of water, containing a very small proportion of nitrobenzene (0.1% on the acid used).

D. WOODROFFE.

Preparation of chamois leather. A. J. CLERMONT (F.P. 595,954, 31.3.25).—Raw goat- and sheep-skins, after liming for 12 hrs. with lime alone, are unhaird, fleshed, coated on the flesh side with a 15% aqueous solution of sodium carbonate, and again after 5 hrs., and piled for 6 hrs. The skins are then rinsed in water, drained for 20 min., partially dried by drumming in sawdust or gypsum, oiled several times with linseed oil, and exposed to sunlight in the open or a warm room at 40°. 24 hrs. are necessary for the tannage. Finally, the skins are freed from surplus oil by drumming in sawdust or gypsum-meal for 3 hrs.

D. WOODROFFE.

Manufacture of tanning agents. I. G. FARBEN-IND. A.-G. (G.P. [A] 438,199, 24.7.24, [B] 438,200, 7.9.24).—(A) Dry powdered wood charcoal containing 9% or more oxygen is mixed with water and treated with nitric acid or an oxide of nitrogen at 70–90° until entirely dissolved, requiring several days. The excess nitric acid or nitrous gases is distilled off by heating for about 30 hrs., avoiding excessive heat. The product is soluble in water and can be used with or without previous neutralisation for tanning. (B) The strongly acid properties of the products obtained by (A) are repressed by mixing them with neutralising agents, e.g., sodium carbonate solution, other synthetic or natural tanning materials.

D. WOODROFFE.

Tanning animal hides with metallic salts. E. CHICOINEAU (F.P. 587,202–3, 19.12.23).—The pelts are treated in neutral, acid, or alkaline solutions with salts of antimony or zinc (e.g., 4–6% of zinc chloride and 5–10% of salt). After penetration, they are horsed up for 1–2 days, treated with water or weak solutions of hydrogen peroxide, and then immersed in a very dilute solution of sodium carbonate or other alkali salt, which should be gradually strengthened until the metallic salts in the pelts are precipitated on the hide fibres.

D. WOODROFFE.

Process of chrome tanning. A. SAUTOT (F.P. 579,207, 2.6.23).—Liquid sodium bisulphite is added to a mixture of sulphuric acid and potassium dichromate solution until the liquor is distinctly green. The product, when cold, is used for tanning pelts.

D. WOODROFFE.

Treatment of horn etc. F. HOMBERG, ASSR. to AMER. NUPLAX CORP. (U.S.P. 1,636,818, 26.7.27. Appl., 30.12.24).—Horn is soaked with hot (but not boiling) acid, then kept, and the treatment repeated until the horn is softened, when it is washed with water, dried, and comminuted.

D. WOODROFFE.

Stuffing of chrome-tanned heavy leather. R. H. PICKARD, D. J. LLOYD, and A. E. CAUNCE (U.S.P. 1,638,877, 16.8.27. Appl., 22.8.25. Conv., 27.8.24).—See E.P. 243,438; B., 1926, 138.

Obtaining solidified granules or pearls [of glue etc.] from liquids. W. WACHTEL, ASSR. to A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL (U.S.P. 1,638,669, 9.8.27. Appl., 29.6.25. Conv., 15.12.24).—See E.P. 244,406; B., 1926, 990.

XVI.—AGRICULTURE.

Soil phosphorus studies. I. Colorimetric determination of organic and inorganic phosphorus in soil extracts and the soil solution. F. W. PARKER and J. F. FUDGE. II. Concentration of organic and inorganic phosphorus in the soil solution and soil extracts, and availability of organic phosphorus to plants. W. H. PIERRE and J. W. PARKER. III. Plant growth and absorption of phosphorus from culture solutions of different phosphate concentrations. F. W. PARKER (Soil Sci., 1927, 24, 109–117, 119–128, 129–146).—I.—For the determination of phosphorus in small quantities the method of Denigès (B., 1920, 781 A) is more sensitive than that of Fiske and Subarrow (A., 1926, 443). The presence of considerable amounts of silica does not affect the accuracy of the method. The technique for determining total and inorganic phosphorus is described. II.—In displaced soil solutions the concentration of organic and inorganic phosphorus averaged 0.47 and 0.09 pts. (PO₄) per million, respectively. Plants absorbed all the inorganic phosphate, but none of the organic phosphate from soil solutions and extracts. The bearing of this unavailability of organic phosphate in the interpretation of plant nutrition experiments is discussed. III.—In culture solutions maximum growth of maize and soya beans was obtained with a concentration of 0.50 pt. (PO₄) per million. It is estimated that one half of this

concentration would suffice if this could be maintained continuously during plant growth. The solid phase of the soil would appear to play an important part in the phosphorus nutrition of plants, but the mechanism whereby a concentration of inorganic phosphate so much greater than that of displaced soil solutions is maintained cannot be explained by the familiar theories of soil chemistry. A. G. POLLARD.

Modification in the solubility of phosphoric acid and in the biological properties of the soil observed during the fallowing of soil previously dried in the air. [I.] A. LEBEDIANTZEF (Compt. rend., 1927, 185, 397—399; cf. B., 1927, 709). [II.] A. T. SCHLÖSING (*ibid.*, 399—400).—I.—Determinations of the phosphoric acid soluble in water, in 2% acetic acid, and in 0.5% oxalic acid on dried and undried samples of different soils (fallow, at 20 cm. depth, triennial rotation at 20 cm., and at 40 cm. depth) show that the desiccation produces marked changes in the phosphoric acid distribution when the samples are left to fallow at the ordinary temperature and with a humidity about 30%. The water-soluble phosphoric acid is increased at first by the drying, but in 25 days decreases to approximately the content of the undried sample, and does not further increase. The phosphoric acid soluble in acetic acid and in oxalic acid shows an increase on the dried sample persisting over a much larger part of the fallow period. The bacteria content of the soil decreases on drying, and remains low during the 2—3 months' fallow period. On the other hand, the production of carbon dioxide is increased by desiccation. II.—The temporary nature of the increase in soluble phosphoric acid content on drying is attributed to (1) the difference in the soils used by Lebediantzef and by Schlösing and Leroux (B., 1927, 343), (2) different methods of washing employed in extracting the phosphoric acid, and (3) the high humidity (30%) in which Lebediantzef's samples were kept after drying. It is concluded that soils kept slightly moist (humidity 6—7%) after desiccation manifest an increased soluble phosphoric acid content effective for crops during a period of three months after desiccation. R. BRIGHTMAN.

Action of superphosphate and Rhenania phosphate on soil. L. VON KREYBIG (Z. Pflanz. Düng., 1927, A 9, 224—235).—The author has investigated layer by layer the effect of superphosphate and "Rhenania" phosphate on the chemical and biological properties of an acid soil. Both fertilisers were applied to the surface at rates usual in practice, and had little effect on the acidity relationships, except that superphosphate increased slightly the hydrolytic acidity of the layer 0—1 cm. The biological activity as evidenced by nitrogen fixation was increased more by the basic Rhenania phosphate than by the superphosphate. The effect of both dressings is confined to the surface layer, and it is shown that the 1% citric acid-soluble phosphate in this layer is greater with Rhenania phosphate than with superphosphate. The author emphasises the localised character of the action of both dressings, and the importance of mode of application and mixing with the soil. G. W. ROBINSON.

Determination of humic acid [in soils]. O. SCHAILL (Landw. Versuchs-Stat., 1927, 105, 209—230).—In a comparison of existing methods for determining humic acid, closest agreement was found between the methods of Tacke and Suchting and of Albert and the titration value of the active acidity. Colorimetric methods depending on the preparation of aqueous soil extracts give results depending on the p_H value of the extracts. The "loss on ignition" method of determining total organic matter in soils yields results closely parallel to those obtained by carbon dioxide measurements. Comber's method is of value in highly organic soils only. The preliminary drying of the soil sample in the Tacke method involves possible alteration in the nature of the organic matter present. A. G. POLLARD.

Determination of the p_H values of soils. K. NEHRING (Landw. Versuchs-Stat., 1927, 105, 231—240).—In preparing soil extracts for p_H determinations the nature of the filter paper frequently affects the results obtained. Only the finest paper should be used, and pre-washing with distilled water is essential. Soil extracts prepared with potassium chloride solution are less liable to be affected in this way. Quinhydrone for electrode work must be highly purified, as many commercial samples have an acid reaction. A. G. POLLARD.

Effect of manuring a crop on the vegetative and reproductive capacity of the seed. B. V. NATH and M. SURYANARAYANA (Mem. Dept. Agric. India, 1927, 9, 85—124).—In experiments with "ragi" (*Eleusine coracana*), "cholam" (*Andropogon Sorghum*), "pani" (*Panicum miliaceum*), and wheat, the seed obtained from manured plots gave higher yields than seed from unmanured plots. In general, seed from plots which had received cattle manure gave better results than when complete minerals were used. The "cattle manure" seed was also found to be superior in animal nutritive value to "mineral manure" and "no manure" seed. This superiority is held to be connected with vitamin content. Experiments with farmyard manure and extracts of it indicate that organic manures, in addition to improving soil texture and supplying plant food, also supply "auximones," or substances which behave towards plants in the same manner as vitamins. Marked increases were obtained with small applications of yeast. It is held probable that the microbial population of the soil contributes to the plant a stimulant which is ultimately passed on to the animal. G. W. ROBINSON.

Influence of manures and organic residues on plant growth. G. A. NEWTON and K. B. DANILOFF (Soil Sci., 1927, 24, 95—101).—The reduced plant growth resulting from the addition to soil of organic material of low nitrogen content may be obviated by the use of sodium nitrate. A. G. POLLARD.

Absorption of nutrients by plants from finely-ground basalt. J. SACHSE (Z. Pflanz. Düng., 1927, A 9, 193—223).—In water and sand cultures with peas, barley, oats, and rye, it was found that whilst the potassium of finely-ground basalt was relatively available, the calcium and phosphorus were only slightly available. The effect of treatment of the ground basalt with hot water, 1% sulphuric acid, and 1% ammonia,

respectively, was also investigated in experiments with peas. The availability of the magnesium, potassium, and phosphorus was increased to the greatest degree by the ammonia treatment, and that of the calcium and silica by hot water treatment. The yield and the absorption of nutrients were increased by addition of calcium carbonate. The results of extraction of the ground basalt with a number of different reagents are given.

G. W. ROBINSON.

Antagonism between chlorides used in large amounts and sulphates in the development of wheat and oats on a large scale. E. BLANCHARD and J. CHAUSSIN (Compt. rend., 1927, 185, 218—220).—Excessive use of fertilisers rich in chlorides is to be avoided, as these cause an almost complete disappearance of sulphur compounds in the plants, and are a danger to the crop in dry seasons.

B. W. ANDERSON.

Experiments with the Neubauer seedling method. T. STECHE (Z. Pflanz. Düng., 1927, A 9, 236—249).—The Neubauer seedling method gives satisfactory results if the amounts of potassium and phosphorus taken up are compared, not with the amounts originally present in the seeds, but with the amounts found in a blank experiment, because in the latter case there is an actual loss of potassium and phosphorus to the soil. In the case of acid soils, it is better to determine the nutrient requirements by the Neubauer method after the acidity has been corrected by liming.

G. W. ROBINSON.

Production of nodules on different parts of the root systems of lucerne plants growing in soils of different reaction. P. E. KARRAKER (Soil Sci., 1927, 24, 103—107).—The poor development of nodules on lucerne roots grown in acid soil is the result of the direct action of the acid soil on the organisms within the nodule. Acid soils do not appear to influence the plant or the free living organisms in the soil in a manner to affect the course of nodule production.

A. G. POLLARD.

Selection for quality of oil in soya beans. L. J. COLE, E. W. LINDSTROM, and C. M. WOODWORTH (J. Agric. Res., 1927, 35, 75—95).—Selective breeding from a commercial variety of soya bean resulted in the isolation of two definite strains—a tall, late type, bearing oil with iodine value 133.7, and a dwarf early type, with oil of iodine value 124.9. High iodine values in soya bean oil appears intimately connected with late maturity in the plant. No correlation exists between the quality of oil from soya plants and the yield per plant. The quantity of oil produced cannot be markedly increased by selective breeding.

A. G. POLLARD.

Lubricating oils as insecticides in dormant spraying. E. L. GREEN (Ind. Eng. Chem., 1927, 19, 931—935).—A series of lubricating oils emulsified by a solution of potash fish-oil soap in crude cresylic acid was tested for insecticidal power by using as sprays in orchards infested with San José scale and orchard leaf roller. The toxicity bears no relation to the viscosity of the oils, but is greatest in the portion that distils between 240° and 300°/40 mm. Owing to the low field temperatures and the high boiling ranges of the effective oils, it is improbable that the oils attack the tissue of the insects

as vapour. Oils with an asphalt or a paraffin base form equally good insecticides, whilst those which have been decolorised with sulphuric acid are likely to be more effective than the untreated oils.

F. R. ENNOS.

Increasing life activities of plants. DENNY.—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Glucose and its industrial preparation by acid and by enzymic saccharification of starch. G. MEZZADROLI and A. NARDELLA (Zymologica, 1927, 2, 49—62, 73—89).—A summary is given of the preparation and properties of commercial glucose, and experimental results are described which show that, by the enzymic saccharification of starch by means of *Mucor*, a greater proportion of the raw material is utilised than when the conversion is effected by acid. In the latter case, appreciable proportions of the starch and of the glucose formed are transformed into humic substances and non-saccharine products, and the unattacked grain residue is almost useless for cattle food. On the other hand, the juices obtained by enzymic saccharification of grain contain only traces of caramelised products, and the residues serve as a valuable feeding material.

T. H. POPE.

Viscosimetric determinations with low-grade sugar refining products. F. DÍAZ AGUIRRECHE (Anal. Fis. Quím., 1927, 25, 178—181).—The author proposes to determine the viscosity of concentrated sugar solutions by measuring the time taken for a float of known density to rise through a given distance in a column of the solution, corrections being applied for temperature.

G. W. ROBINSON.

XVIII.—FERMENTATION INDUSTRIES.

Relationship of ready-formed soluble carbohydrates in malt to extract. H. E. DRYDEN (J. Inst. Brew., 1927, 33, 420—424).—It is suggested that a better idea of the composition of the extract is obtained and that the relationship of all malts can be better compared if the ready-formed soluble sugars, which are arrived at by deducting 4% from the total matters soluble in cold water, are expressed in lb. per 100 lb. of extract and also as a percentage in the analytical report. On this basis foreign malts show a lower proportion of ready-formed soluble sugars, and consequently the composition of the wort from foreign malts is more under control.

C. RANKEN.

PATENTS.

Fermentation and maturation of beer. HANSENA A.-G., and L. NATHAN (E.P. 274,225, 26.4.26).—The Nathan process for the production of beer is improved by carrying out the primary fermentation below 6°, with about 2 litres of yeast per hectolitre, until deposition of the yeast occurs, the attenuation being carried to within 1½—1% of residual fermentable matter. To prevent any further activity on the part of the deposited yeast, the settling space of the fermentation vessel is strongly cooled from the commencement of yeast deposition, preferably to 1°. The maturing or secondary fermentation is effected at 10—25° in the presence of a relatively small quantity of well-aerated, non-germinating

yeast, which may be either the remainder of the yeast produced in the primary fermentation and subsequently aerated, or else taken as fresh yeast from the yeast tank.

C. RANKEN.

Pasteurisation of beer. HANSENA A.-G., and L. NATHAN (E.P. 275,090, 26.4.26).—The finished beer, after expulsion of carbon dioxide by heating at a relatively low temperature, is heated to the pasteurising temperature, subsequently cooled to approximately -1° , and carbonated. It is then bottled and pasteurised again in the bottles at a temperature about 1° lower than that at which the first pasteurisation took place.

C. RANKEN.

Fermentation of cellulose. H. LANGWELL (U.S.P. 1,639,571, 16.8.27. Appl., 7.1.21. Conv., 8.1.20).—See E.P. 161,294; B., 1921, 405 A.

XIX.—FOODS.

Wheat and flour studies. XI. Extraction of proteins from wheat flour. P. F. SHARP and B. L. HERRINGTON (Cereal Chem., 1927, 4, 249—260).—The separation of wheat proteins into four fractions by the official American method is not exact owing to overlapping solubilities. As a basis for formulating a more accurate procedure, the influence of time, concentrations, temperatures, etc. in the successive extraction method has been examined. For the extraction of albumin, globulin, etc., 6 g. of flour and 100 c.c. of 5% potassium sulphate solution were shaken for 1 hr., and centrifuged, and, after filtering, nitrogen was determined in 50 c.c. of the filtrate by the Kjeldahl method. Gliadin was determined in the residue by shaking for 1 hr. with (150— X) c.c. of 70% alcohol (where X is the weight of potassium sulphate solution retained in the residue after filtration), and, after centrifuging, nitrogen is determined in 50 c.c. of the supernatant liquid. Protein-nitrogen in the alcoholic residue was determined by the Kjeldahl method, using 10 c.c. of sulphuric acid, 0.7 g. of mercury, and 10 g. of potassium sulphate, followed by 35 c.c. of sulphuric acid when the frothing had subsided. In calculating the glutenin, allowance was made for the small amounts of protein carried forward by the potassium sulphate solution and alcohol retained in the residues. The protein factor is 5.7 for each fraction. By this method, the alcohol-soluble protein fraction is much lower and the glutenin percentage much higher than is usually recorded. Extracting 4 g. of flour with alcohol of various concentrations showed that the maximum quantity of protein (7.43%) is removed by 50 vol.-% alcohol when shaking is employed, and when refluxing is performed the amount extracted is increased for all concentrations. Varying the ratio of the weight of flour to the volume of alcohol had no appreciable effect. The amounts of protein removed by 5% and 10% solutions of various salts, followed by shaking and by refluxing with 70% alcohol, were also studied. The maximum amount was extracted by magnesium chloride, and extraction by this salt was more complete with the 10% solution; in all other cases investigated, the 5% solutions were the more efficient.

H. J. DOWDEN.

Relation of hydrogen-ion concentration of dough to baking properties. E. GREWE and C. H. BAILEY

(Cereal Chem., 1927, 4, 261—270).—The influence of hydrogen-ion concentration upon the diastatic activity of 17 flours has been investigated. The addition of sufficient lactic acid to reduce the p_H from 5.9 to 5.0 substantially increased the activity in every case, the rate of increase being greatest with flours of high initial activity. The addition of alkali to flours diminishes diastatic activity, and it was observed that increasing the p_H value from 5.9 to 8.5 by adding 3.6 c.c. of 0.1N-caustic soda solution to 10 g. of flour reduced the diastatic activity from 206 to 12 mg. of maltose per 10 g. of flour. The diastatic activity of flours of low initial activity is substantially increased by adding flour milled from sprouted wheat, an addition of 5% being capable of raising the activity of a flour from 125 to 200 units, which is the desirable activity for a flour to be of good baking quality. The loaf volume and crumb texture are also improved by the addition of sprouted wheat flour. The addition of alkali and sufficient sprouted wheat flour to maintain a high diastatic activity demonstrated that the best bread as regards loaf volume and crumb texture is given by doughs in which the p_H has been decreased. These facts indicate that the increase of p_H during dough fermentation results in improved bread because of the accelerated activity of the enzymes. H. J. DOWDEN.

Factors affecting interpretation of experimental baking tests. M. J. BLISH and R. M. SANDSTEDT (Cereal Chem., 1927, 4, 291—299).—The fermentation tolerance of doughs has been investigated by performing the Werner baking test on doughs fermented from $\frac{1}{2}$ —6 hrs. Fermentation tolerance can be accurately judged at one bake, from the crust colour, rise, texture of the bread, etc., provided other influencing factors are maintained constant. Great fluctuations in stability were observed, experimentally milled flours showing "exhaustion" much more quickly than commercially milled flours. To make the results comparable it is recommended that with experimentally milled flours, the sugar content should be increased from 2½% to 5%. Crust colour also gives a valuable indication of the diastatic value. These facts suggest that fermentation tolerance is dependent on the yeast, diastase, or sugar content rather than on the gluten quality or quantity. Oxidising agents act powerfully on gluten, and are largely responsible for the variations in bread-making qualities of flours. A differential baking test, with and without added oxidising agent, indicates readily to what extent the flour can be improved by the addition of these agents. Over-treatment gives symptoms of "age." H. J. DOWDEN.

Value of experimental [flour] milling test. R. S. HERMAN (Cereal Chem., 1927, 4, 270—274).—Experimental milling gives results which are strictly comparable with commercial milling, and is of value in ascertaining the nature of wheat, yield, and flour quality, as regards colour and analytical data. The flour can be used for determining baking characteristics and thus provides guidance in blending.

H. J. DOWDEN.

Washing of gluten from flour. D. W. KENT-JONES and C. W. HERD (Analyst, 1927, 52, 439—443).—A number of chemists determined the gluten content of the same flours, using their own tap water and the special solutions of Dill and Alsberg, and under as nearly as

possible identical conditions. It was found that differences caused by using tap water and the special solutions were very small, but large differences occurred between the results of the various workers. Each, however, obtained essentially consistent results, so that the ratio between the nitrogen of the flour and the dried gluten was approximately consistent for each. D. G. HEWER.

Numerical expression for the colour of flour. D. W. KENT-JONES and C. W. HERD (Analyst, 1927, 52, 443—452).—Definite numerical expressions for the colour factors of flour may be obtained by the successive use of two solvents, viz., a standard grade petrol for extraction of the yellow carotin, and alkaline methyl alcohol for extraction of the reddish-brown pigment, which latter presumably comes from finely powdered offal. The determinations are made against standard solutions in a special colorimeter, and the petrol figure indicates the yellowness, not differentiating high- and low-grade flour, whilst the methyl alcohol figure is the grade figure showing the brightness of the flour and its freedom from minute branny specks. In practice wood spirit may be used for methyl alcohol if the necessary correction for each batch is applied. D. G. HEWER.

Chemical modifications produced in wheat flour by benzoyl peroxide. S. BAGLIONI and L. SETTIMI (Annali Chim. Appl., 1927, 17, 351—356; cf. Javillier, B., 1926, 211).—When benzoyl peroxide acts upon wheat flour it is completely decomposed to give oxygen and benzoic acid, which is lost almost completely at 150°. Its bleaching action consists in conversion of carotin into a leuco-compound; at the same time some non-glutinous proteins are converted into proteoses, a probable cause of the rapid maturing of the flour. Carbohydrates, fats, lecithin, phytosterol, etc. remain unchanged, as do the calorific value and the physical constants. E. W. WIGNALL.

Cacao butter substitutes and their detection. A. W. KNAPP, J. E. MOSS, and A. MELLEY (Analyst, 1927, 52, 452—456).—A critical review of the methods in use for the detection of illipé butters, true and commercial, and shea butter shows that no one method is satisfactory, but by a comparison of results obtained from the titre of the fatty acids, solubility tests, Tate and Pooley's method, and Bywaters' process (B., 1927, 614), together with a determination of the permanence of any green colour, a fair idea of the presence and proportion of cacao butter substitutes may usually be arrived at. The green colour test depends on the comparative resistance of the green colour of Borneo tallow to bleaching; 30 g. of fat are exposed in a Petri dish 15 cm. diam. for 6 hrs. 6.5 in. below a quartz mercury lamp of uroxameter value 28.0, the time of exposure varying with the strength of the lamp. The fat is then filtered into a test tube and the colour compared with that of a set of standard exposed fats. The method was found most useful for mixtures containing less than 20% of Borneo tallow.

D. G. HEWER.

Effect of common salt on lime water used for egg preserving. J. MILLER (Analyst, 1927, 52, 457—458).—Common salt added to lime water increases the progressive diminution of strength of the lime water in propor-

tion to the amount added. Even with excess of lime all the lime is completely carbonated in 6 months in the presence of 100 lb. or more of common salt per 1000 gals. It is suggested that the instructions in Special Report No. 26 of the Department of Scientific and Industrial Research, to use 4 pts. of slaked lime to 20 of cold water might be modified, as the solubility of lime is about 1 in 750, and if tap water is mixed with 4 times the amount of burnt lime necessary to saturate, stirred for 5 min., and allowed to settle, a practically saturated lime solution is obtained without further keeping. The addition of 1 pt. of salt, as instructed, equivalent to 500 lb. per 1000 gals., is not only unnecessary but detrimental.

D. G. HEWER.

Ammonia content of cold-storage eggs. H. C. LYTGOE (Ind. Eng. Chem., 1927, 19, 922—924).—The average monthly content in ammonia of eggs put into cold storage before July 1 showed a gradual increase from 2.4 mg./100 g. in September to 3.2 mg./100 g. in February. Of 1100 cold-storage eggs collected during the entire season for a period of five years, 95% contained between 2.1 and 4.3 mg. NH_3 per 100 g., the former figure probably representing the minimum content at which it is possible to market eggs preserved in this manner.

F. R. ENNOS.

Natural occurrence of boron compounds in cacao and cacao products. S. DODD (Analyst, 1927, 52, 459—466).—Boron compounds occur naturally in cacao and cacao products and are present in chocolate to the extent of about 0.01% (as boric acid) and in commercial samples of cacao beans and cocoa from 0.0217 to 0.0837%, and in coffee beans to about 0.01%. Boric acid may be satisfactorily determined by Thomson's method, but the best indicator is Sofnol indicator No. 1 with p_H 6.5. For qualitative work turmeric paper is used, and it is advisable in the case of oily substances to remove the bulk of the oil before testing.

D. G. HEWER.

Effect of small amounts of chemicals in increasing the life activities of plants [and ripening of fruit]. F. E. DENNY (Proc. Nat. Acad. Sci., 1927, 13, 555—561).—When ethylene is added to the air in which oranges and lemons are stored, a concentration of gas not exceeding 1/5000 doubles the rate of production of carbon dioxide, and any green colour in the peel is rapidly converted into yellow. Too high concentrations of ethylene retarded the change of colour. The evidence shows that the ethylene acts in a physiological and not in a directly chemical manner, raising the general level of life activity in the tissues. The stimulative effects also apply to tomatoes, potatoes, apples, etc. Freshly-harvested potatoes if replanted under conditions favourable to growth do not sprout at once, but require a period of after-ripening. Various chemicals induce early sprouting, ethylene chlorohydrin and alkali thiocyanates being specially effective. The substances causing a stimulation of life activity do not appear to be chemically related, they are not used as food, and the mechanism of their action is not understood.

R. A. MORTON.

Interpretation of bromatological analyses. A. BEYTHEN, C. HARTWICH, and M. KLIMMER (Anal. Oficina

Quím. Prov. Buenos Aires, 1927, 1, 91—100).—The authors give standards for the interpretation of analyses of meat and meat products.

G. W. ROBINSON.

Direct determination of sodium chloride in food-stuffs. F. MACH and W. LEPPER (Landw. Versuchs-Stat., 1927, 105, 333—334).—The sample (5.0 g.) is extracted for 30 min. with about 400 c.c. of water and the extract cleared by precipitation with 40 c.c. of a solution containing 50 g. of phosphotungstic acid, 500 c.c. of nitric acid (*d* 1.4), and water to make 1 litre. In the filtered solution chlorine may be determined by the Volhard method with the addition of ether.

A. G. POLLARD.

PATENTS.

Treatment of milk powder [for packing]. W. G. HAWLEY, Assr. to MERRELL-SOULE Co. (U.S.P. 1,637,311, 26.7.27. Appl., 29.1.27).—The milk powder, enclosed in a container which is air-tight except for a minute opening, is subjected to a high vacuum and then charged with a mixture of carbon dioxide and nitrogen, so that the uncombined oxygen content of the powder and space within the container is less than 5 c.c. per lb. of powder.

F. R. ENNOS.

Production of margarine and like edible fats. B. JIROTKA (E.P. 274,949, 26.4.26).—The margarine mass, after preliminary disintegration, is fed in opposite streams over cooled rollers between which it is kneaded (pressing and spreading action being produced by the different peripheral speeds of the rollers), the fresh films so formed being removed by scrapers.

S. S. WOOLF.

Refining bee honey. W. W. SOMERFORD (U.S.P. 1,636,719, 26.7.27. Appl., 24.8.22).—The natural honey, mixed with a filtering substance and water, is run through heated filter bags and the filtered mass stirred in a vacuum tank until it attains a predetermined consistency.

F. R. ENNOS.

Making concentrated fruit juice. W. M. ZORN (U.S.P. 1,636,890, 26.7.27. Appl., 25.7.24).—The juice extracted from the fruit by pressure is filtered to remove pulpy matter and concentrated by freezing to a snow and centrifuging. The concentrated juice is heated to below its b.p., filtered to remove pectin and other undesirable matter, cooled to just above its f.p., and refiltered.

F. R. ENNOS.

Curing meats. J. ALSBERG, Assr. to B. HELLER & Co. (U.S.P. 1,635,301, 12.7.27. Appl., 12.6.25).—After being charged with a curing liquid containing sodium chloride, a nitrate, and a nitrite, the meat is completely enveloped in a dry curing medium having a predetermined amount of nitrite for 40—60 days.

F. R. ENNOS.

Preservation of food-stuffs etc. C. T. J. VAUTIN and W. G. WHIFFEN (E.P. 274,164, 6.4.26).—Putrefaction is prevented by protecting the exposed surface of the substance from the atmosphere by means of a porous material impregnated with calcium iodate. In certain cases the iodate may be added direct in the form of a powder, or the substance may be immersed in an aqueous solution of the iodate.

C. RANKEN.

Treatment of cereal substances. D. W. KENT-JONES and C. W. CHITTY, Assrs. to WOODLANDS, LTD.

(U.S.P. 1,636,569, 19.7.27. Appl., 5.11.25. Conv., 9.12.23).—See E.P. 228,830; B., 1925, 296.

Egg-preservation process and product thereof. A. J. BELLAMY, Assr. to EGG PATENTS LTD. (U.S.P. 1,639,549, 16.8.27. Appl., 2.9.25. Conv., 13.5.25).—See E.P. 261,194; B., 1927, 92.

Improving the qualities of coffee. O. GEWALT (U.S.P. 1,636,300, 19.7.27. Appl., 16.7.26).—See E.P. 265,865; B., 1927, 315.

Machinery for making aqueous emulsions of dried milk or milk powder. A. C. BAMFORD (E.P. 275,375, 12.6.26).

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

Preparation and properties of diethylene glycol dinitrate. W. H. RINKENBACH (Ind. Eng. Chem., 1927, 19, 925—927).—Diethylene glycol dinitrate, $O[C_2H_4 \cdot NO_2]_2$, is prepared by nitration of pure diethylene glycol with mixtures of nitric and sulphuric acids, the yield, emulsification, and safety of the operation depending on the type of nitrating mixture used. The best yields are obtained by adding the glycol to just sufficient nitrating acid containing 25% of nitric acid and 5—10% of water, keeping the temperature below 10°, pouring the resulting emulsion into three times its weight of ice and water, purifying the ester by washing with ice water and potassium carbonate, and drying over sulphuric acid. Pure diethylene glycol dinitrate is a clear, colourless, odourless liquid of greater fluidity than nitroglycerin; at ordinary temperatures it is completely miscible with nitroglycerin, ethylene glycol dinitrate, ether, acetone, methyl alcohol, chloroform, benzene, toluene, aniline, nitrobenzene, glacial acetic acid, and glycol diacetate. It is immiscible with or only slightly soluble in ethyl alcohol, carbon tetrachloride, and carbon disulphide. Its solubility in water is 4.1 g./litre at 24°, but it is only slightly hygroscopic. At ordinary temperatures it is not saponified by 3% alcoholic potash. It has d_{40}^{25} 1.3908, f.p. —11.3°, n_D^{25} 1.4536, heat of combustion at constant volume 2798 g.-cal./g., at constant pressure 2792 g.-cal./g., vapour pressure at 22.4° 0.007 mm. Diethylene glycol dinitrate can be ignited only by heating above the temperature of the air. It is less sensitive to shock than nitroglycerin or glycol dinitrate, but when mixed with these it can be completely detonated and shows an explosive effect but little less than either of these compounds. The physiological action of diethylene glycol dinitrate on the human system is much less marked than that of nitroglycerin.

F. R. ENNOS.

Determination of cincophen (2-phenylcinchoninic acid) and choice of indicators for its titration. S. PALKIN (J. Amer. Pharm. Assoc., 1927, 16, 632—635).—The material may be extracted from a slightly acid aqueous solution by means of a mixture of chloroform and ether, or it may be treated in acid solution with a mixture of potassium bromide and bromate, and the additive compound extracted with ether. The substance is weighed as the hydrobromide, after removal of the excess bromine by repeated evaporation with acetone.

Electrometric titration indicates a p_H range of 6.5—8.5 for the neutral salts, so that cresol-red, phenol-red, or bromothymol-blue are more suitable indicators for the titration with acids than phenolphthalein.

S. I. LEVY.

Critical study of methods of analysis of antipyrine and pyrimidone. A. BORLOZ (Helv. Chim. Acta, 1927, 10, 543—548).—Antipyrine may be accurately determined by the iodometric methods of Bougault (A., 1917, ii, 344) and Kolthoff (A., 1923, ii, 270), other methods being unreliable. For pyrimidone none of the five methods proposed is good, the best being titration with sulphuric acid in presence of methyl orange (Pegurier, A., 1905, ii, 871), which gives low results and requires special technique. For mixtures of pyrimidone with small amounts of antipyrine, Patein's formaldehyde method (A., 1905, ii, 658) is available for the latter, pyrimidone being extracted with chloroform and determined by titration. In a mixture containing much antipyrine, pyrimidone is first titrated with acid, excess of picric acid is then added to a second sample and the excess titrated (cf. Pegurier, *loc. cit.*).

C. HOLLINS.

Determination of iodine in extract of thyroid gland. J. F. REITH (Pharm. Weekblad, 1927, 64, 777—792).—Experiments carried out to trace the causes of loss of iodine during the burning off show that no loss of iodine occurs when a mixture of potassium iodide with a large excess of fusion mixture is heated, unless a very high temperature is attained. Iodine vapour once formed, however, is not completely absorbed by heated fusion mixture unless the temperature is not less than 400°. Hunter's method (A., 1910, ii, 650) is rapid and accurate, but gives results considerably higher than those obtained by using the method of the Dutch Pharmacopœia; in the latter, iodine is lost in the combustion. The sources of error in the Dutch method are pointed out in detail.

S. I. LEVY.

Factors affecting certain properties of a [tobacco] mosaic virus. H. H. MCKINNEY (J. Agric. Res., 1927, 35, 1—12).

Quantitative and purification methods in [tobacco] virus studies. H. H. MCKINNEY (J. Agric. Res., 1927, 35, 13—38).

Bismuth salts. CHEMNITIUS.—See VII.

Hydnocarpic acid. PERKINS and others.—See XII.

PATENTS.

Production of oxygen-containing aliphatic compounds [keten, acetaldehyde, etc.]. H. DREYFUS (E.P. 273,622 and 273,810, 26.6.25).—(A) Keten is obtained by passing acetaldehyde vapour over a heated hydrogen-splitting catalyst, *e.g.*, nickel, copper, zinc, etc., at not above 400—500° (preferably between 150° and 300°) and preferably at normal pressure; in the absence of a catalyst the reaction is effected at higher temperatures and at 5—50 atm. pressure. The keten may be collected as such, or converted into acetic acid or/and anhydride by treatment with water or/and acetic acid. Acetic acid or/and anhydride are obtained directly by mixing the acetaldehyde with water or/and acetic acid prior to treating it as above. The acetaldehyde may be of any origin or produced by reducing

the vapour of acetic acid (glacial, concentrated, or dilute) according to E.P. 273,810. (B) Dilute or waste acetic acid is reduced to acetaldehyde by passing its vapour, together with hydrogen or gases containing it, with or without carbon monoxide, *e.g.*, water, town or producer gas, etc., over reduction catalysts such as nickel, iron, tin, platinum, palladium, etc. or/and contact substances such as pumice, glass-wool, sodium chloride, etc., singly or mixed. The hydrogen is preferably used in excess of theoretical proportions. *E.g.*, 2 vols. of 15—30% acetic acid are mixed with 1 vol. of hydrogen and passed over finely-divided nickel maintained at 250—300°. The aldehyde is separated by fractional condensation, and may be converted into glacial or concentrated acetic acid by oxidation.

B. FULLMAN.

Manufacturing borneol and isoborneol. G. AUSTRERWEIL (E.P. 258,901, 27.9.26. Conv., 25.9.25).—Nopinene (1 mol.) reacts in neutral solvents (toluene, xylene, pinene, etc.) with 2 mols. of an organic acid (benzoic or dichlorobenzoic acid etc.), yielding only bornyl esters and pinene, without monocyclic terpenes. The reaction is carried out at an acid concentration not exceeding 30% (preferably 20—25%) at 140—150°, heating for 40—50 hrs., according to the acid concentration. The pinene does not react. The reaction may be applied directly to turpentine containing nopinene.

B. FULLMAN.

Manufacture of acetic anhydride. R. MEINGAST and M. MUDGAN, ASSRS. to CONSORTIUM FÜR ELEKTROCHEM. IND. (U.S.P. 1,636,701, 26.7.27. Appl., 21.2.25. Conv., 25.2.24).—See E.P. 230,063; B., 1925, 568.

Production of aromatic aldehydes. A. E. CRAVER, ASSR. to BARRETT Co. (U.S.P. 1,636,855, 26.7.27. Appl., 18.11.21).—See E.P. 189,107; B., 1923, 950 A.

Catalytic oxidation of aromatic hydrocarbons. A. E. CRAVER, ASSR. to BARRETT Co. (U.S.P. 1,636,854, 26.7.27. Appl., 14.11.21).—See E.P. 189,091; B., 1924, 200.

Production of ethyl chloride. H. SUIDA (U.S.P. 1,637,972, 2.8.27. Appl., 3.2.25. Conv., 11.2.24).—See E.P. 229,298; B., 1925, 652.

Compounds of *p*-diamines and sulphur dioxide (E.P. 269,583).—See IV.

***N*-Alkyl-*p*-aminophenol** (E.P. 274,175).—See IV.

Hydrolysis of esters (E.P. 251,290).—See XII.

Condensation products (E.P. 263,853).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Plate sensitivity and fogging agents : methylene-blue. J. SOUTHWORTH (Brit. J. Phot., 1927, 74, 461—463).—A fine-grained, unwashed silver bromide emulsion, supposed to contain no nuclei, fogged on treatment with methylene-blue solution. Addition of thiocarbamides to such an emulsion (an alkaline emulsion) caused an increase in sensitiveness to light and also to methylene-blue, but the increase in the latter case was probably not proportional to the increase in light-sensitiveness. Desensitisation of an unwashed emulsion by a methylene-

blue-acridine-yellow solution does occur, but it is proportionately far less than with a rapid emulsion containing nuclei. A provisional working hypothesis of the action of methylene-blue is as follows:—The fogging action is dependent on the adsorption of methylene-blue to the grains. The desensitising effects are primarily due to adsorption of the dye by the nuclei. Protective dyes, such as acridine-yellow, function by preventing adsorption by the silver halide, but not by the nuclei. The sensitivity substance probably has a greater affinity for methylene-blue than for acridine-yellow. The fogging action of methylene-blue is ascribed to reduction of the dye by the developer to form its leuco-base, which in turn reduces the silver halide to form development nuclei of silver. Alternatively, fogging may be due to chemiluminescence accompanying reduction of the dye to its leuco-base, or else to some unstable fogging oxidation product of the developer.

W. CLARK.

Halogen acceptor theory of sensitivity and the thioanilides. S. E. SHEPPARD and H. HUDSON (Phot. J., 1927, 67, 359—361).—Thioanilides added to a standard emulsion made from relatively inert gelatin have an apparent desensitising effect, which is actually due to reduction of the densities in the toe of the characteristic curve, the % maximum density, and gamma. Fog due to white light is reduced, but the exposure scale is considerably altered. The thioanilides probably have the constitution $\text{HS}\cdot\text{CR}\cdot\text{NR}'$, where R' is an aryl group and R is either an acyl or an aryl group. Complex formation with the silver halides probably takes place through the nitrogen atom, and not the sulphur atom. It appears that if the silver atom can be associated with the sulphur atom, silver sulphide is readily formed, but so long as the complex is anilided this is inhibited. The fact that sensitising is not effected by these compounds is in agreement that silver sulphide is not formed even in presence of alkali. The complex silver halide compound is a very effective halogen acceptor, but since it does not sensitise, the facts are contrary to the halogen acceptor theory of sensitising nuclei. The results are considered to confirm the view that silver sulphide is the effective sensitiser in the case of sensitisers containing sulphur, but they make of doubtful value any generalised halogen acceptor theory. Further, they offer a difficulty for the orientation theory of sensitising and latent image formation.

W. CLARK.

Fogging action of persulphate. E. P. WIGHTMAN (Brit. J. Phot., 1927, 74, 447—448).—It is confirmed that a dilute solution of bromine fogs a photographic plate. All high-speed emulsions contain a trace of free bromide. On treatment of the plate with oxidising desensitisers, free bromine would be liberated from this bromide, and would react slowly with the silver sulphide sensitivity-promoting specks to give metallic silver according to some modification of Hickman's hypothesis (cf. B., 1927, 126), or else to give silver bromide which remains for some time as silver and bromide ions. In either case it would lead to latent fog.

W. CLARK.

Thermodynamic possibilities of the silver sulphide bromine-acceptor hypothesis of latent image

formation. R. H. LAMBERT and E. P. WIGHTMAN (J. Physical Chem., 1927, 31, 1249—1258).—The changes in free energy of the reactions concerned in Hickman's silver sulphide bromine-acceptor hypothesis (cf. B., 1927, 126), and in other reactions, have been calculated, and it is concluded that this hypothesis is thermodynamically sound, although certain of the intermediate reactions advanced by Hickman may be doubtful. It is further suggested that silver sulphide itself in the presence of silver bromide is photo-chemically decomposed, the bromide probably serving as an optical sensitiser, and as the sulphur acceptor giving rise to sulphur bromide and silver. The free energies of formation of silver sulphide and sulphur bromide, respectively, are — 6355 and — 3040 g.-cal.

L. S. THEOBALD.

Photo-colorimeter. TOUSSAINT.—See I.

PATENTS.

Sensitised photographic papers. POLYGRAPHISCHE GES. (E.P. 250,267, 1.4.26. Conv., 3.4.25).—A photographic paper is coated with sensitive emulsion on both sides, and is also provided with at least one independent non-actinic layer between the two layers of emulsion. The non-actinic layer is decolorised during the photographic process, for example, during the development, or during fixing, washing, and drying, and the emulsion layers are strippable.

W. CLARK.

Halation-free photographic plates. Z. Zelizy (G.P. 439,484, 5.4.25. Addn. to G.P. 393,762. Cf. B., 1924, 771).—Before coating with emulsion, both sides of the base are coated with a solution of transparent, chemically pure gelatin or collodion, to which is added a white powder which absorbs ultra-violet light (quinine hydrochloride, morphine, urea, etc.) in transparent aqueous or alcoholic solution. Further, a filter absorbing ultra-violet rays can be used in front of or behind the objective.

W. CLARK.

Manufacture of kinematograph colour films. J. E. THORNTON (E.P. 272,986, 18.3.26).—A two-colour kinematograph film positive is produced by cementing together after printing two thin supports, one having an orange-red colloid relief image, the other a blue-green colloid relief image, the two films being cemented together with the image faces outwards. Two parallel rows of component images are printed on film material of double width, comprising a double-width support, carrying an insoluble substratum on which is coated an image-forming colloid layer, coloured before printing with two parallel stripes of suitable colours, and sensitised with dichromate. After printing, the pairs of differently-coloured images are developed and treated side by side on the double-width base to form a pair of relief images in complementary colours.

W. CLARK.

[Tank] method of development of photographic film or the like and apparatus therefor. H. B. STRINGER and A. STRATTON (E.P. 275,349, 17.5.26).

Photographic films having lenticular elements. SOC. DU FILMEN COULEURS KELLER-DORIAN (E.P. 246,829, 19.1.26. Conv., 31.1.25).

Reproduction of photographic images on films having lenticular elements. SOC. DU FILM EN COULEURS KELLER-DORIAN (E.P. 245,118, 18.12.25. Conv., 27.12.24).

Compounds of *p*-diamines and sulphur dioxide (E.P. 269,583).—See IV.

XXII.—EXPLOSIVES; MATCHES.

Diethylene glycol dinitrate. RINKENBACH.—See XX.

PATENT.

Combustible compositions for pyrotechnic, smoke- and flame-producing, and other like purposes. VICKERS, LTD., and E. E. F. BERGER (E.P. 275,021, 9.8.26).—About 40 pts. by wt. of powdered calcium silicide are ground and mixed with 60 pts. by wt. of potassium nitrate to effect oxidation of the silicide without explosive effect, the mixture being compressed into thin cakes and granulated. The material may be associated as a priming or igniting agent with a mixture of powdered metal, a chlorinated hydrocarbon, and calcium silicide, by making granules of this mixture, coating these with the material by polishing or otherwise, and compressing. W. G. CAREY.

XXIII.—SANITATION; WATER PURIFICATION.

Decomposition of cellulose in fresh sewage solids. H. HEUKELEKIAN (Ind. Eng. Chem., 1927, 19, 928—930).—In studies of the rate of decomposition of cellulose during digestion of limed and unlimed unseeded fresh sewage solids, in which the cellulose contents of the sludges were determined from time to time by use of Schweitzer's reagent, about 36% of the cellulose disappeared in the first week and 79% in 3 weeks in the unlimed material, as compared with 73% in the first week and 96% in 3 weeks in the case of limed mixtures. Lime, by neutralising the acid decomposition products, accelerates also the rate of decomposition of the non-cellulosic organic matter.

W. T. LOCKETT.

Determination of organic matter in sewage and effluent. W. E. ABBOTT (Ind. Eng. Chem., 1927, 19, 919—921).—A study of Adeney's dichromate method for the determination of organic matter in sewage and effluent, more particularly with regard to the conditions required for successful chloride removal, led to the adoption of the following procedure: 25 c.c. of a raw sewage or an equivalent volume of other liquid are placed in a small evaporating basin, 0.3 c.c. of sulphuric acid is added, and the liquid evaporated with occasional stirring until about 0.7 c.c. remains, when the residue is vigorously stirred at intervals during 3 or 4 min. to remove the chloride; 25 c.c. of 0.125*N*-dichromate solution are added, and evaporation is resumed until about 1 c.c. remains; 5—10 c.c. of sulphuric acid are added and the basin is left on the water bath for 2 hrs.; the liquid is then diluted, and the residual dichromate titrated with ferrous sulphate, using potassium

ferriocyanide as external indicator. The liquid in the basins undergoing digestion should be maintained above 94°, and blank determinations are required. Although the oxygen absorbed from dichromate by various organic substances when subjected to the modified test varied from 60 to 99% of the oxygen calculated as necessary to convert them into carbon dioxide, water, and ammonia, the dissolved oxygen absorption (until nitrification commenced) of raw sewage (freed from suspended matter) indicated that the dichromate absorption approximates to the oxygen required to convert all the organic matter present in sewage into carbon dioxide, water, and ammonia. No definite relationship was established between the dichromate absorption and the 5 days' dissolved oxygen absorption. With crude sewage the dichromate absorption is on the average 5.1 times as great as the permanganate absorption (A.P.H.A. 1923 method); with sewage free from suspended matter 4.5 times; and for well purified activated sludge effluents 2.5 times—results which indicate that the dichromate absorption affords a more sensitive index of the course of purification. W. T. LOCKETT.

Repellants for blowflies. R. C. ROARK, D. C. PARMAN, F. C. BISHOPP, and E. W. LAAKE (Ind. Eng. Chem., 1927, 19, 942—943).—Observations as to the number of flies visiting fresh beef liver treated with various repellants and the number visiting untreated fresh beef liver indicated that the most effective repellants are materials, *e.g.*, copper carbonate, which absorb, adsorb, or inhibit the formation of the volatile compounds evolved by decomposing meat which attract the flies to the meat. In addition to various copper compounds, such strong antiseptics as mercuric chloride, potassium permanganate, sodium salicylate, etc. when applied to meat render it almost entirely non-attractive. Some strongly odorous materials are quite effective when undiluted, but lose nearly all their efficacy or become actually attractive when diluted 1 to 9 with an inert vehicle. W. T. LOCKETT.

Lubricating oils as insecticides. GREEN.—See XVI.

PATENTS.

Disinfectant tablets, bricks, etc. BRITISH DYE-STUFFS CORP., LTD., T. H. FAIRBROTHER, and A. RENSHAW (E.P. 274,187, 14.4.26).—Mixtures of auramine with plaster of Paris, diatomite, or a solid volatile medium, with or without the addition of sodium chloride, are moulded into various shapes and suspended in the water of a flush cistern. The auramine diffuses slowly and regularly and confers disinfectant properties to the water. C. RANKEN.

Preparation of water-softening materials. G. W. PRATHER (U.S.P. 1,636,942, 26.7.27. Appl., 10.8.16).—A mineral having a base of hydrated aluminium silicate is dried until practically all the uncombined moisture is driven off and, after being broken to a suitable size, is baked at a high temperature with sodium hydroxide until the latter fuses and permeates the mineral, which is finally washed free of excess of soda. W. G. CAREY.