

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 22, 1928.

I.—GENERAL; PLANT; MACHINERY.

Goldberg spectrodensograph. H. SCHERING (Collegium, 1928, 143—150).—An improved apparatus enables an unskilled operator to obtain in a few minutes graphs showing the colour density between the wavelengths 700—400 μ . The graph is plotted automatically. Non-transparent materials, *e.g.*, leather, paper, fabrics, as well as transparent coloured materials can be examined. The absorption curves are thus obtained without difficulty. The apparatus comprises a monochromator side by side with a photometer. D. WOODROFFE.

Pendulum viscosimeter. ALBRECHT and WOLFF (Petroleum, 1928, 24, 551—555).—The apparatus described is suitable for the rapid determination of the viscosity of any liquid at temperatures up to 300°; 200 c.c. only of the liquid are required. The apparatus consists of a pendulum, part of which is immersed in the liquid to be tested. A mirror is attached to the pendulum at its point of suspension and reflects a ray of light from a fixed source upon a moving photographic film. The liquid is brought to the required temperature, and the pendulum is displaced through a constant angle from its mean position and set in vibration. The oscillations are recorded upon the moving film by the ray reflected from the mirror. The theory of the method is discussed, and an equation for the instrument arrived at, from which the viscosity of the liquid is obtained.

R. C. ODAMS.

Simple method for determining the absolute viscosity of oils. P. E. RAASCHOU (Dansk Tidsskr. Farm., 1928, 2, 134—139).—The apparatus consists of a glass capillary tube with three marks at suitable intervals, fitted into a test tube by means of a stopper, the whole being immersed in a container filled with water. The oil is introduced into the inner test tube, and when the temperature has become constant (15 min.) a little is drawn up the capillary tube to a point above the upper mark, a rubber ball being affixed to the capillary for this purpose. After releasing the ball, the time required for the level of the liquid in the capillary to fall from the upper to the middle mark is accurately noted. The viscosity is then given by the formula $\eta = kt$, where k is a constant for the apparatus, which is found by employing liquids of known viscosity, preferably paraffin or a solution of glycerin. The temperature must be kept constant, and a correction is necessary if the density of the liquid to be determined differs greatly from that used in calibrating the apparatus.

H. F. HARWOOD.

Determination of specific gravity of powders. H. RASQUIN (Farben-Ztg., 1928, 33, 1786—1787).—The

specific gravity of powders insoluble in and heavier than water is determined by introducing 10 g. of the substance through a funnel into 10 c.c. of water in a measuring cylinder, the exit tube of the funnel being kept just clear of the water. The specific gravity is found by dividing 10 by the increase in volume in c.c.

Design of fractionating columns. D. B. KEYES, R. SOUKUP, and W. A. NICHOLS, JUN. (Ind. Eng. Chem., 1928, 20, 464—466).—The graphical determination of the minimum number of plates necessary for a fractionating column for a given duty is effected as follows. A curve is drawn showing the composition relation between the vapour and the boiling liquid; the equal-composition curve (a straight line at an angle of 45°) is drawn between 0 and 100% composition of the more volatile compound; and the compositions of the residue, the feed, and the distillate are drawn as vertical straight lines. Then the number of steps between the two curves from the distillate line to the residue line is counted, and a safety factor to provide for reasonable reflux is applied. Several examples of the calculation are given.

C. IRWIN.

Manometer for vacuum distillation. F. FRIEDRICH (Chem.-Ztg., 1928, 52, 272).—A manometer which is independent of atmospheric pressure and can readily be filled with mercury and freed completely from gas and moisture comprises the usual open and closed parallel tubes enclosed in an outer closed protecting tube, and connected to each other through a small valve on the injector principle. To fill the manometer mercury is poured into the open tube, and the closed tube is heated in an inclined position until the air is displaced and the mercury boils gently. The manometer is then filled with mercury until the metal reaches to the cylindrical part of the open tube, when the apparatus is ready for use.

A. R. POWELL.

Revised psychrometric chart assists high-temperature design [of dryers]. I. LAVINE and R. L. SUTHERLAND (Chem. Met. Eng., 1928, 35, 224—228).—The chart, which is given, comprises a series of curves showing the relation of absolute humidity, latent heat, specific volume, and other humidity data against temperatures from 40° F. to 700° F., this scale being used. In addition, the "humid heat" (heat required to raise 1 lb. of air 1° F. with its accompanying water vapour) is plotted against the absolute humidity, and a series of adiabatic cooling curves is given. The conditions in a dryer are practically adiabatic. An example is given of the use of the chart in determining the conditions of the exit air from a direct-contact coal dryer, the temperatures and the humidity of the entering air being known.

C. IRWIN.

Building of containers for severe service.

T. McL. JASPER (Ind. Eng. Chem., 1928, 20, 466—470).—The building of containers for severe conditions involves a knowledge on the part of the builder of the service conditions proposed, the proper design and protection of the vessel during building, and a suitable test. The strength of steels at high temperatures can only be obtained by long-period tests. These may be carried out in molten lead, the stress-breaking time curve being plotted. Corrosion is greatly increased in many cases by increase of stress or increase of pressure with equal stress. Full-scale tests are desirable and have shown the necessity of reinforcing manholes and the proper design of heads. A riveted or hammer-welded joint is always much weaker than the plate, however, and if such are used reinforced openings etc. are of no value. To avoid strains in fabrication, steel more than 1 in. thick should be formed hot. The author tests vessels before use with gas oil to a pressure of 75% of the calculated yield point pressure. C. IRWIN.

Treatment of distillates. MORRELL and BERGMAN.

—See II. Syntheses under high pressure. MORGAN and others.—See III. Turbidimeter. HAASE and THIELE.—See XI.

PATENTS.

Heating the charge in rotary furnaces. G.

BOJNER and A. P. and A. H. PEHRSON (B.P. 264,825, 12.1.27. Swed., 20.1.26).—The material is heated by a gaseous fluid circulating between the material chamber and an outer chamber rotating with the furnace; in the latter the gas is heated by external means. In some cases the gas may be forced through the material, and if condensable products are given off from the material they may be cooled for the recovery of useful products, the non-condensable part being reheated and recirculated. An intermediate heat-interchanger may be introduced to avoid waste of heat. B. M. VENABLES.

Regenerative furnace. D. B. JONES and F. C.

SWARTZ (U.S.P. 1,667,319, 24.4.28. Appl., 28.4.26).—The longitudinal flues below the chequer chamber are directed towards the stack, and each flue has a line of nozzles, built into the floor, pointing towards the stack. Fluid supplied under pressure to the nozzles thus blows foreign matter into the stack. F. G. CLARKE.

Regenerative heat-exchanging device. AKTIE-

BOLAGET LJUNGSTRÖMS ANGTURBIN (B.P. 275,231, 25.7.27. Swed., 31.7.26).—A regenerative mass is provided with longitudinal passages for the two gases (but no transverse passages) and is shaped as a body of revolution, e.g., two segments of a cylinder with two blanked-off smaller segments between. At each end of the active segments partitions (of which the plane contains the axis) oscillate synchronously, and at the ends of the blanked-off portions are chambers forming the inlets and outlets for the two gases; thus, at the end of a stroke, one segment is giving up heat only and the other receiving only, but during a stroke part of each segment is effecting each purpose. B. M. VENABLES.

Mixing machine. H. H. HACKSTEDDE (U.S.P.

1,666,285, 17.4.28. Appl., 21.6.24).—A mixing bowl furnished with heating (or cooling) jacket is arranged to

swing about a vertical hinge at the side. The inlet for heating medium is in the hinge, and the outlet is through a pipe with a swivel on the same axis as the hinge.

B. M. VENABLES.

Rotary drying apparatus. J. B. VERNAY (B.P.

282,432, 16.12.27. Fr., 18.12.26. Addn. to B.P. 262,464; B., 1927, 719).—As the material passes from one concentric cylinder to the next, it is subjected to a grinding action between teeth formed on the rotating end-plates and teeth on the lower part of the fixed casing. B. M. VENABLES.

Disintegrator. [Emulsifier.] B. CUNIFF, Assr. to COLLOIDAL EQUIPMENT CORP. (U.S.P. 1,666,640, 17.4.28. Appl., 28.4.26).—A pair of discs with intercalating ridges forming a sinuous passage rotate in close proximity at different speeds, the inlet being axial and the outlet for emulsion at the periphery, but one disc is provided with a deep flange which embraces the other disc and causes the final outlet to be comparatively near the axis. B. M. VENABLES.

Settling tanks. G. S., S. W., and M. J. BINCKLEY (B.P. 287,611, 17.12.26).—A settling tank, for material such as activated sludge in which it is desired that the settled material be discharged promptly, is formed of inverted conical shape, the inlet for sludge being over the whole periphery of the tank, and the outlet for clear effluent through a number of central troughs which are just below the surface of the liquid and are preferably radial. A truncated conical partition is hung within the tank, producing an outer or inlet zone which increases in area downwards. Baffles may be placed in the thick pulp zone to prevent turbulence. B. M. VENABLES.

Centrifugal separators. COMP. IND. DES MOTEURS A EXPLOSION (C.I.M.E.), and D. PERRIER (B.P. 283,963, 19.1.28. Ger., 22.1.27).—The fluid containing solid matter to be separated is passed from an axial inlet to an axial outlet at the other end in a sinuous path through concentric double-conical baffles, so that the material is subjected to a number of treatments with the same (*sic*) centrifugal force. B. M. VENABLES.

Centrifugal separators. H. LIST (B.P. 285,061, 10.2.28. Ger., 10.2.27).—A centrifugal apparatus is provided with a cover and feed and discharge passages in the hinge thereof, also a valve in the feed which is open when the cover is closed and *vice versa*.

B. M. VENABLES.

Centrifugal separators etc. F. A. G. NOEL and E. W. BAILY (B.P. 288,746, 24.1.27).—The separated liquid collects in an annular chamber formed by an enlargement of the neck of the bowl, and into this annulus of liquid is inserted the annular slit-like mouth of a fixed bell-shaped device, with double walls. The liquid in the space between the walls of the fixed bell rotates more slowly than the liquid outside, producing a difference in pressure which causes the liquid to discharge inwardly and upwardly to a fixed collecting chamber and spout. The result is discharge of the liquid without aeration. B. M. VENABLES.

Centrifugal. C. SCHAUM, Assr. to FLETCHER WORKS, INC. (U.S.P. 1,667,493, 24.4.28. Appl., 15.1.27).—The driving shaft lies wholly in a stationary tub separated

into two compartments, the lower of which contains turbine blades attached to the shaft and operated by steam, and the upper containing the rotating basket and a governor for regulating the supply of steam to the turbine.

L. A. COLES.

[Rotary] filter. R. A. ELTON (B.P. 287,694, 25.2.27).—The drum of a rotary filter is constructed of two crescent-shaped segments, with longitudinal spaces between so that both the inner and outer surfaces are used for filtering. The filter is operated on the batch system either by pressure with a closed casing or by vacuum with an open casing, and at the start the casing may be completely filled. Detachment of cake is effected by reverse-current flow of liquid or air, and discharge of solid cake by a worm-conveyor in the bottom of the casing, or of sludge, re-formed from the cake and wash liquor, by gravity. Scoops are provided on the edges of the crescents to agitate the pulp and cause a sufficient quantity to flow over the interior filtering surfaces.

B. M. VENABLES.

Rotary drum and vacuum filters. RECKITT & SONS, LTD., and E. H. HALL (B.P. 287,725, 7.4.27).—A rotary drum filter is provided with longitudinal rollers pressing on the cake either directly or through a compression belt, the object being to iron out cracks in the cake. The rollers are given a circumferential movement—preferably reciprocating—over an arc of the filter drum, their position absolute and relative to each other, pressure, and speed being all adjustable.

B. M. VENABLES.

Filters, strainers, etc. V. R. HEFTLER (B.P. 272,236, 7.6.27. U.S., 7.6.26).—A number of washers are assembled on a central bolt with clamping nut; they are stamped in the shape of a wheel with spokes, and alternately with and without a circumferential rim, the latter being thin so that narrow passages will be left for liquid to flow from the spaces between the spokes to the outside, the thickness of the passages being adjustable to a certain extent by compression applied to the pile.

B. M. VENABLES.

Cake former for continuous rotary filters. R. O. BOYKIN, Assr. to N. R. VAIL (U.S.P. 1,666,279, 17.4.28. Appl., 3.3.26).—A pulp distributor is applied to the upper part of a rotary filter drum, comprising a tank of which the bottom is formed by the filtering surface. The distributor is connected to an adjacent pulp reservoir by means which have a certain degree of flexibility.

B. M. VENABLES.

Method of filtering. J. F. WAIT (U.S.P. 1,667,465, 24.4.28. Appl., 10.3.25).—In pressure filtration, a portion of the filter cake formed is removed, the remainder being mixed with the bulk of material within the filter and subjected to further filtration.

H. ROYAL-DAWSON.

Continuous evaporation, distillation, or separation of difficultly distillable liquids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 288,775, 17.2.27).—The liquid is sprayed by a disc rotating rapidly about an inclined axis, which itself rotates more slowly about a vertical axis. The spray impinges on the heated walls of a chamber forming a very thin layer, and the

volatile portion evaporates almost instantaneously. Evaporation of easily decomposable substances may be effected at a temperature below the true b.p. by admixture of a volatile fluid (e.g., benzol may be mixed with tar), by the use of vacuum, or by a stream of inert gas.

B. M. VENABLES.

Treatment of gases, vapours, or liquids. CHEM. WERKE CARBON GES.M.B.H. (B.P. 266,673, 29.11.26. Ger., 24.2.26).—Hard, moulded blocks of active carbon manufactured as described in B.P. 247,241 (B., 1926, 308) are used as an absorber. Twenty-one claims are made relating to the properties of the blocks that may be used.

B. M. VENABLES.

Gas and liquid contact apparatus. C. M. ALEXANDER (U.S.P. 1,667,136, 24.4.28. Appl., 27.11.26).—A rectification tower is provided with bubble plates which seal with the casing and in which the down-flows to each storey are at one side in one vertical line, the flow of liquid being across the plate in contact with the gas and back in compact streams to the next down-flow.

B. M. VENABLES.

Separation of liquids or gases. J. HOWARD (B.P. 286,446, 23.2.27).—An apparatus for separating oil from compressed air or similar purposes comprises a curved pipe in which the mixture flows, the oil emerging through a passage in line with the final direction, but the air emerges at right angles to the plane of the curved pipe. Ribs may be formed on the interior of the pipe to increase the surface.

B. M. VENABLES.

Separation of liquids from gases. J. T. H. WARD (B.P. 288,740, 21.1.27).—An apparatus for separating water and/or oil from steam (and similar purposes) comprises a U-shaped conduit in which the fluid is caused to flow in a sinuous motion by baffles which also form pockets to collect the liquid.

B. M. VENABLES.

Apparatus for storing volatile fluids. SULZER FRÈRES SOC. ANON. (B.P. 263,775, 3.12.26. Switz., 30.12.25).—Volatile liquid such as ammonia which is to be delivered partly as vapour and partly as liquid is stored in a container provided with a heating coil and heat insulation; the liquor space is connected to an evaporator to produce the quantity of vapour required, and the vapour space to a compressor and condenser to liquefy any excess vapour formed.

B. M. VENABLES.

Air classifier. A. H. STEBBINS (U.S.P. 1,666,476—7, 17.4.28. Appl., [A] 14.10.26, [B] 16.3.27).—In (A) a spiral conduit is built round a straight vertical pipe, openings being provided in the floor of the spiral and between the spiral and straight pipes. Suction is applied to the top of the straight pipe, and the material is fed to the top of the spiral. As the material runs down the floor of the spiral the finer particles are picked up by the air drawn in through the openings and carried by it to the central pipe. In (B) a long vertical casing formed with circumferential grooves is provided with a co-axial fan or stirrer with blades extending practically the full length of the casing. An upward current of air is produced by another fan, and the material is delivered into the casing at a point intermediate the ends; the lighter particles are drawn out by the air current and the heavier flung by the stirring fan to the sides of

the casing, down which they slide to an outlet at the bottom.

B. M. VENABLES.

Apparatus for separating dust from gases. A. STIEVENART (B.P. 284,980, 6.2.28. Belg., 4.2.27).—A cyclone separator is provided with vanes which divide the incoming stream of air and with damping means to collect the dust.

B. M. VENABLES.

Gas separator. J. F. BORDEN, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,667,139, 24.4.28. Appl., 20.3.24).—A vertical, cylindrical casing is provided with a number of horizontal, dished partitions with openings at their centres. Through the openings is inserted a shaft upon which are discs rotating below each opening. Fluid is admitted at the top of the casing and suction applied just below each partition.

B. M. VENABLES.

Identification of crystals. F. F. READ (B.P. 288,496, 19.10.27).—A ray of light is introduced into the crystal and the position of the emerging rays recorded in a suitable way on a target. The crystal is adjusted to a standard position such that the central emergent ray is coincident with the incident ray.

B. M. VENABLES.

Treatment of liquids and gases (U.S.P. 1,668,104).—See II. **Filter plate** (U.S.P. 1,669,362).—See VII. **Spraying of molten substances** (B.P. 276,955—6).—See X. **Purification of liquids by distillation** (B.P. 287,607).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Clean coal in the coking industry. R. LESSING (Gas World, 1928, 88, Coking Sect., 58—62).—In the study of the non-removable constituents of coal, disregard of the difference between ash and mineral matter entails considerable errors. By calculations of the loss of water of hydration in clay and shale, of carbon dioxide liberated from carbonate, and of the addition of oxygen to pyrites and other iron compounds with a coal containing about 10% of ash, the carbon in the pure "ash-free dry" coal may be 3% higher than a corrected value. Similarly, the calorific value and volatile matter are liable to error quite apart from the influence on the latter of the catalytic effect of individual mineral components. To study the constitution of individual mineral components before decomposition and oxidation, consideration must be given to their rôle during coal formation. The composition and content of ash in individual coal components are typical and form a method of identification. The ashes of clarain and vitrain, with a high percentage of water-soluble and low acid-insoluble compounds, represent the original plant ash. The ash of durain, 75% insoluble in hydrochloric acid, is clay substance as proved by its alumina: silica ratio being equal to that of kaolinite. Fusain ash is derived from the carbonates and other salts from water diffused into the decomposing plant remains, and is allied to the segregation of the same substance in the cracks of coal seams in the form of cleat and partings. A water-soluble inorganic accessory of many bituminous coals is sodium chloride. Ash content and composition vary within one group according to particle size: thus, the ash in graded fractions shows great friability of

fusain dust, decrease of ash in clarain with decrease in size and increase in its fines, uniformity in durain and increase of ash in dirt with decrease in size. That coking slack consists of such mixtures of clean coal, dust, and refuse is the basis of all cleaning operations. A coke having 10% of ash is equivalent to 6.7—7% of ash in the coal carbonised. Of this, only 1—1.5% is contributed by the inherent ash, the balance being derived from the dirt. The bulk of the inorganic constituents in coke is inert material which reduces the strength and causes the production of breeze.

H. S. GARLICK.

Influence on the coking of coal of the extraction of the bitumen content with tetralin under pressure.

E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 121—122).—The bitumen content of ordinary coking coal has been extracted by treating the coal in an autoclave at 250° with tetralin. The extraction of the bitumen has a marked effect in increasing the density of the resulting coke. The solid bitumen compounds have been shown to have a greater effect in this direction than the liquid ones.

A. E. MITCHELL.

Graphitisation of petroleum coke. Relative catalytic effect of various metals. M. L. SCHLEIFSTEIN (Canadian Chem. Met., 1928, 12, 59—61).—When amorphous carbon is heated at a high temperature in the presence of certain inorganic oxides it is converted into graphite. The effect of the addition of different substances on the conversion of petroleum coke has been studied by making rods of the finely-ground coke and 10% of a special pitch; these rods were heated in an electrical resistance furnace at 2400—2500° for 5 hrs. The change was followed by measurement of the resistance of the product, in ohms/cm.³, at a number of temperatures, and it was found that the effect of calcium, manganese, magnesium, aluminium, iron, nickel, chromium, and boron increased in that order. The graphite formed by the use of boron had a resistance only slightly higher than that of Ceylon graphite and below that of Acheson graphite. Treatment of the coke without addition of any catalyst caused partial graphitisation, which may be due to the heat alone or to the presence of inorganic impurities.

R. H. GRIFFITH.

Correlation of physical and chemical properties of cokes with their value in metallurgical processes.

I. W. T. K. BRAUNHOLTZ and H. V. A. BRISCOE (Gas World, 1928, 88, Coking Sect., 63—67).—An attempt is made to correlate certain properties of cokes with their blast-furnace value. Samples have been drawn from the Cumberland and Durham area to conform with certain requirements, viz.:—(a) manufacturing conditions of the coke to be as regular as possible; (b) information to be available as to blast-furnace behaviour; (c) cokes to be of different blast-furnace values; (d) ash content approximately the same. It is found that, in general, there exists a direct relation between the shatter index of a coke and its value in metallurgical processes. In a series of tests to determine the influence of varying the manipulative details of the test as laid down by the U.S. Bureau of Mines, reliable results were obtained from three successive tests, each with 25 lb.

instead of 50 lb. of coke, and investigations into the influence of size of the shatter machine box on the shatter index are being continued in order to standardise the apparatus and eliminate such secondary influences as cushioning of the coke pieces. Repeated shattering of coke remaining on the screen from a former shatter test shows increase in the index; thus, the previous history of a coke sample is of great influence on the observed shatter index. The average weight of pieces of coke of the same size is approximately the same for all cokes examined. In order to study combustibility a special apparatus was designed and information obtained regarding the behaviour of different cokes when burning with a constant and limited supply of air. Gas analyses are conducted after combustion has started, and fuel consumption is calculated from the ratio of carbon dioxide to monoxide. Average sp. gr. and porosities of the cokes examined are tabulated. Investigation of the capacity for water absorption showed that different pieces of coke may absorb different quantities of water during the initial period, but the subsequent steady rate of absorption is practically the same for different pieces of a given coke, and there is no relation between absorption and shatter index or porosity. Preliminary experiments on a coking test of coal in tins indicates that the "box test" is capable of supplying quantitative information as to mechanical strength of coke, and comparison is being extended to other properties.

H. S. GARLICK.

Sardinian fuels, especially those of the Perdasdefogu basin (Ogliastra). II. R. BINAGHI and A. BRUNDO (*Annali Chim. Appl.*, 1928, 18, 122—134; cf. B., 1926, 82).—This district contains anthracite, the quality of which improves as the depth increases. One of the galleries of the Nuraghe S. Pietro mine yielded a sample showing 8.51% of ash and 0.84% of sulphur. The fuel is suitable for producing metallurgical coke, and could be used for smelting the adjacent iron ore, but the nature of the district involves transport difficulties.

T. H. POPE.

Liquid fuels from coal. R. CHAUX (*Bull. Soc. chim.*, 1928, [iv], 43, 385—411).—A lecture.

Rapid determination of the water contents of solid and liquid fuels. J. TAUSZ and H. RUMM (*Gas- u. Wasserfach*, 1928, 71, 417—420).—The fuel is mixed with excess of tetrachloroethane and the mixture distilled through a short column and condenser, the condensate being collected in a burette over mercury. The distillation is continued until the condensate is clear; on being kept the distilled tetrachloroethane and water separate into two layers and the volume of the water is measured. For great accuracy this measurement is made in a capillary burette, arranged to fit by a ground joint to the usual collecting burette, into which the liquid is forced by the mercury column. The method has been tested against the practice of drying the materials under a vacuum at various temperatures and, in general, the agreement by the two methods is satisfactory. Application of the method to the determination of moisture in material from gas purifiers gave more divergent results. For the method it is claimed that a complete determination

can be made in 20 min., that owing to the large quantities of material that can be employed and the excellent separation of water from tetrachloroethane accurate results are possible, that owing to the non-inflammability of the tetrachloroethane the risks of fire attendant on the xylol method are obviated, and that the total water content, free and combined, of the material is given.

A. E. MITCHELL.

Low-temperature carbonisation. F. SCHWERS (*Chim. et Ind.*, 1928, 19, 583—588).—The Salerni carbonisation process, which is suitable for treatment of shales, lignites, etc., can be used for bituminous coal, although further experience will be necessary before such applications are perfectly successful. Work in this direction is proceeding at Ghent; at Zagreb a local deposit of lignite will be carbonised; at Sarrebruck dust coals of various types, and at Resiutta a bituminous shale are treated. No details as to the yield and nature of the tars are yet available.

R. H. GRIFFITH.

Apparatus used in the distillation of coal, shale, and petroleum products by official methods. F. BORDAS (*Ann. Falsif.*, 1928, 21, 146—154).—The standard distillation apparatus used in France for the grading of petroleum and coal-tar oils is described, and reference is made to the methods employed in other countries.

T. M. A. TUDHOPE.

Removal of hydrogen sulphide from industrial gases by alkaline potassium ferricyanide solutions. F. FISCHER and P. DILTHEY (*Brennstoff-Chem.*, 1928, 9, 122—126).—The factors governing the removal of hydrogen sulphide from industrial gases by alkaline potassium ferricyanide solutions according to the reaction $2K_3Fe(CN)_6 + H_2S + 2KOH = 2K_4Fe(CN)_6 + S + 2H_2O$ have been investigated. A loss of sulphur, the extent of which depends on the alkalinity of the solution, occurs through secondary reactions. This loss was reduced to a minimum by employing solutions containing 1 mol. of sodium carbonate and 2 mols. of sodium bicarbonate to each mol. of the ferricyanide. The difficulty of regenerating the ferrocyanide liquors, encountered by Glud and Schönfelder (B., 1927, 321), has been overcome by using electrolytic methods based on the results of von Hayek (B., 1904, 493) and others. The results form the basis of a regenerative process for the removal of hydrogen sulphide from industrial gases with a maximum recovery of sulphur.

A. E. MITCHELL.

Removal of carbon dioxide from industrial gases with alkali carbonates at normal pressures. F. FISCHER and P. DILTHEY (*Brennstoff-Chem.*, 1928, 9, 138—144; cf. B., 1926, 906).—Several examples of absorption of carbon dioxide by solutions of alkali carbonates are described in the literature, but these are mainly concerned with the preparation of the gas in an air-free condition. A gas containing 21.8% CO_2 was washed with 5% potassium carbonate solution at 23°, and 2 litres of the liquid were found sufficient for 60 litres of gas; the amount of water needed to produce the same effect would be very much greater. The washing becomes less efficient as bicarbonate accumulates in the liquid, but the solution can be

regenerated by boiling or by blowing air through it when warm; passing a stream of air through the solution under reduced pressure is not satisfactory even at 60°. Experiments with granules of solid sodium hydroxide in the presence of limited amounts of water vapour were not successful as the reaction between carbon dioxide and the solid was too slow; it was found that sodium carbonate absorbs less carbon dioxide in a given time than the potassium salt, but could be more rapidly regenerated. Absorption was assisted by warming the solution to about 70–80°, and regeneration proceeded best at 90–95°. A tower for the continuous treatment of gas in this way is 80 cm. high and 8 cm. in diam., and stands immediately above a similar tower, and liquor from the first, which has absorbed carbon dioxide, is revived by an air-blast in the second; regenerated solution is pumped back to treat more gas. The success of the process depends on adjustment of the relative rates of flow of gas, air, and liquor, on the temperature and concentration of the solution, and on the nature of the packing in the tower. R. H. GRIFFITH.

Determination of oxygen in illuminating gas. V. FUNK (Gas- u. Wasserfach, 1928, 71, 443–444).—Analysis of gases containing small amounts of air cannot be accurately carried out by the Lubberger method, and an improved form of absorption vessel has been designed. This is used on the Orsat principle, and can be filled with either pyrogallol solution or with a paste of chromous acetate; the latter has the advantage that preliminary removal of carbon dioxide is not necessary. In order to obtain accurate results it is essential to saturate the liquids with gas before use, and to prevent wide fluctuations in the temperature of the apparatus. Under these conditions oxygen can be determined to within 0.01%. R. H. GRIFFITH.

Synthesis of higher hydrocarbons from water-gas [at atmospheric pressure]. D. F. SMITH, J. D. DAVIS, and D. A. REYNOLDS (Ind. Eng. Chem., 1928, 20, 462–464).—Pure dried water-gas was passed over a heated catalyst and the exit gases were cooled with liquid air. Of various catalysts tried “promoted” iron and cobalt chromate were found inactive, but several mixtures of cobalt oxide with other metallic oxides gave small proportions of liquid hydrocarbons. The oil yield was a maximum at 275°. The products also included carbon dioxide, water vapour, and methane. The highest yield of liquid hydrocarbons found was 66 g./m.³ of gas. From 2 to 3% of the carbon monoxide used appeared as carbon deposited on the catalyst. A very low rate of gas flow was also essential. C. IRWIN.

Determination of free carbon in tars, pitches, etc. E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 137–138).—Extraction of tars with solvents such as benzene, toluene, etc. leaves a residue which consists of a mixture of true “free carbon” and bituminous substances. It is possible to separate the latter only by passing the tar through a suitable sintered glass filter, but this method is laborious, and the use of tetralin under pressure at 250° to extract soluble material is found to give satisfactory concordant results. R. H. GRIFFITH.

Phenol recovery plant avoids waste pollution of streams. H. E. JONES (Chem. Met. Eng., 1928, 35, 215–218).—Trouble has arisen through the discharge of ammonia-still effluents containing phenol into rivers the water from which is used for public supply after chlorination, the taste of chlorophenol even in minute proportions being very marked. In such circumstances the recovery of phenol from crude ammonia liquor by extraction with benzol has been developed. Steel towers in pairs are used with perforated plate and coke distributors and countercurrent feeds in series. Two discontinuous caustic soda washers are used alternately, the sodium phenoxide being decomposed with sulphuric acid. 1800 gals./hr. of benzol are used against 1500 gals. of ammonia liquor. The benzol recovery and the extraction efficiency are each 96%. Benzol losses include a small proportion dissolved in the ammonia liquor and eventually returned to the gas stream and are otherwise minimised by the provision of seals to reduce the effect of the alternate inflow and outflow of air in the system. The part-time attendance of one operator is sufficient. C. IRWIN.

Recovery methods and the ammonia problem. L. H. SENSICLE (Gas J., 1928, 182, 384–390).—A historical review of the development of ammonia recovery and a description of the semi-direct and Otto and Simon-Carves direct recovery processes. An efficient type of saturator and results obtained therewith are described. The possibility of cheapening recovery of ammonium sulphate at gas-works by the adoption of direct-recovery methods is discussed, and an attempt made to determine the conditions necessary for successful operation. Attempts made to eliminate the cost of the sulphuric acid by utilising the sulphur in the gas, which is in excess of that required to provide the sulphate radical required by the ammonia, have met with limited success. Calcium sulphate is used extensively for synthetic sulphate manufacture, but is only suitable for very large ammonia output plant. In a process recently developed by the I. G. Farbenindustrie A.-G. (cf. B.P. 285,999; B., 1928, 325), the ammonia is fixed as thiosulphate and polythionate, the success of the process depending on the market outlet for the mixed salts produced. The direct process of ammonia recovery is shown to have potentialities for the successful solution of the effluents problem since by this process they would be reduced to about one fifth of the amount usually present. The noxious constituents are practically confined to phenolic substances which exist in sufficient concentration to render probable their successful extraction by solvents such as benzol. Further attempts to solve the effluent problem without adding to the present cost of sulphate recovery and to produce sulphate with a saving of probably 5% of the selling price are being undertaken. H. S. GARLICK.

Neutralisation of naphtha-yielding oils and a new dry-refining process. E. T. LEEMANS (Petroleum, 1928, 24, 548–549).—A mid-American oil could not be refined by the usual methods owing to the presence of free naphthenic acids in the crude. The latter was therefore heated to 40–100° in a preheater, neutralised by agitation for 1 hr. with the calculated quantity ($\pm 0.5\%$) of dry, powdered, slaked lime, and

allowed to settle prior to distillation. This was preferably carried out under a high vacuum and with a plentiful supply of steam, when the calcium salts of the naphthenic acids remained in the residual asphalt, the properties of which were not appreciably affected thereby. The asphalt could not, however, be used in the preparation of asphalt emulsions. The lubricating oil fractions, after acid treatment and before neutralisation, were found to be strongly acid, owing to the presence of sulphonic acids in the acid tar dissolved in the oil. The latter (6000 litres) was agitated with a solution (40 litres) of water-glass (9 litres of water; 1 litre of water-glass [d 1.38]), whereby sodium sulphonates and free silicic acid were formed, the latter causing the coagulation of the acid tar particles and, by adsorption, preventing dissolution of the sulphonates in the oil. Precipitation was completed in a flat settling vessel at 40–50°. The oil was then pumped to a bleacher, heated at 100°, and neutralised by agitation with powdered slaked lime (1–2%). A filter-aid was added and the oil filtered through a press. The refined product was orange-yellow to yellow, and showed no tendency to emulsify with water. It was of good odour, and its setting point was lowered from +2° to –6°, probably due to adsorption of impurities by the silicic acid gel formed.

R. C. ODAMS.

Thermal decomposition of organic sulphur compounds. W. F. FARAGHER, J. C. MORRELL, and S. COMAY (Ind. Eng. Chem., 1928, 20, 527–532).—The following organic sulphur compounds, being selected as representative of those types which are considered to be present in petroleum, were dissolved in naphtha made from Pennsylvania crude oil, and the vaporised solutions subjected to an average temperature of 496°: mercaptans, alkyl sulphides, alkyl disulphides, thiophen, and elementary sulphur. The products of the thermal decomposition have been identified. Comparative tests have been made with the products of cracking of solutions of *n*-butyl and *is*oamyl disulphides in gas oil at 437° and 17 atm. The types of sulphur compounds produced were the same as those produced at atmospheric pressure for both disulphides. A suggested mechanism for the thermal decomposition is outlined. E. H. SHARPLES.

Ability of insulating oils to resist oxidising influences. S. FACHINI and C. BORELLA (Chem. Umschau, 1927, 34, 331–334).—Under the influence of atmospheric air and high temperature a gradual change in mineral oils occurs which is indicated by the darkening of the oils, by the formation of asphaltenes insoluble in light petroleum, by the formation of such acidic products as asphaltogenic acids and fatty acids of varying solubility according to the nature of the solvent, and by the formation of such fugitive substances as lower organic acids, water, and carbon dioxide. The formation of such oxidation products is dependent on catalytic influences, on the action of metals, the presence of fatty acids, the chemical nature of the oils themselves and of the extent to which they have been refined, and the influence of electrostatic fields. The various English, American, and Continental methods for testing oils, with a view of judging their ability to resist oxidation, are described. Details are also given of the various tests

applied to determine the amounts of oxidation products, saponifiable material, and acid contents of the oils, the methods including gravimetric, volumetric, and solubility determinations. The destructive action of the oils on the insulating material is also determined.

H. M. LANGTON.

Changes in transformer oils. II. K. TYPKE (Z. angew. Chem., 1928, 41, 418–425).—A review of recent literature dealing with the nature of the changes which take place in transformer oils during use, the products formed, and precautions to be taken to retard the progress of these changes.

A. R. POWELL.

Detection and determination of light petroleum, benzene, alcohol, ether, and tetralin in motor fuels. FORMÁNEK (Chem.-Ztg., 1928, 52, 325–326, 346–348).

—The vat dyes Algol Red BTK and 2G colour benzene, its homologues, and tetralin red, and Lake-Red Ciba B colours them rose-red, whereas light petroleum, gasoline, alcohol, and ether remain practically colourless, so that a close approximation of the benzene content of a motor fuel may be obtained by shaking 20 c.c. of the fuel with 0.02–0.05 g. of the dye for 2 hrs. and comparing the colour with standards produced from mixtures containing known amounts of benzene. Aniline Blue 2B colours alcohols, aldehydes, and ketones a deep blue, but is completely insoluble in water, benzene, ether, or petroleum products. Ether is detected by the blue perchromic acid test. A liquid fuel containing less than 20% of tetralin, benzene, ether, or alcohol may be tested for light petroleum by warming with aniline, which dissolves in the warm liquid, but separates on cooling. Alcohol is determined by shaking 100 c.c. of the fuel with 150 c.c. of water coloured with fuchsin, and measuring the colourless layer which represents the constituents other than alcohol. To determine ether in an alcohol-ether mixture, 100 c.c. of the liquid are mixed with 50 c.c. of light petroleum, and the mixture is shaken with 100 c.c. of coloured water. The volume of the top layer, less 50 c.c., is the volume of ether in the mixture. Tetralin is determined colorimetrically by means of Algol Red BTK in the residue left after distillation of the mixed fuel at 200°.

A. R. POWELL.

Designing equipment for chemical treatment of oil distillates. J. C. MORRELL and D. J. BERGMAN (Chem. Met. Eng., 1928, 35, 210–214).—The orifice column is the most satisfactory device for the continuous mixing of oil with acid or other reagents. Several small holes with a fairly high pressure drop at each orifice plate should be used. The flow in cub. ft./sec. is $0.65A\sqrt{2gh}$, where A is the orifice area and h the head, both in feet. Several methods of construction are described, and experimental data for the pressure drop in columns packed with 1 in. Raschig rings and screened gravel are given. The friction rises more rapidly than the rate of flow. Jet mixers are efficient, but the time of contact is short, and they are not suited to the problem of mixing a large volume of oil with a small volume of reagent. A centrifugal pump with the impeller cut down and running in the reversed direction makes a good mechanical mixer. A baffle-plate column is not an efficient mixer in itself, but is a useful auxiliary to an orifice plate column.

C. IRWIN.

British coking industry and some of its products.

R. RAY (J. Inst. Fuel, 1928, 1, 220—233).

Utilisation of town's refuse and refuse fuels.

A. B. SCORER (J. Inst. Fuel, 1928, 1, 288—300).

Chemical study of processes involving carbonisation of coal by internal heating. M. W. TRAVERS (J. Inst. Fuel, 1928, 1, 272—278).—See B., 1928, 77.

Vertical intermittent chamber ovens for gas manufacture. N. J. BOWATER (J. Inst. Fuel, 1928, 1, 234—245).—See B., 1928, 76.

Liquid fuels other than petroleum. A. E. DUNSTAN and H. G. SHATWELL (J. Inst. Fuel, 1928, 1, 262—271).—See B., 1928, 290.

Viscosity of oils. RAASCHOU.—See I. **Hydrocarbons from fats.** MELIS.—See XII. **Butadiene.** BIRCH.—See XIV.

PATENTS.

Machines for separating coal and other materials.

W. H. BARKER (B.P. 288,786, 1.3.27).—Coal is caused to slide down an inclined plate, the surface of which is made of glass. The coal acquires a higher velocity than the impurities, and therefore can jump a larger gap at the end of the plate. Suitably-placed chutes receive the coal, impurities, and intermediate substances. The end of the inclined plate is made of polished stainless steel or material less resilient than glass, which prevents bouncing of the coal. T. A. SMITH.

[Smokeless] coal-ball manufacture. C. B. WISNER (B.P. 275,602, 25.7.27. U.S., 7.8.26).—Ground bituminous coal is subjected to a preliminary heat-treatment in the presence of air before it is carbonised and converted into balls by passage through an externally-heated rotary retort. During the pretreatment a temperature is maintained just insufficient to distil the hydrocarbons, and by regulating the time of treatment and the quantity and temperature of the air the amount of binder present is controlled, whereby sticking of the charge to the walls of the retort during carbonisation is prevented, and the strength and average size of the product are determined. Complete removal of hydrocarbons is not effected and cracking is avoided. The temperature employed is below that at which the coal ceases to expand, in order to prevent fracture of the product. A strong coking coal, asphalt pitch, or pitch from the oil obtained in the process itself is added when non-coking coal is used, either during the pretreatment or during the carbonisation. Suitable apparatus is described. F. G. CLARKE.

Manufacture of briquettes from low-volatile fuels, fine ores, flue dust, etc. with the aid of binders. KOKS- U. HALBKOKS-BRIKETTIERUNGS-GES. M.B.H. (B.P. 274,876, 20.7.27. Ger., 24.7.26).—By adding sulphite waste liquor or cellulose pitch to the clay binder used in the manufacture of briquettes, the mechanical strength of the latter is considerably increased, without appreciable increase in the ash content, and the good qualities which characterise clay briquettes are retained. C. O. HARVEY.

Manufacture of fuel briquettes. J. S. ROBESON (U.S.P. 1,667,358, 24.4.28. Appl., 26.9.24).—A tubular

briquette formed from fine coal, oil, and lignone, the outer portion being harder than the inner, and containing a lower percentage of volatile matter.

H. ROYAL-DAWSON.

Treatment of fine coal. E. W. WILKINSON, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,667,277, 24.4.28. Appl., 4.8.26).—The fine coal is agitated with water and less than 1% of an organic compound containing sulphur, which assists agglomeration under such conditions that the coal becomes coated with oil and floats. It is separated and freed from excess water. F. G. CLARKE.

Distillation of fine granular coal or bituminous material. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H., L. HONIGMANN, and F. BARTLING (B.P. 289,137, 18.1.27).—Contrary to previous experience with the dry distillation of fine granular coal, peat, etc. by a continuous process in which the material is spread in a layer on a revolving annular horizontal hearth in a furnace, it has been found that, owing to the rapid and complete heating attainable, the throughput may be greatly accelerated, and the output considerably increased by employing shallow layers of material of 1—3 mm. thickness. C. O. HARVEY.

[Batch] distilling apparatus for carbonaceous fuel. Carbonising process. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,668,131—2, 1.5.28. Appl., [A] 14.10.22, [B] 11.3.25).—(A) A vertical, cylindrical distillation chamber contains upon its base an electric grid having upward projections. (B) Charges of fuel introduced into a chamber by means of an endless conveyor are heated therein by a superposed heat-storage element. Gases are removed from the chamber from a point below the charge. F. G. CLARKE.

Retorts for the distillation of lignite and other carbonaceous materials. R. W. EASTON (B.P. 288,700, 11.1.27).—A vertical retort may be tapered and is divided by vertical partitions; both may be of cast iron. The water vapour and poor gases are exhausted at the top, whereas the rich gas is exhausted through horizontal conduits crossing the retort at some point intermediate between the top and bottom. These conduits are of circular form, with openings only on the underside or of inverted U-shape. B. M. VENABLES.

Production of power [from low-grade fuel]. M. J. TRUMBLE (U.S.P. 1,667,403, 24.4.28. Appl., 25.10.22).—Steam is blown through a retort containing low-grade fuel, and the hydrocarbon vapours liberated are passed with the steam to a power unit such as a turbo-generator. The condensable hydrocarbons are recovered from the condenser of the turbine. T. A. SMITH.

Manufacture of fuel gas. I. G. FARBENIND. A.-G. (B.P. 269,594, 19.4.27. Ger., 19.4.26).—Granular fuel is gasified in a horizontal tube lined with refractory material, along which the glowing fuel is carried with a hopping or jumping motion by a current of the gasifying agent; or the fuel may be partially gasified in the above manner and then passed into a separator which is operated as an ordinary gas producer. A. B. MANNING.

Manufacture of water-gas. N. J. BOWATER (B.P. 288,797, 17.3.27).—A downward stream of pulverised fuel reacts with an upward stream of steam introduced at the bottom of a reaction chamber, which contains a heat-radiating surface. The preferred apparatus consists of a vertical chamber, the circular cross-section of which is greater at the top than at the bottom, in order to reduce the speed of the rising gases and thus prevent unburned carbon being carried away. The chamber should be lined and lagged. The heat-radiating surfaces may consist of an earthenware cylinder heated to incandescence by the combustion of gaseous, liquid, or solid fuel in its interior. The products of combustion may be used to preheat the air and fuel used for the combustion or for raising steam. The heat for the reaction may also be supplied by any hot gases or electrically. F. G. CLARKE.

Generation of water-gas from bituminous fuel. O. MISCH (B.P. 288,426, 21.3.27).—A water-gas generator is surmounted by a retort in such a way that the distilled fuel falls by gravity into the generator (cf. B.P. 276,530; B., 1927, 867). The retort is heated externally at its lower end by water-gas, using a specially designed compressed-air burner, by oil or by powdered fuel, in such a way that the temperature is higher than 700° at the lower end of the retort, and falls to about 250° at the charging end. Oil or tar may be injected into the incandescent coke layer in the retort in order to raise the calorific value of the gas. A short intermediate period may be arranged between the "blow" and the "run" in the producer, during which a little steam is passed for scavenging the remainder of the nitrogen and carbon dioxide. A. B. MANNING.

Manufacture of carburetted water-gas. F. B. HAYES, Assr. to WESTERN GAS CONSTRUCTION CO. (U.S.P. 1,667,518, 24.4.28. Appl., 25.9.24).—Up-run water-gas is passed through a carburettor where it is enriched by admitting oil. The down-run blue water-gas is by-passed with respect to the carburettor, which during this part of the cycle is employed to make oil-gas. The blue water-gas, carburetted water-gas, and oil-gas are finally mixed in a holder. T. A. SMITH.

Gas producers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 288,491, 8.10.27).—Finely powdered or granular fuel is gasified in a disc-shaped chamber set with its axis horizontally. The fuel and the gasifying media (air and steam) are admitted tangentially, through a number of openings or jets in the periphery, in such a way that the fuel particles and gases rotate with a high velocity and the fuel is rapidly gasified. The gaseous products and ash are led away through a central opening in one side of the chamber. The combustion and reduction zones may be separated by an annular wall within the chamber. The producer may be used as a burner, the air for combustion being supplied to the gas at its point of exit from the chamber. A horizontal extension pipe, preferably with openings for the admission of additional gases, may be attached to the discharge opening of the chamber. A. B. MANNING.

Water-gas plant. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 288,463, 6.7.27).—The plant

includes a steam superheater and waste-heat boiler, the superheater being of the U-tube type and having its tubes covered with non-metallic protective material of high heat-storage capacity; this material serves also as an igniter for the mixed "blow" gases and air. The water space near the top of the boiler tubes is made of larger diameter than the rest of the boiler, and a plurality of pipes is provided between the steam drum and the top of the boiler in order to facilitate the removal of steam. A. B. MANNING.

Manufacture of hydrogen or gas mixtures containing hydrogen from hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 288,662, 15.11.26; cf. G.P. 403,049; B., 1925, 71).—Hydrocarbons are partially burned by means of oxygen or air enriched with oxygen, possibly with the addition of water vapour, and the resulting gases treated with activated catalysts which facilitate the conversion of a mixture of hydrocarbons, carbon monoxide, and steam into a mixture containing hydrogen and carbon monoxide or dioxide. When carbon monoxide is not desired in the product, steam is added during the second stage and suitable catalysts are used to give a mixture of hydrogen and carbon dioxide, the latter being finally removed, if desired. More than one catalyst may be employed; e.g., the preheated gases may first meet a heat-resisting catalyst and then a more active catalyst. The partial combustion may be effected by means of flame or by surface combustion, with employment of solid materials upon which catalysts may be deposited. The temperature of the partial combustion is reduced by freeing the hydrocarbons from organic and inorganic sulphur compounds. Instead of subjecting the whole of the gases to partial combustion, a portion may be burned and the products mixed with the remaining hydrocarbons. Thermal decomposition may be employed in the first stage of the process instead of partial combustion. The hydrocarbons may be passed through externally heated tubes and the resulting carbon collected; or, in a discontinuous process, the gases are passed in contact with hot fireproof materials, which are reheated at a subsequent stage by burning the deposited carbon. The decomposition may be facilitated by adding carbon dioxide, oxygen, or steam. F. G. CLARKE.

Washing or cleaning of gas. H. A. BRASSERT & CO. From H. A. BRASSERT (B.P. 289,118, 24.12.26).—A gas washer suitable, for instance, for blast-furnace gases, and of robust and inexpensive construction, consists of a tower filled with bundles of wooden hurdles and provided with water sprays. The hurdles are of graded mesh, those at the base of the tower having the largest apertures. The arrangement is such that the gases follow a tortuous path, and are given a preliminary treatment involving alternate compression and expansion. C. O. HARVEY.

Bituminous emulsions. J. A. MONTGOMERIE (B.P. 288,821, 23.4.27).—The process of B.P. 226,032 (B., 1925, 91) is modified by the use of solutions of alkali carbonates or bicarbonates in place of the hydroxides, whereby precipitation of calcium and magnesium salts, present in the water used, is effected, and breaking of

the emulsion due to the formation of insoluble organic compounds of these metals is avoided.

C. O. HARVEY.

Coating of material [paper] with bituminous substances. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 273,281, 16.6.27. Holl., 28.6.26).—The paper pulp is mixed with asphalt in a "gel" form, obtained by treating an emulsion of asphalt with an electrolyte in presence of a protective colloid such as albumin.

C. O. HARVEY.

Recovery of ammonia from ammoniacal liquor. SOUTH METROPOLITAN GAS CO., P. PARRISH, F. C. SNELLING, and O. W. WEIGHT (B.P. 288,436, 13.4.27).—Waste gases from the retort settings pass through a waste-heat boiler and then through a preheater. The ammoniacal liquor is passed through the preheater on its way to the ammonia still, which is operated by steam from the waste-heat boiler.

A. B. MANNING.

Manufacture of motor fuels. I. G. FARBENIND. A.-G. (B.P. 278,341, 2.10.26. Ger., 5.11.25).—The fuels consist of mixtures of natural benzene, aliphatic alcohols (e.g., methyl alcohol), and products obtained by destructive hydrogenation. Such products, unlike pure natural benzenes, are readily miscible with the lower alcohols (cf. B.P. 259,944; B., 1928, 222).

C. O. HARVEY.

Motor fuel. T. MIDGLEY, JUN., ASSR. to GEN. MOTORS CORP. (U.S.P. 1,668,022, 1.5.28. Appl., 6.1.25).—The fuel contains a hydrocarbon, a volatile compound of a metal, and a halogenated lubricating oil.

H. ROYAL-DAWSON.

Manufacture of synthetic liquid fuels. SOC. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 275,585, 12.7.27. Fr., 3.8.26).—Petroleum and similar fuels are produced by hydrogenation of the vapours obtained by distillation of materials such as lignite, peat, shales, tars, heavy oils, etc. in a chamber containing no catalyst. The hydrogen is rendered active by treatment with a catalyst in a separate chamber, fouling of the catalyst being thus avoided (cf. B.P. 274,846).

C. O. HARVEY.

Gasification of liquid fuels. GASIFIER CO., ASSEES. OF G. L. REICHELME (B.P. 277,604, 4.1.27. U.S., 16.9.26).—Liquid fuel is gasified in a specially designed apparatus by injecting air and fuel into an enclosed space and supplying air to promote an enshrouding zone of complete combustion (indicated by a blue flame). A catalysing medium situated in this zone effects gasification of the fuel.

C. O. HARVEY.

Manufacture of lubricating oils. F. W. HALL, ASSR. to TEXAS CO. (U.S.P. 1,668,313, 1.5.28. Appl., 5.10.23).—Sulphuric acid is added to cylinder oil, and the treated oil mixed with a lighter petroleum oil and allowed to settle from the resulting acid sludge.

H. ROYAL-DAWSON.

Decolorisation of [lubricating] oils at high temperatures. P. W. PRUTZMAN, ASSR. to CONTACT FILTRATION CO. (U.S.P. 1,667,984, 1.5.28. Appl., 6.12.26).—A stream of oil containing an absorbent is heated to a temperature above that of vaporisation of the oil, and is subsequently cooled below that temperature, so that no vapours are lost.

C. O. HARVEY.

Manufacture and treatment of lubricating grease for use with grease guns etc. C. BREWER (B.P. 288,796, 17. and 22.3.27 and 13.5.27).—A lubricating grease which can be moulded into bars and remains sufficiently solid to be handled under any climatic conditions is made by mixing a lubricating distillate from a paraffin or other suitable base-oil with stearine or a stearine compound (tallow, soap, etc.), distilling the mixture with superheated steam and a current of oxygen, and re-combining distillate and residue after freeing the latter from asphalt.

C. O. HARVEY.

Treatment of hydrocarbon oils. W. M. CROSS, ASSR. to GASOLINE PRODUCTS CO. (U.S.P. 1,667,883, 1.5.28. Appl., 20.7.22. Renewed 5.3.28).—The oil is fractionally distilled and the fractions are subjected to processes of refining and redistillation. Portions of the residues and higher fractions are cracked under pressure, and the resulting cracked liquids are recirculated through the fractional distillation plant.

C. O. HARVEY.

Removal of sulphur and its compounds from hydrocarbon oil. G. L. WENDT, ASSR. to STANDARD OIL CO. (U.S.P. 1,668,225, 1.5.28. Appl., 16.7.24).—Lower mercaptans are added to the oil, which is then treated with "doctor solution."

H. ROYAL-DAWSON.

Treatment of hydrocarbons. W. HESS (U.S.P. 1,668,275, 1.5.28. Appl., 7.9.26. Ger., 25.9.25).—The hydrocarbons are pretreated with gaseous and then with liquid sulphur dioxide.

H. ROYAL-DAWSON.

Oxidation of mineral hydrocarbons. J. H. JAMES (U.S.P. 1,667,419, 24.4.28. Appl., 6.12.21).—A mixture of the vaporised hydrocarbons and a gas containing oxygen is brought into contact with a catalyst at a temperature sufficiently high to bring about partial oxidation.

C. O. HARVEY.

Refining of petroleum products. F. B. THOLE, S. F. BIRCH, and W. S. G. P. NORRIS (B.P. 288,931, 13.10.26).—Petroleum fractions boiling within the benzene-kerosine range are decolorised and deodorised by a refining process, the novel step in which is a treatment with a solution of an alkali hypochlorite containing not less than 1.5% of free caustic alkali. The objectionable sulphur compounds are thus rendered easily removable by subsequent acid-alkali refining and distillation. The fractions may also undergo partial refining prior to the hypochlorite treatment.

C. O. HARVEY.

Apparatus for treating and combining hydrocarbon liquids and gases and other liquids and gases. N. H. COLWELL (U.S.P. 1,668,104, 1.5.28. Appl., 29.4.24).—The apparatus consists of a chamber into which liquids may be pumped under pressure through vaporising nozzles.

C. O. HARVEY.

Removal of wax from oil. G. D. GRAVES, ASSR. to STANDARD OIL CO. (U.S.P. 1,668,239, 1.5.28. Appl., 27.7.25).—The waxes which hinder filtration or sweating of ordinary wax from oil distillates are removed prior to the final de-waxing process by filtration at a temperature at which the ordinary wax remains in solution, but below that temperature at which crystallisation of the objectionable waxes occurs.

C. O. HARVEY.

Fractionation of oils. C. M. ALEXANDER (U.S.P. 1,667,340, 24.4.28. Appl., 12.1.26).—The vapours obtained by distillation of the oil are brought into contact with reflux condensate of which the final b.p. is lower than that of the highest-boiling constituent of the vapours. The vapours are then brought into contact with a higher-boiling condensate.

C. O. HARVEY.

Distillation [of various substances] and generation of [high-pressure] steam. T. CHMURA (B.P. 259,214, 29.9.26. Ger., 29.9.25).—Benzene vapour, which has a heat capacity at 350° of about 4½ times that of superheated steam and a decomposition temperature of about 800°, is used as a circulating heat-carrier in the distillation of the high-boiling fractions of petroleum, lignite, oil shales, etc., or for heating a boiler for extra-high-pressure steam. The benzene may give up its heat through a separating wall or directly to the material. For the latter case, methods, *e.g.*, rectification, are described for purifying and avoiding loss of benzene. To avoid decomposition of benzene, the heater and conduits may be lined with tin or ferrous sulphide, or a proportion of hydrogen may be introduced. Low-value heat carriers such as steam, carbon dioxide, or natural gas may also be admixed to a moderate extent.

B. M. VENABLES.

Manufacture of fuel briquettes. A. M. HART, Assr. to HART CARBON FUEL CO., LTD. (U.S.P. 1,668,643, 8.5.28. Appl., 9.8.27. U.K., 22.4.26).—See B.P. 279,140; B., 1928, 79.

Treatment of pulverised vegetable fuels. S. SHIMAMOTO (U.S.P. 1,668,660, 8.5.28. Appl., 1.11.26).—See B.P. 273,556; B., 1927, 693.

Carbonisation of coal. W. RUNGE, Assr. to INTERNAT. COAL CARBONIZATION CO. (U.S.P. 1,669,023—4, 8.5.28. Appl., 6.11.24).—See B.P. 242,622; B., 1926, 428.

Composition to be used in connexion with anti-knock compounds. W. GAUS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,693, 17.4.28. Appl., 17.4.25).—See B.P. 252,019; B., 1927, 357.

Treatment of emulsions [of hydrocarbon oils and water]. C. F. PESTER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,669,379, 8.5.28. Appl., 20.3.23).—See Can. P. 247,810; B., 1927, 403.

Evaporation etc. of liquids (B.P. 288,775). Treatment of gases etc. (B.P. 266,673)—See I. **Lead sludge** (U.S.P. 1,667,550). **Production of gas** (U.S.P. 1,667,272).—See VII. **Cement from oil shale** (U.S.P. 1,665,993).—See IX.

III.—ORGANIC INTERMEDIATES.

Syntheses under high pressure. Interaction of carbon monoxide and hydrogen. G. T. MORGAN, R. TAYLOR, and T. J. HEDLEY (J.S.C.I., 1928, 47, 117—122 τ).—A study has been made of the interactions occurring between carbon monoxide and hydrogen. The preparatory experiments were made in a static apparatus consisting of a hydraulic compressor through which the mixed gases were forced at 200 atm. into small copper-lined steel reaction vessels charged with

the catalytic mass and heated in a metal bath to 280—410°. The relative efficacy of each catalyst was ascertained by noting the time required for a drop in pressure of from 10 to 20 atm. These catalysts had zinc oxide as a foundation, and this basic oxide was employed either alone or in combination with the acidic oxides of chromium, manganese, molybdenum, tungsten, uranium, and vanadium. For special purposes other basic oxides such as those of cobalt and copper were added to the zinc oxide. These tests indicated that a catalytic mass containing zinc and chromium oxides would probably prove to be an efficient catalyst when employed at 350—400° and 200 atm. Flow-through experiments were then made in apparatus consisting of a Reavell three-stage compressor, a Lennox circulatory pump, a set of three copper-lined steel reaction vessels heated in a bath of molten lead, and an efficient cooling and condensing plant. Each vessel was fitted with inlet and outlet tubes, each of the latter communicating with its own condenser and receiver. Since the circulating gases retained an appreciable amount of the more volatile products of reaction, these gases, after passing from the condensers, circulated through tubes cooled in brine by a sulphur dioxide "frigorifère" refrigerator which operated automatically in reducing the temperature. In this flow-through plant condensations were effected both with a basic zinc chromate catalyst and with admixtures of this material with cobalt chromate. With the latter catalyst, in addition to the main product, methyl alcohol, there was obtained a notable proportion of ethyl alcohol together with such higher primary alcohols as propyl and isobutyl alcohols. Moreover, the organic distillates contained acetaldehyde, propaldehyde, butaldehyde, and other higher aldehydes. The chemical changes involved may be regarded as arising from aldolisation of formaldehyde which leads to acetaldehyde, whereupon this aldehyde can react with more formaldehyde in either single or two-fold aldolisation, thus giving rise to higher aldehydes. These aldehydic substances are subsequently hydrogenated to a series of primary alcohols.

Lead acetate for glycerin determination by the dichromate method. J. KELLNER (Chem. Umschau, 1927, 34, 330).—After describing the preparation of lead acetate according to the I.S.M. 1911 and the purification of a good crude glycerin by the addition of zinc sulphate to the glycerin solution previously made slightly alkaline by caustic soda solution, the effect is described of the basicity of the lead acetate solution on the accuracy of the analytical results. Lead acetate solutions to which varying amounts of lead monoxide have been added are prepared, and from the analytical data recorded it follows that the glycerin found decreases with the increasing basicity of the lead acetate solution. It is not recommended for analytical purposes to use more than 300 g. of lead monoxide with 1 litre of 10% lead acetate solution.

H. M. LANGTON.

Production of vanillin from safrole. A. WAGNER (Chem.-Ztg., 1928, 52, 379).—Vanillin is prepared by heating safrole in an autoclave with absolute alcohol and then with metallic potassium under 16 atm. pressure, the vanillin being isolated from the mixture by a method

similar to that described by Boedecker (B.P. 285,156; B., 1926, 360), whose patent appeared subsequent to the author's work. E. H. SHARPLES.

Synthesis of phosgene. GIORDANI.—See VII. **Furfuraldehyde as ionising medium.** MCKEE and others.—See XI.

PATENTS.

Production of additive products of unsaturated hydrocarbons of the acetylene series. VER, F. CHEM. IND. A.-G., and H. WALTER (B.P. 288,707, 11.1.27).—Acetylene or a homologue (allylene), mixed with excess of vapour of the substance with which it is to react, is passed into a liquid containing a mercury catalyst and mineral acid if desired. Examples are: acetaldehyde from acetylene and steam; ethylidene diacetate or dipropionate from acetylene and acetic acid or propionic acid; vinyl trichloroacetate from acetylene and trichloroacetic acid; α -diisobutoxyethane from acetylene and isobutyl alcohol; $\alpha\beta$ -ethylidenedioxyethane [glycol ethylidene ether] from acetylene and glycol; acetone from allylene and steam or acetic acid; propenyl isobutyl ether from allylene and isobutyl alcohol. C. HOLLINS.

Carrying-out the catalytic oxidation of organic compounds in the gaseous or vapour state. I. G. FARBENIND. A.-G. (B.P. 272,557, 13.6.27. Ger., 14.6.26).—Pure products are obtained in the oxidation of vaporised organic compounds in the presence of non-volatile catalysts, if the reaction mixture coming from the reaction chamber is purified by bringing it into contact with solid adsorbents of large surface (*e.g.*, pumice, earthenware sherds, silica gel, etc.), maintained at a temperature which prevents deposition of the reaction product. The adsorbent must not melt, volatilise, or decompose. It may be impregnated with solutions of salts or bases, or coated with salts or metallic oxides. *E.g.*, commercial naphthalene is oxidised as above, and the gaseous product passed over granulated pumice at 260°. White phthalic anhydride of more than 99% purity is obtained on cooling the purified vapour. B. FULLMAN.

Manufacture of hydrocarbons of the benzene series. I. G. FARBENIND. A.-G. (B.P. 261,393, 11.11.26. Ger., 12.11.25).—Olefines, particularly ethylene, are converted in two stages into benzene hydrocarbons; in the first stage the olefine is passed at high pressure (*e.g.*, 40 atm.) over a hot catalyst (*e.g.*, charcoal at 400°), and the liquid hydrocarbons so formed are then dehydrogenated above 500°, *e.g.*, by passing the vapour over ammonium molybdate at 600° under 5 atm. pressure or by adding phenols and passing the mixed vapours over tinned iron turnings at 650°. Benzene and its homologues are obtained. C. HOLLINS.

Manufacture of dinitrohalogen-aryls [halogeno-dinitro-benzenesulphonamides and -benzamides]. I. G. FARBENIND. A.-G. (B.P. 279,134, 18.10.27. Ger., 18.10.26).—Secondary aromatic amines are condensed with 2- or 4-chloro-3 : 5-dinitro-benzenesulphonyl or -benzoyl chloride in presence of water or other diluent and of an acid-binding agent (*e.g.*, excess of the amine) to give the corresponding benzenesulphonamides or

benzamides. 4-Chloro-3 : 5-dinitrobenzenesulphonyl-methylanilide, m.p. 161°, the 2-chloro-isomeride, 4-chloro-3 : 5-dinitrobenzenesulphonylbenzylanilide, and 4-chloro-3 : 5-dinitrobenzylmethylanilide are mentioned.

C. HOLLINS.

Manufacture of 1-methyl-2 : 5 : 6-trichloro-3-aminobenzene- [2 : 5 : 6-trichloro-*m*-toluidine]-4-sulphonic acid. I. G. FARBENIND. A.-G. (B.P. 277,372, 12.9.27. Ger., 11.9.26).—2 : 5 : 6-Trichlorotoluene-4-sulphonic acid is nitrated with mixed acid at 70° and the nitro-compound is reduced to 2 : 5 : 6-trichloro-*m*-toluidine-4-sulphonic acid. C. HOLLINS.

Manufacture of chlorine-substitution products of 1-amino-2 : 4-dimethylbenzene [*m*-4-xylidine] I. G. FARBENIND. A.-G. (B.P. 278,729, 6.10.27. Ger., 6.10.26).—*m*-4-Xylidine is chlorinated in concentrated sulphuric acid in presence of ferric chloride at 0° to give 6-chloro- (m.p. 97—98°) and 2-chloro- (m.p. 47°) derivatives, separable by freezing out; or at the ordinary temperature with more chlorine to give 2 : 6-dichloro-4-*m*-xylidine, m.p. 56—57°, which is isolated as sulphate. C. HOLLINS.

Manufacture of 2 : 4-dichloro-1-aminoanthraquinone and intermediate products from *m*-dichlorobenzene and phthalic anhydride. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN (B.P. 288,884, 15.8.27).—*o*-2' : 4'-Dichlorobenzoylbenzoic acid, m.p. 100—101°, prepared from phthalic anhydride, *m*-dichlorobenzene, and aluminium chloride, is nitrated with mixed acid to give the 5'-nitro-compound, m.p. 174°, which is reduced to the 5'-amino-compound, m.p. 164°. Cyclisation of this with 100% sulphuric acid at 150—160° yields 2 : 4-dichloro-1-aminoanthraquinone, m.p. 205—206°. C. HOLLINS.

Manufacture of 3 : 4-diaminobenzoyl-*o*-benzoic acids. R. ADAMS, J. M. DAVIDSON, and J. GUBELMANN, Assrs. to NEWPORT Co. (U.S.P. 1,663,229, 20.3.28. Appl., 13.9.26).—3 : 4-Diaminobenzoyl-*o*-benzoic acid from the corresponding 4-amino-3-nitro-compound by reduction with powdered iron and acetic acid has m.p. 176—177°, and by treatment with concentrated sulphuric acid loses water with the formation of a mixture of 1 : 2- and 2 : 3-diaminoanthraquinone. A. R. POWELL.

Manufacture of condensation product from olefines and hydrocarbons of the naphthalene series. R. MICHEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,667,214, 24.4.28. Appl., 21.6.27. Ger., 4.2.26).—Hydrocarbons of the naphthalene series are treated with olefines at above 50° under raised pressure, in the presence of metal halides having catalytic properties. L. A. COLES.

Manufacture of diacyl derivatives of naphthalene and acenaphthene. I. G. FARBENIND. A.-G. (B.P. 279,506, 24.10.27. Ger., 23.10.26).—A monoacyl-naphthalene or -acenaphthene additive compound with aluminium chloride, or the ketone itself in presence of aluminium chloride, is condensed with an acyl chloride. Phenyl α -naphthyl ketone with benzoyl chloride gives 60% of 1 : 5-dibenzoylnaphthalene, m.p. 186.5°, and

20% of the 1:8-isomeride, both boiling about 300°/12 mm. 5:6-Dibenzoylacenaphthene, m.p. 143°, and 5:6-diacetylacenaphthene, m.p. 146°, are similarly obtained in yields of 50% and 70—80%, respectively.

C. HOLLINS.

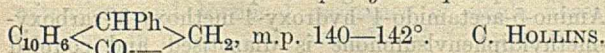
Manufacture of halogen-substituted tertiary aromatic amines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,665, 10.12.26).—Halogenation of tertiary amines of the benzene and naphthalene series in presence of mineral acid (hydrochloric acid) leads mainly to *ortho*-substitution and little *para*. The preparation of 2-chlorodimethyl-*p*-toluidine, b.p. 225—228°, the 3-bromo-compound, b.p. 237—238°, 1-chlorodimethyl- β -naphthylamine, b.p. 185—188°/22 mm., the 1-bromo-compound, b.p. 195°/40 mm. (decomp.; hydrobromide, m.p. 130°), mixed 2- and 4-chlorodimethylaniline, b.p. 206—239°, 2:4-dichlorodimethylaniline, and 3:3-dichloro-4:4-tetramethyldiaminodiphenylmethane, b.p. 276—277°/36 mm., and the 3:3-dibromo-compound is described.

C. HOLLINS.

Manufacture of a 2:3-dichloronitro-1:4-naphthaquinone. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,927, 22.11.27).—2:3-Dichloro-1:4-naphthaquinone is nitrated, without appreciable oxidation, by mixed acid (30% HNO₃) at 40—50°, giving dimorphic 2:3-dichloro-5-nitro-1:4-naphthaquinone, m.p. 175°, in 90—95% yield.

C. HOLLINS.

Manufacture of cyclic ketones of the aromatic series [indanones]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,441, 26.4.27).— α -Unsaturated carboxylic acids or their hydrogen halide additive products are condensed with aromatic hydrocarbons or their halogen derivatives in presence of aluminium chloride to give α -indanones, or in presence of sulphuric acid to give β -arylpropionic acids which are converted into α -indanones by way of the acid chlorides. Benzene and β -chloropropionic acid with aluminium chloride yield α -indanone, m.p. 40°, toluene gives a mixture of 4- and 6-methylindan-1-ones, chlorobenzene a separable mixture of 4-chloro- (m.p. 91—92°) and 6-chloro- (m.p. 79°) -indan-1-ones. From β -chlorobutyric acid are obtained 3-methylindan-1-one, b.p. 255°, its 4-chloro- (m.p. 47°) and 6-chloro- (b.p. 275°) derivatives, and 3:4:6-trimethylindan-1-one, b.p. 250°. Crotonic acid gives with chlorobenzene the same 4- and 6-chloro-3-methylindan-1-ones, with *m*-dichlorobenzene the 4:6-dichloro-compound, m.p. 77—80°, with *o*-dichlorobenzene an oil, b.p. 200°, and with bromobenzene a solid bromomethylindanone, m.p. 83°, and an isomeride, b.p. 260°. Naphthalene condenses with cinnamic acid in nitrobenzene in presence of 85% sulphuric acid at 45° to give β -phenyl- β -1-naphthylpropionic acid, which is converted by phosphorus pentachloride and aluminium chloride into 1-phenyl- α -naphthindan-3-one



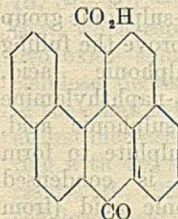
Manufacture of alkylpyrazolanthrones. I. G. FARBENIND. A.-G. (B.P. 264,503, 12.1.27. Ger., 13.1.26).—Pyrazolanthrones are alkylated by heating (e.g., at 170°) with alkyl sulphates, alkyl arylsulphonates, or

mixtures of alcohols with sulphuric acid, in presence of acid condensing agents (e.g., sulphuric acid). Methylpyrazolanthrone, m.p. 221—224°, and 6-chloro-2-methylpyrazolanthrone, m.p. 225—227° (from the pyrazolanthrone derived from 1:5-dichloroanthraquinone and hydrazine), give red vat dyes by alkaline fusion.

C. HOLLINS.

Manufacture of new vat dyes and new intermediate compounds. W. CARPMAEL. I. G. FARBENIND A.-G. (B.P. 288,666, 10.12.26).—1:1'-Dinaphthyl-8:8'-dicarboxylic acid is first treated with an acid condensing agent (e.g., concentrated sulphuric acid at 60°, or zinc chloride in boiling acetic acid) and the resulting benzobenzanthrone-13-carboxylic acid (annexed formula), m.p. 278°, is heated with potassium hydroxide in alcohol, pyridine, etc. to give a dicarboxylated dibenzanthrone or isodibenzanthrone, which, by cyclisation with an acid condensing agent (concentrated sulphuric acid), yields a greyish-green-blue vat dye. Analogous compounds are obtained from bromo-, dibromo-, and chloro-1:1'-dinaphthyl-8:8'-dicarboxylic acid, the corresponding benzobenzanthronecarboxylic acids melting at 288—290°, above 300°, and 268—269°, respectively.

C. HOLLINS.



Manufacture of bromo-derivatives of 1:1'-dinaphthyl-8:8'-dicarboxylic acid. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,957, 10.12.26. Divided from B.P. 288,666; preceding).—Bromination of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in acetic acid gives a yellow monobromo- and, with excess of bromine, an orange dibromo-compound, both melting above 300°.

C. HOLLINS.

Manufacture of an intermediate for [thioindigoid] dyes. I. G. FARBENIND. A.-G. (B.P. 279,436, 14.10.27. Ger., 23.10.26).—3:5-Dichloro-*o*-toluidine is converted by Sandmeyer's reaction into 3:5-dichloro-*o*-tolunitrile, which is hydrolysed with 65% sulphuric acid to give 3:5-dichloro-*o*-toluic acid. The 3-chlorine atom may be replaced by a thiol group and the product used for synthesis of 6:6'-dichloro-4:4'-dimethylthioindigo.

C. HOLLINS.

Manufacture of additive compounds of unsaturated hydrocarbons. H. WALTER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,669,384, 8.5.28. Appl., 18.1.27. Ger., 21.1.24).—See B.P. 288,707; preceding.

Manufacture of phenylglycine or compounds thereof. G. IMBERT, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,668,964, 8.5.28. Appl., 26.10.20. U.K., 2.7.20).—See B.P. 173,540; B., 1922, 170 A.

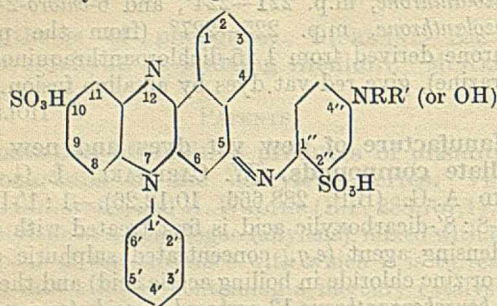
Acetylene as anæsthetic (B.P. 264,826).—See XX.

IV.—DYESTUFFS.

PATENTS.

Manufacture of acid dyes of the phenolphthalein series. J. R. GEIGY A.-G. (B.P. 265,986, 11.2.27. Ger., 13.2.26).—Level blue to green-blue

shades, fast to light and to alkali, are obtained by using azine dyes of the general formula :



containing, if desired, a third sulphonic group and other substituents; the effect of the 2'-sulphonic group is to make the shade greener and to improve the fulling fastness. Diethylisorosinduline-10-sulphonic acid, obtained by oxidation of phenyl- β -naphthylamine together with 4-aminodiethylaniline-2-sulphonic acid, is oxidised in presence of sodium bisulphite to form the 5:10-disulphonic acid, which is condensed with 5-aminoethyl-*o*-toluidine-4-sulphonic acid (from methyl-*o*-toluidine-4-sulphonic acid by coupling with a diazo component and reducing), giving a pure blue wool dye. Diethylisorosinduline-11-sulphonic acid is similarly converted into the 5:11-disulphonic acid and condensed with 4-aminodiethylaniline-3-sulphonic acid for a blue, or with 5-amino-*o*-cresol-4-sulphonic acid for a reddish-blue; 4'-acetamidodiethylisorosinduline-2:5:11-trisulphonic acid (from *p*-acetamidophenyl- β -naphthylamine-7-sulphonic acid and 4-aminodiethylaniline-3-sulphonic acid followed by bisulphite) with 5-aminoethyl-*o*-toluidine-4-sulphonic acid for a greenish-blue. If an acetyl-*p*-diamine is used for the final condensation the product must be hydrolysed in order to attain the desired properties. C. HOLLINS.

Manufacture of acid wool dyes. I. G. FARBENIND. A.-G. (B.P. 282,409, 14.12.27. Ger., 14.12.26).—4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with *p*-aminoacetalkylanilides (*e.g.*, *p*-aminoacetmethylanilide) to give acid wool dyes of redder shade than the non-alkylated compound. C. HOLLINS.

Manufacture of mordant dyes. J. R. GEIGY A.-G. (B.P. 265,203, 26.1.27. Ger., 28.1.26).—Bright chrome printing reds and violets are obtained by diazotising an aminodiarylmethane, prepared by condensing an arylamine (having a free *o*- or *p*- position) with a chloromethyl-salicylic or -cresotic acid, and coupling with any coupling component. Thus, 4'-amino-4-hydroxy-3-methyldiphenylmethane-5-carboxylic acid, prepared by boiling 5-chloromethyl-*o*-cresotic acid with aniline and water, is diazotised and coupled with α -naphthol-3:6-(or 3:8)-disulphonic acid to give a chrome printing red fast to soap, carbonate, and chlorine, or with 1-(2'-chloro-5'-sulphophenyl)-3-methylpyrazolone for a yellow wool dye (after-chromed, greenish-yellow) suitable also for lakes. The diarylmethanes from 5-chloromethyl-*o*-cresotic acid and *o*-toluidine, cresidine, and metanilic acid are mentioned in other examples; the cresidine compound gives with acetyl-H-acid a chrome printing heliotrope; the

metanilic acid compound, with Schaffer acid a chrome printing orange-yellow. C. HOLLINS.

Dyes and dyeing [soluble leuco-esters of vat dyes]. J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 288,673, 31.12.26).—The soluble esters of leuco-vat dyes described in prior patents (B.P. 186,057, 202,630, 202,632, 203,681, 212,546, 218,649, 220,964, 231,889, 245,587, 247,787, 248,802, 251,491, 258,626, 260,638, 260,647, and 261,139) are obtained by using pyrosulphuryl chloride, $S_2O_5Cl_2$ (Grignard and Muret, A., 1926, 1113, 1218) in place of chlorosulphonic acid etc. A small amount of water is not harmful in this case as it converts the reagent into chlorosulphonic acid. C. HOLLINS.

Dyes and dyeing [dry dye and leuco-vat dye preparations]. B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 288,358, 29.12.26 and 13.7.27).—Dye pastes are incorporated with anhydrous or partially hydrated compounds capable of absorbing the water from the dye paste, giving a dry mixture of dye and hydrated substance. The process is applicable to leuco-esters of the Soledon type (in which alkalinity is an advantage) and to dyes in general. Examples are: the leucoflavanthrone ester paste of B.P. 247,787 (B., 1926, 403) with anhydrous sodium carbonate, acetate, sulphate, or disodium hydrogen phosphate; dichloroisodibenzanthrone paste with anhydrous sodium carbonate; 1-amino-2-methylantraquinone paste with anhydrous sodium sulphate. C. HOLLINS.

New azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,788, 2.3.27).—2-Amino-5-acyl-(oralkyl)amino-4'-hydroxy-3'-carboxydiphenylsulphones (B.P. 267,366; B., 1927, 360) are used as diazo components in azo dyes. The acetyl compound, *e.g.*, is diazotised and coupled with 2-methylindolesulphonic acid (reddish-yellow), acetoacetic 4-sulphoanilide (greenish-yellow), bisacetoacetyltolidine (pure yellow), 1-(2':5'-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow), H-acid (reddish-violet, chromed), methyl- β -naphthylamine-7-sulphonic acid (bluish-red), β -naphthylamine-6- or -7-sulphonic acid (yellowish-red), benzylethylaniline or sulphobenzylethylaniline (yellowish-red), 2-methylamino-6-naphthoic acid (red), γ -acid (coupled acid; bluish-red), methyl- γ -acid (violet), *p*-tolyl- γ -acid (brownish-violet), *N*- β -acetamidoethyl-H-acid (brown). The acetyl group in the diazo component may be replaced by other acyl groups, such as chloroacetyl, oxalyl, carbethoxyl, β -chloroethanesulphonyl, carbamyl, carbonyl, benzoyl, anisoyl, phthaloyl, *p*-toluenesulphonyl, salicyl-5-sulphonyl, 6- or 4-chloro-3-nitrobenzenesulphonyl, etc. The *p*-toluenesulphonyl compound, diazotised and coupled with β -naphthylamine-7-sulphonic acid, gives a pure red chrome dye. 2-Amino-5-acetamido-4'-hydroxy-4-methoxy-3'-carboxy-5'-methyldiphenylsulphone is diazotised and coupled with methyl- γ -acid (violet) or γ -acid (bordeaux-red). 2-Amino-5-acetamido-4'-hydroxy-3'-carboxy-4-methyldiphenylsulphone with γ -acid gives a chrome bluish-red. 2-Amino-5-dimethyl(or diethyl)amino-4'-hydroxy-3'-carboxydiphenylsulphone gives with γ -acid a violet, with methyl- γ -acid a bluish-red, with H-acid a blue.

The 5-formamido-compound diazotised and coupled with γ -acid and hydrolysed with 10% sulphuric acid gives a violet. The 5-acetamido-compound is diazotised and coupled with Cleve's acid, rediazotised and coupled with naphthionic acid, hydrolysed with alkali, diazotised again and coupled with Schäffer acid for a direct blue, becoming reddish-blue on coppering. The various dyes obtained show good fastness properties, are usually little changed in shade on chroming, and are suitable for wool, chrome printing for pigments, and for conversion into chromium-containing dyes by boiling with chromium fluoride. Couplings may be made on the fibre in suitable cases.

C. HOLLINS.

Manufacture of [trisazo direct] dyes. SOC. CHEM. IND. IN BASLE (GES. CHEM. IND. IN BASEL) (B.P. 267,162, 8.3.27. Switz., 8.3.26).—Trisazo dyes of the type: dehydrothio-*p*-toluidinesulphonic acid \rightarrow resorcinol \leftarrow usual non-hydroxylated middle component (B) \leftarrow arylamine (A), give level violet to brown shades on cotton, some of which are fast to light. Suitable dyes may be further diazotised on the fibre and developed. Examples are: (i) B = Cleve's acid, A = sulphanilic acid (brown), aniline (red-brown), *p*-aminoacetanilide (hydrolysed, red-brown; developed with β -naphthol, violet-brown), Cleve's acid (brown-violet), or *p*-nitroaniline-*o*-sulphonic acid (yellow-brown); (ii) B = α -naphthylamine, A = β -naphthylamine-4:8-disulphonic acid (violet-brown).

C. HOLLINS.

Production of [chrome]-azo dyes containing two hydroxynaphthalene nuclei. F. STRAUB and H. SCHNEIDER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,669,069, 8.5.28. Appl., 7.4.26. Switz., 25.4.25).—See B.P. 251,266; B., 1926, 702.

Manufacture of trisazo dyes derived from dehydrothio-*p*-toluidinesulphonic acid and resorcinol. B. MAYER and J. GRIMMER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,667,327, 24.4.28. Appl., 24.2.27. Switz., 8.3.26).—See B.P. 267,162, preceding.

Vat dyes (B.P. 288,666).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Attack of cotton by mineral oils at higher temperatures. H. STÄGER (Helv. Chim. Acta, 1928, 11, 377—386).—A comparison is made of the attack of cotton wool by turpentine and transformer oil mixed with various organic acids. The results indicate that the disintegration of the insulation of transformers is not due to acids formed by oxidation of transformer oil, and support the view that interfacial forces are the important factor.

F. J. WILKINS.

Tensile testing of single wool fibres. P. KRAIS (J. Text Inst., 1928, 19, 32—36 T).—The "Deforden" apparatus for measuring the tensile strength, breaking extension, and torsional resistance of single fibres is described in detail, and examples of its application to technical problems are given. There is no difference in strength between wool and "bristly" hairs of the same diameter, but the latter have the higher percentage extension at the breaking point.

B. P. RIDGE.

"Knight test" for feathers. H. F. KNIGHT (Analyst, 1928, 53, 278—279).—To determine the degree

of cleanliness of feathers, a sample (20 g.) is shaken with 1 litre of distilled water, left with occasional shaking for 1 hr., pressed to the side of the bottle, and to 100 c.c. of extract filtered through glass wool is added 1 c.c. of dilute sulphuric acid (1:5), and 0.1*N*-potassium permanganate is run in from a burette two drops at a time until the pink colour persists for 1 min. The standard adopted by the Air Ministry is 40 pts. of oxygen absorbed per 100,000, but 15 pts. per 100,000 both for oxygen and chlorine are easily obtained by proper purification.

D. G. HEWER.

Influence of preliminary treatment with alkali in the preparation of cellulose by means of gaseous chlorine. F. GIORDANI (Annali Chim. Appl., 1928, 18, 87—90).—Mutti and Venturi (B., 1927, 904) found that, when esparto is treated successively with sodium hydroxide solution and gaseous chlorine, the yield of cellulose decreases continuously as the concentration of the alkali is increased, whereas the α -cellulose content at first increases and then decreases, the maximum occurring with 4% sodium hydroxide solution. The author's results (B., 1927, 627) show, on the other hand, that the cellulose yield at first increases and then diminishes as the strength of the alkali is increased, the maximum being obtained with 3% sodium hydroxide. Subsequent experiments indicate that the results obtained depend on the relative proportions of alkali and chlorine employed, and further work in this direction is in progress.

T. H. POPE.

Utilisation of "Kaoliang" stalk. I. Digestion of the stalk by the soda process and effect of preliminary treatment on the digestion. R. YAMAMOTO (J. Cellulose Inst., Tokyo, 1928, 4, 53—66).—A relatively easy-bleaching soda pulp can be obtained from the stalk of "Kaoliang" (*Andropogon Sorghum*, Broth.) if the material is pretreated with hot water. Thus, by digestion first with water at 100—120° for 0.5—1 hr. and then, after removal of the aqueous liquor, with 12% of caustic soda at 130—140° for 3—4 hrs., a 46% yield of light brown pulp was obtained which bleached with 2.97% of available chlorine giving a 36% yield of bleached pulp. It is estimated that the annual production of stalk is about 14,400,000 tons.

D. J. NORMAN.

Cellulose fibre. FEUCHTER.—See XIV.

PATENTS.

Treatment of silk. J. C. MACKEY and J. S. KAUFMAN, Assrs. to VAN RENSSLAER LANSINGH (U.S.P. 1,666,501, 17.4.28. Appl., 19.8.25).—Silk is weighted with tin phosphotungstate. The weighted silk shows the same properties as regards dyeing and printing as does unweighted silk.

D. J. NORMAN.

Degreasing of raw wool and other textile materials. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,230, 16.12.26).—*cyclo*Hexanone or a homologue thereof is used either alone or in admixture with other water-soluble organic solvents. Any solvent remaining in the extracted wool may be washed out with water and recovered.

D. J. NORMAN.

Treatment of animal fibres. I. G. FARBENIND. A.-G. (B.P. 264,146, 22.12.26. Ger., 7.1.26).—Sulphite-cellulose waste liquor, or a salt of ligninsulphonic acid,

is used for the protection of animal fibres during treatment with solutions containing chromium compounds for purposes other than dyeing or mordanting (cf. B.P. 188,632; B., 1923, 652 A). D. J. NORMAN.

Disintegration of vegetable fibre bundles. I. G. FARBENIND. A.-G. (B.P. 271,524, 23.5.27. Ger., 21.5.26).—The material is treated, before or after retting, for about $\frac{1}{4}$ hr. at 10–15° with a solution containing a dialkyl-naphthalenesulphonic acid or a salt thereof (0.1–0.5%), optionally with addition of emulsifying agents and other desizing agents. D. J. NORMAN.

Electrically insulating composition of matter for the construction of partitions etc. I. G. FARBENIND. A.-G. (B.P. 280,178, 11.10.27. Ger., 6.11.26).—Asbestos or other fibrous inorganic material, optionally in admixture with inorganic fillers, is impregnated before or after moulding with a solution containing nitrocellulose, a softening agent (preferably a phenol ester of phosphoric acid), and natural and/or artificial resins. The product is non-hygroscopic and does not become conductive in the event of the combustible material therein coking. D. J. NORMAN.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 268,289, 14.12.26. Ger., 24.3.26).—Improved results are obtained in the manufacture of cellulose esters if the cellulose is pretreated with lactic acid, preferably of about 80% strength. *E.g.*, 1 pt. of cotton is stirred with 20 pts. of 80% lactic acid for about 3 hrs. at 80°. The treated material is then centrifuged, washed, and dried. D. J. NORMAN.

Production of viscose silk fabrics. S. TODA (B.P. 287,807, 22.4.27).—Freshly coagulated viscose threads are loosely twisted (not more than 50 twists per m.) and are then starched and woven into fabric, in which form they are desulphurised and bleached. This procedure permits the filaments to swell during desulphurisation, whereby the covering power of the yarn is increased. D. J. NORMAN.

Production of acetylcellulose compounds and the artificial silk, bands, films, and other artificial textile products, plastic masses, etc. manufactured therefrom. BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 263,771, 29.11.26. Holl., 24.12.25).—Cellulose is acetylated in a bath the water content of which has previously been reduced by allowing the acetic acid-acetic anhydride mixture to remain, with the exclusion of water or water vapour, for at least 2 hrs. before and 1 hr. after the addition of the catalyst. Since the viscosity of the cellulose acetate increases as the quantity of water present in the reaction mixture is reduced, it is possible, by using an acetylating bath which is practically anhydrous, *i.e.*, which has been kept for about 10 hrs., and adjusting the moisture content of the cellulose, to control to a large extent the viscosity and other properties of the final product. D. J. NORMAN.

Manufacture of sheets or films of compositions containing cellulose esters or ethers. SPICERS, LTD., and H. J. HANDS (B.P. 287,635, 23.12.26).—The last traces of volatile solvent are removed from freshly-prepared cellulose ester or ether films by passing the

film under slight tension through a tunnel or similar device into which low-pressure steam is continuously introduced in sufficient quantity to maintain a warm, damp atmosphere without substantially wetting the film. This procedure prevents subsequent cockling and distortion, and removes any electrical charge which may have accumulated on the film during manufacture. D. J. NORMAN.

Cellulosic materials and products obtained therefrom. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 288,657, 8.10.26).—Cellulosic materials are made more reactive to esterifying agents by pre-treating them with small quantities, *e.g.*, 10–30% on the weight of cellulose, of formic acid of 50–100% (preferably 70–90%) strength, optionally in admixture with acetic acid. The solution may conveniently be applied by spraying, and the impregnated material is kept for a period up to 12 hrs. at the ordinary temperature or for a shorter time at a raised temperature. It is unnecessary to remove the formic acid before esterification, but an extra quantity of acetic anhydride must be added to the acetylating bath to compensate for the wastage due to the presence in the cellulose of formic acid and any water associated therewith. D. J. NORMAN.

Treatment of yarns, fabrics, films, etc. H. DREYFUS (B.P. 284,798, 12.11.26. Addn. to B.P. 281,084; B., 1928, 227).—The following compounds may be used, either alone or in admixture, as swelling agents in the weighting of organic substitution derivatives of cellulose by the process of the earlier patent: thiocarbimides, cyanates, or carbimides, particularly those of the alkali metals and ammonium; urea, urethanes, thiourea, thiourethanes, guanidine, or their alkyl, aryl, or aralkyl substitution products. D. J. NORMAN.

Utilisation of lignin. R. GRIESSBACH and J. EISELE, Asss. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,696, 17.4.28. Appl., 18.10.27. Ger., 22.10.26).—Material rich in lignin is extracted with an organic solvent containing a halogen. A. R. POWELL.

Manufacture of paper which possesses properties closely resembling those of hand-made paper. C. MOES (B.P. 282,620, 30.5.27. Holl., 24.12.26).—A continuous web of paper is made on the substantially uninterrupted surface provided by a number of separate, wire-cloth covered suction boxes mounted on a moving endless belt. The suction boxes and couch roll are given a lateral shake as a whole, relative displacements of the boxes during the formation of the sheet being prevented. The finished web shows weak cross-zones, and when torn at these points a "deckle-edge" effect is obtained. D. J. NORMAN.

Proofed paper. J. REID, Assr. to SCUTAN CO. (U.S.P. 1,667,691, 24.4.28. Appl., 26.11.26).—Strong, close-grained, flexible paper is passed through a bath of a bituminous proofing agent of relatively high m.p. (above 121°). Impregnation is limited to the surface layers of the paper by adjusting the duration of the treatment and maintaining the bath at such a temperature that the proofing material is liquid but still viscous. D. J. NORMAN.

Manufacture of artificial textile products. H. DREYFUS (U.S.P. 1,669,158, 8.5.28. Appl., 26.5.23. U.K., 6.7.22).—See B.P. 209,125; B., 1924, 213.

Apparatus for the manufacture of [twisted] yarns or threads. BRIT. CELANESE, LTD., W. A. DICKIE, and R. MARTIN (B.P. 289,321, 22.10. and 8.12.26).

Spinning-pump arrangements for machines for manufacturing artificial textile thread. H. KINDERMANN (B.P. 289,497, 23.11.26).

Apparatus for treatment [jigging] of fabrics in industrial processes. WHITEHEAD & POOLE, LTD., and J. WAGSTAFF (B.P. 289,616, 5.4.27).

Increasing the capacity of drying installations [for paper-making]. AKTIEBOLAGET SVENSKA FLÄKTFABR., Assees. of J. G. OLSSON (B.P. 281,339, 23.11.27. Swed., 29.11.26).

Briquetting of fuels etc. (B.P. 274,876). **Coated paper** (B.P. 273,281). **Bituminous emulsions** (B.P. 288,821).—See II. **Fatty material from emulsions.** (B.P. 289,350).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Affinity of amidated and pyridinated fibres for acid substances. P. KARRER and S. C. KWONG (Helv. Chim. Acta, 1928, 11, 525—529).—The amounts of seven organic and inorganic acids and five acid dyes which combine with amidated and pyridinated cotton fibres (cf. B., 1926, 659) have been determined. Using an amidated fibre of nitrogen content 0.71%, about 0.05 g.-equiv. of each acid is fixed, whilst the value for a fibre of nitrogen content 0.21% is 0.015—0.02, even if the acid bath is varied from 0.1N to 0.5N. Both amidated and pyridinated fibres combine with acid dyes. There appears to be no relationship of the amount fixed and the nitrogen content of the fibre as is the case with acids. H. BURTON.

PATENTS.

Stabilising of bleaching liquors [soap powders etc.]. T. BENCKISER, A. REIMANN, and A. REIMANN, JUN. [J. A. BENCKISER], and F. DRAISBACH (B.P. 289,156, 21.1.27).—The action of alkali pyrophosphates in stabilising solutions containing peroxides and per-salts is improved by adjusting the hydrogen-ion concentration of the solution to p_H 7—8.5 in the absence of soap, or to p_H 7—10 in the presence of soap, by adding mineral acids or acid salts to the solutions, or acid salts to the dry mixtures used in preparing the solutions.

L. A. COLES.

Production of resists on animal fibres. K. DAIMLER, C. E. MÜLLER, and H. GÄRNER, ASSYS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,668,144, 1.5.28. Appl., 12.2.26. Ger., 20.2.25).—See B.P. 248,007; B., 1927, 628.

Spool or cop cores for spool or cop dyeing. J. ANNICQ (B.P. 289,708, 10.10.27).

Dyeing (B.P. 288,358 and 288,673).—See IV.

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

Modified circulation system improves operation of chamber [sulphuric acid] plant. E. L. LARISON

(Chem. Met. Eng., 1928, 35, 229).—In operating three Gay Lussac towers in series with regard to acid feed as well as gas, a larger amount of acid is circulated through these towers than is fed to the Glover tower, the excess simply overflowing from one tank to another at the bottom. The best conditions for Glover operation (restricted feed of nitrous vitriol) are thus combined with a desirably ample feed for complete absorption of nitrous gases in the Gay Lussac towers. A considerable economy in nitre was obtained.

C. IRWIN.

Development of the process and equipment for the manufacture of chemically pure acid by electrical distillation. **Hydrochloric and nitric acids.** C. M. HOFF (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 24 pp.).—The development of an improved commercial process for the production of chemically pure hydrochloric acid by distillation of the crude commercial acid is outlined. In the process finally adopted and operated by the Grasselli Chemical Co. a continuous distillation system using electrical heating is employed and has led to considerable reduction of operating costs and breakage, whilst the working conditions and the quality of the product have improved. The crude acid is fed at a controlled rate into a granite retort, and in it are submerged two graphite electrodes between which an A.C. is passed. These electrodes are plates drilled with several small holes to ensure easy circulation of the acid, and if the current density does not exceed about 300 amp./ft.², there is no evidence of electrolysis or disintegration of the electrodes. Granite was adopted as the material for retort construction after unsuccessful trials with earthenware, glass, and silica. To the top of the retort are connected four pyrex glass condensers by way of an earthenware manifold with four outlets luted with phosphoric acid. From the manifold the condensers slope upwards for a short distance in order to permit of some refluxing and then bend over forming a long vertical section down which the acid passes into a collecting vessel and thence through a "gravity jar" into carboys. At given power input the concentration of the product, as shown by the gravity jar, can be readily controlled by adjusting the rate of flow of crude acid into the retort. A loose packing of glass wool in the end of the condenser joining the retort was found effective in preventing contamination of the distillate with traces of iron. About one seventh of the acid entering the retort is run off to waste in order to keep down the concentration of impurities. One kw.-hr. produced as much chemically pure acid as 3.45 kg. of coal by the old process. Details of plant construction are described and the application of the process, with certain modifications as to materials of construction, to the distillation of crude nitric acid is discussed. H. J. T. ELLINGHAM.

Modification of Ridsdale's method for determining phosphoric acid. A. S. DODD (Analyst, 1928, 53, 276—278).—Certain modifications of the details given by Cameron and Dow (cf. B., 1927, 919) are suggested, chief among which is the use of a simple wash-bottle device, fitted with cotton wool pad, for siphoning off supernatant liquid during the washing of the precipitate. The whole determination is made in one

vessel, and centrifuging is unnecessary; strong nitric acid with 2 vols. of water is used in place of acid of d 1.20; 0.5*N*-sulphuric acid replaces the nitric acid and *N*-sodium hydroxide the 0.5*N*-alkali; and 5 c.c. of dilute nitric acid (1:3) is used instead of 4 c.c. of nitric acid (d 1.20).
D. G. HEWER.

Prediction of boiling points of concentrated caustic-salt [sodium hydroxide-sodium chloride] solutions. F. W. ADAMS and C. W. RICHARDS (Ind. Eng. Chem., 1928, 20, 470—471).—The b.p. of sodium hydroxide solutions of varying concentrations and saturated with sodium chloride were determined for absolute pressure of 50—760 mm. of mercury. If the temperature of the solution, whether of sodium hydroxide or of the latter with sodium chloride, is plotted against the temperature of pure water for varying pressures, a series of parallel straight lines is obtained corresponding to different concentrations. This indicates that within this range Dühring's rule holds good, and if the b.p. at two pressures is known it can be predicted for any other pressure. The data are required for the design of multiple-effect evaporators.

C. IRWIN.

Reduction of sodium sulphate with carbon. P. P. BUDNIKOV and A. N. SYSOIEV (Z. anorg. Chem., 1928, 170, 225—232).—In the reduction of sodium sulphate with carbon at 800—950°, the main reaction is represented by the equation $\text{Na}_2\text{SO}_4 + 2\text{C} = \text{Na}_2\text{S} + 2\text{CO}_2$; carbon monoxide in the gaseous products of reaction appears to be formed by the reaction $\text{CO}_2 + \text{C} = 2\text{CO}$. The formation of carbonyl sulphide, presumably by the reaction $\text{Na}_2\text{S} + 2\text{CO}_2 = \text{Na}_2\text{CO}_3 + \text{COS}$, can also be detected. Further, if hydrocarbons are associated with the carbon, water is produced, which reacts with the sulphide liberating hydrogen sulphide, and forming sodium hydroxide, the latter then combining with carbon dioxide. When the reduction is carried out under a pressure of 15 mm., separation of free sulphur occurs. In an atmosphere of nitrogen at the ordinary pressure, reaction starts at 750—880°, depending on the kind of carbon used, and takes place at first entirely between solids. With sufficient excess of carbon, a yield of up to 95% of sulphide may be obtained in 2—3 min. at 850—1000°. It therefore appears probable that the present hearth process of manufacture might profitably be replaced by a process in which the reaction is distributed over a large volume, *e.g.*, by blowing the finely-divided sulphate into flames of pulverised fuel or carbon monoxide.
R. CUTHILL.

Influence of the "conversion" yield and the temperature of condensation on the purity of the product and on the "condensation yield" in the synthesis of phosgene. F. GIORDANI (Annali Chim. Appl., 1928, 18, 90—96).—The relationships between (1) the proportion, x , of the equimolecular mixture of carbon monoxide and chlorine in the feed gases, (2) the conversion yield, y , which is the fraction of the equimolecular mixture reacting, (3) the condensation yield, ρ , and (4) the gross yield η , when phosgene is produced under different conditions, are considered. As x diminishes, ρ and η also diminish, and the content, τ , of phosgene in the resulting liquid increases slightly;

diminution of y influences appreciably both τ and also the yields, and in practice y should not be below 0.96. A slight excess of carbon monoxide in the initial gases results in greater purity of the phosgene produced. As the temperature of reaction falls, the purity also falls, but ρ and η increase rapidly and tend to assume an asymptotic course. In practice, no lower temperature than -20° should be employed, except in cases where the presence of chlorine in the product is unimportant.

T. H. POPE.

Colloidal behaviour of lime. RAY and MATHERS.—See IX. **Equilibria in reduction of ferric oxide.** FURNAS and BROWN.—See X. **Iron in red lead.** HEINRICHs.—See XIII.

PATENTS.

Manufacture of hydrocyanic acid from crude cyanides. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,667,838, 1.5.28. Appl., 25.3.24).—Crude cyanides containing alkaline-earth compounds are treated with water, any hydrocyanic acid formed being removed from the zone of reaction before any objectionable quantity of azulmic compounds are formed.

H. ROYAL-DAWSON.

Manufacture of phosphoric acid. I. HECHENBLEIKNER, Assr. to CHEM. CONSTRUCTION Co. (U.S.P. 1,667,549, 24.4.28. Appl., 1.4.24).—Dilute phosphoric acid is added to phosphate rock during grinding, and the overflow, which carries in suspension the finer particles of rock, is treated with sulphuric acid.

L. A. COLES.

Filter plate. Cellular silica product. H. L. WATSON, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,669,362—3, 3.5.28. Appl., [A] 31.1.24, [B] 15.8.27).—(A) Particles of silica are caused to adhere to one another without the aid of a binder and to expand into an intumescent, cellular condition. (B) Highly porous silica is prepared by heating crystalline silica at about 1700°, cooling it to below 230°, impregnating the product with a decomposable binder, and reheating rapidly to about 1750°.

F. G. CROSSE.

Ammonia synthesis process and catalyst. A. T. LARSON, Assr. to LAZOTE, INC. (U.S.P. 1,667,322—3, 24.4.28. Appl., [A] 30.7.25, [B] 16.2.26).—Particles comprising an intimate mixture of (A) iron oxide, potassium oxide, and magnesium oxide, or (B) iron oxide, an alkali oxide, and an alkaline-earth oxide, are treated at 300—700° with a gas containing hydrogen and free from catalyst poisons.

L. A. COLES.

Purification of sodium sulphide. R. BOTSON and SOC. IND. DES APPL. CHIM., SOC. ANON. (I.N.D.A.C.) (B.P. 289,354, 14.11.27).—Commercial sodium sulphide is liquefied in a digester by pressure steam, and purified by the addition of 1% of terpinolene. After dilution with water it is decanted and filtered. The density of this liquor is then raised to 1.15 by means of caustic soda (d 1.32) and the product filtered, concentrated, and crystallised out to give pure sodium sulphide suitable for photographic work.

W. G. CAREY.

Production of nitrate of lime. APPAREILS & EVAPORATEURS KESTNER (B.P. 289,370, 13.3.28. Fr., 6.5.27. Addn. to B.P. 279,037; B., 1928, 230).—Sandstone spheres, hollowed or pierced, are added with

the limestone in the proportion of 2% by vol. in order to keep constant the resistance offered by the charge to the nitric acid and gaseous currents, and an inclined grating is provided at the base of the tower to facilitate their removal.

W. G. CAREY.

Manufacture of calcium arsenate. W. C. PIVER (U.S.P. 1,667,490—1, 24.4.28. Appl., [A] 18.1.24, [B] 5.7.24).—(A) Arsenic acid solution of suitable density is added with stirring to dry calcium hydroxide, prepared by adding to calcium oxide just sufficient water to hydrate it, at such a rate that the heat evolved drives off any water present, yielding a dry powdered product. (B) Arsenic acid is added to partially hydrated calcium oxide containing sufficient free calcium oxide to combine with the free water, yielding a dry commercial product.

L. A. COLES.

Manufacture of salts of the alkaline-earth metals from alkaline-earth sulphides. J. Y. JOHNSON.

From I. G. FARBENIND. A.-G. (B.P. 289,196, 14.2.27).—Alkaline-earth sulphides are triturerated, *e.g.*, in an edge-runner mill, with the ammonium salt of the desired acid, the mixture being heated and/or sprayed with water during the process.

L. A. COLES.

Production of potassium ferrocyanide. G. H. BUCHANAN and C. M. HULINGS, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,667,839, 1.5.28. Appl., 27.3.24).—Calcium ferrocyanide is added in excess to a ferrocyanide mother-liquor, then commercial potassium chloride. The insoluble constituents are removed from the solution and treated with sufficient potassium carbonate to decompose any double salt of potassium and calcium ferrocyanide, but insufficient to react with any calcium sulphate present. Potassium ferrocyanide is then obtained from the filtered solution.

H. ROYAL-DAWSON.

Manufacture of copperas [ferrous sulphate crystals]. H. C. STEWART, Assr. to WESTMORELAND CHEM. & COLOR Co. (U.S.P. 1,667,693, 24.4.28. Appl., 3.5.23).—A solution of ferrous sulphate (*d* 1.32—1.38) is allowed to fall in a thin stream against a transverse current of air to atomise the solution and effect crystallisation of the contained salt.

A. R. POWELL.

Manufacture of lead salts [from lead chloride]. S. C. SMITH (B.P. 289,105, 20.10.26).—An aqueous suspension of "active" lead chloride, prepared by pouring a hot solution saturated with sodium chloride and lead chloride into about an equal volume of cold water and collecting and washing the precipitated lead chloride, is treated at the boil with the acids of arsenic, antimony, chromium, tungsten, molybdenum, or uranium for the production of the lead salts of these acids.

L. A. COLES.

Conversion of [recovery of lead compounds from] lead sludge. J. B. HILL, Assr. to ATLANTIC REFINING Co. (U.S.P. 1,667,550, 24.4.28. Appl., 16.7.27).—The sludge is treated with an alkali hydroxide solution in the presence of a gas containing oxygen.

L. A. COLES.

Zinc oxide catalysts. G. BLOOMFIELD, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,838, 8.5.28. Appl., 21.6.26. Renewed 12.10.27).—Zinc oxide is treated with a solution of ammonium nitrate, and the product ignited.

F. G. CROSSE.

Production of basic aluminium sulphate. D. B. BRADNER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,435, 20.3.28. Appl., 19.3.25).—Finely-ground bauxite is digested at 90—105° with a solution of aluminium sulphate containing 7—12% Al_2O_3 and with a reducing agent (*e.g.*, aluminium dross or sulphur dioxide) which reduces the iron to the ferrous condition. The resulting solution contains about 11% of Al_2O_3 , of which 8.5% is present as aluminium sulphate and 2.5% as colloidal hydroxide.

A. R. POWELL.

Preparation of oxygen-evolving preparations suitable for use in respiratory apparatus etc. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 280,554, 9.11.27. Ger., 10.11.26).—Alkali peroxides are stirred and cooled with small quantities of atomised water (preferably 6—7%), oxygen catalysts (*e.g.*, oxides of iron, manganese, etc.) having been incorporated before or after conversion into peroxides.

W. G. CAREY.

Production of gas. H. H. SMITH, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,667,272, 24.4.28. Appl., 14.9.22).—A gas consisting mainly of hydrogen sulphide is produced by passing steam and a restricted amount of air through a heated mixture of carbon and a sulphur-bearing material.

F. G. CLARKE.

Production of mixtures of hydrogen and nitrogen. W. GAUS and W. WILD, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,694, 17.4.28. Appl., 5.2.25. Ger., 14.11.22).—A gas producer is operated with steam and with two different oxygen-nitrogen mixtures, one richer and one poorer in oxygen than atmospheric air and both derived from the rectification of liquid air. The proportions of steam and the two gas mixtures passed over the incandescent fuel are so regulated that the gas issuing from the producer consists of a mixture of 1 vol. of nitrogen to 3 vols. of hydrogen and carbon monoxide together, and that the producer is kept in continuous operation without intermediate heating periods. The producer gas is then subjected to treatment adapted to substitute hydrogen for carbon monoxide, and the carbon dioxide formed in this operation is removed by known methods.

A. R. POWELL.

Apparatus suitable for the gradual production of gases. W. E. KEMMERICH (U.S.P. 1,668,371, 1.5.28. Appl., 1.4.25).—A receptacle, half full, for example, of hydrochloric acid and infusorial earth, has a close-fitting cover which comprises an annular trough having overflow passages leading into the receptacle and supports a central vertical tube the upper end of which is closed. *E.g.*, the tube with its lower end protruding into the receptacle may contain potassium permanganate and sand; water poured into the trough will cause slow evolution of chlorine.

F. G. CLARKE.

Regeneration or reactivation of catalysts. M. LARSSON, Assr. to PHOSPHORUS HYDROGEN Co. (U.S.P. 1,668,539, 1.5.28. Appl., 11.1.26. Swed., 29.9.25).—Catalysts used in the decomposition of steam by means of phosphorus are regenerated by treatment with hydrogen at such temperatures that the inert coating is converted into a substance which will react with steam.

F. G. CLARKE.

Storage of volatile liquids (B.P. 263,775).—See I.
Hydrogen from hydrocarbons (B.P. 288,662).—See II.

VIII.—GLASS; CERAMICS.

Preparation of water-glass [sodium silicate]. K. VON REIBOLDT (Chem.-Ztg., 1928, 52, 345—346).—Practical hints are given for improving the quality and output of fused sodium silicate and for converting the product into water-glass. A. R. POWELL.

Iron in red lead. HEINRICHS.—See XIII.

PATENTS.

Manufacture of composite glass. H. J. MALLABAR, and NON-INFLAMMABLE FILM CO., LTD. (B.P. 288,782, 24.2.27).—A relatively thin layer of cellulose acetate, capable of being cast from solution in continuous rolls, is fastened by gelatin between sheets of glass. Bonding is secured by drying the gelatin layer with which the surfaces are coated, treating with a softening liquid, and uniting under pressure. Before applying the gelatin layer to the surface of the cellulose acetate, the latter is suitably treated, as by partial hydrolysis or with a solvent liquid, whilst the glass surface is prepared by coating with a gelatin solution containing a hardening agent. The gelatin layer for bonding is made by dissolving this substance in a water-acetic acid-methyl alcohol-acetone mixture. The glass may be rendered colourless by imparting a bluish-violet tint to the cellulose acetate, gelatin, or glass. A. COUSEN.

Enamels based upon barium and strontium compounds and adapted for the enamelling of cast and wrought iron, copper, and other metals. SOC. MIGEOT FRÈRES & ARNOULD (B.P. 273,260, 19.5.27. Fr., 26.6.26).—A transparent enamel for applying to metals previously coated with opaque enamels is free from lead and titanous acid, and contains 5—50% of baryta or strontia. A suitable composition contains 21% of borax, 11.25% of boric acid, 25% of baryta, 8.75% of felspar, 11% of alkali, 11.5% of lime, 10% of zinc oxide, and 1.5% of alumina. Fluorine compounds and colouring agents may also be added. L. A. COLES.

Glazes for the manufacture of ceramic ware. J. W. MELLOR, and CERAMIC PATENT HOLDINGS, LTD. (B.P. 288,679, 4.1.27).—A "leadless" glaze, capable of maturing at low temperatures, consists of approximately 2 pts. of Cornish stone (or its equivalent) and 1 pt. of borocalcite or an insoluble or very sparingly soluble calcium borate, and it contains no artificially fritted material. Other constituents, designed to improve dipping properties and tendency toward settling, may be added. A. COUSEN.

Manufacture of ceramic products. L. KERN (U.S.P. 1,666,936, 24.4.28. Appl., 13.10.25).—A mixture of sand, hydrated lime, and silicic acid is moulded under pressure and treated under pressure with a gas which hardens the product by reacting with it. F. G. CLARKE.

Drying of sanitary pipes and other clay goods. G. O. LAWTON, S. DOWTHWAITE, and ALBION CLAY CO., LTD. (B.P. 289,225, 15.3.27).

IX.—BUILDING MATERIALS.

Colloidal behaviour of lime. K. W. RAY and F. C. MATHERS (Ind. Eng. Chem., 1928, 20, 475—477).—The plasticity of a lime putty made by slaking quicklime with excess water as compared with a putty made by adding water to the dry hydrate is believed to be due to the formation of an irreversible colloid during slaking. This colloid is destroyed during the drying of the hydrate and not again formed. Exceptional cases are due to the presence of magnesia which is not completely slaked during hydration. In order to confirm this hypothesis, the variation in the rates of settling of suspensions of different limes under an electric field was determined. Considerable evidence of the existence of electrically charged particles in plastic limes was obtained. The effect of these colloid particles in conferring plasticity or non-drying qualities on putties may be explained by supposing the charges to hold a film of water round the particles more firmly. It is shown that this must be the case if the electrical charges are due to the selective absorption of ions, an osmotic pressure being set up against which the drying must be performed. C. IRWIN.

Waste heat in Portland cement manufacture. H. POOLEY, JUN. (Engineering, 1928, 125, 497—498, 562—563).—In view of the greater uniformity of the final product, Portland cement is being made increasingly by means of the "wet process," though the recuperation of heat obtainable on the average wet-process plant is much less than that necessary to run the mechanical side of the works, in contrast to conditions prevailing on the "dry-process" system. Methods which tend to eliminate the wastage of a large amount of heat leaving the kiln at about 450° include those which artificially increase the temperature of the exit gases until they contain the heat necessary for the works' plant, or, in the reverse direction, cooling the gases to a greater extent by useful work before leaving the kiln. Thus, a rotary kiln normally about 200 ft. long might be shortened by 40 ft., and the waste gases, now at about 700°, provide all heat required by the power plant; the coal consumption would be reduced from 42% (expressed as a percentage of the cement produced) to a comparative value of 35%. The effective drying zone of the wet-process kiln can be increased by, e.g., spraying the slurry and generating the necessary power independently, the estimated total coal consumption then being 37%. In a process in which the slurry is dewatered from, say, 38% to 20% of contained water, as obtains at the plant of the Ford Motor Co., the consumption of coal may be expected to be 29—30% of the cement produced. Other operating disadvantages may then be expected, though possibly the atomiser system would show the greater relative advantage when the water content of the slurry is high. Conservation of heat from the cooling clinker is best effected by a current of air which later is used in the kiln combustion, the cooling taking place in what is essentially an extension of the lower end of the kiln—a practice already in common usage. C. A. KING.

PATENTS.

Production of unsintered binding means [hydraulic cement] from oil shale and limestone.

O. TETENS (U.S.P. 1,665,993, 10.4.28. Appl., 29.12.24. Ger., 21.5.22).—Raw shale is mixed with limestone, the mixture distilled to remove volatile oils, the residual mass of coke and lime burnt in the same kiln, and the product ground (cf. U.S.P. 1,536,165; B., 1925, 549).

A. R. POWELL.

Cement compositions. E. C. PALM and H. T. THORSSON (B.P. 288,903, 28.9.27).—The compositions comprise Portland cement and a powdered mineral containing hydrated magnesium silicate, *e.g.*, chlorite or serpentine, but practically free from asbestos.

L. A. COLES.

Manufacture in the cold state of tiles for walls and slabs for floors. A. FEIST and F. L. KOPP (B.P. 288,867, 18.7.27).—A hardening agent, *e.g.*, a mixture of bituminous material with silicofluorides, is incorporated into a pasty mixture of magnesium chloride lye with an intimate mixture of finely-ground burnt magnesite, sand, talc, and colouring material, and the product is allowed to harden in moulds.

L. A. COLES.

Coated paper (B.P. 273,281).—See II.

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

Equilibria in the reduction of ferric oxide. C. C. FURNAS and G. G. BROWN (Ind. Eng. Chem., 1928, 20, 507—510).—The authors have obtained thermal data for iron and its oxides for temperatures up to 800°, including heats of transformation, absolute entropies, and heat capacities. From these, with the help of extrapolation, the relation of heat of formation to temperature for the two oxides is shown graphically up to 1800°, and that for carbon dioxide, carbon monoxide, and water vapour is also expressed graphically. Equilibrium constants were then obtained for the various possible reactions in the blast furnace from the relation $\Delta F^\circ = -RT \log k$ at temperatures from 100° to 1500°. They indicate that the reduction of ferric oxide with solid carbon can scarcely take place below 1000°, and that whilst the oxidising effect of steam on iron decreases with rise of temperature, the oxidising effect on magnetic oxide of iron increases. Very low dissociation pressures of ferric oxide are indicated even at 1500°. The effect of energy changes which may occur in the formation of solid solutions is not known, and no thermal data are available for ferrous oxide.

C. IRWIN.

Application of the nitridation of certain special steels. L. GUILLET (Compt. rend., 1928, 186, 1177—1180; cf. B., 1926, 410).—Chromium-aluminium steels hardened by nitridation have been used successfully for the cylinders of internal-combustion engines and for propeller-shafts. In the former case wearing is reduced to a minimum by the polishing effects of the pistons, and the normal oil consumption of the motor remains at its initial figure even after prolonged usage. In the latter the polished surfaces obtainable have resulted in a gain of 10% for a shaft working at 3000 r.p.m.

J. GRANT.

Deposition of metals on copper from cyanide solutions. I. Separation and determination of small amounts of lead. B. S. EVANS (Analyst, 1928,

53, 267—275).—Lead, especially in the presence of ammonium oxalate, will deposit quantitatively in the cold from cyanide solutions. Tin, antimony, zinc, cadmium, and nickel do not interfere in the cold. The apparatus used is as for the determination of mercury (cf. Evans and Clarke, A., 1926, 704). A 5 g. sample is dissolved in 60 c.c. of nitric acid (*d* 1.2) (or if tin is present 35 c.c. of nitric acid and 25 c.c. of hydrochloric acid are used), excess of acid is boiled off somewhat after dissolution, about 9 g. of potassium bitartrate are added, the solution is cooled, the liquid made slightly alkaline to litmus with sodium hydroxide, and saturated potassium cyanide solution added until the precipitate has re-dissolved, the necessary excess of this reagent being then adjusted (2—3 c.c. for copper, about 5 c.c. for bronze, 10 c.c. for brass and zinc, and 30 c.c. for nickel and cupro-nickel) at the point where the precipitate is almost dissolved. The deposition tube is prepared much as for the determination of mercury. Percolation is carried on for 2 hrs., the lead completely removed from the copper by glacial acetic acid, the liquid treated with dilute sulphuric acid and a little nitric acid, and the lead in the impure lead sulphate determined by matching it as lead chromate solution against a potassium dichromate solution. The effect of small amounts of elements liable to occur in commercial copper was found to be negligible; results for brass are more uneven than for copper or zinc, owing to segregation of the lead; nickel and iron do not interfere, and phosphates exert a slight restraining influence. Lead can by this process be quantitatively separated from at least 10,000 times its weight of copper, 1000 of tin, 5000 of nickel or zinc, 20,000 of sulphuric acid, or corresponding weights of alkali or ammonium sulphates. An adaptation of the process is described for making it universally applicable in the presence of large amounts of tin, phosphates, or mechanically interfering substances. D. G. HEWER.

Melting sterling silver in high-frequency induction furnaces. R. H. LEACH (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 5 pp.).—Data are given for the melting of sterling silver scrap in two types of Ajax-Northrup high-frequency furnace, (1) the type energised by high-frequency current obtained from a Tesla oscillator with a mercury discharge gap, and (2) that energised by high-frequency current produced by a motor generator set of special design. In type (2) the much lower frequency causes a large percentage of the inductive heating to occur in the metal instead of in the crucible, and produces a vigorous stirring action. Large units of this type are now in use. In a unit of 100 kw. rated capacity supplied with single-phase 480-cycle current at 1800 volts and using water-cooling of the coil, a charge of 432 kg. of the silver alloy is melted in 50 min. with an energy input of 115 kw.-hr. Comparative data for operation with an air-cooled coil are given. There are 48 condensers, of which 12 can be thrown in or out of circuit to control the power factor. Operating costs compare favourably with the oil-fired furnaces previously used.

H. J. T. ELLINGHAM.

Detection of zinc in presence of iron. W. H. HAMMOND (Chemist-Analyst, 1928, 17, 14).—For the

detection of zinc in spent electrolyte, 10 c.c. are mixed with 2 c.c. of 85% phosphoric acid and 1 drop of 0.5% copper sulphate solution containing 0.5% of concentrated sulphuric acid. On stirring with 4 drops of a solution containing mercuric chloride (8%) and ammonium thiocyanate (9%), zinc yields a violet precipitate which is compared colorimetrically with that from a solution containing 0.0025% Zn.

CHEMICAL ABSTRACTS.

Behaviour of plating baths and anodes during electrodeposition of chromium. R. SCHNEIDEWIND and S. F. URBAN (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 29 pp.).—The behaviour of chromium plating baths has been studied with special reference to anodic influences. During electrolysis of chromic acid baths containing potassium sulphate and various amounts of chromic hydroxide using steel anodes, the concentration of trivalent chromium increases or decreases so as to approach an equilibrium value, C_e , which, under given conditions of electrolysis, is practically independent of its initial value. During electrolysis iron dissolves from the anodes at a rate which is constant at given current density. Increase of current density or temperature increases C_e , but increase of current density appears to cause a slight decrease in the rate of anode corrosion (g./amp.-hr.). The effect of current density on C_e becomes very small when the former exceeds 5.4 amp./dm.² at the anode. Increasing the sulphate content of the bath increases C_e and the rate of anode corrosion according to practically linear relations. Intermittent electrolysis increases C_e notably without materially affecting the rate of anode corrosion (calculated on actual time of plating). With lead anodes C_e is much lower than with steel under the same conditions, and the value is hardly affected by intermittent electrolysis. At high temperatures lead dissolves more rapidly than iron or steel. The presence of iron or trivalent chromium in the electrolyte does not notably affect the cathodic current efficiency, but it reduces the range of current densities within which satisfactory deposits can be obtained and it decreases the conductivity of the bath. The coating of lead chromate of high resistance which tends to form on lead anodes can be removed by treatment with a saturated sodium chloride solution slightly acidified with hydrochloric acid. The relative merits of lead and steel anodes for chromium plating are discussed.

H. J. T. ELLINGHAM.

Effect of trivalent chromium and iron on chromic acid chromium plating baths. R. SCHNEIDEWIND, S. F. URBAN, and R. C. ADAMS, JUN. (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 9 pp.).—From experiments on the electrolysis of a bath containing 234 g./litre of chromic acid and 4.3 g./litre of potassium sulphate it is shown that the current efficiency of chromium deposition is a linear function of the logarithm of the current density. The position of this line and, to some extent, its slope are affected by the temperature, an increase of temperature lowering the current efficiency at given current density. Bright deposits of chromium up to 0.0007 mm. thick on copper were obtained when the relation between current density and temperature was such as to give

a current efficiency between 5 and 20%, but the optimum current efficiency, which permits of the production of heavy deposits, is about 13%. A diagram shows the current density required to produce any given current efficiency at any given temperature. The relation between current density and current efficiency at 25° is shown to be quite unaffected by the presence of considerable amounts of trivalent chromium and iron in the bath, but the range of current densities within which bright deposits are obtained is notably reduced thereby. It is believed that trivalent chromium is mainly responsible for this effect (cf. Schneidewind and Urban, preceding).

H. J. T. ELLINGHAM.

Hardness and structure of deposited chromium. L. E. and L. F. GRANT (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 11 pp.).—Chromium was deposited from a solution containing 209 g. of chromic acid, 23 g. of chromic oxide, and 6.4 g. of chromic sulphate per litre on steel specimens which had been polished, brass plated, buffed, and then nickel plated. The hardest deposits, according to tests with the Bierbaum apparatus, were obtained at relatively high current densities and the softest at low current densities. At about 20 amp./dm.² good deposits were obtainable over the temperature range 30–60°; the deposit produced at 45° was harder than those at 30° or 60°. The hardest deposits obtained were produced at 45° and 33.3 amp./dm.² The occurrence of a network of fine cracks was observed in some of the deposits, and in some cases a series of networks could be seen underlying the surface network. Preliminary observations on specimens exposed to corrosive conditions suggest that corrosion of the underlying metal occurs more readily along the lines of the network.

H. J. T. ELLINGHAM.

Photomicrographic study of rough or nodulised electrodeposited nickel. C. UPTHEGROVE and E. M. BAKER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 24 pp.).—A carefully cleaned steel surface was plated successively with about 0.01 mm. of flash nickel, 0.10 mm. of copper, and 0.02 mm. of nickel. Nodules developed on a particular specimen were examined and numerous photomicrographs of horizontal and vertical sections through the nodules are reproduced. The nodules appear always to be the result of outgrowths originating in the normal flash nickel deposit, and the extent of the outgrowths determines the size of the nodule. In no case could the presence of the nodules be related to defects or irregularities of the steel surface or to the method of cleaning, for nodules may start to develop after some thickness of nickel has been deposited. But the flash nickel outgrowths were invariably of a dirty or porous character, and it is believed that in general the nodules are caused by suspended particles of impurities in the bath coming into contact with the metal surface after deposition has started. Hence the use of clear solutions is important for the avoidance of nodular deposits.

H. J. T. ELLINGHAM.

Electroplating on aluminium and its alloys. H. K. WORK (Amer. Electrochem. Soc., April, 1928, 53.

Advance copy. 24 pp.).—An extensive investigation has been made into the conditions under which commercial aluminium and its alloys can be successfully electroplated. For plating on relatively smooth aluminium surfaces, deposition of zinc from a cyanide bath containing free ammonia proved most satisfactory. Nickel, copper, cadmium, or brass can then be deposited over this initial layer of zinc. The plating so produced adheres well, but fails rapidly in corrosion tests, so that it is only satisfactory for dry, indoor service. Improved resistance to corrosion can be attained by heat-treatment of aluminium which has been plated with zinc followed by copper. After heat-treatment the surface may be buffed and nickel plated. Travers' process (B., 1918, 272 A), in which aluminium is plated directly with nickel and then heat-treated at relatively low temperatures for a long time was also tested. As a result of these tests and of trials of other processes described in the literature, it is concluded that plating on smooth aluminium is of doubtful value unless followed by heat-treatment, whilst processes involving heat-treatment are subject to several objections in other ways. Attention was, therefore, given mainly to methods of plating on a surface of aluminium or of its alloys which had been roughened by treatment with a suitable etching solution. It is found that the solutions most suitable for preliminary degreasing of the metal surface and for subsequent etching vary with the chemical and physical character of the alloy. The compositions of such solutions and the period of dipping suitable for various commercial alloys are tabulated. When the surface has been etched, nickel appears to be the most satisfactory metal for the initial plating, and nickel baths of very varied types are suitable for this purpose. The adhesion and corrosion-resistance of electrodeposited coatings 0.013 mm. thick on commercial aluminium and various alloys are tabulated, and photomicrographs showing the mode of adherence of the plating to the etched surface are given. H. J. T. ELLINGHAM.

Electrodeposition of thallium. O. W. BROWN and (Sister) A. MCGLYNN (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 9 pp.).—The electrolysis of baths containing thallium sulphate, fluosilicate, or perchlorate was investigated and the perchlorate type shown to be most promising. Conditions for the deposition of thallium from perchlorate baths were then examined in detail. Good deposits were obtained at room temperature from solutions containing 40–120 g./litre of thalious perchlorate with 10 g./litre of free perchloric acid, 10 g./litre of peptone, and 5 c.c./litre of cresylic acid at 0.4 amp./dm.² Addition of cresylic acid gives smooth, instead of spongy, crystalline deposits, and peptone prevents the formation of thallic oxide on the thallium anode. Electrolysis at a higher temperature (50–60°) improves the deposit, and current densities up to 1.8 amp./dm.² can then be used with concentrated perchlorate solutions containing as much as 60 g./litre of free perchloric acid. H. J. T. ELLINGHAM.

Metallurgical cokes. BRAUNHOLTZ and BRISCOE. **Sardinian fuels.** BINAGHI and BRUNDO.—See II. **Aluminium electrolytic condensers.** SEGMUND.—See XI.

PATENTS.

Ore concentrating machines. J. R. BROADLEY (B.P. 289,178, 28.1.27).—In concentrating machines of the Wilfley type, the table is given a controlled longitudinal motion by means of an attached yoke driven by toggle levers, cams, etc. against the resistance of springs. C. A. KING.

Precious-metal recovery machine. C. LOVE-RIDGE and A. E. COX (U.S.P. 1,668,162, 1.5.28. Appl., 6.10.25).—The machine has flat reciprocating screens arranged with jump-gaps, and material passing through the latter falls upon a stratifying table placed below. Material passing over the gaps is discharged. F. G. CLARKE.

Flotation of ores. G. G. GRISWOLD, JUN., and G. E. SHERIDAN (U.S.P. 1,668,202, 1.5.28. Appl. 6.3.24).—Prior to being subjected to a froth flotation process, an ore containing sulphides of lead, copper, zinc, and iron is treated with sufficient chlorine to inhibit the subsequent flotation of the sulphides of zinc and iron. F. G. CLARKE.

Mechanical [ore-roasting] kilns. ERZRÜSTUNG GES.M.B.H., and G. BALZ (B.P. 288,823, 25.4.27).—In a kiln of the multiple-shelf type the stirring vanes are attached to a carrier arm of relatively light construction which is supported at both ends. The arms may be rotated by mechanical means operating either on the central shaft or on supporting rings at the peripheral ends of the arms. C. A. KING.

Reduction of metallic oxides. W. H. SMITH (B.P. 266,289, 20.10.26. U.S., 17.2.26).—A mixture of iron ore or other metallic oxide and a reducing agent is caused to pass continuously through horizontal, closed, rectangular retorts arranged in banks. The retorts are heated to about 1100° by means of separate heating flues arranged between the retort benches. Heat is recuperated from the cooling metal. C. A. KING.

Preparation of pig iron in highly-heated mixers. K. STOBRAWA (U.S.P. 1,666,428, 17.4.28. Appl., 22.1.27. Ger., 16.2.26).—Pig iron rich in carbon is pre-heated on the flat hearth of a highly-heated mixer by the addition of scrap iron with a low carbon content which has been heated nearly to its m.p. in a separate furnace. The scrap iron is immediately carbonised and its m.p. reduced, while the carbon, silicon, and phosphorus content of the pig iron is reduced without substantially reducing the manganese content. A. R. POWELL.

Manufacture of steel. KABUSHIKI KAISHA NIHON SEIKOSHO (B.P. 273,248, 12.3.27. Japan, 22.6.26).—In the heat-treatment of steel to remove internal stresses and to increase toughness, the steel is quenched from a temperature above the Ac₃ point, reheated to rather above the Ac₁ point, and slowly cooled to rather below the Ar₁ point. The metal is then annealed between Ac₁ and Ar₁ and slowly cooled. C. A. KING.

Ferrous alloys. B. D. SAKLATWALLA (B.P. 288,861, 30.6.27).—An alloy containing 1–8% Cr, 0.5–5% Cu, 1–6% Si, with or without carbon up to 3.5%. C. A. KING.

Steel alloys. F. KRUPP A.-G. (B.P. 276,317, 25.7.27. Ger., 23.8.26).—Heat-resisting steel alloys

contain 15–25% Cr, 15–25% Ni, and less than 0.2% C. C. A. KING.

Coating of metallic articles to protect them from corrosion. R. NOVELLY (B.P. 288,674, 1.1.27).—The metal sheets are provided on one side or on both sides with a thin sheet of an alloy of iron with 6–25% Cr, 20–0.5% Ni, and up to 1% C. A. R. POWELL.

Purification of antimonial lead alloys. H. WADE. FROM U.S. SMELTING, REFINING, & MINING Co. (B.P. 288,939, 21.12.27. Addn. to B.P. 236,782; B., 1925, 727).—Arsenic may be removed from antimonial lead or lead-tin alloys by blowing the molten alloy with a current of air under a flux of molten sodium hydroxide at 335–480° (preferably 450–460°). A. R. POWELL.

Manufacture of alloys. W. M. GROSVENOR and V. P. GERSHON (U.S.P. 1,168,642, 8.5.28. Appl., 3.2.25).—An alloy, having the colour of platinum and capable of being cast or rolled for jewellery, is obtained by melting a base-metal alloy (about 80% Ni and 20% Zn) with 3 pts. by wt. of fine gold. F. G. CROSSE.

Manufacture of metal catalysts. I. G. FARBERIND. A.-G. (B.P. 282,410, 14.12.27. Ger., 14.12.26. Addn. to B.P. 281,218; B., 1928, 235).—Metallic catalysts such as nickel, cobalt, and copper, or mixtures thereof, may be made by precipitation from their ammoniacal solutions by the action of hydrogen or a gas containing hydrogen, preferably under pressure, in the presence or absence of a carrier. The precipitation may be accelerated by the presence on the carrier of a metal having a catalytic action such as nickel, cobalt, or copper.

M. E. NOTTAGE.

Bimetallic [thermostatic] element. H. SCOTT, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,665,935, 10.4.28. Appl., 27.12.26).—The elements, having different temperature coefficients of expansion, consist of a ferrous alloy containing 42% Ni and of a ferrous alloy containing nickel and 1% Mn.

A. R. POWELL.

Electrolytic extraction of tin from alkaline lyes. SIEMENS & HALSKE A.-G. (B.P. 286,673, 7.3.28. Ger., 8.3.27).—In order that the tin may be removed in a compact form, the electrolyte or the initial material, before electrolysis, is treated with an additional and relatively powerful oxidising agent such as ozone, hydrogen peroxide, or the peroxides of the alkali or alkaline-earth metals, until practically all the tin is present in the form of stannate; a cathodic-attractive colloid such as gelatin is then added.

M. E. NOTTAGE.

Electrolytic recovery of pure tin from impure stanniferous materials. BERZELIUS METALLHÜTEN GES.M.B.H. (B.P. 271,521, 23.5.27. Ger., 21.5.26).—Tin may be recovered from tin alloys containing less than 80% Sn by electrolysis of the alloy in the form of fine shavings or granules produced by pouring the molten alloy at a high speed into a quenching liquid. The particles should preferably have a thickness not exceeding 2 mm. and are loosely held together in a permeable container which forms the anode in a sodium hydroxide electrolyte.

A. R. POWELL.

Method and alloy for galvanising metal articles. J. H. LAWES (B.P. 288,741, 21.1.27).—Instead of using

chloride fluxes above the galvanising bath, the bath itself is treated from time to time with small quantities of an alloy of 1 pt. of aluminium with 40 pts. of zinc.

A. R. POWELL.

Apparatus for the spraying of molten substances [metals]. C. J. JUNG (B.P. 276,955–6, 29.4.27. Holl., 3.9.26).

Production of [catalytic] platinum contact bodies. T. VON ARTNER (B.P. 265,938, 18.1.27. Ger., 15.2.26).—See G.P. 440,338; B., 1927, 813.

Briquetting of ores (B.P. 274,876).—See II.
Enamels for metals (B.P. 273,260).—See VIII.

XI.—ELECTROTECHNICS.

Caustic soda primary battery. M. L. MARTUS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy, 17 pp.).—The construction of primary cells of the Lalande type used by the American railroads on signal and track service is described. The copper-copper oxide electrode (cathode) is either of the "loose element type" consisting of powder, granules, or flakes of cupric oxide in a perforated or porous container, or of the "compressed oxide element type" prepared by mixing finely-ground cupric oxide with a binder, such as a solution of sodium or potassium hydroxide or copper sulphate, placing in forms of various shapes, compressing, baking, and treating to produce partial superficial reduction. The latter type is now more widely used, and the electrode is generally in the form of a flat plate or a hollow cylinder. The importance of setting up these cathodes and the zinc anodes in the upper part of the electrolyte is indicated by capacity data quoted. Results are given showing the effect of temperature and the electrolyte concentration on the capacity of the battery expressed as the number of amp.-hrs. obtained during discharge at 3 amp. rate until the voltage fell to 0.5. The higher the concentration of sodium hydroxide, within certain limits, the more efficient is the cell at high temperatures and the less efficient at low temperatures. The variation of capacity with temperature is least when the concentration of the electrolyte is about the standard value, 1050 g. of sodium hydroxide in 4400 c.c. of water. The cells are manufactured commercially in sizes from 75 to 1000 amp.-hr. capacity.

H. J. T. ELLINGHAM.

Adsorption phenomena in primary cells. E. O. JEGGE (Amer. Electrochem. Soc., April, 1928, 53. Advance copy, 8 pp.).—Capacity for adsorbing ammonia from solution is an important property of manganese dioxide for use in the manufacture of dry cells. Thus, pyrolusite from Montana, although it contains only 70–72% MnO₂ is now preferred to the richer Caucasian and Javanese ores, since, owing to its greater adsorptive power for ammonia, it keeps down the alkalinity of the electrolyte and thus lowers the polarisation of the cell during discharge. On the other hand a cell of the Leclanché type can be set up in which charcoal pressed around a carbon rod constitutes the cathode system and no manganese dioxide is used. With such a cell containing 600 c.c. of a saturated solution of ammonium chloride as electrolyte, a zinc anode, and a cathode weighing about 100 g., discharging through

10 ohms down to 0.9 volt gave 45—50 amp.-hrs. After renewing the electrolyte and, if necessary, the zinc, a further discharge can be obtained, and by repeated renewals of electrolyte and zinc and repeated discharges down to 0.8 volt as much as 400 amp.-hrs. may be obtained before the pores of the charcoal become clogged with salts. The theory of the action of the cell is discussed with reference to data for the adsorptive capacity of the charcoal. H. J. T. ELLINGHAM.

Drying and storage of secondary battery plates in the charged state. I. Characteristics of plates dried in carbon dioxide in the charged condition. S. MAKIO (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—Positive and negative plates were fully charged, washed, and dried in carbon dioxide, and then wrapped in paper and stored for 6 months. On setting up batteries with these plates in sulphuric acid (*d* 1.20), a constant terminal voltage somewhat over 2 volts was attained in about 1 hr., and the capacity of the battery, calculated from the first discharge, has an average value of 79% of the rated amount. H. J. T. ELLINGHAM.

Theory of the lead storage battery. L. JUMAU (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—The theory put forward by Féry (cf. B., 1925, 997) as to the chemical changes occurring during the operation of the lead accumulator is discussed critically. Féry's own experimental results on the change in weight of the positive plate during discharge are corrected for change in buoyancy due to change in volume of the active material, and are then found to agree closely with Gladstone and Tribe's classical double-sulphation theory and not at all with Féry's theory. Moreover, Féry's assumption that plumbous sulphate is formed at the negative plate has no sound basis. It is concluded that the double-sulphation theory accounts fully for the reactions occurring in the lead accumulator. H. J. T. ELLINGHAM.

Physico-chemical aspects of the Leclanché dry cell. F. DANIELS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 20 pp.).—The reactions occurring during the discharge of a Leclanché dry cell are discussed. Measurements of single potential were made on electrodes of manganese dioxide electrodeposited on platinum. The potential was found to depend on the p_H value of the solution according to the relation $e = e' - 0.075p_H$ over the p_H range 5—10, where e' is a constant, but to be independent of whether ammonia and ammonium chloride or citric acid and disodium phosphate buffer mixtures were used as the electrolyte. From these data and other evidence the suggestion is made that the fundamental depolarisation reaction in the Leclanché cell is $[Mn(OH)_3] + \ominus = [Mn(OH)_2] + OH'$, the ions of the former type being produced by hydrolysis of manganese dioxide. With manganese dioxide electrodes which had been partially reduced by heating or cathodic treatment, extremely base values of the single potential were obtained at first, but they soon became more positive and eventually approached the normal value corresponding with the p_H value of the solution. The base values are attributed to the presence of manganous hydroxide, and it is

believed that this substance may be produced when very heavy currents are taken from a dry cell or when a cell has run down. The conditions for the precipitation of $ZnCl_2 \cdot 2NH_3$ are examined. After much electricity has been taken from the cell, so that the ammonium chloride concentration has fallen considerably, precipitation of $OH \cdot ZnCl$ may also occur. The factors determining the voltage relations during discharge under various conditions are discussed and the question of shelf deterioration is considered.

H. J. T. ELLINGHAM.

Furfural[dehyde] as a possible ionising medium. J. R. MCKEE, C. A. MANN, and G. H. MONTILLON (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 17 pp.).—The electrical conductivity of various inorganic salts dissolved in furfuraldehyde, stabilised by the addition of 0.1% of pyrogallol, was determined. Only the halides, and especially the iodides, are soluble to any great extent in furfuraldehyde, and solutions of ammonium iodide, potassium iodide, zinc iodide, ammonium bromide, ammonium chloride, and ferric chloride had appreciable conductivities. The conductivity of furfuraldehyde stirred with ferrous, chromic, or cuprous iodides was unchanged, but cuprous and zinc iodides are good conductors in solutions of ammonium iodide in furfuraldehyde. Cuprous iodide dissolves more readily in furfuraldehyde solutions than in aqueous solutions of ammonium or potassium iodide, and the solubility and conductivity of cuprous iodide in furfuraldehyde solutions of the ammonium halides increases with the mol. wt. of the halide. Experiments were made to determine whether metals could be electrodeposited from furfuraldehyde solutions of their salts. Electrolysis of a solution of cuprous and ammonium iodides in furfuraldehyde at about 0.5 amp./dm.² with a copper anode and platinum cathode gave a copper deposit with a current efficiency of 56.5%. From a solution of silver and potassium iodides in furfuraldehyde, silver was deposited as a loose, granular deposit at very high current efficiency. But attempts to deposit zinc from iodide solutions were unsuccessful owing to the formation of tar on the electrodes and to notable reduction of the furfuraldehyde. Electrolytic corrosion of a tungsten anode in a furfuraldehyde solution of ammonium iodide did not occur, and the furfuraldehyde was changed to a rubbery tar.

H. J. T. ELLINGHAM.

Aluminium electrolytic condenser. H. O. SIEGMUND (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 25 pp.).—The electrical properties of aluminium anode films, the sources of energy loss in aluminium condensers, and the conditions affecting their life are discussed and the construction of the commercial form of the apparatus is described and illustrated. The positive electrodes consist of corrugated aluminium plates. The higher the purity of the aluminium, the more rapid is the formation of the film and the lower the resultant leakage current, but failures due to corrosion are decreased by the use of aluminium containing a notable proportion of silicon. Data for the specific resistance of solutions containing ammonia and boric acid in various proportions are given, and the effect of electrolyte composition on the performance and life

of the condenser is referred to. Curves showing the effect of corrosion of the anodes on capacity and leakage current are recorded, and the variation of the capacity of the condenser during operation over a period of several years is also illustrated. H. J. T. ELLINGHAM.

Photoelectric turbidimeter. L. W. HAASE and H. THIELE (Gas- u. Wasserfach, 1928, 71, 414—417).—The turbidities of liquids have been satisfactorily determined relative to distilled water by examining them in a cylindrical cell closed at one end by a parallel-sided glass plate illuminated with constant energy, and at the other by a selenium cell. This selenium cell is connected to form one arm of a Wheatstone bridge, and its *E.M.F.* is balanced in the usual way against a constant *E.M.F.* from an accumulator. A. E. MITCHELL.

Attack of cotton by oils. STÄGER.—See V. **Distillation of acids.** HOFF.—See VII. **Melting silver.** LEACH. **Chromium plating baths.** SCHNEIDEWIND and others. **Chromium.** GRANT and GRANT. **Nickel.** UPTHEGROVE and BAKER. **Plating on aluminium and its alloys.** WORK. **Thallium.** BROWN and MCGLYNN.—See X. **Cane juice defecation.** BALCH and PAINE.—See XVII.

PATENTS.

High-frequency electric induction furnace. R. DUFOUR (B.P. 275,611, 28.7.27. Belg., 4.8.26).—An inductor element, separate from and not attached to the furnace walls, projects into the heating chamber through an opening in the base. J. S. G. THOMAS.

Electrodes [for electric arcs]. W. IRBY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,669,129, 8.5.28. Appl., 7.1.27).—The electrode consists of ilmenite, chromite, magnetite, sodium fluoride, and lithium fluoride.

F. G. CROSSE.

Activation of electron-emission material [tungsten filaments]. J. W. MARDEN and J. E. CONLEY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,665,636, 10.4.28. Appl., 21.2.23).—Tungsten powder is mixed with a small proportion of tungsten carbide, and the mixture pressed into rods which are sintered, swaged, and drawn into filaments. A. R. POWELL.

Electromagnetic bodies. SIEMENS & HALSKE A.-G. (B.P. 271,106, 13.5.27. Ger., 17.5.26).—Ferromagnetic monocrystalline substances, e.g., iron, produced by the decomposition of iron carbonyl at a temperature between 100° and 500° under a pressure less than atmospheric, and possessing practically negligible hysteresis losses, are used as cores for electromagnetic relays, transformers, etc. J. S. G. THOMAS.

X-Ray tube. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,665,637, 10.4.28. Appl., 18.6.27).—An X-ray tube having a molybdenum cathode and a uranium anode is claimed. Uranium has better ray-emitting properties than tungsten. A. R. POWELL.

Insulating composition (B.P. 280,178).—See V. **Recovery of tin** (B.P. 271,521).—See X.

XII.—FATS; OILS; WAXES.

Composition of fatty acids of palm oil. A. RAYNER and S. G. CAMPBELL (J.S.C.I., 1928, 47, 149—150 t).—The general properties of several grades of

palm oil are recorded including the contents and properties of the solid acids. It is shown that (1) the titre and iodine value and solid acid content of palm oils may vary to a considerable extent according to the source of supply of the oil; (2) the difference in these characteristics is due to variations in the amount of solid acids present, and not to any great variation in the composition of the latter; (3) the proportion of stearic acid in the solids is considerable, and varies from 12.5% to 18.5%; (4) the liquid acids calculated from the iodine value contain, on an average, 20.0% of linoleic and 80.0% of oleic acid.

Rational selection of soapmaking properties by means of a new chemical control. S. PRAGER (Chem. Umschau., 1927, 34, 327—330; cf. E. T. Webb, "Modern Soap and Glycerin Manufacture").—By means of the *INS* factor (iodine value — saponif. value) and *SR* (solubility ratio) it is possible to control the hardness, solubility, and lathering properties of soap. For linseed oil the *INS* factor is 15, for palm kernel oil 235, and for coconut oil 250. The relative hardness which an oil imparts to a soap is measured by the *INS* factor, this value varying directly with the hardness. Suitable blending of soft and hard oils enables a soap of any desired hardness to be made (preferably with *INS* factor of 150 for the mixed fats), and simple numerical equations afford a means of calculating the percentage of each oil necessary to give any desired *INS* factor in the mixture. The solubility ratio is determined by dividing the *INS* factor ($\times 100$) of the mixed fats by the sum of all the *INS* factors above 130 (excluding palm kernel and coconut oils) multiplied by the percentages of the respective constituents. For *INS* factors between 130 and 160 the *SR* value should not be below 1.50, though it may be above 2. The solubility ratio is a measure of the solubility and the lathering power of soap: the higher this value the greater are these properties. H. M. LANGTON.

Transformation of fats into hydrocarbons. B. MELIS (Annali Chim. Appl., 1928, 18, 108—114).—Results are given of the fractional distillation of the hydrocarbon oil obtained when a mixture of the sodium and calcium salts prepared from animal and vegetable fats is subjected to dry distillation (B., 1925, 235). The percentage composition of the oil is: b.p. below 150° *d* 0.706—0.759, 20.99; b.p. 150—310°, *d* 0.786—0.843, 45.33; b.p. above 310°, *d* 0.860, 22.66; tar, 7.00; loss, 4.02. These numbers correspond closely with those furnished by the oil obtained by dry distillation of fats in presence of zinc chloride (cf. Mailhe, B., 1925, 534), and differ from those given when natural petrol-oleums are distilled mainly in the higher proportion of light oils. The oil contains aromatic and terpenic products and, indeed, members of all the series represented in natural petrol-oleums; it would probably correspond more closely with the latter if the dry distillation were carried out under pressure, so as to favour rehydrogenation. T. H. POPE.

Determination of iodine values by means of aqueous solutions. A. HANSEN (Dansk Tidsskr. Farm., 1928, 2, 89—133).—Winkler's method for the determination of iodine values (cf. B., 1916, 305) has been critically

examined. The addition of the halogen proceeds rapidly at first, but the rate diminishes greatly after about 80–90% of the total halogen has entered into combination, and shaking the reaction mixture does not appear to accelerate the reaction. Losses due to the volatility of bromine can be prevented by employing 5 g. of potassium bromide instead of the 1 g. originally proposed by Winkler. The mixture with 1 g. of bromide gives a somewhat more rapid addition, of halogen, and yields results which are slightly higher than those obtained by using 5 g., but both mixtures give iodine values which are in good agreement with those obtained by Hübl's method. Using 5 g. of potassium bromide the reaction is complete in 2 hrs. in the case of non-drying oils, but requires 20 hrs. in determinations of drying or partially-drying oils. Prolongation of the time of reaction beyond these limits has no appreciable effect on the result. The iodine values obtained are influenced by the excess of halogen present in the reaction, the best values being given when an excess of 40–50% over and above the theoretical amount is present. It is consequently recommended to use an amount of the substance under examination corresponding to the formula weight w (grams) = $33 \div$ presumed iodine value of substance. Winkler's solutions exposed to a light of 600 c.p. in a special apparatus for varying periods at 20° showed no liberation of bromine, but a series of similar experiments with the addition of olive oil gave irregular results for the iodine value, varying from 90.25 to 102.6. This is ascribed to the reaction taking place in two stages consisting first in the addition of halogen, and then of substitution. During the first period the mixture is not sensitive to light, but becomes so during the later stage, mixtures containing 1 g. of potassium bromide being more sensitive than those containing 5 g. The use of 5 c.c. of ether with 5 c.c. of carbon tetrachloride as a solvent, and 25 c.c. of a 0.2*N*-solution of potassium bromate containing 0.415 g. of potassium iodide and 0.3 g. of potassium bromide as reaction mixture diminishes the sensitivity of the process to light, and shortens the time required for the reaction, but cannot be recommended, as low results are obtained unless the excess of halogen amounts to at least 40%. The following modification of Winkler's original method is finally recommended as the most satisfactory:—A quantity of the substance in grams equal to $33 \div$ presumed iodine value is weighed out into a flask fitted with a ground-in glass stopper, and dissolved in 10 c.c. of pure carbon tetrachloride, 5 g. of potassium bromide in 15 c.c. of water and 25 c.c. of 0.2*N*-potassium bromate solution are added, followed by 10 c.c. of 10% hydrochloric acid. The stopper is quickly inserted and the flask rotated several times to cause the bromine to dissolve in the tetrachloride. The flask is then placed in a metal canister, painted black internally and externally, and rendered light-tight, and left there for from 2 to 20 hrs. according to the nature of the substance under examination. The covering and stopper are then removed and, without taking the flask out of the canister, 10 c.c. of 10% potassium iodide solution are poured in, and, after an interval of a few minutes, 150 c.c. of water are added. The liberated iodine is then titrated with 0.1*N*-thio-sulphate, the liquid being shaken well during the

titration so as to ensure complete decolorisation of the tetrachloride layer. H. F. HARWOOD.

Detection of refined olive oil in virgin olive oils. A. BAUD and COURTOIS (*Chim. et Ind.*, 1928, 19, 602–603).—See B., 1928, 201.

Viscosity of oils. RAASCHOU.—See I. **Determination of glycerin.** KELLNER.—See III. **Wax from sugar cane.** BARDORF.—See XVII.

PATENTS.

Extraction of oils and the like from bodies containing the same. L. J. SIMON, and SIMON EXTRACTING MACHINE SYND., LTD. (B.P. 288,670, 14.12.26. Cf. B.P. 255,923; B., 1927, 49).—Oils, fats, and waxes are extracted by subjecting the material to repeated flushings with solvent while disposed in a basket rotating in a closed extraction vessel from which the solution is withdrawn and re-introduced into the centre during each flushing. This process provides a more expeditious method of manipulating the solvent and solutions from the various flushings. E. HOLMES.

Production of oils containing vitamins. BRIT. DRUG HOUSES, LTD., and F. H. CARR (B.P. 289,187, 7.2.27).—A solution of vitamins suitable for pharmaceutical purposes, or for use in food products, may be obtained by extracting mammalian livers with vegetable, animal, or mineral oils, with or without raising the temperature to 55–60°. E. HOLMES.

Soap powder and the like. LEVER BROS., LTD., and R. THOMAS (B.P. 288,654, 4.10.26).—A washing and bleaching agent capable of exerting its bleaching action in the presence of water at ordinary temperatures may be prepared by incorporating with soap flakes, powder, or bars, varying amounts of a double compound of trisodium phosphate and sodium hypochlorite, with or without sodium carbonate and other usual soap-powder ingredients. If kept substantially dry the double salt is stable at least for several months even in the presence of the organic matter of the soap. E. HOLMES.

Manufacture of rosin soap. C. C. KESLER, ASSR. to PINE INSTITUTE OF AMERICA, INC. (U.S.P. 1,663,764, 27.3.28. Appl., 30.7.25).—The resene component of the rosin is removed after the acid component has been converted into an alkali hydrogen salt, and the salt is used in the manufacture of soap. L. A. COLES.

Production of aqueous solutions of organic compounds insoluble or difficultly soluble in water. I. G. FARBENIND. A.-G. (B.P. 261,720, 7.10.26. Ger., 19.11.25).—Aqueous solutions of sulphonic acids having soap-like properties (especially aralkylated sulphonic acids, e.g., the isopropylated acids of G.P. 336,558; B., 1921, 630 A) or their salts have their property of dissolving insoluble organic compounds, e.g., oils, fats, hydrocarbons, etc., considerably increased by the addition of soluble salts, such as Glauber salt. [Stat. ref.] B. FULLMAN.

Production of sulphuric esters of poly[hydr]oxy-fatty acids. H. FLESCH (B.P. 287,076, 4.8.27. Ger., 14.3.27).—The laboratory preparation of dihydroxystearic acid (cf. Grün, B., 1907, 57) can be adapted to the production of sulphuric esters of dihydroxy-

trihydroxy-, and polyhydroxy-fatty acids on the technical scale by treating the cooled and actively agitated hydroxyfatty acid with sulphuric acid in the form of a very fine spray, and with sulphuric acid containing 5–10% of sulphuric anhydride. E. HOLMES.

Production of sulphonated oils and fats with a high content of organically combined sulphuric acid. H. FLESCH (B.P. 282,626, 4.8.27. Ger., 23.12.26).—The interaction of unsaturated oils, fats, and fatty esters, and glacial acetic acid with sulphur trioxide or chlorosulphonic acid gives high-grade sulphonates which have a higher content of organically combined sulphuric acid and a higher wetting power than Turkey-red oils. E. HOLMES.

Fluid composition [metallic soap] applicable as a covering, colouring, or dispersing agent, etc. C. J. J. FOX (B.P. 289,122, 15.1.27).—A dispersion medium consisting essentially of a strong ammonium base and the soap of a fatty acid or mixture with metals such as zinc, copper, or nickel, which give ammonia-soluble hydroxides, but which are insoluble in water alone, may form the basis of numerous technically useful products. Such a dispersion may, for example, be emulsified with pigments, insect poisons, or rubber latex to give sheep-marking fluids, insecticides, and water-proofing liquids, respectively. Such products are water-resistant after the base has evaporated, yet may be readily removed with dilute ammonia, ammonium chloride, or sodium carbonate. E. HOLMES.

Recovery of fatty material from emulsions. SOC. FRANÇ. DE CENTRIFUGATION, and J. A. L. ROZIERES (B.P. 289,350, 25.10.27).—In an apparatus for the extraction of fatty materials from such emulsions as wool scourings, by means of a counter-current of a volatile solvent which is subsequently removed by centrifuging, special provision is made to prevent clogging of the apparatus by solid matter, as pulverised wool. E. HOLMES.

Bleaching liquors (B.P. 289,156).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colloid chemistry of paints, varnishes, and their components. R. S. MORRELL and W. E. WORNUM (J. Oil and Colour Chem. Assoc., 1928, 11, 49–62, 93–112).—A digest of the literature on the colloid chemistry of paints, drying oils, resins, and varnishes, covering the period 1920–1927 (cf. "Third Report on Colloid Chemistry and its General and Industrial Applications," Dept. Sci. Ind. Research, 1920).

S. S. WOOLF.

The red lead question. [Action of red lead in rust-protecting paints.] H. WOLFF and R. SINGER (Farben-Ztg., 1928, 33, 1909–1913).—Rahder's statement (B., 1928, 99) that red lead accelerates the corrosion of iron is true only for the system red lead–iron–water, and does not hold for paints in which red lead forms the pigment. The oil adsorbed by highly dispersed red lead may vary very considerably, and is of more importance in determining the rust-protecting capacity of a paint than is the total oil content. The oil content, degree of dispersion of the pigment, and the

swelling capacity of a paint are not interdependent functions. The last-named depends on the free oil content, i.e., on the oil which is not adsorbed on the surfaces of the particles of pigment. The protective action of the paint is due to a thin, almost molecular film in which the oil is present as an extraordinarily strong adsorption complex with the pigment and the base. The swelling capacity of the upper layers cannot therefore be a measure of the protective action of the paint, which is much more likely to be due to the formation of a protective film on the surface of the iron.

A. R. POWELL.

Determination of iron in red lead. H. HEINRICH (Z. angew. Chem., 1928, 41, 450–453).—The usual method of determining iron in red lead by dissolving the sample in nitric acid, precipitating the lead as sulphate, and weighing the iron as oxide gives low results owing to the insolubility of part of the iron in nitric acid and to adsorption of ferric sulphate by the lead sulphate precipitate. Correct results may be obtained by the following colorimetric method: 10 g. of the sample are mixed with 25 c.c. of a cold saturated solution of hydroxylamine hydrochloride, and the mixture is boiled for 10 min. with 50 c.c. of hydrochloric acid (*d* 1.19). The lead chloride is dissolved completely in 400 c.c. of boiling water and the solution set aside overnight. The liquid is decanted, evaporated to 100 c.c., and again allowed to crystallise. The second filtrate is treated with ammonia, the ferric hydroxide precipitate collected and dissolved in hydrochloric acid, and the iron determined colorimetrically with thiocyanate. A blank test should be carried out using the same quantities of reagents as in the analysis. A small, grey, spongy residue after dissolving the sample in the acid mixture is metallic lead, the presence of which is objectionable in red lead used for glass frits.

A. R. POWELL.

Bleaching of lac. M. VENUGOPALAN (J. Indian Inst. Sci., 1928, 11A, 17–22).—Lac may be bleached by dissolving it in 2.5% sodium carbonate solution at 60–70° and, after filtration and cooling to air temperature, adding a solution prepared by passing chlorine into a solution containing 12.5% of caustic soda and 2.5% of sodium carbonate. The latter should contain 6–8% of available chlorine and, if of p_H 10–10.5, does not require storing in a cool place. The amount of such a solution necessary for bleaching indicates a chlorine requirement of 10–14% on the weight of lac, and a yield of 93–95% is obtained. The bleached lac may be recovered by the slow addition, with stirring, of 1:20 sulphuric acid, the precipitate being then collected, washed, and dried *in vacuo* over sulphuric acid. The product is freely soluble in cold 97% alcohol, and the solubility does not alter on prolonged storage in air. The bleached material contained 2.3–3.1% of moisture, 0.98–3.52% Cl, and had saponification value 236.0–256.7, acid value 70.68–83.52, and iodine value 3.9–5.0.

F. E. DAY.

PATENTS.

Manufacture of white oil paints from lithopone and manufacture of lithopone. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,153, 21.1.27).—Thickening of white oil paints manufactured from litho-

pone is hindered by the presence of at least sufficient free fatty acids to prevent or minimise the thickening action of the zinc oxide present. Linseed oil may be used containing a sufficiently high proportion of the fatty acids, or these may be added in colloidal suspension or dispersion to the lithopone. L. A. COLES.

Steam turpentine still. J. O. REED, Assr. to U.S.A. (U.S.P. 1,667,168, 24.4.28. Appl., 28.7.27).—Steam is introduced into the bottom of the still through a vertical supply pipe which communicates with radial jets, and between the supply pipe and the jets is a valve operated by the steam pressure. F. G. CLARKE.

Enamel for coating cans [to contain foodstuffs]. R. H. LUECK, Assr. to U.S.A. (U.S.P. 1,667,212, 24.4.28. Appl., 23.3.26).—A neutral oleoresin varnish is incorporated with compounds of metals forming sulphides which are not dark in colour. L. A. COLES.

Plastic material. Flexible moulded articles from condensation products. E. L. AIKEN, Assr. to CARBOLOID PRODUCTS CORP. (U.S.P. 1,668,444—5, 1.5.28. Appl., 23.9.21. Renewed [B] 25.4.24).—(A) Phenol and glycerol are condensed by a dehydrating agent. (B) The product is condensed with "methylene," and the final material shaped without pressure and heated. B. FULLMAN.

Synthetic resin. C. E. BURKE and H. H. HOPKINS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,667,189, 24.4.28. Appl., 28.4.27).—A mixture of pentaerythritol, a polybasic acid, and a drying oil acid is heated to a temperature at which reaction takes place. A. R. POWELL.

Titanium pigment. J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (Re-issue 16,956, 8.5.28, of U.S.P. 1,504,673, 12.8.24).—See B., 1925, 999.

Fluid composition (B.P. 289,122).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preparation of butadiene. S. F. BIRCH (Ind. Eng. Chem., 1928, 20, 474).—By bromination of the gases obtained on distillation of 8 gals. of "railway hydrocarbon" (the liquid obtained during the preparation of oil gas), 1250 g. of solid butadiene tetrabromide and 560 g. of the liquid tetrabromide can be obtained. Pure butadiene can be prepared from the solid derivative by Thiele's method (A., 1900, i, 2) with zinc dust and alcohol. Butadiene obtained similarly from the liquid tetrabromide is probably contaminated with traces of high-boiling olefines. E. H. SHARPLES.

Rubber fibre and cellulose fibre. Recognition of the elastic linking. H. FEUCHTER (Kautschuk, 1928, 4, 73—75; cf. B., 1928, 377).—The "shell-aggregation" theory of the structure of rubber is reviewed and its confirmation by the phenomena of "racking" is indicated. The analogy between the structure of rubber and cellulose extends to the persistence of the structural principle after vulcanisation in the case of racked rubber and after nitration or acetylation in the case of cellulose. D. F. TWISS.

Specifications as to composition and selling conditions for rubber goods. W. ESCH (Kautschuk,

1928, 4, 75—83).—A discussion of the value of specifications fixing the composition of rubber articles for various purposes. In addition to unavoidable variation arising from imperfections in the mixing operations and in subsequent chemical analyses, a disadvantage of such specifications is that they fail to ensure the qualities essential to good service, and, indeed, tend to check the development of newer methods of compounding capable of giving better results. Examples are cited.

D. F. TWISS.

XV.—LEATHER; GLUE.

Extractor for vegetable tanning materials. B. SCHWARZBERG (Collegium, 1928, 151—154).—A cylindrical glass vessel is connected from the bottom to another similar glass vessel containing the tanning material. Both stand in the same water-bath. A tube from the second vessel carries the tanning infusion so that it siphons into a litre flask. The first vessel contains 100—200 c.c. of distilled water, which is continually replenished from an inverted litre flask mounted in it and containing distilled water. A rubber connexion and clip in the delivery tube for the extract enables the rate of flow to be regulated. The vessel containing the tanning material can easily, if desired, be fitted up like a Procter extractor, with inverted thistle funnel. The whole apparatus stops automatically in case of choking. D. WOODROFFE.

Gelatins of different origin as emulsifying agents. J. C. KERNOT and J. KNAGGS (J.S.C.I., 1928, 47, 96—100 T).—The contradictory results obtained by different authors while studying the efficiency of gelatins as emulsifying agents are due to the presence of both organic and inorganic impurities, and to the fact that the p_H of the gelatin solutions has not been considered. By working with highly-purified gelatins, it was found that the previous treatment of the precursors has a marked effect on the emulsifying power of the gelatins obtained from them. The viscosity of the gelatin solutions also plays a great part in its emulsifying power. The viscosity also depends on the previous history of the precursor.

PATENT.

Manufacture of [vegetable] glue. P. KREISMANN (U.S.P. 1,667,073, 24.4.28. Appl., 28.3.21).—A mixture of potato starch, clay, water, and an alkali is heated until semi-fluid. H. ROYAL-DAWSON.

XVI.—AGRICULTURE.

Comparison between soil examinations by the seedling method and that of Mitscherlich. H. NEUBAUER, W. BONEWITZ, and A. SCHOTTMÜLLER (Landw. Versuchs-Stat., 1928, 107, 131—142).—Arrangement of a series of soils according to their content of available potash and phosphate as determined by the two methods showed a general parallelism. The average potash values by the seedling method were double, and those for phosphate one half those obtained by the Mitscherlich method. Differences may be partly explained by the difference in the growth period in the two processes. The important difference, however, is that in Mitscherlich's method crop increases result from

added fertilisers which differ in the chemical condition, capability of movement, and distribution in the soil mass from the nutrients present in relatively stable equilibrium in untreated soils as used by Neubauer. The seedling method is not advocated for recently fertilised soils.

A. G. POLLARD.

Effect of dehydration of soils upon their colloid constituents. I—III. J. L. STEENKAMP (Soil Sci., 1928, 25, 163—182, 239—251, 327—332).—I. Dehydration caused a definite decrease in the clay fraction owing to cementing together of small particles. Determinations of the sum of the absorbed bases actually present in the soil, and the degree of saturation in respect to bases at differing stages of dehydration, indicate that on drying an increase in the unsaturated clay acids and in the exchangeable bases present occurs.

II. An increase in acid salt-forming bases takes place on dehydration.

III. Only air-dried soil of high humus content showed reversibility of the colloidal condition after 21 days. The bearing of the above experiments on cultural conditions is discussed.

H. J. G. HINES.

Decomposition of hydrogen peroxide in soils.

K. SCHARRER (Landw. Versuchs-Stat., 1928, 107, 143—187).—No definite relationship exists between the catalase activity of soils and their reaction, although, in general, acid soils are less active than neutral or alkaline ones. The catalytic power of soils increases with the amount of iron and manganese compounds present. Chalky soils are very active, and a general relationship exists between catalase activity and the amount of calcium extracted with ammonium chloride solution. Sandy soils are usually more active than loams and clays, and humus soils than mineral soils of similar composition and reaction. Catalytic power in soils is more closely associated with dispersed clay than with the larger soil particles, although these have a definite influence. Mineral salts affect catalase activity according to their ability to flocculate or deflocculate the clay. Boiling or moderate heating of soil reduces its activity and further reduction occurs after ignition. Catalase activity is primarily the result of inorganic salt catalysis, and is not materially influenced by biological factors.

A. G. POLLARD.

Deflocculation and plasmolysis of soils. B. GANOSSIS (Compt. rend., 1928, 186, 1234—1236).—In addition to the alkali carbonates (cf. Dumont and Ganossis, B., 1928, 63), solutions of the hydroxide, chloride, or nitrate of sodium act as de-coagulants to soil and render it impermeable to water. Solutions of calcium sulphate (0.2%) or nitrate, potassium chloride or nitrate, and hydrochloric or nitric acid behave like calcium chloride (*loc. cit.*) and re-establish the permeability, the nitrate and sulphate of calcium being the most and least effective, respectively. Solutions of lime-water (saturated), potassium sulphate or hydroxide (0.1N) have no effect in the latter case, but potash applied to the soil may react as potassium carbonate. When potassium nitrate or chloride is used, there is evidence of exchange of the potassium ions with the calcium ions in the soil.

J. GRANT.

Buffering in soils. A. UHL (Forts. Landw., 1927,

2, 645; Bied. Zentr., 1928, 57, 104—106).—Jensen's method for measuring "buffer values," "buffer areas," etc. is discussed. The unsuitable development in this method lies in the determination of lime requirements, on the basis of the ratio lime added/change in p_H . Experimentally, changes in lime units are additive, whereas unit change in p_H values represents a multiplication, and the mathematical value of the fraction becomes distorted. For the purpose of determining lime requirements measurements of "hydrogen capacity," *i.e.*, the quantity of acid (or alkali) required to be added to a soil to produce a definite p_H value, are suggested. The hydrogen capacity corresponds to the maximum of the buffer formula, log acid added/acid remaining.

A. G. POLLARD.

Origin of red soils. E. BLANCK and F. GIESECKE [with A. RIESER and F. SCHEFFER] (Chem. Erde, 1927, 3, 44; Bied. Zentr., 1928, 57, 97—104).—Analyses of numerous red soils of varying types are recorded and discussed from the point of view of the correlation of chemical composition and mode of formation, age, and origin.

A. G. POLLARD.

Laboratory apparatus for the measurement of carbon dioxide evolved from soils. F. W. MARSH (Soil Sci., 1928, 25, 253—261).—In an apparatus consisting essentially of a filter flask surmounted by a cylindrical percolator and in direct communication therewith, purified air is drawn both over and through the soil, thus approximating more nearly to natural conditions.

H. J. G. HINES.

Calculation of soil respiration. H. LUNDEGÅRDH (Biochem. Z., 1928, 194, 453).—A misprint in the author's formula ("Kreislauf der Kohlensäure in der Natur," Jena, 1924, 146) for determining the production of carbon dioxide by soils is corrected, the formula becoming: $[(a - b) \times 1.858 \times V \times 60/t] / 10Y$ grams of carbon dioxide per hr. per m.² of surface, where a is the original and b the final percentage value of the carbon dioxide content of the bell-jar, t is the time in min., 1.858 the weight of 1 litre of carbon dioxide at 15° and 760 mm., V the volume of the bell-jar in c.c., and Y the surface in cm.² covered by the bell-jar.

P. W. CLUTTERBUCK.

Influence of heavy applications of dry organic matter on crop yields and on the nitrate content of the soil. A. W. BLAIR and A. L. PRINCE (Soil Sci., 1928, 25, 281—287).—Pot experiments extending over three years afford confirmatory evidence that the depressing effect of dry organic matter on crop yield is due to the locking up of available nitrogen by soil organisms.

H. J. G. HINES.

Effect of growing plants on solubility of soil nutrients. W. H. METZGER (Soil Sci., 1928, 25, 273—280).—Measurements of the bicarbonate concentration in soil in proximity to roots and in soil more remote gave higher values for the former, indicating that plants are able to exert a solvent action by excreting carbon dioxide.

H. J. G. HINES.

Manuring of the potato crop. T. J. SHAW (J. Min. Agric., 1928, 35, 36—38).—Experiments embodying the use of ammonium sulphate and potassium sulphate at four different rates, *viz.*, 0, 1, 2, and 3 cwt. per acre,

and in all possible combinations, were carried out in 1927. Each treatment was quadruplicated, and the arrangement was in four randomised blocks of sixteen plots. The experiments were set out on both dunged and undunged land. On the former, without fertilisers, the yield was 4 tons 17 cwt.; on the latter 3 tons 4 cwt. Ammonium sulphate alone, on the dunged area, gave increases of 27 cwt. and 33 cwt. for the first and second cwt., respectively, of fertiliser applied. The third cwt. of ammonium sulphate was only effective in the presence of 3 cwt. of potassium sulphate, when the mean yield reached 9 tons. On the undunged land the maximum yield was attained by the use of 2 cwt. of ammonium sulphate in conjunction with 1 cwt. of potassium sulphate, whereby a crop of 7 tons 10 cwt. was obtained.

H. J. G. HINES.

Calcium as a factor in soya bean inoculation.

R. W. SCANLAN (Soil Sci., 1928, 25, 313—325).—The general effect of calcium was to increase nodulation both in soil and in water cultures. H. J. G. HINES.

Determination of phosphoric acid. DODD.—See VII.

PATENTS.

Manufacture of fertilisers. R. C. FLUCK and A. THEIL (B.P. 289,284, 1.7.27).—Fertiliser material in faecal matter is removed by treatment with peat, lignite, etc. By plural filtration through a mass of peat, suspended matter is removed to form an organic fertiliser. Ammoniacal matter in solution may be obtained by boiling, either before or after filtration, and phosphates obtained from the liquor by precipitation. Alternatively the faecal matter may be boiled with 3—5% of peat, and stirred with an agitator. The mud after settlement may be centrifuged and prepared as a fertiliser. The clear liquor may be treated as above for preparing phosphate and ammoniacal fertilisers.

A. G. POLLARD.

Manufacture of fertilisers. E. L. PEASE (U.S.P. 1,668,464, 1.5.28. Appl., 11.3.26. U.K., 16.4.25).—See B.P. 253,291; B., 1925, 685.

XVII.—SUGARS; STARCHES; GUMS.

Desiccation (De Vecchis) process of beet sugar manufacture. C. S. GARRETT (Chem. and Ind., 1927, 46, 1014—1020).—The working of the De Vecchis process (B., 1925, 256; 1924, 991), which has been in operation in one or two Italian factories for several campaigns, is described. The beet cossettes are dried to a moisture content of about 3% at 90—100° and the heating is prolonged for 1—2 hrs. after the removal of moisture to ensure coagulation of proteins and other constituents. The dried cossettes occupy about 120—140 cub. ft. per ton, and can be stored for long periods without deterioration. The extraction of the sugar is carried out at 60—70° with the minimum amount of water in a battery of 10—12 lixiviators, and yields juices of 40—50° Brix. In open vessels, operating under gravity, extraction may occupy 4 hrs., but with closed vessels worked under pressure the period can be reduced to 2—2½ hrs., and juices of 60° Brix have been obtained. The purity of the juice is about 2.5—3.5% higher than that of the pressed juice of the original beets, which,

however, are of low quality. It is defecated at 80° with 0.25—0.3 pt. of lime per 100 pts. of fresh roots, followed by about twice as much superphosphate. Filtration yields a hard filter-press cake much smaller in amount than that from diffusion factories, and of high fertilising value. The defecation effects an increase of 2—3.2% in the purity of the juice from low-grade Italian beets. The purified juice, of 40—50° Brix, requires no multiple-effect evaporation, but is brought to 60° Brix by the re-melting of second-product sugars, and boiled to grain in the vacuum pan. The amount of molasses produced is not greater than in the diffusion process, nor are the sugar losses in exhausted cossettes and filter-press cake; also the waste waters from the exhausted cossettes are less liable to putrefaction than those from the diffusion process. It is estimated that in England the relative capital costs of plant and erection for diffusion and desiccation factories for the same annual output are about 40:26.5, and the working costs for the De Vecchis process are lower in spite of the extra fuel required for the drying process. It is suggested that the output of existing diffusion factories could be economically increased by the installation of drying plant, the present extraction plant serving, with slight modifications, for the recovery of the sugar during the period when the factories are now idle.

J. H. LANE.

Desiccation of sugar beet and the extraction of sugar. B. J. OWEN (Rep. Min. Agric., 1927, 84 pp.).—A full report is given of an investigation into the De Vecchis process (B.P. 209,738 and 211,113; B., 1925, 256; 1924, 991) carried out at Eynsham. The conditions necessary for drying beet cossettes were examined, first in laboratory experiments and then in three types of dryers, viz., (a) a cylindrical mass dryer similar in principle to that of B.P. 235,273 (B., 1925, 648); (b) a moving-belt dryer of the type used by De Vecchis, and (c) a tray dryer with three hot-air compartments, one perforated tray with cossettes being placed over the first, another over the second, and two over the third, these positions representing successive stages of drying. Among the factors studied were the practicable thickness of the cossette layer (8—12 in.), the reduction in bulk (about 50%) and in resistance to air during drying, and the varying sensitiveness of the sugar to high temperatures at different stages of drying. It was completely established that the drying can be carried out on a large scale without inversion of sugar or caramelisation. So long as the cossettes were moist the temperature of the applied air (38—127°) had no effect on the sugar, but loss of sugar occurred from the use of high temperatures (above 113°) on cossettes having a moisture content below 20%. It was accordingly considered inadvisable to continue heating after the moisture was expelled, no benefit being found to result from the prolonged heating advocated by De Vecchis for the coagulation of proteins. The drying process could thus be completed in less than 1 hr. The tray dryer was adopted for most of the work as being easy to manipulate and control. Cossettes containing 3—5% of moisture suffered no deterioration on storage; after long exposure to the air the moisture content rose to 11—13%, but not beyond, and in large piles only the outermost layers of cossettes (to a depth

of about 6 in.) lost their original crispness. Extraction of the dried cossettes in a battery of small diffusion vessels, each provided with a calorisor for heating the juice passing from one vessel to the next, yielded juices of 45–50° Brix with purities of about 90%. These thick juices had excellent physical characteristics, being light in colour and clear, and not darkening perceptibly on exposure to air. The difficulty experienced in obtaining gravities above 50° Brix, together with microscopical evidence and osmotic experiments, led to the conclusion that the cells of the beets are not ruptured by the drying process, and that the subsequent extraction of the sugar is an osmotic phenomenon as in the case of fresh beets. A continuous diffuser in the form of a vertical cylinder with a helical conveyor to raise the cossettes against the descending juice (cf. B., 1927, 537) was also used successfully for the extraction of the sugar. Serious filtration difficulties were experienced with De Vecchis' method of purifying the juice with lime and superphosphate. Satisfactory working was attained, however, by a mechanical removal of suspended impurities in a centrifugal clarifier, either before or after liming, the precipitate produced by superphosphate being then easily filterable. The colour of the purified juice can, if necessary, be further improved by treatment with active carbons. Without the latter treatment, however, a strike of white sugar could be obtained from the juice reinforced by a proportion of second-product sugar. The amount of effluent from flumes and beet washing is, of course, the same for the desiccation process as for the diffusion process, but that from the working of the cossettes is less for the former than the latter, and would in a normal desiccation factory be distributed over a long working period. An appendix dealing with beet factory effluents describes a revolving double screen by which the coarse and finer suspended matters can be separately removed from the waste waters from flumes and washers, and some suggestions are made for the treatment of process effluent. J. H. LANE.

Plant for the drying of cossettes in the De Vecchis process of beet sugar manufacture. G. W. RILEY (Chem. and Ind., 1927, 46, 1036–1042, 1080–1084).—The author describes the design and construction of a form of dryer intended primarily for beet cossettes, in which the material is exposed to air at a given temperature, e.g., 100–105°, until most of the moisture is removed, and then to dry air at a somewhat lower temperature for a further period, e.g., 1–2 hrs., to effect coagulative changes. The first stage is carried out in an upper compartment with steel wire moving belt supports for the material, whilst a lower compartment with similar belts travelling more slowly serves for the coagulation stage. The air entering is heated by direct admixture with adjustable proportions of furnace gases, and high thermal efficiency is attained by a system of recirculation in which a portion of the air most heavily moisture-laden is exhausted from the upper chamber at each circuit. The relatively dry air from the lower chamber is exhausted by fans into the furnace gases, and other fans control the admixture of furnace gases with the drying air, and the exhaust from the upper compartment. A plant drying 70 tons of beets

per day consumed in all-day runs oil fuel equivalent to 6.86–8.4 tons of coal of 13,500 B.Th.U. per 100 tons of washed roots. J. H. LANE.

Factors influencing char filtration [of sugar solutions]. II. E. W. RICE and G. W. MURRAY, JUN. (Ind. Eng. Chem., 1928, 20, 276–277).—A portion of bone-char was washed for 3 days with distilled water, and, after drying, a solution of invert sugar was percolated upward through it, and simultaneously through a similar portion of unwashed char. On examining the filtered material, the results showed no appreciable differences for the two chars, the conclusion drawn being that the ammonia known to be present in re-activated char does not affect the selective adsorption of levulose previously observed (cf. B., 1927, 263). Adsorption tests with monocalcium phosphate were found to be vitiated owing to the presence of calcium carbonate in the char. Mixtures of sodium sulphate and calcium acetate with sugar were filtered through char, the values obtained showing an increase in the adsorption of the salt as the percentage of sugar was increased. When, therefore, the maximum of ash is to be removed by char in refining raw sugars, the liquors should be kept as dense as possible. J. P. OGILVIE.

Influence of p_H and of lead salts on invertases, and preservation of beet pulp for analysis. E. PARISI (Annali Chim. Appl., 1928, 18, 99–103).—Vondrák's method (B., 1925, 254) of preserving beet juice and pulp by either heating them to 82° or by rendering the samples faintly alkaline by addition of lime and transporting them to the analysts at –5° to –10° is impracticable and not always effective; Change of polarisation may, however, be prevented by mixing 26 g. of the pulp or juice with 177 c.c. of 4% basic lead acetate solution. The loss in polarisation of the untreated material is due to the action of invertase, which is usually regarded as of bacterial origin, but the author finds that beet pulp from the freshly sliced roots, if placed at once in a vessel with toluene and kept tightly closed, soon undergoes a fall in polarisation. The presence of invertase in the beet, thus indicated, may play a part in the destruction of sucrose in the sliced roots. T. H. POPE.

Influence of phosphate and colloid contents of cane juice on defecation. H. S. PAINE, J. C. KEANE, and M. A. MCCALIP (Ind. Eng. Chem., 1928, 20, 262–267).—The phosphate content of the raw juice showed an approximately linear relation to the colloid elimination. Increasing the p_H at which the juice was limed increased the colloid elimination, but also the calcium content of the defecated juice, which latter (per 100° Brix) showed a definite relation to the content of reversible colloids in the defecated juices. Excess of lime in defecation apparently has a peptising effect on the gummy substances in the juice, as there is an increase of reversible colloids proportional to the increase in calcium salts, a portion of which is of a colloidal character. By adding phosphoric acid to raw juices deficient in that constituent a decided increase in colloid elimination was exhibited, together with a distinct reduction in the calcium salts. J. P. OGILVIE.

Factory operation of automatic electrometric p_H control of cane juice defecation. R. T. BALCH and H. S. PAINE (Ind. Eng. Chem., 1928, 20, 348—353).—Using a recording potentiometer, tungsten and calomel electrodes, continuous flow chamber, and temperature compensator for the automatic measurement and recording of p_H values, the control of the addition of the lime used for defecation has been successfully applied in a Porto Rican factory over a period of four months. If full automatic control of the liming process is not required, it is still very advantageous to adopt means of automatically recording the p_H value of the juice, using similar equipment. This could be applied to batch as well as to continuous methods of liming.

J. P. OGILVIE.

[Sugar-works' deposits.] E. O. VON LIPPMANN (Ber., 1928, 61, [B], 222—223).—Calcium citrate and calcium α -gluconate were obtained from a deposit on the walls of a sugar magazine which had been coated with lime and subsequently used for storage of inferior sucrose for a long time and at a rather high temperature. Calcium tricarallylate was deposited in the juice preheaters during the working-up of some very unripe beet; the unusually pronounced odour of ammonia suggests that the acid was present originally as the amide or imide.

H. WREN.

Use of the Einhorn fermentation saccharimeter for the determination of sugar. T. UMBACH (Chem.-Ztg., 1928, 52, 273).—It is pointed out that as the capacity of the Einhorn saccharimeter varies from 6 to 10 c.c., whereas the graduations on the closed limb in c.c. and percentage of sugar are constant, the instrument is unsuitable for the determination of sugar in a liquid, e.g., urine.

A. R. POWELL.

Influence of the determination of the sugar content on fresh slices [of beetroot] by different methods of the total loss. A. DOLINEK (Z. Zuckerind. Czechoslov., 1928, 52, 329—333).—It is recommended that the official pipette method, employing the single normal weight of pulp and 308 c.c. of dilute basic lead acetate for the determination of sucrose in fresh slices, should be abandoned in favour of either of the following two: (1) that in which double the normal weight of pulp is treated with 354 c.c. of dilute basic lead acetate; or (2) that in which a proportionate amount of pulp is mixed with 308 c.c. of dilute basic lead acetate from an automatic pipette. Both give satisfactory results, compared with the Herles hot water digestion process, whereas if the above official method be employed in control the losses indicated may be too high by 0.13% of the sugar in the beet.

J. P. OGILVIE.

Fermentation of molasses. BIRCKNER and PAINE.—See XVIII.

PATENTS.

Manufacture of sugar. C. G. PETREE (B.P. 287,590, 18.10.26).—To reduce the viscosity of massecuite in the centrifuge and facilitate the purging of the crystals, the massecuite is heated, preferably by admixture with hot molasses, immediately before or during its introduction into the centrifuge. Apparatus for carrying out the process is also claimed.

J. H. LANE.

Oxidation of aldoses. G. B. ELLIS. From CHEM.

WORKS, FORMERLY SANDOZ (B.P. 289,280, 17.6.27).—See U.S.P. 1,648,368; B., 1928, 137.

XVIII.—FERMENTATION INDUSTRIES.

Surface of yeast as a factor in fermentation. C. RANKEN and J. R. BELL (J. Inst. Brew., 1928, 34, 265—274; cf. Ranken, B., 1927, 200).—The coating of yeast with peptone-tannin favours the production of high and coherent heads of yeast and generally retards the rate of fermentation. The amount of reproduction except in the very latest stages is diminished. The retardation is greatest during the earlier stages of fermentation, but, owing to the progressive solubility of the coating during fermentation, its effect on the time required to reach the attenuation usual in brewery practice is of small magnitude. The physical condition of the coating is influenced by traces of iron in solution which cause the yeast heads to become less coherent, with an accompanying acceleration of the rate of fermentation. With a low seeding rate, the rate of fermentation of yeast coated with calcium oxalate is retarded and the reproduction is smaller. If the amount of calcium oxalate deposit is increased, or if the rate of seeding is high, the rate of fermentation is accelerated.

C. RANKEN.

Invertase-free yeasts and their application in the selective fermentation of final cane molasses. V. BIRCKNER and H. S. PAINE (Ind. Eng. Chem., 1928, 20, 267—275).—A process in which the reducing sugars present in molasses are fermented with the production of alcohol (cf. U.S.P. 1,572,359; B., 1926, 337), thus obtaining a liquor which may be submitted to a suitable desaccharification operation, is stated to have given results that have been "fairly successful."

J. P. OGILVIE.

Drying of hops. VI. Institute of Brewing Research Scheme. Report of the sixth season's work at the experimental oast, 1926. A. H. BURGESS (J. Inst. Brew., 1928, 34, 248—264).—The time of drying is related to the air speed and depth of loading according to the expression $T = kD/a^x + M$, where T is the time of drying in min., D the depth in inches, a the air speed in ft. per min., and M is the minimum time in min. D may be substituted by the loss of water in oz. per sq. ft. of kiln floor. The colour value and aroma of the hops decrease as the temperature of drying is raised from 15° to 100°. There is a decrease in the content of α -acid when the hops are dried at 80° and above, whereas the β -fraction is unaffected by the temperature. Up to 90° the antiseptic value is unaffected, and is very slightly reduced by drying at 100°. Burning sulphur under the hops gives an inferior colour to that obtained by the use of sulphur dioxide, but the antiseptic value is higher. Sterilisation of the hops by heat and subsequent storage in the absence of air preserve and retain the antiseptic power to a greater extent than does drying.

C. RANKEN.

Conservation of the food value of barley during the process of brewing. A. A. D. COMRIE (J. Inst. Brew., 1928, 34, 284—286).—It is calculated that 100 g. of barley of calorific value 272.1 kg.-cal. will produce, after conversion into malt, 419 c.c. of beer of calorific

value 43.7 kg.-cal. per 100 c.c., *i.e.*, a calorific value, as beer, of 183.1 kg.-cal. Accordingly, 67.3% of the human-food value of the barley is conserved in the finished beer. The sources of loss are the respiration and rootlet growth on the germinating floor, the flocculation of protein matter in the copper, and the conversion of sugar into alcohol and carbon dioxide during fermentation.

C. RANKEN.

Sulphurous acid in wine manufacture. L. MOREAU and E. VINET (Ann. Falsif., 1928, 21, 130—136; cf. B., 1927, 665).—The application of sulphurous acid in the preservation of grape juice and white wines is discussed. The minimum quantity of sulphur dioxide necessary for the effective preservation of grape must is stated to be 150 mmg. of free SO₂ per litre, and of sweet white wines 40—70 mmg. according to strength.

T. M. A. TUDHOPE.

Micro-determination of phosphate in wine and other fermented liquids. G. DENIGÈS (Ann. Falsif., 1928, 21, 136—142).—A rapid colorimetric method is described. The reagent employed is a sulphuric acid solution of ammonium molybdate which has been partially reduced by the action of metallic copper. A blue colour develops on heating a solution containing phosphate ions with the reagent. The intensity of the colour is proportional to the amount of ionised phosphate present. Standards are prepared containing known amounts of phosphate. The total phosphate, including that organically combined, can be determined by first evaporating to dryness with nitric acid, and then boiling with dilute sulphuric acid. 5 c.c. of a 2% solution of the wine is sufficient for the analysis. T. M. A. TUDHOPE.

Examination of brandies. R. COHN (Pharm. Zentr., 1928, 69, 241—246, 261—265).—Methods for the determination of the alcoholic strength and ester content of spirits are described. The influence of a small amount of dissolved extract on the sp. gr. of spirits is also discussed.

T. M. A. TUDHOPE.

Adulterated spirits and the Micko distillation. C. BREBECK (Chem.-Ztg., 1928, 52, 377—379).—The Micko distillation test for detecting the origin and adulteration of spirits is discussed. The test is based on the odour and taste of the distillate prepared under definite conditions, and is the only method by which the origin of spirits can be determined. E. H. SHARPLES.

Methyl alcohol in various alcoholic drinks. W. SEIFERT (Oesterr. Chem.-Ztg., 1928, 31, 65—68, 74—77).—By the use of Denigès' method, the methyl alcohol content of a number of wines and brandies was found to be: commercial grape wines less than 0.11 to 0.25, fruit wines 0.08—0.2, wine brandies less than 0.4, fruit brandies 2.3 and 4.1, grape and fruit brandies from whole mash or residues 3.2—5.8 c.c./litre. The differences between the various classes of material examined were more marked when the proportion of methyl alcohol was calculated on the total alcohol, and the author concludes that a greater amount of methyl alcohol is present in wines from American varieties of grape and their hybrids. Experimental mashes of such grapes gave wines containing less than 0.1 to 0.58 c.c. of methyl alcohol per litre, reduced to below 0.3 by heating the mash to 70—75° before fermenting. The methyl

alcohol thus appears to be derived from the pectin, and is no longer found after the destruction of pectase by heat. In expressed must, increase of pressure slightly increases the methyl alcohol content of the wine. Some methyl alcohol may be present in the wine as esters. Literature relating to pectin and to the physiological action of methyl alcohol is reviewed, and it is suggested that brandies from American varieties of grape and from residues may contain toxic amounts. F. E. DAY.

Presence of glycuronic acid in wines from diseased or rot-infested grapes. D. CHOCHAK (Ann. Chim. anal., 1928, [ii], 10, 97—98).—See B., 1928, 281.

PATENTS.

Purification of liquids by distillation. E. C. R. MARKS. From U.S. INDUSTRIAL ALCOHOL CO. (B.P. 287,607, 29.11.26).—High-strength alcohol is produced by rectification in the presence of benzol, the constant-boiling mixed vapour from the rectifier being separated into its constituents (which are re-used) by several stages of settling into layers and rectifications. The process is applicable to other liquids besides those mentioned. B. M. VENABLES.

Butyl [alcohol]-acetone fermentation. D. A. LEGG, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,814, 8.5.28. Appl., 1.10.26).—See B.P. 278,307; B., 1928, 32.

XIX.—FOODS.

Effect of severe weathering on certain properties of wheat. W. O. WHITCOMB and A. H. JOHNSON (Cereal Chem., 1928, 5, 117—128).—Progressive weathering, as observed over a period of two years (1925—6), lowered the grade of the wheat, bleached it, and increased the proportion of damaged kernels. The test weight per bushel and the weight per kernel were decreased. The most noticeable effect was the lowering of the germination. After an initial reduction during the first two months the protein content remained practically constant. The ash of the wheats decreased during weathering, whilst that of the flours milled from them increased. The yield, *p_H* value, and viscosity of the flour, and the quality of the gluten were unaffected. The flours from weathered wheats decreased more rapidly in viscosity during auto-digestion, but pteoclastic activity as measured by the Sørensen titration or Van Slyke amino-nitrogen method was the same in flours from weathered wheat as in those from unweathered wheat. The colour and texture of the bread produced from weathered wheat was normal in 1925 and slightly inferior to that produced from unweathered wheat in 1926. The loaf volume showed no significant variation. Weathering reduced the fermentation time required in baking tests, and this held not only for wheat harvested at different times, but for wheat from the inside and outside of the shock. W. J. BOYD.

Effect of delayed harvesting on quality of wheat. A. F. BRACKEN and C. H. BAILEY (Cereal Chem., 1928, 5, 128—145).—It is shown that a kernel does not change in weight when dried to a uniform moisture content after wetting, but that the decreased weight to a measured volume is due to increased volume. Microscopical examination showed that this is due to cracking of the kernel

and development of air spaces. The opacity of wheat which has been wet and the occurrence of a high percentage of broken kernels in threshing such grain are to be attributed to the presence of these air spaces and cracks. High density is shown to be associated with high protein content. Wetting produces bleaching of the grain. Titratable acidity and pH showed no change as a result of delayed harvesting. Chemical and microscopical tests gave no indication of increased diastatic or proteoclastic activity. Baking tests showed no indication of deterioration as determined by loaf volume and loaf texture. It is concluded that dark hard wheat of the Turkey-red type does not deteriorate in quality through delayed harvesting apart from bleaching and increase in volume of the grain. W. J. BOYD.

Wheat and flour studies. XIV. Factors influencing the viscosity of flour-water suspensions. III. Effect of small quantities of carbon dioxide in water used for the extraction of electrolytes. A. H. JOHNSON and B. L. HERRINGTON (Cereal Chem., 1928, 5, 105—116).—The viscosities of acidulated flour-water suspensions previously extracted with water containing carbon dioxide were higher than those of similar suspensions extracted with neutral distilled water. These differences increased as the quantities of the respective waters used for extraction were increased. No greater quantities of electrolytes were extracted with water containing carbon dioxide than with neutral water. Although differences in the pH value of the extracts were found, these do not explain satisfactorily the variations in viscosity. When carbon dioxide was added to the suspensions after extraction no increase in viscosity resulted. Greater quantities of protein were extracted when water containing carbon dioxide was used. It is concluded that certain proteins of wheat flour which operate to depress the viscosity of acidulated suspensions are removed more readily by water containing carbon dioxide than by neutral distilled water, and that their removal raises the viscosity of the suspension. W. J. BOYD.

Proteoclastic activity of flour. A. CAIRNS and C. H. BAILEY (Cereal Chem., 1928, 5, 79—104).—Eight chemical methods for measuring progressive proteolysis in flour suspensions were studied. Of these, Sørensen's formol titration method was found to be the most suitable. The amount of proteolysis which occurs, as measured by the amino-nitrogen formed when suspensions of high-grade flour milled from sound wheat are digested for 48 hrs. at 37° , is of the order of a few mg. per 10 g. of flour. When yeast is added the amount of proteolysis is still small, but a multiple of that which occurs without yeast. From a comparison of different flour streams from the same hard spring wheat it is deduced that the more highly refined flours milled from purified middlings will undergo less proteolysis when mixed into a dough and fermented than will the less highly refined break flours and tailings flours. Whilst flour from sprouted wheat had a much higher rate of proteolysis than flour from ungerminated wheat, the addition of 2—3% of sprouted wheat to the wheat mixture modified the proteolytic activity of the flour very slightly. Ash content and proteolytic activity tend to increase at about the same rate. No relation

between proteolytic activity and wheat variety or region of origin could be discerned. Decreased viscosity of incubated flour suspensions is associated with increased proteoclastic activity. The presence of protease in flour was demonstrated by adding safranin solution to a flour extract, collecting the precipitate, and testing its proteolytic action on casein. W. J. BOYD.

Chemistry of bread. D. W. KENT-JONES (J.S.C.I., 1928, 47, 143—149 T).—An account is given of the manufacture of bread through all its stages from the wheat to the finished product, particular attention being given to the application of chemical control. The chemistry of bread making is discussed, and the essential features of a satisfactory baking flour are stated to be sufficient diastatic activity to produce sugar for the yeast, and sufficient protein of good quality.

Standard experimental baking test [for bread]. Report of Committee of the American Association of Cereal Chemists. M. J. BLISH (Cereal Chem., 1928, 5, 158—161).—A statement of the formula, method, and equipment to be employed in carrying out this test.

W. J. BOYD.

Determination of starch in bread. P. FLEURY and G. BOYELDIEU (Ann. Falsif., 1928, 21, 124—130).—A method for the determination of starch in breads, especially those prepared for diabetic patients, is described. After hydrolysis with dilute sulphuric acid, the proteins are precipitated by means of an acid solution of mercuric sulphate, which is shown to be more effective for this purpose than lead acetate. The dextrose remaining in the solution is then determined by polarimetric or reduction methods. T. M. A. TUDHOPE.

Ripening of cheese of "Sbrinz" type. M. SOLARI (Anal. Ofic. Quim. Prov. Buenos Aires, 1927, 1, 107—140).—Analyses of "Sbrinz" cheese were made at intervals during ripening over a period of 11 months. Figures are given of the variation in content of moisture, fat, nitrogenous material (ammonia, amines, soluble and insoluble albumins), ash, sodium chloride, and acidity. A notable feature was the fall of acidity to zero after four months, followed by a rapid rise apparently connected with degradation of the fat. Ammonia, soluble albumin, and amino-compounds showed an increase parallel with the degree of ripeness. Lactose had already disappeared when the observations were started. The results are discussed. R. K. CALLOW.

Acidity in fruit juices and preserves. C. F. MUTTELET (Compt. rend., 1928, 186, 1299—1301).—The removal of pectins in fruit juices etc. before determining their organic acid content may be satisfactorily effected by first hydrolysing the pectins to pectic acid and then precipitating either the free pectic acid or its barium salt. B. W. ANDERSON.

Commercial tomato preserves. C. FERRI (Anal. Ofic. Quim. Prov. Buenos Aires, 1927, 1, 141—191).—The chemical and microscopical examination of tomatoes preserved in various forms is outlined, and a series of representative analyses is given. The essential determinations are of poisonous metals, preservatives, colouring matter, and starch. Over 6% of starchy material indicates adulteration, and sodium chloride over 1% an addition of salt. Microscopical examination

yields evidence of the efficiency of sterilisation and of the presence of other pulps, such as papaw or carrot.

R. K. CALLOW.

Changes produced in meat extracts by the bacterium *Staphylococcus aureus*. Application of the alcohol titration methods. F. W. FOREMAN and G. S. G. SMITH (Dept. Sci. Ind. Res., Food Invest., 1928. Spec. Rept. No. 31. 97 pp.).—The extract used was that of ox heart made with plain tap water and concentrated to one fourth or one tenth of its original volume. The growth of the bacteria in this medium at 37° was compared over extended periods with the variation in the proportion of amino-acids, volatile bases, volatile and non-volatile acids, etc., as determined by the alcohol titration method (cf. Foreman, A., 1928, 448). The organisms exhibited various periods of growth, each of which was characterised by a particular chemical change. In the first period the rate of growth reached a maximum on the fourth day and resulted in the production first of volatile bases, then of volatile and non-volatile acid radicals, and finally of non-volatile acid radicals alone. A second maximum occurred on the twenty-eighth day, when non-volatile acid radicals were converted into the equivalent amount of volatile acid radicals. During a third period of growth the number of organisms gradually diminished, whilst the decline in the non-volatile acid radicals continued practically to zero with production of carbon dioxide, the volatile acid radicals remaining approximately constant. During the later stages of prolonged incubation the organisms were found to destroy the volatile acid radicals at room temperature, but at 37° they attacked the amino-acids and non-volatile amines in preference.

F. R. ENNOS.

Control of reaction in cultures and enzymic digests. Comparison of the effects of certain salts on changes in p_H and changes in absolute $[H^+]$ with reference to enzyme action. Rôle of creatinine in the control of reaction in cultures. F. W. FOREMAN and G. S. G. SMITH (Dept. Sci. Ind. Res., Food Invest., 1928. Spec. Rept. No. 32, 27 pp.).—Examination of the p_H value and titratable acidity of ox-heart extracts in which *Staphylococcus aureus* has been growing for varying periods indicates that phosphates and creatinine, which are present in the ratio of 1.7:3.0, are largely responsible for the resistance to changes of p_H on addition of acid. The influence of salts on the activities of enzymes and bacteria in media should be considered from the point of view of their effect not only on the p_H values (buffer action), but also on the absolute $[H^+]$ and $[OH^-]$ (depressor effect). In the region of $[H^+]$ where phosphates have little depressor effect, the action of creatinine in facilitating the continued growth of acid-producing organisms is demonstrated.

F. R. ENNOS.

Determination of dextrose in presence of proteins [in gluten bread]. P. FLEURY and G. BOYELDIEU (Bull. Soc. Chim. biol., 1928, 10, 568—575).—See B., 1928, 383.

PATENTS.

Preparation of a pectin product. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 259,948, 5.10.26.

U.S., 17.10.25).—The crude pectin is rendered suitable for jelling of fruit juices either by washing with an alcoholic solution of a salt of a strong base and a weak acid, e.g., sodium bicarbonate, or by mixing with sufficient sodium bicarbonate with or without the addition of an organic acid, e.g., citric or tartaric acid, so that the finished product has a p_H value of from 3.4 up to but not including 7.0.

F. R. ENNOS.

Soluble food product having fresh kola as a base. A. and E. CHALAS (U.S.P. 1,669,326, 8.5.28. Appl., 24.4.25. Fr., 7.5.24).—See B.P. 233,695; B., 1926, 213.

[Machines for] the manufacture of biscuits etc. T. & T. VICARS, LTD., and E. W. CROSLAND (B.P. 289,240, 29.3.27).

Preservation of [carcases for] food. P. C. LAVENDER and A. E. SHERMAN (B.P. 289,653, 24.5.27).

Oils containing vitamins (B.P. 289,187).—See XII. **Cans for foodstuffs** (U.S.P. 1,667,212).—See XIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of potassium iodide in tincture of iodine. P. MANICKE and W. POETHEKE (Pharm. Zentr., 1928, 69, 257—261).—The tincture is evaporated to dryness on the water-bath. Water is then added, and the solution is again evaporated to dryness in order to remove any hydrogen iodide which has been formed. The iodide in the residue is determined iodometrically or by Volhard's method.

T. M. A. TUDHOPE.

Esterification of ethyl alcohol in citric acid solution. C. W. CORNWELL (Pharm. J., 1928, 120, 391).—A flavouring mixture, originally containing about 25.5% of citric acid and 49.8% by vol. of alcohol, on examination after a period of 5 years, contained 38.9% of alcohol and 17.16% of ethyl citrate. In the removal of alcohol and water from a mixture containing ethyl citrate the ester is not appreciably hydrolysed, and the alcohol may be determined by direct distillation if alkali and volatile oils are absent or by direct separation with brine and distillation from a neutral solution if essential oil is present. Ethyl citrate cannot be separated from alcohol by light petroleum.

E. H. SHARPLES.

"Calcium glycerinophosphoricum solubile" and some glycerophosphates. G. KOGAN (Pharm. Zentr., 1928, 69, 49—53).—The pharmaceutical preparation "calcium glycerinophosphoricum solubile" contains in addition to calcium glycerophosphate the less soluble diglycerophosphate; the product is rendered soluble by addition of citric acid. It therefore cannot be analysed by the method of titration by acid to the acid salt, followed by back titration to the neutral salt. A preliminary titration of free citric acid (which may be detected by extraction with ether) is required; the content of diglycerophosphate is found by determination of ash (calcium meta- and pyro-phosphates). Magnesium glycerophosphate is similarly analysed; for the iron salt the iodometric method is used.

E. W. WIGNALL.

Glycerophosphates and inositolphosphates. G. LOCATELLI (Boll. Chim. Farm., 1927, 66, 737—738).—*Calcium magnesium inositolphosphate*, $C_6H_6O_{24}P_6Ca_3Mg_3$, is contained in the husk of cereals to the extent of about

8%, and is easily extracted by dilute acid; for pharmaceutical purposes it can advantageously replace calcium glycerophosphate. E. W. WIGNALL.

Caucasian thuja oils. B. RUTOVSKI and K. GUSSEWA (Riechstoffind., 1927, 185; Chem. Zentr., 1927, ii, 2723).—Fractionation of the oil of *Thuja occidentalis* yielded α -pinene, α -thujone, fenchone, and esters. That from the leaves of *T. Varreana* (d^{20} 0.9078, α_D -1.23° , n_D^{20} 1.4550, acid value 1.5, ester value 16.36, ester value after acetylation 30.36) yielded sabinene, α -thujone, and thujyl alcohol. That from the shoots of *T. gigantea* var. *semperaurea* (d^{20} 0.9145, α_D -1.21° , n_D^{20} 1.4552, acid value 2.34, ester value 26, ester value after acetylation 47.15) contained α -thujone, α -pinene, and thujyl alcohol. A. A. ELDRIDGE.

Russian mint oil. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst., 1923, No. 4, 10—13).—The oil examined had $[\alpha]_D$ -20° to -27° , d_4^{18} 0.914—0.915; menthol (esters) 6—15%, menthol (free) 41.51%, menthone 16—18%, acid value 0.7—1.0. CHEMICAL ABSTRACTS.

Russian fennel oil. B. N. RUTOVSKI and P. P. LEONOV (Trans. Sci. Chem.-Pharm. Inst., 1923, No. 4, 16—24).—The oil contained anethole, *d*-fenchone, *d*- α -pinene, dipentene, α -phellandrene, camphene, and anisaldehyde. CHEMICAL ABSTRACTS.

Essential oil from *Gastrochilus panduratum*, Ridl. A. J. ULTÉE (Proc. K. Akad. Wetensch. Amsterdam, 1928, 31, 62—64).—On steam-distilling the oil from the rhizomes of the above plant and fractionating the distillate, methyl cinnamate (b.p. 255—260°) was obtained, probably to the extent of 4.32—4.46% of the oil. A fraction (b.p. 180—200°) forming about 32% of the oil gave the tests for cineole. A small fraction distilled under a reduced pressure of 35 mm. at 120—140° consisted mainly of camphor. The lower-boiling fraction from the vacuum distillation, which had been freed from cineole by shaking out with resorcinol, was repeatedly distilled from sodium in an atmosphere of carbon dioxide. An olefinic terpene was obtained as a mobile oil which had b.p. 62—64°/13 mm., d^{17} 0.8253, n_D^{15} 1.4843, α_D^{15} $+11^\circ 42'$. The b.p. at ordinary pressures, in an atmosphere of carbon dioxide to avoid resinification, was 174° at 753 mm., but it altered quickly. The liquid was not ocimene. On reduction with sodium and alcohol, steam distillation, and distillation at ordinary pressure from sodium, a fraction was obtained having b.p. 168—169°/754 mm., d^{14} 0.805, n_D^{12} 1.4553, and α_D^{12} $+12^\circ 54'$, with an odour similar to hydromyrcene. With bromine in acetic acid, the olefinic terpene gave hydrogen bromide and became coloured, but no solid compound was obtained. The lower fractions obtained by distillation of the terpene appeared to contain still another terpene. M. S. BURR.

Apparatus for the determination of volatile oil. J. F. CLEVINGER (J. Amer. Pharm. Assoc., 1928, 17, 345—349).—An apparatus for the direct determination of volatile oil in plants and plant products consists of a round-bottomed flask containing the material mixed with water and connected with a separator and condenser. The mixture of oil and water obtained on distillation is collected in the separator, and the water is

automatically returned to the flask. Comparative determinations of the yields of oil and their physical constants obtained by the above and the U.S.P. methods show satisfactory agreement.

E. H. SHARPLES.

Absolute essence of *Salvia sclarea*. Y. VOLMAR and A. JERMSTAD (J. Pharm. Chim., 1928, [viii], 7, 390—395).—See B., 1928, 284.

Viscosity of oils. RAASCHOU.—See I.

PATENTS.

Preparation of acetylene for anæsthetising purposes. A. BOEHRINGER [C. H. BOEHRINGER SOHN] (B.P. 264,826, 17.1.27. Ger., 25.1.26. Addn. to B.P. 205,240; B., 1923, 1199 A).—Technically pure acetylene is freed from residual impurities and solvent (e.g., acetone) by passage through solid adsorbents such as active charcoal, silica gel, etc., these being pretreated with chromic acid, water, etc. if desired. Where impurities and solvent are removed simultaneously, the adsorbent should be of an acid character or be pretreated with an acid or acid substance, a small quantity of oxygen or air may be added to the acetylene, and adsorbent and acetylene should be dry. B. FULLMAN.

Manufacture of mixed alkaloid salts. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,279, 14.6.27. Ger., 26.6.26).—By treating equivalent mixtures of two *Solanaceæ* alkaloids (e.g., scopolamine with hyoscyamine or with atropine) in a solvent (water, alcohol, ether) with camphoric, phthalic, meconic, malonic, tartaric, saccharic, sulphuric, or other dibasic acid, and removing the solvent in a vacuum, mixed alkaloid salts of therapeutic value are obtained.

C. HOLLINS.

Manufacture of readily-soluble organic salts of C:C-[5:5]-dialkyl- and arylalkyl-barbituric acids. ÉTABL. POULENC FRÈRES (B.P. 271,837, 14.4.27. Fr., 25.5.26).—5:5-Dialkyl- or 5-aryl-5-alkyl-barbituric acids are dissolved in water with the addition of an equivalent of a β -aminoethyl alcohol, e.g., β -amino- or β -diethylamino-ethyl alcohol, or $\beta\beta'$ -dihydroxydiethylamine. Solutions of 5—10% concentration are readily obtained and are stable below 100°. C. HOLLINS.

Pharmaceutical product [cyclic amino-metalmercapto-compounds]. W. SCHOELLER, A. FELDT, M. GEHRKE, and E. BORGFWARDT, Asss. to CHEM. FABR. AUF ARTIEN (VORM. E. SCHERING) (U.S.P. 1,667,052, 24.4.28. Appl., 1.6.26. Ger., 2.6.24).—See B.P. 234,806; B., 1925, 692.

Specific antidiabetic principle [purified insulin]. H. W. DUDLEY (U.S.P. 1,669,328, 8.5.28. Appl., 20.3.24. U.K., 27.3.23).—See B.P. 216,978; B., 1924, 654.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Viscosimeter. ALBRECHT and WOLFF.—See I.

PATENTS.

Manufacture of photographic silver halide emulsions. W. DIETERLE, O. MATTHIES, and J. REITSTÖTTER, Asss. to I. G. FARBENIND. A.-G. (U.S.P. 1,667,589,

24.4.28. Appl., 12.7.26. Ger., 7.10.25).—Substances which have been extracted from proteins by dialysis are added to silver halide emulsions during manufacture.
A. R. POWELL.

Production of photo-prints and photo-copies. I. G. FARBENIND A.-G. (B.P. 286,233, 28.2.28. Ger., 28.2.27).—Surfaces coated with salts of 1:2-naphthaquinone-4-sulphonic acid are exposed to light, *e.g.*, under a drawing on tracing cloth, and then treated with an organic compound containing one or more amino-groups. The portion of the salt undecomposed by the light is converted into a coloured anil, a positive picture being produced.
B. FULLMAN.

Sodium sulphide (B.P. 289,354).—See VII.

XXII.—EXPLOSIVES; MATCHES.

Analysis of residual acid from nitroglycerin manufacture. W. YOUNG (J.S.C.I., 1928, 47,126—130 T).—Total acidity determination does not include the whole of the nitric acid combined as nitroglycerin. For the direct determination of sulphuric acid a method is described in which nitrogen acids are distilled out in steam, the denitrated acid is oxidised with 0.1*N*-permanganate, and residual acid is titrated with *N*-caustic soda after treatment with hydrogen peroxide. A correction is applied for the alkali equivalent of the permanganate used. For total nitrogen acids the nitrometer was used and a direct method employing steam distillation is described. The latter method was also used in determining total nitrogen acids in a solution of nitrosylsulphuric acid in 97% sulphuric acid, in check work on the determination of nitrous acid in this solution by potassium dichromate. Nitrous acid in residual acid was determined by permanganate. Nitroglycerin was determined by difference between the dichromate and permanganate values and by wet combustion.

PATENT.

Ignition of explosives. W. ESCHBACH (B.P. 276,962, 30.6.27. Ger., 1.9.26).—A thermite mixture, preferably forced into caps with or without the addition of a primer, *e.g.*, nitrocellulose or thiocyanogen-perchlorate priming, ignited by a fuse or by electrical means, is used for the ignition of explosive charges. [Stat. ref.]

L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Water softening with barium salts. C. J. RODMAN (Chem. Met. Eng., 1928, 35, 221—223).—The use of barium salts in softening is advantageous with waters of high sulphate content. One method is to add a determined amount of lime-water to a softener containing a large excess of (insoluble) barium carbonate. A newer process provides for the preliminary treatment with steam of a mixture of barium carbonate and lime, the material actually added to the water thus including a proportion of barium hydroxide. Barium carbonate alone will not remove more than 85% of dissolved calcium sulphate from water. The use of lime-water improves on this, but for total elimination the presence of sodium sulphate or chloride is necessary, the electrolytes precipitating barium sulphate from the

colloidal state. The second process, unlike the first, frees the water from sodium sulphate and calcium hydroxide, sodium hydroxide being formed by interaction with barium hydroxide. It has the further effect of reducing the solubility of calcium carbonate. A minimum concentration of 1 pt. of sodium chloride in 70,000 pts. of water is to be maintained for successful working. A very thin scale only is formed and priming disappears.
C. IRWIN.

Chlorination of water. L. W. HAASE (Gas- u. Wasserfach, 1928, 71, 385—390).—For the purpose of this investigation freshly prepared chlorine water was used, it having been ascertained that even in the dark and at 0° it cannot be kept more than 5 days without alteration in p_H value. This value for treated water was determined electrically, the effect of chlorine on a platinum electrode being negligible for a short time. No indicators are suitable for use in presence of chlorine, but a rough alternative method is to remove the latter with its equivalent of sodium thiosulphate and titrate. The gradual increase of acidity in natural water with addition of chlorine was traced, this being due to the conversion of calcium carbonate into calcium bicarbonate. The same result is shown with water treated with calcium hydroxide of p_H 9.5—10.0. This is supersaturated with calcium carbonate and becomes turbid on being kept, but the turbidity is cleared by addition of chlorine. Water containing organic matter behaved similarly showing increased acidity, also a reduction in the oxygen absorption and an increase in the (small) permanent hardness. The organic matter acts as a catalyst. Such waters if low in lime may well be rendered corrosive.
C. IRWIN.

Microdetermination of iodine in potable waters. III. **Oxidation method.** M. SETTIMI (Annali Chim. Appl., 1928, 18, 104—107).—Further experiments (cf. B., 1927, 958) show that microtitration of iodine with sodium thiosulphate gives satisfactory results if the dilutions of the two solutions are not greater than 0.002*N* and if the temperature does not differ greatly from the ordinary air temperature. Previous treatment of the water, or of any liquid rich in salts, with chlorine water or sodium hypochlorite leads to erroneous results.

T. H. POPE.

Pollution in streams. JONES.—See II.

PATENTS.

Evaporation and distillation [of feed-water make-up]. J. S. FORBES (U.S.P. 1,666,777, 17.4.28. Appl., 26.7.21).—A stream of exhaust steam is divided, a portion being directly condensed in a main condenser; the other portion is reheated (*e.g.*, by live steam in an ejector compressor) and passes to a smaller condenser or heat-interchanger, where it is itself condensed and raises the temperature of a portion of the main warm cooling water to such an extent that the latter will partially vaporise; this additional quantity of vapour also passes to the main condenser and forms an additional quantity of feed water.
B. M. VENABLES.

Settling tanks (B.P. 287,611).—See I. **Preparations for respirators** (B.P. 280,554).—See VII. **Fertilisers** (B.P. 289,284).—See XVI.