

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 20, 1929.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Furnaces. J. F. O. STRATTON (U.S.P. 1,713,566, 21.5.29. Appl., 28.8.25).—In a furnace in which comminuted fuel slips down the imperforate sloping walls of a combustion chamber which converge to an oscillating air-inlet grate at the bottom or throat, the forced draught to the grate is first caused to pass over the outside of the walls of the throat to cool it. The upward air currents are adjusted to keep the lighter particles of coal (mostly coked) in suspension while the heavier fused particles of ash drop through the grate.

B. M. VENABLES.

Furnaces and furnace grates. T. O. WILTON, and CHEM. ENG. & WILTON'S PATENT FURNACE CO., LTD. (B.P. 313,380, 19.9.28).—An inclined grate is provided through which air may be forced in at the back of the grate of a Lancashire boiler or the like, so as to produce a swirling motion of the partly-burnt gases in the neighbourhood of the white-hot firebridge. A method of construction is described by which standard parts may be used for several different sizes of furnace.

B. M. VENABLES.

Soaking-pit furnaces. F. W. MANKER, ASSR. to SURFACE COMBUSTION CO., INC. (U.S.P. 1,711,273—4, 30.4.29. Appl., [A] 15.7.25, [B] 17.11.27).—(A) A furnace of the pit type has tapering combustion chambers in opposite walls, with gas burners situated in the smaller parts; the products of combustion are exhausted through passages situated below each combustion chamber. (B) A number of soaking pits are arranged side by side, and each has independent firing and exhausting means, but passages are provided between adjacent pits, so that products of combustion can be passed from one pit to another.

B. M. VENABLES.

Heat exchangers. SUPERHEATER CO., LTD. From COMP. DES SURCHAUFFEURS SOC. ANON. (B.P. 312,470; 4.5.28).—This invention may be applied to the heating of feed water by live steam. The feed water (or other fluid to be heated) is contained in a drum which also contains one or more heating units comprising superposed inlet and outlet headers connected by U-shaped heating tubes which fall all the way, and the headers are also connected directly by a smaller drainage hole or passage; the result is that condensate is cleared so rapidly that the heating agent may be live steam taken from a boiler and circulated back to the same boiler.

B. M. VENABLES.

Heat-exchange apparatus. G. D. MANTLE, ASSR. to MANTLE ENGINEERING CO. (U.S.P. 1,712,892, 14.5.29. Appl., 9.4.26).—The fluid to be heated flows in passages transverse to the flow of heating gases in a zig-zag

manner, the resultant general direction being counter-current. The fluid to be heated is, at entry, divided into two streams each controlled by a valve and flowing in separate parallel passages to the exit, where they recombine. At the hot end one of the streams makes additional transverse passes, and this stream only is subjected to the highest temperature.

B. M. VENABLES.

Heat-exchange apparatus comprising [brass] tubes having an electrolytic [chromium] deposit on the interior surface. F. G. MARTIN and W. RAMSAY (B.P. 299,100, 22.4.27).—The corrosion of brass condenser tubes is prevented by coating them internally and externally with electrolytically deposited chromium.

A. R. POWELL.

Heat-exchanging devices. C. MCNEIL (B.P. 313,780, 11.7.28).—The inner fluid passes through a bundle of tubes which is rotated in a fixed drum containing the outer fluid. The headers for the tubes are radial and are connected to hollow hubs and trunnions forming the inlet and outlet. The outlet hub is divided into compartments, one for each spoke or radial header, to prevent undue choking by the outlet fluid, which may be condensed steam.

B. M. VENABLES.

Heat-transferring devices, such as air preheaters. W. A. WHITE (B.P. 313,998, 21.3.28).—The preheater described is built up of unit cells formed from corrugated plates, the heating and heated gases passing at right angles through the cells.

B. M. VENABLES.

Heat-treatment apparatus. A. VUILLEUMIER, ASSR. to NEW DEPARTURE MANUF. CO. (U.S.P. 1,715,317, 28.5.29. Appl., 7.5.27).—The chamber or mixer for holding the articles to be treated is shaped as a wide-mouthed beaker flask and is rotated about a normally horizontal axis, but is preferably tiltable together with the surrounding heating chamber to discharge the articles. The heating chamber is preferably closed and electrically heated.

B. M. VENABLES.

Dryer. D. E. WASHINGTON, ASSR. to W. B. CONNELL (U.S.P. 1,715,729, 4.6.29. Appl., 25.7.27).—A revolving drum for the material is provided with an interior heating barrel which is revolved independently.

B. M. VENABLES.

Material dryer. M. MADSEN, ASSR. to MADSEN IRON WORKS (U.S.P. 1,715,852, 4.6.29. Appl., 11.10.26).—A rotating-cylinder dryer is provided with a hopper and stack at the feed end, with a stationary cowl surrounding the drum at the other end, and a stationary firebox (within the drum) in the form of a brick-lined cylinder with open ends. Fluid fuel is supplied through the closed end of the cowl to the interior of the firebox, and the air for combustion is preheated in the space

between the cowl and the drum and firebox. The material discharges through the space between the firebox and the drum, to which is connected a shoot extending through the cowl.
B. M. VENABLES.

Desiccating apparatus. B. BOUDA and G. G. SKELTON, Assrs. to COLLIS Co. (U.S.P. 1,717,491, 18.6.29. Appl., 23.11.27).—A rotating heated drum is provided with a scraper bar bearing heavily on it and reciprocated in a direction parallel to the axis of the drum.

B. M. VENABLES.

Drying system for use with annular kilns. O. BUSCH (U.S.P. 1,718,190, 18.6.29. Appl., 14.5.27. Ger., 21.9.26).—A number of parallel drying chambers are arranged at opposite sides of the kiln, and are provided with upper and lower series of flues, so connected that combustion gases and hot air from the kiln may be used for direct and indirect heating of the chambers, and to permit moist air to be returned to the flues for admixture with fresh air.

B. M. VENABLES.

Extraction of moisture from air or other gases. A. E. SHERMAN and I. MAYNARD (B.P. 314,592, 15.10.28).—The drying medium, *e.g.*, calcium chloride, is used in two stages, the incoming gas passing first in contact with partially spent calcium chloride liquor (which is used till it is completely spent) and finally in contact with solid calcium chloride, the liquor dripping from which is used in the first stage. A suitable apparatus is described, including light-weight flap valves to close automatically the final drying chamber when not in use.

B. M. VENABLES.

Pulveriser. A. F. THOMPSON (U.S.P. 1,715,724, 4.6.29. Appl., 13.6.27).—In a stamp mill constructed so as to be light and easily dismantled, and not to need much water, there are no dies, and the square shoes of the stamps are close together and operate directly on a floor plate that slopes downwards from the feed end. A cover plate is provided for the mortar box; the stamps are spring-loaded and a special form of lifting cam is described.

B. M. VENABLES.

Apparatus for pulverising materials. W. T. DOYLE, Assr. to STURTEVANT MILL Co. (U.S.P. 1,715,123, 28.5.29. Appl., 1.10.28).—In a closed-circuit grinding system the net feed is caused to vary inversely as the circulating feed to compensate for variation in the fragility of the material.

B. M. VENABLES.

Grinding plate for attrition mills. W. H. MECHLIN, Assr. to BAUER BROS. Co. (U.S.P. 1,715,772, 4.6.29. Appl., 26.9.27).—The segmental grinding plates are held in the end of the mill by bolts the heads of which are in pockets in the back of the plates, with openings to the periphery for insertion of the bolts.

B. M. VENABLES.

Mixing apparatus. R. W. SHAFOR, Assr. to GILCHRIST & Co. (U.S.P. 1,701,164, 5.2.29. Appl., 13.2.25).—The liquids or substances to be mixed are circulated in a closed system, *e.g.*, by propeller stirring, centrifugal pump, etc., through a series of baffle plates arranged to divide the stream of liquid into a series of channels, the separate streams being further divided and intermingled, and finally united in a common stream. The process may be continuous or discontinuous and the direction of flow

changed repeatedly in the operations. Several such mixing tanks may be arranged in series. R. BRIGHTMAN.

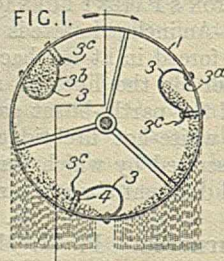
Mixing apparatus. J. M. BRINKLEY (U.S.P. 1,708,493, 9.4.29. Appl., 29.5.26).—A rotatable shaft projects from the lid of the apparatus, and a head bar, adjustable horizontally and vertically in relation to the shaft, carries a mixing frame having a pair of diamond-shaped side blades connected by a triangular bottom blade.

W. G. CAREY.

Mixing machine. W. B. RANNEY, Assr. to W. WRIGLEY, JUN., Co. (U.S.P. 1,713,554, 21.5.29. Appl., 2.8.26).—An elongated open receptacle is formed from a number of parallel transverse troughs in each of which is rotated a mixing blade of distorted Z-shape; the mixers run at equal speeds, but in opposite directions in alternate compartments. The feed is at one end and outlet at the other, but no other means is provided for creating longitudinal flow, so that the motion is rapidly undulating, with very slow longitudinal travel when used continuously.

B. M. VENABLES.

Screening or sifting devices. A. S. D. CRONE (B.P. 302,829, 2.2.28).—The interior of a rotary drum screen or trommel is provided with a number of scoops,



so that the descending lower quarter of the circumference is effective for screening, as well as the ascending quarter.

B. M. VENABLES.

Screening of materials. WOODALL-DUCKHAM (1920), LTD., and S. SYMINGTON (B.P. 312,743, 26.3.28).—The screening surface comprises alternate sections of fixed spaced bars and rotating fingers; the latter lift the material from the set of fixed bars above and deposit on the set of fixed bars below.

B. M. VENABLES.

Centrifugal machine. C. V. BOUILLON (U.S.P. 1,717,753, 18.6.29. Appl., 14.7.28. Fr., 6.6.28).—A centrifugal separator which is provided with continuous decanting means for muddy liquid, comprising a number of tapered chambers with an orifice in the apex of each, has a revolving eccentric blade inside each orifice, the blades being pressed towards the orifice by centrifugal force and rotated by gearing while the separator is spinning.

B. M. VENABLES.

Reduction of froth formation at centrifugal liquid separators etc. G. W. RUDA, Assr. to AKTIEBOLAGET BALTIC (U.S.P. 1,718,081, 18.6.29. Appl., 24.9.27. Swed., 25.8.27).—The separated liquid flows over a weir in the neck of the bowl into a circumferential groove which is provided with a dam to cause the out-flowing liquid to flow the opposite way round to the rotation, towards the final outlet. The weir extends

round the whole circumference except at the final outlet.

B. M. VENABLES.

Separation of comminuted matter from the liquid in which it is immersed. [Thickener.] C. H. NORDELL (U.S.P. 1,718,871, 25.6.29. Appl., 19.8.26).—The feed pulp is caused to flow, with as little disturbance as possible, inwardly through a number of superposed shallow circular cells, clear liquid being exhausted from the centre and the settled sludge moved outwards mechanically.

B. M. VENABLES.

Gas-washing apparatus. H. A. BRASSERT, C. E. DOUGAN, and E. VON MALTITZ, Assrs. to H. A. BRASSERT & Co. (U.S.P. 1,707,548, 2.4.29. Appl., 9.3.27).—The apparatus comprises a series of washing chambers supplied with sprays for injecting washing liquid and arranged in a casing between two horizontal diaphragms one above the other. The diaphragms extend inwards about halfway across the diameter of the casing, and are midway between two circular discs supported by a central shaft and extending outwards from the shaft to a point about midway between the walls and the inner ends of the diaphragms. The space between discs and diaphragms is closed by annular rows of interacting disintegrator elements which break up the gas stream between the various compartments.

A. R. POWELL.

Washing flue gases. J. T. BARON and J. B. CLARKE (B.P. 314,110, 22.3.28).—A chimney for the washing and discharge of the whole of the flue gases flowing without mechanically induced draught is constructed so that the flue gases enter sideways part way up; above that point are situated a number of stories of mist-producing sprinklers, each story offset to the next, the grit and water (or other fluid) being carried straight down past the inlet to the lower part of the chimney which is adapted to collect the mud produced.

B. M. VENABLES.

Air filter. L. L. DOLLINGER (U.S.P. 1,714,854, 28.5.29. Appl., 21.3.27).—A number of filtering units are suspended below holes formed in a plate within the upper part of a casing which converges above the plate to form a single outlet. The filter units consist of perforated plates or wire mesh pleated into cylinders the walls of which are in the form of radial pockets. The mesh work is covered by filter medium and the flow of air is inwards, the lower ends of the cylinders being closed by plates which also secure the filters to the plate above by means of V-brackets and screwed rods within.

B. M. VENABLES.

Preparation and consumption of compressed gases. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 287,486, 20.3.28. Ger., 21.3.27).—A vehicle for the transport of liquefied gases is provided with a compressor, an expansion machine, a liquefier, and a vaporiser. One use of the apparatus is that when the material, e.g., oxygen, is to be delivered in a gaseous form additional liquid oxygen may be made from the atmosphere.

B. M. VENABLES.

Rotary chemical furnace. L. TOCCO and M. LANDI, Assrs. to FOUR CHIMIQUE ROTATIF SOC. ANON. (U.S.P. 1,708,526, 9.4.29. Appl., 22.6.26. Italy, 26.6.25).—See B.P. 278,774; B., 1927, 927.

II.—FUEL; GAS; TAR; MINERAL OILS.

Material and morphological behaviour of lignin-rich conifer tissues in the formation of forest humus and brown coal. W. GROSSKOPF (Brennstoff-Chem., 1929, 10, 161—167, 213—217; cf. B., 1926, 939).—The chemical and morphological changes accompanying the transformation of pine needles and pine wood into peat, lignite, and brown coal have been studied. All conifer needles possess a high lignin content, usually greater than 35% of the cell-wall material. In the transformation of pine needles into peat a close relationship exists between the rate of disappearance of the lignin and the rate of appearance of the humus. The cellulose:lignin ratio in the plant tissue gives a measure of its ease of decomposition. Morphological examination of decomposing pine needles shows that the parenchyma rich in cellulose is more rapidly destroyed than are the more highly lignified tissues. In the transformation of conifer wood into peat and brown coal the autumn wood presents a greater resistance to decomposition than the spring wood; this, however, is due, not to its greater lignin content, but probably to the different distribution of lignin within the tissue. Chemical examination of samples of pine-wood peat and lignite shows that, compared with the unchanged wood, the changes in composition are greater than is apparent from the morphological examination. Thus the pinewood peat contains only half the cellulose (24%) of the undecomposed wood and 20% of humus, whilst the Senftenberg lignite contains only 5% of cellulose and 70% of humus. The excess of the decrease in the cellulose over the increase in the humus is proof of the complete disappearance of cellulose in these decomposition processes. Attention is directed to the probable importance of conifer needles as the starting material from which brown coals have been formed.

A. B. MANNING.

Reactivity of coke. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 128—131, 148—153, 168).—The reactivity is determined by passing air at a rate of 20 litres/hr. over the powdered coke contained in a platinum boat within an electrically heated furnace, and measuring the rate of formation of carbon dioxide. After placing the boat containing the sample in the furnace the air current is passed until temperature equilibrium is attained (2 min.) and the exit gases are then passed for a definite time (1—5 min.) through a weighed absorption tower charged with potassium hydroxide. The reactivity is expressed as mg. of carbon dioxide per sq. cm. of coke surface per 5 min. Taking the same total volume of material each time, and using sugar charcoal, the reactivity was found to be independent of particle size. Experiments carried out with four different cokes and with different lengths of coke surface in the boat showed that the alteration in the surface during the determination did not appreciably affect the relative reactivities. A length of 0.5 cm. was taken as standard. At 600° the reactivities of a number of cokes varied from 17 for a foundry coke and 22 for a blast-furnace coke to 125 for a coke made from a Polish flaming gas coal. At 700° these differences became smaller, and above 800° all the cokes gave the same value for the reactivity. Graphite and diamond

possessed relatively low reactivities. A study of the influence of catalysts on the reactivity and the form of the reactivity-temperature curve confirms the assumption that the true reactivity of the material is being determined, independently of its relative surface area. The reactivity as determined by the passage of the air current through a column of the coke involves the surface area as well as the actual reactivity; it is suggested, therefore, that a comparison of the results of the two methods should give a means of determining the relative surface areas of different cokes. By determining the reactivities at temperatures below 500° and by extrapolating to zero reactivity the ignition temperatures of the cokes may be determined. The slope of the curve, however, often renders accurate extrapolation difficult, and it is suggested that the relative ignition temperatures be taken as those at which the reactivity possesses a small definite value, *e.g.*, $10 \text{ mg./cm.}^2/5 \text{ min.}$ These values varied from 338° for a brown-coal coke to 730° for graphite.

A. B. MANNING.

Reactivity of coke. F. G. HOFFMANN (Brennstoff-Chem., 1929, 10, 287—288; cf. Kreulen, preceding abstract).—Kreulen's assumption that the rate of reaction measured in his experiments is independent of the particle size of the coke is criticised.

A. B. MANNING.

Reactivity of coke. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 288—289; cf. preceding abstract).—Further experiments are quoted to show that the influence of particle size on the rate of reaction, if appreciable at all, is very small. In comparing reactivities, however, it is agreed that coke of a definitely limited range of size should be chosen.

A. B. MANNING.

Reducing power of brown-coal coke. F. SEIDENSCHNUR and A. JÄPPELT (Brennstoff-Chem., 1929, 10, 195—198).—A current of carbon dioxide at 5 litres/hr. was passed through a 5-cm. layer of the coke (2—3 mm. size) in an electrically heated porcelain tube and the exit gases were analysed. The results are shown as curves in which the volume of carbon monoxide produced from 100 vols. of carbon dioxide is plotted against the temperature, which ranged from 400° to 1000° . Low-temperature cokes from brown coal possessed a greater reducing power than either wood charcoal or active charcoal, which in turn were more reactive than cokes from a bituminous coal. The reducing power of the brown-coal cokes fell off as the temperature of carbonisation was raised. Treatment with methane at 800° or 1030° reduced the reactivity of the brown-coal cokes, as did also extraction of the ash with hydrochloric acid.

A. B. MANNING.

Practical apparatus for Fischer's coking test. O. KELLER (Chem.-Ztg., 1929, 53, 517).—The apparatus described by Schaefer for the determination of water in coals (B., 1928, 557) may be used for the determination of gas, water, tar, and coke in bituminous coals by providing it with a side tube terminating in a flask with side tube for removing the gas. The tar collects in the side tube of the heating apparatus and the water in the flask; the sum of the two is obtained by noting the increase in weight of the tube and flask, and the water is then determined by the xylene method of Schaefer.

A. R. POWELL.

Retardation of carbonisation in coke-oven flues.

C. A. BAGLIN (Gas World, 1929, 90, Coking Sect., 58—59).—The combustion of coal gas with a normal excess of air gives a short flame which delivers more heat to the lower part of the charge. The speed of combustion in the flues may be reduced by diluting the combustion air with products of combustion, thereby obtaining a long flame; various methods of diluting the combustion air are discussed and criticised, and experiments are described whereby products of combustion are introduced into reversible hairpin flues in which combustion takes place in two stages.

C. B. MARSON.

Manufacture of domestic coke in the coke oven.

ANON. (Gas World, 1929, 90, Coking Sect., 54—57).—Factors which influence the reactivity of coke are discussed. Highly reactive cokes are obtained by the low-temperature carbonisation of any coal carbonised below, say, 700° and by the high-temperature carbonisation of non-coking or poorly coking coals; fuels possessing the property of high reactivity do not become plastic during carbonisation. It is impossible to carbonise coals in the coke oven which do not become plastic because the resulting coke cannot be discharged, but coking and non-coking coals can be blended so that a coke is produced which is sufficiently strong to be discharged and robust enough to withstand transport conditions. Similar results may be obtained by (a) pre-oxidation of a portion of the coal before coking, (b) the coking of low-temperature coke with coking coal, and (c) the addition of mineral substances such as iron oxide and lime to the coal before carbonisation. The combustibility of coke is greatly reduced if the coke is heated to too high a temperature or if it is exposed unduly to a moderate temperature. It is suggested that the best conditions for the manufacture of a coke for domestic use are (i) treatment of the coal before carbonisation by one of the methods described above, (ii) manufacture of the coke in an oven in which the carbonisation is completed simultaneously at the top and bottom of the charge, and (iii) pushing of the charge with the centre slightly "green," so that overheating is avoided and a little volatile matter is retained in the coke.

C. B. MARSON.

Activation of carbon by means of steam. P. P. KOSAKEVITSCH and N. A. ISMAILOV (Kolloid-Z., 1929, 48, 241—246).—A method is described for activating carbon for adsorption purposes by passing superheated steam over the carbon, which is heated in a porcelain tube in an electrical resistance furnace. Examination of the influence of varying the conditions of activation shows that between 700° and 800° the activity of the carbon increases with the duration of activation and falls when the velocity of the current of steam is reduced. At 850° the activity reaches a maximum after activation for 15 min., and subsequently falls off rapidly; the appearance of the maximum is delayed by reducing the rate of the current of steam; the maximum is even more strongly marked at 900° . As a measure of activity, the adsorption of phenol from a 1% aqueous solution was taken; similar results were obtained when a basic substance (ethylamine) was used for adsorption. Some experiments were also carried out on the influence

of various common constituents of the ash of charcoal on the activity. This was effected by impregnating the charcoal with salts of iron, aluminium, or calcium, precipitating the hydroxides by means of ammonia, and heating the products before activation by means of steam. Calcium oxide was found to increase the activity of the carbon, ferric oxide diminished the activity, whilst the effect of aluminium oxide was irregular.

E. S. HEDGES.

Products of combustion from typical gas appliances. IV. 21ST REP. OF JOINT RES. COMM., INST. GAS ENG. AND UNIV. LEEDS (Gas J., 1929, 186, 740—744; cf. B., 1928, 557).—A number of tests were made with a 2½-gal. geyser, a surface-combustion griller, and a griller and ring-burner of a typical domestic cooker. It is shown that (a) the permissible gas rate increases as the flue length of a geyser is increased, and it is therefore recommended that the baffle should be placed as near as practicable to the ceiling; (b) causes tending towards a high carbon monoxide production with the geyser examined are: (1) smothering of the outer ring of flames if too short a flue pipe is used, and (2) the flames becoming ragged and blowing out intermittently (due to excessive gas pressure and rate on individual burners, together with excessive velocity of air and products through the combustion chambers); (c) production of carbon monoxide from a surface-combustion griller is remarkably low; (d) a typical domestic griller, having a single central burner and fixed iron frets, produces larger quantities of carbon monoxide, but the quantity produced can be materially lessened by raising the frets; carbon monoxide production is greatly increased by immersing a solid substance in a flame; (e) a drilled burner, when employed for heating a pan of water, and if not used above its normal gas rate, does not produce carbon monoxide in any harmful amount; this is probably because the considerable distance between the pan and the burner head allows adequate admixture with secondary air, while only the tips of the flames are chilled by the pans; (f) disc burners, under similar conditions, give relatively high carbon monoxide production (but the quantity is not influenced so greatly by the gas rate as with the drilled burners); this may be due to the greater chilling of the flame by the vessel or to the less favourable conditions for secondary aeration offered by a thin film of flame exposed to the air on one side only. It is concluded that the carbon monoxide production of a disc burner may be kept reasonably low by paying careful attention to details of design, without interfering with its higher thermal efficiency (compared with a drilled burner).

C. B. MARSON.

Purification [of coal gas from hydrogen sulphide]. G. C. PEARSON (Gas J., 1929, 186, 797—807).—An historical survey is made of the development of the process for the removal of hydrogen sulphide from crude gas by means of iron oxide in box purifiers, and the design and layout of a typical plant are described in full detail. The use of various liquid purifying agents is discussed, and the results of a number of experiments are given.

C. B. MARSON.

Higher alcohols from petroleum hydrocarbons.

I. S. PILAT and J. WINKLER. II. S. PILAT, W. J. PIOTROWSKI, and J. WINKLER (Przemysl Chem., 1929, 13, 185—195, 208—220).—I. The highest yields of olefines, as well as of benzine, are obtained from the cracking of gasoline at 600—670°. The olefine gases are converted into a mixture of higher secondary alcohols, from which isopropyl, *sec.*-butyl, and *sec.*-amyl alcohols are isolated.

II. The gaseous products of the cracking of gasoline, using the Cross system, contain 10—12 vol.-% of olefines (propylene and homologues). In order to convert these olefines into alcohols, the gas must first be freed from water, sulphur, and gasoline. The first two objects are best achieved by passing the gas through two towers, the first of which is packed with a mixture of calcium oxide, sodium hydroxide, and wood shavings, whilst the second contains granular ferric hydroxide in place of the lime in the first tower. Gasoline is best removed by adsorption on active charcoal, which can, provided that the gas has previously been properly desulphuretted, be used repeatedly. Absorption of olefines increases with concentration of sulphuric acid, from 32% in 75% acid to 83.7% in 96% acid, but the yield of alcohol diminishes with increasing concentration of acid, from 84.2% of the theoretical yield using 75% acid to 36.3% using 96% acid, various oily highly polymerised products being obtained in the latter case. The best yields of alcohol are obtained using 80—85% sulphuric acid, which at 15—20° and 2 atm. absorbs about 70% of the olefines present. Absorption is more complete if for each volume of acid in the absorption towers 2 vols. of solar oil are present. The highest yields of alcohols are obtained by adding 1.5—2 vols. of water to the absorption acid, and then distilling until the residual acid is about 50%. This residual acid may be further concentrated to 80%, when it may, after being freed from sulphur dioxide, be again used for absorption. The distillate contains 35—40% of alcohols, and, after neutralisation with lime, gives on rectification 86—88% alcohol. Practically anhydrous alcohol is obtained from this distillate by saturation with sodium chloride, and this product may be further purified by acidifying with sulphuric acid and adding 1% of potassium permanganate, agitating, and alkalisng with sodium silicate, when the silica gel formed adsorbs most of the empyreumatic impurities present. The gasoline adsorbed on the charcoal during purification of the olefine gas amounts to about 200 g. per m.³ of gas, and consists of 56% of paraffin, 6% of aromatic, 2% of naphthene, and 35% of unsaturated hydrocarbons. The last-named fraction consists chiefly of amylenes, hexylenes, and heptylenes, and may be converted by the action of sulphuric acid into a mixture of *iso*amyl and higher alcohols. The residue may by appropriate rectification be converted into benzine containing only aliphatic hydrocarbons.

R. TRUSZKOWSKI.

Treatment of bitumens with "sulphosil." V. TOKMANOV (Nef. Choz., 1928, 15, 73—74).—Silica gel is used for the determination of neutral tars and soft asphalts, and is followed by treatment with "sulphosil" (activated silica gel), whereby the unsaturated compounds and aromatic substances are extracted, leaving paraffins and naphthenes.

CHEMICAL ABSTRACTS.

Bunsen flames of unusual structure. F. A. SMITH and S. F. PICKERING (Bur. Stand. J. Res., 1929, 3, 65—74).—See B., 1928, 880.

PATENTS.

Apparatus for dehydrating crude [petroleum] oils. W. L. PALMER, Assr. to REGAN FORGE & ENG. Co. (U.S.P. 1,696,859, 25.12.28. Appl., 31.1.25).—The oil, after removal of natural gas, is heated and sprayed into a horizontal, rotating, elongated cylinder. The water and other foreign substances are discharged by valve-controlled peripheral outlets, and the oils remaining in the central portion of the cylinder are sent to storage.

R. BRIGHTMAN.

Treating hydrocarbon oil. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,710,063, 23.4.29. Appl., 31.8.22. Renewed 13.9.26).—Cracked petroleum distillate is treated successively with sulphuric acid, sodium hydroxide solution, *d* 1.045—1.091, and sodium plumbite solution, and after distillation is mixed with 50—60% of refined bottoms or high-boiling residues and again distilled.

R. BRIGHTMAN.

Purification of hydrocarbons. J. C. BLACK and W. H. LOW, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,696,377, 25.12.28. Appl., 17.3.27).—Hydrocarbons are purified (desulphurised) by counterflow treatment with cadmium hydroxide in presence of sodium hydroxide.

R. BRIGHTMAN.

Purifying petroleum oils. J. C. BLACK, W. D. RIAL, and R. T. HOWES, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,709,203, 16.4.29. Appl., 26.4.26).—The oil is treated with dilute sodium hydroxide under pressure and agitation with steam, then with an alkaline-earth hydroxide and an adsorbent, agitated, and heated to 150°.

R. BRIGHTMAN.

Process of purifying hydrocarbons. J. C. BLACK and M. L. CHAPPELL, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,710,205, 23.4.29. Appl., 28.5.27).—Petroleum distillate, *e.g.*, gasoline stock, preferably after treatment with sodium hydroxide or hydrochlorite, is cooled and treated with about 25% of liquid sulphur dioxide at 8°, then passed in countercurrent with fuming sulphuric acid (3—15% SO₃), and the remainder of the sulphur dioxide removed by heat, and cooled and compressed for re-use. Suitable heat-exchanging cycles are claimed.

R. BRIGHTMAN.

Removing sulphur compounds from petroleum oils. W. N. DAVIS and W. H. HAMPTON, Assrs. to STANDARD OIL Co. (U.S.P. 1,705,809, 19.3.29. Appl., 15.10.24. Renewed 2.6.28).—The oil is cooled by heat exchange with treated oil and treated at -10° to 5° in countercurrent with 15% fuming sulphuric acid.

R. BRIGHTMAN.

Treatment of residual [petroleum] oils. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,199, 12.3.29. Appl., 22.10.23).—Residual oil from cracked petroleum oil is withdrawn hot from the expansion chamber, agitated with sulphuric acid, and settled. The treated oil may be returned to the cracking still, the precipitated matter being used, *e.g.*, for asphalt.

R. BRIGHTMAN.

Cracking of [petroleum] oils. J. R. MARDICK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,706,629, 26.3.29. Appl., 5.5.23).—Petroleum oil, *e.g.*, gas oil, b.p. about 250°, is treated at about 300° with 0.25—1% of dry chlorine previously activated by electrical means and supplied under, *e.g.*, 5 lb./in.² Alternatively, petroleum oil may be refined with 0.1% of the activated chlorine at 60—75°.

R. BRIGHTMAN.

Treatment [cracking] of hydrocarbons. J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,706,396, 26.3.29. Appl., 12.8.25).—Light cracking oil, *e.g.*, gas oil, is passed through a series of heating coils after successive heat exchange with residuum from the evaporator and hot oil passing from the coils to the evaporator. The gas oil is raised to cracking temperature (400—480°) in the first two coils and is mixed with heavy cracking oil, also previously heated (320—400°), in primary and secondary coils, and passed into a final coil maintained at the mean temperature or above, the heat supplied in the secondary heaters balancing losses by radiation and absorption in the endothermic cracking reaction. From the third coil the oil passes through the heat exchanger to an evaporator the vapours from which pass up a fractionating tower, and the residuum is run into storage. Condensate from the fractionating tower passes through a steam still where heavy naphtha distils off, the residue being used to spray the fractionating tower or is returned to the heavy oil supply. Light naphtha vapours from the fractionating tower are condensed and collected in the usual way.

R. BRIGHTMAN.

Treatment of compounds preferably of a hydrocarbon nature. W. J. KNOX, Assr. to PETROLEUM CONVERSION CORP. (U.S.P. 1,715,239, 28.5.29. Appl., 9.9.22. Can., 24.8.22. Renewed 16.4.29).—After a partial fractional distillation, hydrocarbons are cracked by treatment with a highly heated gas containing hydrogen or a lower hydrocarbon, *e.g.*, methane. The hot cracked gases are used to effect the partial distillation by passing them in countercurrent to the incoming oils. The vapours thus distilled are passed in part to the cracking chamber, and in part to the heater, *e.g.*, a hot-blast stove, which supplies the hot gas for effecting the cracking process.

F. G. CLARKE.

Conversion [cracking] of petroleum oils. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,707,348, 2.4.29. Appl., 16.8.22. Renewed 2.6.27).—The oil is fed into the dephlegmator and passes out with the phlegms in countercurrent with the entrant vapours to a vaporising chamber containing a partition which prevents the reflux from mixing with the heated vaporising oil, and directs its flow through a header into the heating tubes, from which it returns through a second header to the other end of the vaporiser. The second header extends sufficiently far into the vaporiser to prevent carbon draining back into the heating zone.

R. BRIGHTMAN.

Separation of liquids [hydrocarbon oils] having different b.p. B. TORREY, JUN., and G. R. SANFORD, Assrs. to SEMET-SOLVAY Co. (U.S.P. 1,701,988, 12.2.29. Appl., 13.11.22).—Crude light oil is washed and distilled, preferably continuously with direct steam. The water-

white condensate, containing oils of lower b.p. than xylenes, is passed continuously through a series of dephlegmating columns such that in each column only the liquid of lowest b.p. is completely distilled off. The residual phlegm from each column, free from that product of lower b.p., is fed into the succeeding column. First runnings (benzene, toluene, and xylenes) are drawn off pure from successive condensates. Phlegms from the final (xylene) column are returned to the reservoir for high-boiling residues from the preliminary distillation.

R. BRIGHTMAN.

Fractional distillation [of hydrocarbon oils].

J. PRIMROSE, ASSR. to FOSTER WHEELER CORP. (U.S.P. 1,707,369, 2.4.29. Appl., 30.1.24).—The oil is fed into a steam-heated storage tank and is heated progressively by circulation through a tubular heater, the oil and vapour returning to a separating tower, whence the vapours escape and are condensed in the usual manner, while the oil returns to the storage tank. Alternatively, the oil and vapours may be returned direct to the storage tank, the vapours escaping with evaporation vapours through a vapour column. R. BRIGHTMAN.

Distillation of mineral oil. A. E. PEW, JUN., ASSR. to SUN OIL Co. (U.S.P. 1,707,448, 2.4.29. Appl., 22.12.26).—Water is de-aerated by evaporation and condensation in a high vacuum while flowing in shallow streams over the evaporator shelves, converted into steam in a boiler below atmospheric pressure, and injected into the oil, also under vacuum. The oil vapours and steam from the still pass into air-jacketed towers, the lubricating oil and lighter oil fractions being successively and separately condensed, while the uncondensed steam passes out to a barometric injection condenser and is condensed by injected water, water vapour and non-condensable still gases being ejected by a wet or dry vacuum pump.

R. BRIGHTMAN.

Treatment of [mineral] oils for distillation.

J. M. McCLAVE, ASSR. to CONSERVO Co. (U.S.P. 1,703,158, 26.2.29. Appl., 8.12.25).—The crude oil is treated with manganese oxide (pyrolusite) and hydrochloric acid prior to and at the time of distillation.

R. BRIGHTMAN.

Refining of mineral oils. A. LACHMAN, ASSR. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,709,315, 16.4.29. Appl., 8.12.26).—Vapours from the cracking still are passed upwards through a tower down which a stream of 85% phosphoric acid is directed by a spreader over brickwork or resistant material, the concentration of the acid being maintained constant by the injection of steam at the base of the tower. The gasoline vapour passes to a condenser, and after settling from entrained water is agitated for 5–10 min. with a solution containing 18% of sodium hydroxide and 9% of sodium hypochlorite.

R. BRIGHTMAN.

Refining used [lubricating] oils. R. V. AYCOCK and W. D. HARRIS, ASSRS. to REFINOIL MANUF. CORP. (U.S.P. 1,707,671, 2.4.29. Appl., 27.7.28).—Used mineral or lubricating oil is settled from water, agitated at 50° with about 2% of sulphuric acid, *d* 1.84, for 15–30 min., again settled, and drawn off from the resultant sludge to a still where it is heated to about 230° in a relatively high vacuum to vaporise diluents.

Fuller's earth (15–18%) is added to the heated oil so that the oil passes upwards through the reagent, which settles into a bed, through which the oil is finally filtered.

R. BRIGHTMAN.

Clarifying, decolorising, and neutralising oils.

D. R. MERRILL, ASSR. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,695,198, 11.12.28. Appl., 1.2.26).—The oil, *e.g.*, petroleum lubricating oil, is agitated with a suspension of 4–20% of raw clay or mineral earth in about half its weight of water containing a strong mineral or organic acid in amount equal to 0.25% of the weight of oil treated, *e.g.*, at 160°, then, after separating the earth, with 4–16% of fresh clay and about 2–8% of a dilute alkali solution containing not more than about 0.5% of alkali on the oil treated, together with a salt of an acid affording an insoluble calcium salt, *e.g.*, oxalate or fluoride.

R. BRIGHTMAN.

Process for treating emulsions. C. FISCHER, JUN., and W. J. REDDISH, ASSRS. to KONTOL Co. (U.S.P. 1,710,159, 23.4.29. Appl., 2.8.26).—The emulsion is heated with "sludge layer mineral oil aluminium sulphonate" and sodium hydroxide.

R. BRIGHTMAN.

Submerged-combustion process. C. ELLIS, ASSR. to ELLIS-FOSTER Co. (U.S.P. 1,716,433, 11.6.29. Appl., 17.3.25).—A pool of hydrocarbon oil is distilled by heat generated from the combustion of a hydrocarbon fuel within the pool.

H. ROYAL-DAWSON.

Apparatus for recovering gasoline. H. B. BERNARD, ASSR. to SINCLAIR OIL & GAS Co. (U.S.P. 1,713,323, 14.5.29. Appl., 14.11.24).—Vapours from a preliminary still are introduced under reduced pressure into the lower part of an exhausting tower, while liquid from the still is introduced into the upper part. The gases and vapours pass to a dephlegmator, where they are treated with a liquid absorbing medium.

H. S. GARLICK.

Manufacture of motor fuels and similar products.

D. J. YOUNG (U.S.P. 1,706,686, 26.3.29. Appl., 13.4.27).—Water-gas plant consisting of a fuel heating-chamber containing a bed of solid fuel, and a primary and secondary heat exchanger consisting of at least one shell containing checker brick is used. The fuel chamber is air-blasted, and the resulting heat stored by combustion in the primary heat exchanger and superheater to establish material temperature differences in the heat exchangers. The heated fuel bed is steam-blasted intermittently and reversely, and liquid hydrocarbons are admitted selectively at the top of the fuel chamber or at the top or bottom of the primary or secondary heat exchangers, the combustible gas being withdrawn through the fuel bed and passed through a scrubber or condenser.

R. BRIGHTMAN.

Motor fuel and its manufacture. L. KIRSCHBRAUN and C. B. BELKNAP (U.S.P. 1,707,019, 26.3.29. Appl., 13.1.20. Renewed 17.8.28).—A mixture of gasoline, kerosene, and gas oil, obtained, *e.g.*, by distilling crude petroleum until the gas oil has vaporised, is emulsified mechanically with, *e.g.*, 3–5% of water, in presence of a saturated cyclic hydrocarbon or crude naphthenic acid derivative as emulsifier, the hydrocarbon being in excess of 70% by vol. in the final product.

R. BRIGHTMAN.

Lubricating composition. G. W. GRAY, Assr. to TEXAS CO. (U.S.P. 1,716,310, 4.6.29. Appl., 8.6.27).—The rails of the curved sections of railway tracks are lubricated with a mixture which comprises approx. 60% of cylinder stock residuum and 40% of a residual oil from an asphalt-base crude petroleum.

H. S. GARLICK.

Pressure cracking treatment of liquid hydrocarbons. E. S. L. BEALE, G. H. COXON, and A. E. DUNSTAN, Assrs. to ANGLO-PERSIAN OIL CO., LTD. (U.S.P. 1,708,782, 9.4.29. Appl., 9.5.28. U.K., 15.3.27).—See B.P. 293,889; B., 1928, 701.

Apparatus for pulverising coal or other fuel. J. MULLIN (B.P. 313,245, 2.4.28).

Arrangement for admitting steam through the bottom cover of vertical-chamber ovens for gas manufacture. CHAMBER OVENS, LTD., Asses. of PINTSCH & DR. OTTO GES.M.B.H. (B.P. 289,058, 18.4.28. Ger., 21.4.27).

Apparatus for handling and quenching coke. R. DEMPSTER & SONS, LTD., and W. H. HANDLEY (B.P. 313,020, 3.2.28).

Installations for dry-cooling of incandescent coke. SULZER FRÈRES SOC. ANON. (B.P. 312,033, 17.9.28. Switz., 18.5.28).

Pulverised fuel burners. H. E. YARROW (B.P. 313,368, 24.8.28).

Burners for pulverised fuel. H. A. MARSTON (B.P. 312,972, 3.3.28. Addn. to B.P. 285,314).

Powdered fuel burners. H. E. HAZLEHURST and O. MARGETSON (B.P. 313,100, 7.12.27).

Liquid fuel burners. B. POWELL-BRET (B.P. 313,262, 19.4.28).

Burner for liquid fuels. J. C. NILSSEN (B.P. 313,830, 16.10.28).

Burners for powdered or other fuel. R. V. WHEELER and J. BRASS (B.P. 312,588, 23.2. and 5.4.28).

Liquid seals with special reference to gas producers or generators. HUMPHREYS & GLASGOW, LTD., and J. C. STELFOX (B.P. 313,754, 22.6.28).

Removal [by suction] of solid combustion residues from internal-combustion engines operated with pulverulent fuel. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 313,781, 13.7.28).

Production of acetylene and apparatus therefor. H. GEBHARDT (B.P. 313,669, 26.3.28).

III.—ORGANIC INTERMEDIATES.

Alcohols from petroleum hydrocarbons. PILAT and others.—See II.

PATENTS.

Continuous manufacture of vinyl esters. G. O. MORRISON, Assr. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,710,181, 23.4.29. Appl., 8.11.26).—Acetylene is passed into an aliphatic carboxylic acid, e.g., acetic acid, in presence of a mercury orthophosphate. When the reaction slackens fresh catalyst is similarly prepared from mercuric oxide in, e.g., acetic

acid and molecular equivalents of orthophosphoric acid and acetylene are passed into it at, e.g., 78° until formation of vinyl acetate commences. The active catalyst mixture is then added to the reaction vessel and spent catalyst or sludge removed. R. BRIGHTMAN.

Manufacture of coloured polymerised styrene and its homologues. I. OSTROMISLENSKY, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,705,264, 12.3.29. Appl., 31.3.27).—Polymerised styrene is coloured with heavy metals in colloidal solution by forming a solution of the metallic salt (e.g., silver nitrate) in unpolymerised styrene containing 1–3% of polymerised styrene as protective colloid and polymerising at 140–180°.

R. BRIGHTMAN.

Reduction of aromatic nitro-compounds. I. G. FARBENIND. A.-G., and A. CARPMAEL (B.P. 314,573, 29.3.28. Addn. to B.P. 263,376; B., 1927, 156).—In the process of the prior patent a finely-divided oxide or hydroxide of a ter- or quadri-valent metal (aluminium, cerium, iron) is suspended in the reaction mixture.

C. HOLLINS.

Manufacture of phenol from chlorobenzene. I. G. FARBENIND. A.-G. (B.P. 288,308, 4.4.28. Ger., 6.4.27).—Chlorobenzene is boiled with aqueous alkali and the vapours of chlorobenzene and water are passed over active silica gel at 350°, condensed, and returned to the boiler until all chlorobenzene has been converted.

C. HOLLINS.

Manufacture of 4-amino-1-oxybenzene [*p*-aminophenol] and *N*-derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 293,792, 23.6.28. Ger., 12.7.27).—Alkyl ethers of *p*-aminophenol are dealkylated by heating with 60–75% sulphuric acid. *N*- β -Hydroxyethyl-*p*-phenetidine at 160° gives *N*- β -hydroxyethyl-*p*-aminophenol, m.p. 96–97°; *diN*-(β -hydroxyethyl)-*p*-aminophenol, m.p. 140°, *p*-aminophenol, and *N*-methyl-*p*-aminophenol are similarly obtained from their methyl or ethyl ethers.

C. HOLLINS.

Production of salts of carboxylic acids. G. H. BUCHANAN, G. BARSKY, and K. D. ASHLEY, Assrs. to AMER. CYANAMID CO. (U.S.P. 1,717,353, 18.6.29. Appl., 18.12.23).—A solution containing mainly calcium cyanamide, obtained by treating a mixture of calcium cyanamide and cyanide with water, is digested under pressure at above 60°. The liberated ammonia is removed and the aqueous formate separated from the precipitated oxalates.

F. G. CLARKE.

Manufacture of oxygenated organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,388, 24.2.28).—Liquid non-aromatic hydrocarbons, b.p. below 180°, with or without addition of a catalyst (manganese acetylacetonate), are passed in counter-current with a stream of air through a packed tube at about 145° and 20–50 atm. Hexane or benzene, b.p. 70°, gives fatty acids, C₂–C₈, and their esters.

C. HOLLINS.

Manufacture of condensation products from alicyclic ring-ketones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,421, 7.2.28).—Arylamines having free *para*-position are condensed with cyclohexanones in presence of acid at 140–220°. The diarylcyclohexane so formed is converted into a hydro-

generated diphenyl derivative by heating alone or with an acid agent (sulphuric acid, zinc chloride). *cyclohexanone* and aniline yield (a) 1:1-*di-(p-aminophenyl)cyclohexane*, m.p. 111°, b.p. 275—276°/12 mm., convertible into (b) 1-*p-aminophenylcyclohexane*, b.p. 170—171°/12 mm. Analogous compounds are obtained from *cyclohexanone* and *o-chloroaniline* (a, m.p. 126—128°; b, m.p. 32°, b.p. 196—198°/16 mm.), *o-toluidine* (a, m.p. 166°; b, b.p. 183—185°/15 mm.); *acetyl* derivative of b, m.p. 153°, *o-anisidine* (a, b.p. 289°/12 mm.; b, m.p. 50°, b.p. 185—187°/12 mm.), *ethyl-o-toluidine* (a, m.p. 118—120°; b, b.p. 180—185°/12 mm.), or *dimethylaniline* (a, m.p. 158—160°; b, b.p. 152—153°/3 mm.). 4-Methyl*cyclohexanone* and *o-toluidine* yield 1:1-*di-(4-amino-3-methylphenyl)-4-methylcyclohexane*, m.p. 145°, and 1-(4-amino-3-methylphenyl)-4-methyl*cyclohexane*, b.p. 194—196°/13 mm. (*acetyl* derivative, m.p. 163°). C. HOLLINS.

Manufacture of condensation products from hydroxybenzenes [phenols] and hydroaromatic ring ketones [cyclohexanones]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,825, 28.1.28).—Phenol or *o-cresol* is condensed with *cyclohexanone* or an alkyl*cyclohexanone* in presence of hydrogen halides in water or preferably in glacial acetic acid. 1":1"-*Di-(p-hydroxyphenyl)cyclohexane*, m.p. 186° (cf. Schmidlin and Lang, A., 1910, i, 836), and its 3:3'-*dimethyl* and 4"-*methyl* derivatives, m.p. 186° and 179°, respectively, are described. C. HOLLINS.

Manufacture of hydrogenated hydroxy-derivatives of the diphenyl series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 310,832, 28.1.28. Cf. B.P. 310,825; preceding).—1":1"-*Di-(p-hydroxyphenyl)cyclohexane* when heated, e.g., in a vacuum or with hydrogen chloride or zinc chloride at 200°, loses 1 mol. of phenol and yields 4'-hydroxy-2:3:4:5-tetrahydro-diphenyl [1-*p-hydroxyphenylcyclohexane*], m.p. about 124°, together with hydroxyhexahydrodiphenyl [*p-hydroxyphenylcyclohexane*], m.p. 128—129°, b.p. 180—210°, the latter being the main product when a catalyst is used. The condensation product from *o-cresol* and technical methyl*cyclohexanone* behaves similarly. C. HOLLINS.

Manufacture of aniline-2:5-disulphonic acid. I. G. FARBENIND. A.-G. (B.P. 285,488, 15.2.28. Ger., 18.2.27).—Metanilic acid is sulphonated with oleum at 160° and the resulting mixture of di- and tri-sulphonic acids is boiled with dilute sulphuric acid to yield pure aniline-2:5-disulphonic acid. C. HOLLINS.

Manufacture of solid stable diazo compounds. KALLE & Co. A.-G. (B.P. 294,248, 16.7.28. Ger., 21.7.27).—Stable double salts are precipitated by addition of cadmium chloride, with or without sodium chloride, to diazo solutions, e.g., diazotised *p*-aminodimethylaniline or its *o*-carboxylic acid. C. HOLLINS.

Manufacture of organic [quinoline] bases [from arylamines and acetylene]. I. G. FARBENIND. A.-G. (B.P. 296,423, 9.3.28. Ger., 2.9.27. Addn. to B.P. 283,163; B., 1929, 275).—Quinaldines are obtained by passing an arylamine vapour with acetylene at 300—350° over the catalysts of the prior patent, indoles being formed (in small amount) only when secondary

amines are used. Aniline gives quinaldine; *o*-toluidine, 2:8-dimethylquinoline; ethylaniline, 1-ethylquinaldine (*picrate*, m.p. 152°), quinaldine, and 1-ethylindole; and *o*-chloroaniline, 8-chloroquinaldine. C. HOLLINS.

Manufacture of condensation products of the benzodiazine [quinazoline] series. I. G. FARBENIND. A.-G. (B.P. 287,179, 16.3.28. Ger., 18.3.27).—2- or 4-Halogeno- or 2:4-dihalogeno-quinazolines are condensed with phenols or aromatic hydrocarbons in presence of aluminium chloride, or with halogenated hydrocarbons by means of sodium, or with a Grignard reagent. 2:4-*Di-(4'-hydroxy- α -naphthyl)quinazoline*, decomp. 240—250° without melting, and 4-(4'-hydroxy- α -naphthyl)quinazoline, m.p. 230—232°, are obtained from 2:4-dichloro- and 4-chloro-quinazoline, respectively, with α -naphthol and aluminium chloride. 4-Hydroxy-2-phenylquinazoline, m.p. 235°, from 2-chloro-4-hydroxyquinazoline, and 2-phenylquinazoline, m.p. 100—101°, b.p. 305°, are similarly prepared. 2-Chloro-6-nitro-4-hydroxyquinazoline, naphthalene, and aluminium chloride yield 6-nitro-4-hydroxy-2- α -naphthylquinazoline, reducible to an amine which when diazotised and coupled with G-acid gives a bluish-red dye. From 2-chloro-4-phenylquinazoline, chlorobenzene, and sodium, 2:4-diphenylquinazoline, m.p. 120—121°, is prepared; this is also obtainable from 2:4-dichloroquinazoline and magnesium phenyl bromide. 2-Phenyl-4-methylquinazoline results from the successive action of magnesium methyl and phenyl halides on 2:4-dichloroquinazoline. C. HOLLINS.

Manufacture of cyclic compounds containing aldehydic groups. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,208, 6.2.28).—Cyclic compounds having suitably reactive $\cdot\text{CH}\cdot$ groups, e.g., *m*-xylene, anthracene, ethylcarbazole, pyridine, quinoline, etc. are condensed at 15—80° in presence of phosphorus chlorides, sulphuryl chloride, thionyl chloride, or aluminium chloride, with formamide or its *N*-alkyl or -aryl derivatives. In the case of keto-enolic compounds when the formyl derivative of a secondary arylamine is used the enolic hydroxyl is at the same time replaced by chlorine. Examples are: anisaldehyde from anisole and *N*-methylformanilide; 2-ethoxy- α -naphthaldehyde; 4-methoxy-*ar*-tetrahydro- α -naphthaldehyde, m.p. 59—60° (*aldazine*, m.p. 210—211°); the 2-methoxy-compound, m.p. 52—53° (*aldazine*, m.p. 237—238°), and 2-ethoxy-compound, m.p. 62—63° (*aldazine*, m.p. 215°); 9-*aldehydoanthracene*, m.p. 104°; 2:4-dimethylbenzaldehyde; 1(or 4)-chloro-9-*aldehydoanthracene*, m.p. 97—104°; 2-hydroxy- α -naphthaldehyde, m.p. 81—82° (oxime, m.p. 157°); the 4-hydroxy-compound; 2:7- and 4:8-*dihydroxy- α -naphthaldehydes*; 2-hydroxy-1-*aldehydo-3-naphthoic acid* and its *amide* (*aldazines*, m.p. above 300°); 4-hydroxy-2:5-dimethylbenzaldehyde; β -resorcyaldehyde; vanillin; 6-chloro-2-*aldehydo-4-methylthioindoxyl* (*aldazine*, m.p. 277—278°); 6-ethoxy-2-*aldehydothioindoxyl* (*aldazine*, m.p. 248°) from 6-ethoxythioindoxyl and formamide, or the 3-chloro-compound, m.p. 166°, when methylformanilide is used; 2(?)-*aldehydo-N-ethylcarb-azole*, m.p. 87°; 6-*aldehydonaphthastyril*, m.p. 223°; 3:6-dichloro-2-*aldehydo-4-methylthioindoxyl*; 9-chloro-10-*aldehydoanthracene*, m.p. 216°, from anthrone; the 2:9-dichloro- (m.p. 174°) and 1:5:9-trichloro- (m.p. 197°)

derivatives; 9-chloro-3:4-dimethoxy-10-aldehydoanthracene, m.p. 172°; 9-chloro-2:6-dimethoxy-10-aldehydoanthracene, m.p. 233°. C. HOLLINS.

Manufacture of 2:3-aminonaphthoic acid. I. G. FARBENIND. A.-G. (B.P. 284,998, 7.2.28. Ger., 7.2.27).—2:3-Hydroxynaphthoic acid (1 pt.) is added to molten zinc chloride-ammonia (2 pts.), or zinc chloride-ammonia is added to the molten acid, and the mixture is heated at 240–250° for 10–12 hrs. (Cf. Fierz and Tobler, A., 1922, i, 869.) C. HOLLINS.

Manufacture of compounds of the anthracene series [nitrotetrahydroanthraquinones]. I. G. FARBENIND. A.-G. (B.P. 295,943, 20.8.28. Ger., 18.8.27).—Tetrahydroanthraquinones are nitrated, chiefly in the *ar*- α -position, by nitric and sulphuric acids at 40–50°. The preparation of 5-nitro-1:2:3:4-tetrahydroanthraquinone, m.p. 192°, the 6-isomeride, m.p. 133–134°, 5-nitro-6-methyl-1:2:3:4-tetrahydroanthraquinone, m.p. 130°, and 5-nitro-8-acetamido-1:2:3:4-tetrahydroanthraquinone, decomp. 185°, is described. C. HOLLINS.

Manufacture of basic derivatives of anthraquinone [amination and alkylamination of hydroxylated anthraquinones]. BRIT. CELANESE, LTD., J. HALL, H. C. OLPIN, G. REEVES, and E. W. KIRK (B.P. 310,784, 31.1.28).—Reduced hydroxylated anthraquinones, *i.e.*, the leuco-compounds or the anthrones, are treated with ammonia or alkylamines in presence of caustic alkali. The products are re-oxidised, *e.g.*, with alkali and perborate. C. HOLLINS.

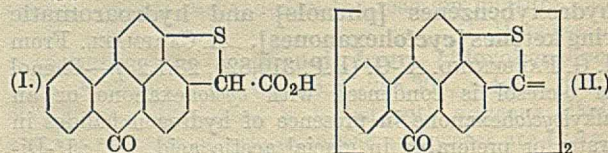
Preparation of *oo'*-dicarboxydiphenyldiaminoanthraquinones. W. L. RINTELMANN and R. J. GOODRICH (B.P. 312,678, 31.1.28).—A dihalogenoanthraquinone (especially 1:5) is heated with potassium anthranilate and basic copper acetate in methyl or ethyl alcohol at 135–140° and under 150 lb. Decarboxylation is thus avoided. C. HOLLINS.

Manufacture of benzanthrone derivatives. I. G. FARBENIND. A.-G. (B.P. 286,685, 4.2.28. Ger., 9.3.27. Addn. to B.P. 268,830; B., 1928, 666).—In the process of examples 13, 15, and 16 of the prior patent an unsaturated carboxylic acid (or ester) of the type CHR:CR':CO₂H, in which R = hydrogen, alkyl, or aryl, and R' = hydrogen, aryl, or carboxylic ester group, may be employed. The intermediate additive compound, X·CHR·CHR'·CO₂H (where X = *ms*-anthronyl), is cyclised by acid reagents to give the desired benzanthrone; if R = Ph, oleum or chlorosulphonic acid, diluted with glacial acetic acid to avoid sulphonation, must be used. The resulting 3-hydroxybenzanthrone is converted by aqueous ammonia and zinc chloride into 3-aminobenzantrones, from which the amino-group may be eliminated in the usual way by diazotisation and boiling with alcohol. The following compounds are described: β -*ms*-anthronylpropionic acid, m.p. 181°, and 3-hydroxybenzanthrone, from anthrone and acrylic or β -chloropropionic acid; β -*ms*-anthronyl-*n*-butyric acid, m.p. 160°, and 3-hydroxyl-1-methylbenzanthrone, m.p. 287°, from anthrone and crotonic acid; β -phenyl- β -*ms*-anthronylpropionic acid, m.p. 197°, and 3-hydroxy-1-phenylbenzanthrone, m.p. 320°,

from anthrone and cinnamic acid or from methyl (phenyl-*ms*-anthronylmethyl)malonate. C. HOLLINS.

Manufacture of benzanthrone-*peri*-[3:4]-dicarboxylic acid or its derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,985, 16.4.28).—4-Benzoyl-1:8-naphthalic anhydride is heated with aluminium chloride at 190–210° to give benzanthrone-3:4-dicarboxylic acid (anhydride, m.p. above 300°). 4-*p*-Chlorobenzoyl-1:8-naphthalic anhydride and the derivatives obtained by condensing these compounds with ammonia, amines, or *o*-diamines, behave similarly. C. HOLLINS.

Manufacture of condensation products [thiazoles] of the benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,047, 16.3.28).—3-Benzanthroneylthioglycollic acid fused with alkali gives the compound (I), which is readily decarboxylated and oxidised to a bluish-green vat dye (II). In place of the thioglycollic acid its ester, amide, or nitrile may be



used. Similar products are obtained from substituted derivatives, *e.g.*, from the 9-chloro-3-benzanthroneylthioglycollic acid, m.p. 212–214°, obtained from 9-chloro-3-bromobenzanthrone, m.p. 258–259°. The alkaline condensing agent may be potassium hydroxide (alone or with sodium hydroxide) with or without water, alcohol, or pyridine. C. HOLLINS.

Manufacture of chemical compounds [di(halogenoacylated)diphenyl ethers]. O. VON SCHICKHE, ASST. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,717,424, 1.26.29. Appl., 28.2.28. Ger., 9.3.27).—See B.P. 286,688; B., 1929, 673.

IV.—DYESTUFFS.

PATENTS.

Manufacture of mordant [azo] dyes. I. G. FARBENIND. A.-G. (B.P. 285,097, 11.2.28. Ger., 11.2.27).—Chrome-printing brown azo dyes are obtained by coupling a resorcinol with 2 mols. of a diazotised aminosalicic acid. Examples are: 5-aminosalicylic acid (2 mols.) \rightarrow resorcinol, α - or β -resorecylic acid; 5-aminosalicylic acid \rightarrow cresidine or α -naphthylamine \rightarrow resorcinol \leftarrow 5-aminosalicylic acid; [5-aminosalicylic acid \rightarrow cresidine]₂ \rightarrow resorcinol. C. HOLLINS.

Manufacture of new basic azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 313,293, 23.5.28).—Azo dyes of the type dehydrothio-*p*-toluidine or -*p*-xylydine \rightarrow unsulphonated coupling component are alkylated with a toluene-*p*-sulphonic ester or an alkyl sulphate; alternatively, alkylation may precede coupling, the amino-group of the thiazole being first protected by acylation and the acyl group removed after alkylation. As coupling components the examples mention β -naphthol (yellowish-red on mordanted cotton), phenylmethylpyrazolone (yellowish-red), resor-

cinol (orange-brown), 2:7-dihydroxynaphthalene (yellowish-scarlet), α -naphthylamine (reddish-violet), ethyl- β -naphthylamine (violet), cresidine (claret-violet; \rightarrow resorcinol or *m*-tolylendiamine, brown), acetoacetanilide (greenish-yellow). 6-Amino-2-methylbenzthiazole is coupled with phenylmethylpyrazolone and methylated (reddish-yellow). C. HOLLINS.

Manufacture of [direct dis]azo dyes [for cotton and viscose silk]. I. G. FARBENIND. A.-G. (B.P. 285,812 and 286,226, [A] 15.2.28, [B] 27.2.28. Ger., [A] 21.2.27, [B] 28.2.27).—(A) Dyes possessing good solubility and good cotton affinity are obtained by coupling an *oo'*-dicarboxytetrazo compound with 2 mols. of an acetoacetic sulphonylamide; alternatively, a *p*-nitro-*o*-carboxydiazo compound is coupled with the sulphonated arylamide, the nitro-group reduced, and the product treated with carbonyl chloride, thiocarbonyl chloride, or carbon disulphide. Examples are: 4:4'-diaminodiphenylcarbamide-3:3'-dicarboxylic acid with acetoacetic *o*-chlorosulphonylamide (yellow on viscose; greenish-yellow on cotton, yellow when coppered), or with disulphonated acetoacetic α -naphthylamide (orange-yellow on viscose; yellow on cotton, brownish-yellow when coppered); 5-nitroanthranilic acid with acetoacetic sulphonyl-anisidide, reduced and phosgenated (golden-yellow on viscose or cotton, pure yellow when coppered). (B) A tetrazotised *pp'*-diaminodiphenylcarbamide is coupled with a sulphonated acetoacetic arylamide, or with an acetoacetic arylamide and subsequently sulphonated; alternatively, the corresponding sulphonated monoazo dye is phosgenated. Examples are: 4:4'-diaminodiphenylcarbamide-3:3'-disulphonic acid \rightarrow 2 mols. of acetoacetic sulphonyl-anisidide (yellow on cotton, golden-yellow on viscose; deep yellow lake on alumina); *p*-nitroaniline-*o*-sulphonic acid \rightarrow acetoacetic *o*-chloroanilide, sulphonated, reduced, and phosgenated (yellow on cotton or viscose or cuprammonium silk); 5-nitro-*o*-anisidine-4-sulphonic acid \rightarrow acetoacetic sulphonyl-anisidide, reduced and phosgenated (yellow). C. HOLLINS.

Thiazoles of benzanthrone series (B.P. 311,047).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Oxidation of alkali-cellulose by ageing and its importance in the manufacture of artificial silk. G. KITA and I. SAKURADA [with, in part, Y. NAKAMURA, K. SAKURADA, J. ONAHARA, and R. TOMIHISA] (Cellulose-chem., 1929, 10, 113—120).—Different effects are produced by ageing alkali-cellulose in the squeezed-out and steeped conditions. Oxidation through the action of the air occurs in both cases, but in the former the oxidation products remain in the mass, and are used in the subsequent preparation of viscose, whilst in the latter they are removed by dissolution in the sodium hydroxide liquor. Comparison is made between the properties of steeped and of pressed alkali-cellulose; the effect on the copper number of ageing for different times, the effects of ageing in a closed atmosphere of air and in an atmosphere of hydrogen, and of time of steeping in the production of the alkali-cellulose on the spinning properties of the fibres are discussed, and

differences in spinning properties, viscosity, and surface tension of viscoses obtained from alkali-celluloses prepared in different ways are described. An increase of copper number with increasing time of ageing results not only for material aged in closed or open atmospheres of air, but also for that aged in an atmosphere of hydrogen. There is no great difference between the properties of viscoses prepared from pressed and steeped alkali-celluloses. The longer is the time, or the higher the temperature of ageing in a closed atmosphere of air, the less viscous is the viscose, the thicker are the fibres, and the longer is the time of ripening necessary to give a spinnable viscose. Ageing in an atmosphere of hydrogen is a slower process than that in air; the viscose obtained is more viscous, more easily spun, and gives stronger fibres. Under certain conditions long steeping followed by a short period of ageing gives fibres as strong as those obtained by ageing in an atmosphere of hydrogen, whilst good fibres can also be obtained by ageing the alkali-cellulose for a short time only in a closed atmosphere of air.

B. P. RIDGE.

Manufacture and properties of regenerated cellulose films. W. L. HYDEN (Ind. Eng. Chem., 1929, 21, 405—410).—Transparent cellulose sheets and films, e.g., Cellophane, are made by the viscose process, a final treatment with glycerin being given to impart softness and pliability. Plain transparent Cellophane permits the rapid penetration of water vapour and of water-soluble gases, e.g., ammonia, carbon dioxide, but those sparingly soluble in water, such as hydrogen, diffuse through it slowly. A moisture-proof material has recently been developed, and is particularly useful as a wrapper for food products. Of the other properties of Cellophane described, the most interesting is its relatively high transparency to ultra-violet light, which is not decreased by long exposure. F. R. ENNOS.

Influence of atmospheric humidity on paper. L. E. WALTER (Papier-Fabr., 1929, 27, 369—371).—Absorption and desorption of moisture take place more rapidly in the case of strips of paper cut transversely than in that of lengthwise strips, although the same final values are reached in both cases. At low R.H. the rate of absorption of water by paper is slower than, at average values approximately proportional to, and at high humidities more rapid than, that of increase of humidity. The extension produced is approximately proportional to the quantity of water absorbed. Absorption and desorption at ordinary room temperatures are completely reversible processes; extension is irreversible. B. P. RIDGE.

Influence of drying on the strength of paper. E. RIETH (Papier-Fabr., 1929, 27, 385—387).—Samples of a uniform paper from unbleached sulphite-cellulose were dried under different conditions and their moisture content, tearing length, extension, etc. were measured. The same material varies in tearing length between wide limits according to the conditions of drying. Not only is the manner of drying (e.g., in the air, on the cylinder, etc.) important, but the time of contact with the cylinder has a considerable effect; the longer is this time the greater is the tearing length of the paper

and the lower are its moisture regain, extensibility, and bursting test number. Strength is dependent on the degree of swelling of the fibres, and this swelling is influenced both by the duration of drying and the nature of the grinding process.

B. P. RIDGE.

PATENTS.

Manufacture of [chloroform-soluble] acetylcellulose. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 291,001, 7.3.28. Ger., 23.5.27).—Chloroform-soluble cellulose acetates of satisfactory mechanical properties are obtained with zinc chloride as catalyst and a reaction time of 3–6 hrs. if the esterification is conducted in the early stages at 55° and is then completed at about 40°.

D. J. NORMAN.

Manufacture of cellulose esters. H. DREYFUS (B.P. 312,095—6, 17.12.27).—(A) Cellulose that has been treated with organic acids, *e.g.*, formic or acetic acid, is esterified by the action of a fatty acid anhydride in the presence of a ferric halide, particularly ferric chloride, preferably 10–30% on the weight of cellulose. The reaction is best conducted below or not substantially above the ordinary temperature. (B) A stannic halide, in the presence or absence of hydrogen halide, and optionally in mixtures with halides of iron, manganese, copper, nickel, or cobalt, is used as the condensing agent in the esterification of cellulose. Suitable quantities are 7–25% of stannic chloride or 5–15% each of stannic chloride and hydrochloric acid on the weight of the cellulose, and the parent material may, if desired, be pretreated with the lower fatty acids to increase its reactivity.

D. J. NORMAN.

Production of cellulose esters and cellulose ester products therefrom. H. DREYFUS (B.P. 312,098, 14.1. and 7.6.28).—Cellulosic material is more easily esterified if it is pretreated with 2–50%, preferably 5–10%, of hydrogen halide, alone or in conjunction with the lower fatty acids and/or ferric, stannic, manganese, cobalt, nickel, or copper halides. The pretreating agent may be used in gaseous form or in aqueous solution, and the reaction, which preferably takes place at the ordinary temperature, may proceed for 1–24 hrs. depending on the nature of the cellulosic material, the concentration of the pretreating agent, the temperature, and the degree of reactivity desired in the cellulose.

D. J. NORMAN.

Manufacture of mixed esters of cellulose and of conversion products thereof. I. G. FARBENIND. A.-G. (B.P. 283,595, 14.1.28. Ger., 15.1.27).—Mixed nitric acid-fatty acid esters of cellulose are made (1) by esterifying cellulose by means of an organic acid anhydride and adding nitric acid, preferably mixed with the fatty acid during the process when esterification has reached a more or less advanced stage; (2) by pretreating cellulose in a suitable diluent, *e.g.*, an organic acid, with a mixture of nitric acid and a catalyst (sulphuric acid) and finally esterifying by the addition of the acid anhydride.

F. R. ENNOS.

Treatment of artificial silk. Soc. D. O. (B.P. 305,622, 25.9.28. Fr., 8.2.28).—In order to increase its tenacity and elasticity, artificial silk of any type is immersed in an aqueous bath heated at 40–50° and containing

ordinary white soap, pure olive oil, sulphuric acid (*d* 1.83), and caustic soda (*d* 1.33).

F. R. ENNOS.

Celluloid compound. R. ROLAND, ASSR. to ROLAND FIREPROOF CELLULOID CORP. (U.S.P. 1,713,482, 14.5.29. Appl., 2.3.28).—A mixture of 25 pts. of acetylcellulose, 25 pts. of alcohol, 10 pts. of chlorobenzene, 10 pts. of tetrachloroethane, 10 pts. of benzylbenzoate, 10 pts. of triacetin, and 10 pts. of dichlorohydrin is heated at 75° until the acetylcellulose dissolves and the mixture becomes viscous. On evaporation a product capable of forming flexible, transparent, and non-inflammable sheets or films is obtained.

F. R. ENNOS.

Treatment of paper pulp. R. G. SMITH (U.S.P. 1,715,310, 28.5.29. Appl., 11.4.25).—Sodium sulphate and sulphuric acid are added to the pulp after digestion and before it is beaten. The solvent action is increased by the addition of sulphurous acid, and advantageously the three reagents are formed *in situ* by adding sodium sulphate and treating with excess sulphuric acid.

F. G. CLARKE.

Preparation of coconut fibres for spinning and weaving. F. L. C. BARBOUR. From B. G. H. VAN DER JAGT (B.P. 313,016, 5.12.27).

Drying of textile fibres or material. H. HAAS (B.P. 301,057, 22.8.28. Ger., 25.11.27).

Seaming compound fabrics containing rubber. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 292,170, 16.6.28. Ger., 16.6.27).

Spinning cans or boxes for artificial silk. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 297,468, 20.9.28. Holl., 22.9.27).

Production of artificial silk by the stretch-spinning process. J. P. BEMBERG A.-G. (B.P. 300,953, 31.8.28. Ger., 21.11.27).

Winding of artificial silk. S. DUNLOP. From SOIE DE CHATILLON (B.P. 313,425, 5.3.28).

Apparatus for drying cellulose films. FELDMÜHLE, PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,296, 11.5.28. Ger., 12.5.27).

Apparatus for continuous drying of shrinking films, particularly for drying of viscose films. H. HAMPEL (B.P. 313,829, 13.10.28).

[Roller] apparatus for treating with liquids artificial films prepared from viscose. FELDMÜHLE, PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,232, 10.5.28. Ger., 10.5.27).

Papermaking [machines]. A. KOLITSCH (B.P. 298,653, 13.10.28. Can., 13.10.27).

[Roller machine for] colouring, sizing, impregnating, or otherwise treating paper [on one surface]. I. G. FARBENIND. A.-G. (B.P. 293,000, 22.6.28. Ger., 29.6.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cause of uneven dyeing of cotton yarn. H. J. KIST and C. W. ZAHN (Textilber., 1929, 10, 471–472).—Uneven results obtained in the dyeing (large-scale) of skeins of Egyptian and American cotton were traced to a previous uneven exposure of the skeins to frost. A

normal air-dry skein of cotton after exposure to -10° while in a sealed waterproof box had an increased affinity for direct dyes. When the same skein was again similarly frozen together with another normal skein, both gained an equal affinity for dyes. A. J. HALL.

Discharges [using resorcinol] on cellulose acetate silk. SCHEURER, LAUTH & CIE., and L. DISERENS (Sealed Note No. 2662, 23.6.26. Bull. Soc. Ind. Mulhouse, 1929, 95, 349—352). Report by A. WOLFF (*Ibid.*, 352—354).—The difficulty of obtaining clear white discharges on cellulose acetate silk owing to poor penetration of the silk by the sodium hyposulphite used in such discharge processes is largely overcome by adding resorcinol to the discharge paste. Although resorcinol is a solvent for cellulose acetate its action in the discharge paste is not due to this property alone since other solvents such as phenol and acetone are not effective. When cellulose acetate is heated with a 50% solution of resorcinol, addition of ferric chloride fails to give the characteristic violet coloration, thus suggesting that a reaction between resorcinol and cellulose acetate occurs which is similar to that between acetic anhydride and phenol. In assisting discharges resorcinol is effective with a larger range of dyes than a thiocyanate (cf. B.P. 262,254; B., 1927, 71) under similar circumstances, and yields purer discharges. Wolff reports favourably on the use of resorcinol, but finds its utility restricted as in the case of a thiocyanate. If the quantity of resorcinol used exceeds 20 g. per kg. of discharge paste the silk is liable to deterioration. Attention is drawn to a similar process described by Schneevogt (B., 1926, 662). A. J. HALL.

Detection of indigosols on the fibre. J. C. LIVINGSTON (Bull. Soc. Ind. Mulhouse, 1929, 95, 230).—It is possible to determine whether cotton fabric coloured with an indigoid dye has been dyed by the usual vat method or by means of an indigosol, by stripping it with an alcoholic solution of a hyposulphite, then boiling it in a 0.025% solution of methylene-blue, washing, and drying; fabric dyed with an indigosol has a definite affinity for the basic dye whereas fabric dyed in the vat has not. This affinity for methylene-blue is independent of the method of developing the indigosol, and is not due to the presence in the fabric of oxycellulose; it is suggested that the sulphuric acid formed by hydrolysis of the indigosol simultaneously combines with the cotton, thereby conferring on it an affinity for the basic dye. A. J. HALL.

[Production of] crêpe effects on delaines. C. FAVRE (Sealed Note No. 2284, 8.10.13. Bull. Soc. Ind. Mulhouse, 1929, 95, 359—360). Report by A. WOLFF (*Ibid.*, 360).—In treating a large number of pieces by the method previously described (B., 1926, 123) it is difficult to obtain evenness of crêpe. It is found preferable to treat each piece separately in a tub, allowing 20 min. per piece. Those portions of the fabric previously printed with a solution of sodium silicate (d 1.384) resist the crêping action of the sulphuric acid used. Wolff confirms the resist action of sodium silicate, and states that whilst solutions of albumin and sucrose are equally effective, that of a gum containing barium chloride is not. A. J. HALL.

PATENTS.

Dyeing and printing [of mixed textiles]. A. THAUSS and A. GUENTHER, Assrs. to GRASELLI DYE-STUFF CORP. (U.S.P. 1,711,002, 30.4.29. Appl., 10.11.27. Ger., 3.12.26).—In the dyeing of mixed fabrics, the wool or silk is first dyed and the direct cotton dye is then applied from a bath containing sulphonated colophony, which resists the wool or silk against the direct dye. Tin salts may be added to assist the result.

C. HOLLINS.

Ageing or reducing printed or dyed fabrics. A. E. WHITE. From DELTEX Co. (B.P. 312,423, 13.3.28).—In an ageing machine for fabrics printed or dyed with vat dyes (especially in the case of discharge printing) the steam is kept wet by supplying water in regulated quantity at points in the immediate vicinity of the runs of fabric. A machine is described.

C. HOLLINS.

Solution for dyeing purposes. A. GUENTHER, A. THAUSS, and G. MAUTHE, Assrs. to GRASELLI DYE-STUFF CORP. (U.S.P. 1,709,976, 23.4.29. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 307,777; B., 1929, 639.

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

Organic inhibitors in the acid corrosion of iron. J. C. WARNER (Amer. Electrochem. Soc., May, 1929. Advance copy. 10 pp.).—The rate of dissolution of iron in an aqueous solution may be diminished not only by substances which produce passivity at anodic areas, but also by substances which increase the hydrogen overvoltage at cathodic areas. This conclusion from the local-element theory is supported by experiments on the effect of gelatin, petroleum bases, and coal-tar bases on the rate of dissolution of a low-carbon iron in dilute sulphuric acid and on the hydrogen overvoltage at the metal surface in this solution. But aniline and quinoline, which definitely retard dissolution, actually lower the hydrogen overvoltage measured at 10 milliamp. per cm^2 . Overvoltage measurements at lower current densities might remove this anomaly.

H. J. T. ELLINGHAM.

Action of carbonic acid under high pressures on iron. E. MÜLLER and H. HENECKA (Z. anorg. Chem., 1929, 181, 159—171).—Under high pressures of carbon dioxide, *e.g.*, 20—50 atm., carbonic acid reacts on aqueous suspensions of metallic iron to produce soluble ferrous hydrogen carbonate. As the concentration of the hydrogen carbonate ion increases the hydrogen-ion concentration of the medium decreases and the rate of dissolution of iron approaches zero. At temperatures above 30° partial decomposition of ferrous hydrogen carbonate into insoluble ferrous carbonate occurs. The dissolution-time curves for these temperatures hence show maximum values. Above 30° the eventual quantity of soluble iron decreases with increasing temperature. The eventual amount of soluble iron increases with increasing pressure of carbon dioxide, but is almost independent of pressure for pressures greater than 30 atm.

J. S. CARTER.

Corrosion of steel in dilute acids. H. ENDO and K. NAKAWAGA (J. Study Met., 1928, 5, 301—313).

Addition of cobalt, nickel, molybdenum, chromium, titanium, vanadium, phosphorus, or much silicon to steel (containing 0.3–0.6% C) increases the resistance to 5% sulphuric, hydrochloric, or nitric acid, whilst the resistance is decreased by carbon, manganese, or sulphur. The relation between the annealing temperature after quenching and the loss of weight in 1% sulphuric acid was studied, the results being explained in terms of Honda's theory of quenching. CHEMICAL ABSTRACTS.

Tensile tests [on alloy steels] at elevated temperatures. L. GUILLET, J. GALIBOURG, and M. SAMSOEN (Compt. rend., 1929, 188, 1328–1330).—Earlier experiments (B., 1929, 601) have been extended to include tensile tests on nickel and nickel-chromium steels at 450°, and determinations of their elasticity and Brinell hardness numbers. The values obtained depend on the previous thermal treatment of the metal; e.g., oil-tempering produces a higher instantaneous extension limit than when air is the tempering medium.

J. GRANT.

Tungsten steel. W. ZIELER (Arch. Eisenhüttenw., 1929–30, 3, 61–78; Stahl u. Eisen, 1929, 49, 1083–1084).—The system iron-carbon-tungsten at the iron corner of the ternary diagram comprises the following constituents: cementite, iron tungstide, Fe₂W, two double carbides I and II distinguished from one another by their stability on annealing and their behaviour on etching with sodium picrate, and tungsten carbide. Normal cooling of alloys with 10–20% W favours the formation of a double carbide, but prolonged annealing above a critical temperature causes the separation of WC; this decomposition is retarded by quite small amounts of chromium. The metastable double carbide is coloured brown by sodium picrate and is strongly etched, whereas the stable double carbide is only slightly attacked, but is coloured dark brown. In steels with less than 0.3% C the A3 transformation point is progressively raised by addition of tungsten and its intensity decreased. In steels with 0.7–1.4% C, on the other hand, the Ac1 and Ac3 points are fairly constant with all proportions of tungsten, but the Ar1 and Ar3 points are lowered. The hardness of steels with 0.3% C, after quenching in oil or water, is at a maximum with 5% W; with 0.7% C steels after oil-quenching that with 1% W has the maximum hardness. A high silicon content tends to reduce the hardness. Steels with more than 1.1% C have maximum hardness with 20% W after quenching from about 1000°; the alloy with 1.4% C and 20% W, after this treatment, has a hardness of 750, which is increased still further by the presence of 0.6–0.7% Cr. A. R. POWELL.

Determination of gases in metals, especially oxygen in iron and steel, by the hot-extraction method. H. DIERGARTEN (Arch. Eisenhüttenw., 1928–9, 2, 813–828; Stahl u. Eisen, 1929, 49, 1053–1054).—A furnace is described and illustrated for the determination of gases in iron and steel by heating *in vacuo* at 1500–1800°. It comprises a water-cooled cylindrical vessel containing a graphite heating coil enclosing a graphite crucible and provided with a water-cooled cover having openings for charging the crucible, for exhausting the furnace, and for removing the gases

evolved. The oxygen is recovered as carbon monoxide, but the results are low when more than 0.7% Mn is present owing to absorption of the gas by the volatilised manganese. A. R. POWELL.

Rapid determination of vanadium in alloyed and unalloyed steels. K. RIES (Chem.-Ztg., 1929, 53, 527).—The steel (1–2 g.) is dissolved in dilute sulphuric acid, 10 c.c. of phosphoric acid being added if tungsten is present, the iron oxidised with nitric acid, and the solution evaporated until fumes of sulphuric acid begin to be evolved. After cooling and dilution, a few c.c. of 3% ferrous sulphate solution are added to reduce the vanadic acid to vanadyl sulphate, the excess of iron is oxidised with ammonium persulphate, and the cold solution titrated with permanganate.

A. R. POWELL.

System nickel-iron. G. J. SIZOO and C. ZWIKKER (Z. Metallk., 1929, 21, 125–126).—Single-crystal rods of iron-nickel alloys have been prepared by drawing the molten alloy by suction up a quartz tube at 50° above the m.p. of the alloy, and subsequently removing the polycrystalline skin by etching with nitric acid. The iron used contained less than 0.1%, and the nickel less than 0.4%, of impurities, and both were free from manganese. Using these alloys wires of 0.25 mm. diameter were prepared, and their sp. electrical conductivity and the temperature coefficient were determined. The conductivity curve has two rather ill-defined maxima at compositions corresponding with Fe₃Ni and FeNi₂, and two minima at Fe₂Ni and about 90% Ni. The temperature coefficient curve has maxima and minima at the same points, but in all cases they are very well defined. A. R. POWELL.

Influence of aluminium, lead, iron, and zinc in brass. First report of a Committee of investigation of the effect of impurities in brass (Z. Metallk., 1929, 21, 152–159).—Addition of up to 4% Pb to brass with 57, 60, or 65% Cu has little effect on the yield point or breaking strain, but it decreases the elongation of the 57% Cu alloy and the reduction in area, bending strength, and torsion of all the alloys; on the other hand addition of 1.8% Pb to the 57% Cu alloy renders it much more readily workable on the lathe. Iron up to 2% in 60 and 62% Cu brass tends to increase the strength but reduce the ductility, although the results obtained are usually very irregular. Aluminium increases the tensile strength of 68 and 62% Cu brass, but reduces the ductility and the Erichsen value. Substitution of part of the zinc in 82% Cu brass by the equivalent quantity of tin improves all the tensile properties, especially the yield point. A. R. POWELL.

Influence of sulphur dioxide on bronze and copper. P. RÖNTGEN and G. SCHWIETZKE (Z. Metallk., 1929, 21, 117–120).—When bronze with 10% Sn is melted in an atmosphere containing sulphur dioxide and then allowed to cool no porosity is observed, whereas under the same conditions copper becomes very porous. Prolonged heating of bronze in the presence of sulphur dioxide results in the formation of a sulphide layer on top of the metal, but this does not occur with copper. These results indicate that the gas is not really soluble

in the molten metal, but reacts with it forming sulphide and oxide; in the case of copper the reverse reaction proceeds with sufficient velocity during cooling of the metal to cause appreciable amounts of sulphur dioxide to be re-formed, thus giving rise to porosity. On the other hand, with a bronze the primary reaction is $\text{SO}_2 + 2\text{Cu} + \text{Sn} = \text{Cu}_2\text{S} + \text{SnO}_2$, and this reaction is irreversible at lower temperatures. Aluminium bronzes, brass, and nickel silver should behave similarly to tin bronze. A. R. POWELL.

[Solid] solubility of copper in silver. M. HANSEN (Z. Metallk., 1929, 21, 181—184).—The curve showing the boundary of the α -solid solution field in silver-copper alloys has been determined by microscopical examination of alloys after prolonged annealing at various temperatures and quenching. Up to 250° the solubility is 1.7% Cu, at 400° 2.2%, at 500° 3.2%, at 600° 5%, at 700° 7.2%, and at the eutectic temperature, 779°, 9.0%. The solidus of the α -phase is a straight line joining the m.p. of silver to the eutectic line at 9% Cu. These results are in agreement with Norbury's observations on the age-hardening of quenched silver-copper alloys (cf. B., 1928, 267). A. R. POWELL.

Rapid method of analysis of bearing-metal alloys. J. F. ILTSCHENKO and R. M. STACHORSKI (Ukraine Chem. J. [Tech.], 1928, 3, 237—240).—Analysis of alloys consisting of antimony, tin, lead, and copper is described. Antimony after conversion into the sulphate is determined volumetrically by titration with a solution of potassium permanganate, and the presence of salts of tin, lead, bismuth, zinc, and of small quantities of copper does not affect the result. Tin is determined iodometrically after conversion into the sulphate and separation from antimony and copper. Antimony, copper, arsenic, bismuth, iron, nickel, and zinc may be present without affecting the results. Lead is determined gravimetrically, and copper colorimetrically as copper ammonium salt. A. FREIMAN.

Determination of tin and antimony in bearing-metal alloys. A. P. AFANASIEV (Ukraine Chem. J. [Tech.], 1929, 4, 11—17).—On heating a sample of the alloy with nitric acid, then diluting with water, filtering, and igniting the residue, a mixture of stannous and antimony oxides is produced, in which the antimony is determined as sulphate by titrating its solution with standard potassium permanganate. The tin is found by difference. Lead present as impurity is determined as lead sulphate and copper as copper oxide. The procedure to be adopted when an excess of either alloy component is present is also fully discussed. A. FREIMAN.

Determination of bismuth [in ores]. G. J. HOUGH (Chemist-Analyst, 1929, 18, No. 2, 3—4).—The ore (0.5 g.) is evaporated nearly to dryness with nitric acid, 2—3 c.c. are added in excess, the solution (50 c.c.) is warmed and filtered, ammonia added to opalescence, followed by 1 c.c. of 6*N*-hydrochloric acid and 200 c.c. of cold water. After stirring and keeping for several hours (or 1 hr. at 100°), the precipitate is decanted with hot water and washed through the filter with 25 c.c. of hot water containing ammonium chloride (5 g.) and 7.5*N*-sulphuric acid (1 c.c.). The filtrate and

washings are diluted to 150—200 c.c., boiled for 30 min. with aluminium foil, and decanted, the bismuth being dissolved by warming with saturated ferric chloride solution (10—15 c.c.). After addition of cold water (200 c.c.) and syrupy phosphoric acid (5 c.c.) the ferrous iron is titrated with permanganate.

CHEMICAL ABSTRACTS.

Determination of manganese by the Volhard method. F. J. WATSON (Chem. Eng. Min. Rev., 1929, 21, 352—353).—The electrometric determination of the end-point of manganese titrations by the Volhard method is slow, owing to the slowness of the reactions at the end of the titration. Good results may be obtained by adding a slight excess of permanganate followed by sufficient thiosulphate to destroy this excess, and then electrometrically titrating the excess of thiosulphate at 80° with permanganate. A. R. POWELL.

Use of Ganelin's method in the treatment of Altai lead ores. N. N. EFREMOV and E. M. YAKIMETZ (J. Chem. Ind. Moscow, 1927, 4, 662—664).—A preliminary examination of the applicability of Ganelin's method (G.P. 97,943/1897, 124,846/1899) to the treatment of concentrates containing Pb 45.3, Zn 12.70, Cu 0.77%, and a little silver and gold. A. R. POWELL.

CHEMICAL ABSTRACTS.

Importance of the method of sampling for the analysis of alloys. ZWICKER (Chem.-Ztg., 1929, 53, 546—547).—Examples are given of the variation in composition to be expected in large ingots of red bronze or bearing metals. For sampling consignments of the former the ingots should be sawn right through at two places and all the sawings thoroughly mixed for analysis. White metal ingots may be sampled by taking drillings from several parts of the ingot, melting them in a carbon crucible, and casting in a large iron mould so that solidification is rapid. Drillings from this small ingot should represent the bulk. A. R. POWELL.

Reagent for macroscopic metallography. G. D'HUART (Rev. Mét., 1929, 26, 300—306).—An etching reagent for developing the macrostructure of cast iron, mild steels, and copper alloys comprises a mixture of solutions containing (a) 16 g. of nickel chloride dissolved in 100 c.c. of hydrochloric acid, *d* 1.18, and 50 c.c. of water, and (b) 40 g. of chromic acid in 50 c.c. of water. This reagent reveals the segregation of sulphur and phosphorus, the presence of fissures, and lines of slip in highly deformed mild steels. For enhancing the development of these lines in slightly worked steels 8 g. of copper chloride should be added to solution (a). A. R. POWELL.

Method of electrolytic gold refining adopted by the Hitachi copper works. T. KIMATA and T. YAMAMOTO (Japan. J. Min., 1928, 44, 785—814).

Electrolytic recovery of metals. G. EGER (Chem. Fabr., 1929, 323—324, 333—335, 351—352).—See B., 1929, 560.

PATENTS.

Annealing furnaces. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 291,437, 8.5.28. Ger., 3.6.27).—The furnace is provided with an inner jacket of heat-insulating material and a gas-tight outer jacket preferably of sheet metal;

in both jackets are apertures which can be closed or opened independently of one another, and the cooler is mounted between them and within the outer casing, thus preventing any escape of gas from the annealing chamber to the immediate vicinity of the furnace. Alternatively, the furnace has a removable cover with a hood over it, the edge of the latter being immersed in a sealing groove filled with liquid, whilst the cooler is arranged around the edge of the cover under the hood. M. E. NOTTAGE.

Continuous annealing process. H. M. NAUGLE and A. J. TOWNSEND, Assrs. to COLUMBIA STEEL CO. (U.S.P. 1,714,040, 21.5.29. Appl., 4.4.24. Renewed 13.10.28).—For the continuous bright annealing of ferrous metal wire or strip the material is passed into a long tube provided with water seals at both ends and containing a non-oxidising atmosphere. The first portion of the tube serves as the heating furnace and the remainder as a cooler in which the temperature of the annealed strip is reduced to below 100° before it passes the second water seal. A. R. POWELL.

Manufacture of alloy pig iron. H. P. PARROCK (U.S.P. 1,716,181, 4.6.29. Appl., 24.12.26).—A portion of the pig iron tapped from the blast furnace is transferred while molten to an open-hearth furnace, there treated with the requisite amount of previously melted alloy metal, and cast, the remainder of the pig iron from the blast furnace being cast separately. A. R. POWELL.

Production of cast iron. E. PIVOVARSKY, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,715,509, 4.6.29. Appl., 17.6.27. Ger., 15.6.26).—Pig iron of such a composition that it exhibits a grey fracture when cast in the usual sand moulds is cast into chill moulds having a cross-sectional area of at least 100 cm.²; cooling is hastened by spraying the castings with water, and the metal is remelted at a high temperature to redissolve any residual graphite. Subsequent castings have a high-grade white structure. A. R. POWELL.

Bearing metal and its manufacture. R. J. SHOEMAKER, Assr. to S. & T. METAL CO. (U.S.P. 1,717,469, 18.6.29. Appl., 25.3.26).—Lead is melted, together with a metal which prevents oxidation, at a temperature above the m.p. of the second metal, allowed to cool, and treated with sodium at a temperature between the m.p. and b.p. of sodium. A. R. POWELL.

Extraction of mercury from cinnabar. W. C. BAXTER (U.S.P. 1,718,103, 18.6.29. Appl., 13.4.26).—Ores containing cinnabar are leached with a solution containing an alkali sulphide and hydroxide, and the resulting liquors are electrolysed to recover the mercury and regenerate the solution. A. R. POWELL.

Treatment of light-metal [magnesium] alloys. J. A. GANN, Assr. to DOW CHEM. CO. (U.S.P. 1,712,988—1,712,990, 14.5.29. Appl., [A] 28.11.21, [B] 13.1.27, and [C] 29.3.27).—(A) A magnesium alloy containing 8% Al is annealed at 482—493° for about 1 hr., whereby the greater part of the eutectic goes into solid solution. (B) A magnesium alloy containing a metal which forms both a solid solution and eutectic with magnesium is annealed at a temperature just below the m.p. of the

eutectic for a sufficient time to cause all the eutectic to enter into solid solution. (C) A magnesium alloy containing zinc and aluminium is annealed at 340° until the zinc enters completely into solid solution, then at 490—500° to cause the greater part of the aluminium to dissolve. A. R. POWELL.

Aluminium-beryllium alloy and its [heat] treatment. R. S. ARCHER and W. L. FINK, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,716,943, 11.6.29. Appl., 22.11.26).—Aluminium containing beryllium is annealed at a temperature slightly below the m.p. of the eutectic to cause the greater part of the beryllium to pass into solid solution, quenched, and subsequently aged at such a temperature and for such a time as to produce the desired hardening effect. A. R. POWELL.

XI.—ELECTROTECHNICS.

PATENTS.

Electrolyte condenser. TELEGRAPH CONDENSER CO., LTD., and W. J. COLE (B.P. 314,160, 18.4.28).—Metal sheets, e.g., of aluminium 0.001—0.004 in. thick, interleaved with sheets of absorbent fibrous material are rolled spirally, and the absorbent material impregnated, preferably *in vacuo*, with an electrolyte containing dextrin, gelatin, agar-agar, sugar, gum arabic, or glycerin. J. S. G. THOMAS.

Electrolytic condenser. STANDARD TELEPHONES & CABLES, LTD. FROM WESTERN ELECTRIC CO., INC. (B.P. 314,565, 29.3.28).—Electrodes, of which one at least is made of tantalum, and an electrolyte of jelly-like consistency composed of sodium silicate and dilute sulphuric acid are arranged in a container having a non-liquid seal, e.g., of cork impregnated with paraffin, which is normally gas-tight but becomes pervious to gases before the gas pressure in the container becomes excessive. J. S. G. THOMAS.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Kauri resin. J. R. HOSKING (Rec. trav. chim., 1929, 48, 622—636).—An attempt is made to estimate and compare the constituents of kauri resins of varying age. Four varieties of the resin have been examined, viz.: (1) fresh from the tree *Agathis australis*, (2) the same after remaining in air or in casks for a long period, (3) fossil resin ("range gum"), and (4) from peat moors. (1) The thick, milk-white liquid was distilled with steam until all volatile oils had passed over (average yield 8.4%); the distillate, d_4^{25} 0.8588, n_D^{25} 1.4673, $[\alpha]_{5461}^{25} +17.47^\circ$, by fractionation afforded a 92% yield of d - α -pinene, b.p. 154—158°, d_4^{25} 0.8535, n_D^{25} 1.4658, $[\alpha]_{5461}^{25} +22.26^\circ$, but no trace of β -pinene could be detected. The aqueous liquors which remained in the distilling apparatus yielded (1.1%) a gum by concentration and precipitation with excess of alcohol, which was initially bright red, but rapidly became black in air. The colour change is due to the presence of small quantities of manganese derivatives which undergo oxidation. By shaking the aqueous solution of the gum with kaolin and reprecipitating it with alcohol, a nearly colourless product which no longer reduces Fehling's solution was obtained; after

drying in air it contained 14.3% of water and yielded 5.1% of ash, mainly calcium carbonate, giving a positive reaction for manganese. The aqueous solution of the purified gum was inactive. Oxidation with nitric acid (d 1.15) furnished a 13% yield of mucic acid; hydrolysis with 5% sulphuric acid at 100° for 18 hrs., followed by destruction of galactose with yeast, afforded an uncrystallisable syrup, dextrorotatory in solution, from which an osazone, m.p. 158°, and a *p*-bromophenylhydrazone, m.p. 160° (decomp.), were obtained, and which was therefore probably *l*-arabinose. The mother-liquors from the precipitation of the gum after concentration and treatment with a solution of basic lead acetate gave a strongly dextrorotatory solution which yielded an uncrystallisable syrup (osazone, m.p. 186—190°) which possibly contained mainly *d*-glucose. The insoluble resin remaining from the steam-distillation (77.6% of the original resin) was powdered, and separated from ligneous matter by dissolution in an ether-alcohol mixture; the product, obtained by removal of the solvent, gave an equivalent of 103 by titration, and values from 104 to 108 were obtained by hydrolysis for varying periods at 15° or at 100°; the resin therefore contained no esters. The powdered product was extracted thrice by shaking with ether, and the acid fraction of the extract separated by treatment with alcoholic potassium hydroxide, followed by acetic acid. This " α "-resin acid was purified through the potassium salt, the product so obtained constituting 63% of the crude steam-treated resin. Further purification by repeated conversion into the sodium salt yielded the " α "-resin acid as a white, amorphous powder, softens 98°, m.p. 121—124°. The neutral portion of the ethereal extract yielded a yellow solid, m.p. 60—70°, representing 10% of the crude steam-treated resin, and corresponding with the "kauroresin" of Tschirch and Niederstadt (A., 1901, i, 398). The " β "-resin acid was obtained by shaking the residue from the ether extraction with alcohol, and isolated as the potassium salt; this salt by treatment with alcoholic hydrochloric acid gave the free " β "-resin acid as a white, gelatinous mass, converted by drying into a brown powder, softens 210°, m.p. 233—236°. The " β "-resin acid constitutes 19% of the crude steam-treated resin. The original resin contained 3.1% of impurity, and some water (water + loss = 9.8%). The other varieties, by similar treatment, gave the following results: (2) The specimens examined were 2 and 5 years old, respectively, and gave 6.8, 4.85% of volatile oil; 0.45, 0.3% of gum; and 89.4, 92.65% of resin. The oil from the older specimen contained 89% of *d*- α -pinene, d_4^{25} 0.8529, n_D^{25} 1.4633, $[\alpha]_{5461}^{25}$ +10.08°, whilst the gum contained manganese and closely resembled that obtained from the fresh resin (1). The resin from the older specimen (titration value 76.6) yielded an " α "-resin acid, m.p. 120—124° (39%), a " β "-resin acid, m.p. 235—237° (45%), together with 9% of neutral products. (3) The specimen afforded 4.0% of volatile oil, 0.15% of gum, 94.05% of resin; fractionation of the oil gave a trace of a solid, m.p. 128°, which was acid to litmus, a 63% yield of *d*- α -pinene, d_4^{25} 0.8622, n_D^{25} 1.4664, $[\alpha]_{5461}^{25}$ +10.96°, and a 6% yield of a fraction, b.p. 190—215°, which was probably

mainly fenchyl alcohol. The gum was obtained only in small quantity and in an impure state; it contained manganese. The resin (titration value 64.9) yielded an " α "-resin acid, m.p. 120—125° (35%), a " β "-resin acid, m.p. 232—234° (48%), and 9% of neutral products. (4) The specimen gave 0.85% of volatile oil, 0.15% of gum, and about 85% of resin. The crude steam-distilled oil (titration value 1.9; ester value zero), by acetylation and hydrolysis, had an alcohol content of 12.9%, calculated on the assumption that the alcohol present was fenchyl alcohol. By fractionation were isolated: *d*- α -pinene, b.p. 155—156°/758 mm., d_4^{25} 0.8632, n_D^{25} 1.4653, $[\alpha]_{5461}^{25}$ +11.24° (30.5%); dipentene containing some limonene (38.5%); fenchyl alcohol, m.p. 39—41° (fenchyl hydrogen *o*-phthalate, m.p. 145—146°, $[\alpha]_{5461}^{25}$ +15.08°; fenchoxime, m.p. 163—164°) (12.9%, of which 7.5% was isolated). The amount of gum was very small, but contained manganese. The resin (titration value 63.5) was not further examined. The variation with age of the components isolated and their probable mode of origin is discussed; in general, the amount of volatile oil and gum decreases, and the amount of resin increases, with increasing age.

C. W. SHOPPEE.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rational classification of the principal accelerators of vulcanisation [of rubber]. R. THIOLLET and G. MARTIN (Caoutchouc et Gutta-Percha, 1929, 26, 14494—14497).—In grading vulcanisation accelerators it is desirable to consider, in addition to mere activity, the following features:—times necessary at different temperatures for "fixation" of mixtures, and for vulcanisation to maximum mechanical properties, ageing, plastifying effect, and influence of other ingredients on the activity of the accelerator. The point of "fixation" is reached when the mass can be just removed cleanly from the mould, but cut pieces are still capable of being pressed together; immediately before arrival at this point the mass softens if heated rapidly, whereas just after this point it becomes stronger. A mixture of two accelerators, e.g., mercaptobenzthiazole and diphenylguanidine, may cause fixation in a shorter time than the same total quantity of either constituent alone, but this is not always so.

D. F. TWISS.

XV.—LEATHER; GLUE.

Analysis of artificial bating materials [for leather]. II. V. KUBELKA and J. WAGNER (Collegium, 1929, 247—262; cf. B., 1929, 140).—The authors' method is fully described. It differs from the Schneider-Ulcek method (B., 1927, 662) in principle, in the concentrations used, and in the mode of expression of the results. The enzyme activity of the samples is determined by the Schneider-Ulcek method only after they have been brought to the same ammonium salt content, whereas in the authors' method the bate is tested as it is. Temperature of extraction affects the analysis; 37° is used by the authors and 18° in the Schneider-Ulcek method.

D. WOODROFFE.

"Syntan" [synthetic tannin] tannage. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1929, 21,

698—701).—Experiments to determine the amount of tannin fixed by hide powder from solutions of quebracho and 1, 5, and 15% respectively of a synthetic tannin, quebracho and sulphuric acid, quebracho, sulphuric acid, and sodium sulphate, and quebracho alone, showed that the addition of synthetic tannin increased the initial rate of fixation of tannin. The amount of tannin fixed in a given time from the different quebracho liquors was increased by small additions, but diminished by larger amounts of synthetic tannin, which can be explained by the lowering of the p_H value of the liquors. The tannin fixed from wattle liquors was decreased by the addition of synthetic tannin, but was greater than when sodium sulphate was added. The greater fixation from tan liquors containing synthetic tannin and of the same p_H value as tan liquors containing sodium sulphate pointed to some other effects of the synthetic tannin besides acidity. The tanned powder was more "leathery" and softer. D. WOODROFFE.

Tannin analysis. Report of a committee of the International Association of Leather Trades' Chemists. M. BERGMANN (Collegium, 1929, 233—247).—Higher results for the total soluble matter were obtained by the use of calcium chloride instead of sulphuric acid or phosphorus pentoxide in the desiccators. Quicker evaporation was obtained in metal basins than in porcelain, but the same weight of dry residue was obtained in each. The greatest degree of concordance of the results of different analysts on different tanning materials respectively was obtained by the Grasser-Allen method of extraction (B., 1911, 909) as compared with the Koch methods of extracting 1 or 2 litres respectively. Solutions of extracts have been prepared by the proposed official international method, by dissolving in boiling water and diluting to 1 litre with boiling water, and also by dissolving in 100 c.c. of boiling water, heating on the water-bath for 30 min., and diluting to 1 litre with boiling water. The results obtained by these three methods on different extracts were very similar. A suggestion to moisten the ground tanning material before determining the moisture was proved unnecessary. The total soluble matter in a solid untreated quebracho extract was best determined by using a Berkefeld filter candle and suction. Ready-chromed powder gives as good results as freshly-chromed hide powder. Good results have been obtained with the Darmstadt (B., 1929, 140), Freiberg (B., 1928, 722), and Keigueloukis (B., 1929, 221) apparatus for avoiding the handling of the hide powder. Comparisons of the "filter-bell," old official "shake," and provisional international methods of analysis have shown that the greatest degree of concordance was obtained by the "filter-bell" and the least by the international method. D. WOODROFFE.

XVI.—AGRICULTURE.

Use of alcoholic salt solutions for the determination of replaceable bases in calcareous soils. O. C. MAGISTAD and P. S. BURGESS (Ariz. Agric. Exp. Sta. Tech. Bull., 1928, 20, 481—497).—The ammonium chloride method is inaccurate on account of dissolution of organic matter and of calcium from calcium carbonate,

and owing to mechanical losses. Ethyl-alcoholic (68%) barium chloride solution (0.1N) is therefore used as displacing agent. The alcohol is removed by evaporation, the barium precipitated as chromate, and the calcium, magnesium, and other bases are determined in the supernatant liquid. Alkali salts, if present in the soil, must first be extracted with water.

CHEMICAL ABSTRACTS.

Apparatus for measurement of p_H . A. UHL (Fortschr. Landw., 1928, 3, 973—975; Chem. Zentr., 1929, i, 133).—An apparatus for field work is described.

A. A. ELDRIDGE.

Soil of the forest areas of E. Norway and the Trondhjem district. H. GLÖMME (Publ. Norwegian Forestry Res. Service, 1928, 3, No. 10 [i]; Proc. Internat. Soc. Soil Sci., 1929, 4, 97—99).—Interrelationships of soil properties, surface vegetation, forest growth, and the nature of the humus formed are examined. The humification of acid material poor in lime and nitrogen (*e.g.*, heather lichens and pine needles) mainly by the action of fungi is slow and leads to a poor acid humus. Examination of forest profiles is recorded.

A. G. POLLARD.

Distribution of phosphorus compounds in soils, with reference to soil-surface formation. F. TERLICHOWSKI and S. MICHNIEWSKI (Rocz. nauk. Roln. i Lesnych, 1927, 18; Proc. Internat. Soc. Soil Sci., 1929, 4, 57).—The changes occurring in the form of combination of soil phosphorus and its ultimate assimilation by the plant are largely dependent on the nature and general direction of soil solution movements, and these in turn are controlled by the mechanical and chemical composition of the soil. By the examination of the distribution of phosphorus in different soil horizons certain relationships are established.

A. G. POLLARD.

Preservation of soil profiles. K. SCHLACHT (Z. Pflanz. Düng., 1929, 13A, 426—431).—A sheet of celluloid or other suitable material is coated with a solution of a colourless water-soluble condensation product of urea and formaldehyde. This is pressed against the vertical, smoothly-faced profile wall. After 5—10 min. the sheet is peeled off with a thin layer of soil adhering; when dry a permanent "monolith" of the profile is obtained. Negative reproductions can be made with paraffin wax casts and subsequently transferred to plaster of Paris positives. A. G. POLLARD.

Denitrification in uncultivated soils. A. KARLSEN (Bergens Museum Årbok, 1927. Naturv. rekke No. 4; Proc. Internat. Soc. Soil Sci., 1929, 4, 28—29).—Denitrifying organisms were present in numerous cultivated, forest, heath, and moorland soils. No relationship between the intensity of denitrification and the nature of the flora or geological strata was observed.

A. G. POLLARD.

The phosphate question [and crop yields]. O. ARRHENIUS (Z. Pflanz. Düng., 1929, 14A, 121—140).—On the basis of Mitscherlich's growth curves a close relationship is established between the amount of soil phosphate soluble in 2% citric acid solution and crop yields. The results of field trials combined with simultaneous analysis of the citric-soluble phosphate allow

of the preparation of soil charts from which the phosphate requirement of crops may be calculated.

A. G. POLLARD.

Conditions of application and of action of phosphates [on chernozem soils]. V, VI. **Reversion of phosphates in soils.** VII. **Organic parts of the exchange complex.** M. A. EGOROV (Ukraine Chem. J., 1928, 3, 241—262; cf. B., 1928, 618).—V. It is shown that the specific action of sodium phosphates on soils is not due only to their alkalinity. The amount of organic matter extracted by a sodium phosphate solution increases with time, but the rate of increase for even the same soil type, e.g., chernozem, varies with the locality. Time and degree of humidity of the soil are of great importance in determining the amount of organic matter extracted. For chernozem soils there exists an optimum humidity, but this again varies from locality to locality. Also the concentration of the sodium phosphate solution used is of importance; in some cases this concentration is sharply defined, and in others less so.

VI. Shaking of a soil with solutions of the different sodium phosphates gives the corresponding calcium salts. These are considered to be the separate steps in the reversion process taking place in the soil, and they are manifested by the different colour intensities of the water extracts of the soils. The reversion process, however, does not in all cases preserve its initial intensity or its continuity. In some cases both the continuity and the intensity remain unchanged, in others it remains continuous but less intense, whilst again in others both factors suffer.

VII. It is confirmed that non-chernozem soils on extraction with a sodium phosphate solution furnish more organic matter than does a chernozem soil. Again, the fraction due to crenic and apocrenic acids in the total organic matter extracted is particularly small in the case of the chernozem soils, whereas the humic acid fraction is largest in chernozem soils and smallest in other types.

A. FREIMAN.

Nitrates in soil and plant as indexes of the nitrogen need of a growing crop. B. E. GILBERT and J. B. SMITH (Soil Sci., 1929, 27, 459—468. Cf. B., 1927, 825; A., 1928, 1162).—The artificial maintenance of the nitrate contents of the soil and plant solutions above the "sub-optimum" values for a number of market-garden crops yielded uniformly increased crops. The nitrogen requirement of soil for young growing crops can be calculated from the nitrate content of the soil, but the nitrate content of the plant solution is also necessary in the evaluation of the nitrogen requirement for the total crop.

A. G. POLLARD.

Fertilising action of some potash salts used singly and in mixtures. H. LIESGANG (Z. Pflanz. Düng., 1929, 8B, 62—65).—Comparison is made of the efficiency of the chloride, sulphate, and nitrate of potassium in pot-culture experiments with oats. Contrary to the conclusions of Hellriegel, mixtures of the chloride and sulphate or chloride and nitrate produced no greater crops than equivalent amounts of potassium applied as nitrate or sulphate alone. In the relatively high concentrations used, potassium chloride yielded definitely

smaller crops than the sulphate or nitrate applied singly.

A. G. POLLARD.

Liming as a factor in the mobilisation of phosphoric acid in podsol soils. D. L. ASKINASI and S. S. JARUSSOV (Wiss. Inst. Düngemittel, 1928, No. 57; Z. Pflanz. Düng., 1929, 13A, 294—295).—The liming of podsoles increases the energy of decomposition processes and the mineralisation of humus. The latter is connected with the enrichment of the soil solution in inorganic nitrogen and phosphorus. Following liming, the crop yield and also its nitrogen and phosphate content increased. These effects gradually decrease as the effect of the lime is obliterated during a period of about ten years. The changing condition of phosphate nutrition during this time is confirmed by examination of Neubauer's method. The slow-acting phosphates of aluminium and iron and the organic phosphorus compounds existing in acid soils are assumed to be converted into calcium phosphate by liming.

A. G. POLLARD.

Agricultural value of Nile silt held fallacious. E. MCK. TAYLOR (Eng. News-Rec., 1929, 102, 993—995).—Field trials extending over a number of years show that Nile silt has not the fertilising power usually ascribed to it. The essential factor in the maintenance of the soil fertility of the Nile basin is the summer fallow, and the decline in crop yields in recent years is attributed to the elimination of this from the system of agriculture rather than to the decreased amounts of silt reaching the land during irrigation.

A. G. POLLARD.

Significance [to crop yields] of the solubility in ammonium citrate of Rhenania phosphate and basic slag. A. WILHELMJ [with H. K. and S. GERICKE] (Z. Pflanz. Düng., 1929, 8B, 42—61).—In pot-culture experiments with oats there was no relationship between the crop yields and the ammonium citrate solubility of a number of samples of Rhenania phosphate and basic slag. The nutrient value of these two fertilisers is best expressed by their solubility in citric acid. The alleged superiority of Rhenania phosphate over basic slag could not be substantiated.

A. G. POLLARD.

Plant nutrition studies in relation to the triangular system of water cultures. H. F. CLEMENTS (Plant Physiol., 1928, 3, 441—458).—Length, weight, and content of ash, nitrate, or soluble carbohydrates do not adequately indicate the best balance of salts for the growth of plants.

CHEMICAL ABSTRACTS.

Mud fertiliser from sewage fields. HEINE (Landw. Jahrb., 1928, 68, Suppl. I, 355—356; Chem. Zentr., 1929, i, 130).

Effect of lime materials on the outgo of sulphur from Hagerstown silt loam soil. W. B. ELLETT and H. H. HILL (J. Agric. Res., 1929, 38, 697—711).—The results are recorded of the examination of the drainage waters in lysimeter experiments with soil treated with various materials containing lime. Such treatment did not markedly increase the leached sulphur, but stimulated the biological transformation of sulphur in soils. The addition of subsoil to surface soil decreased the sulphur losses in the drainage.

A. G. POLLARD.

Is sulphur a limiting factor of crop production in some Utah soils? J. E. GREAVES and W. GARDNER

(Soil Sci., 1929, 27, 445—457).—A relationship is established between the crop-producing power of Utah soils and their sulphur content as the latter decreases during continuous cropping. The addition of sulphur-carrying salts increases bacterial activity in these soils, particularly nitrogen fixation. A. G. POLLARD.

Comparative acid tolerance of some southern legumes. G. JANSSEN (Soil Sci., 1929, 27, 469—497).—The growth of a number of legumes in sand cultures was examined, and in some cases good plants were obtained in moderately acid media provided nitrate was present. In soil the optimum growth reaction occurred at p_{H} 6.0—6.8 for the various species. Sweet clover grew better in acid soils neutralised with soda than when lime was used. A. G. POLLARD.

Nitrogen problem in sugar cane culture in Java O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, III, No. 3, 91—152; Proc. Internat. Soc. Soil Sci., 1929, 4, 87—89).—The rate of intake of nitrogen by sugar cane reaches a maximum after the 30th week, and the total assimilated increases with the concentration of nitrogen in the nutrient. Optimum crop yields are attained with concentrations of 10.5—31.5 mg. of nitrogen per litre, corresponding to a total consumption of 640—800 kg./hectare. Low nitrogen concentrations in the early growth period of cane have an unfavourable effect on the crop, but in later stages low concentrations are tolerated without loss. The nitrate-producing power of soils is more important for sugar cane culture than the actual amount of nitrate present at any one time. Ammonium sulphate manuring was profitable on soils the nitrate-producing capacity of which was less than 10 mg. NO_3 per kg. The inverse relationship between nitrate-producing capacity and nitrogen deficiency is sufficiently close to allow of the calculation of the latter from the former in practice. A. G. POLLARD.

The chlorine question in sugar cane culture in Java O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, I, No. 3, 90—100; Proc. Internat. Soc. Soil Sci., 1929, 4, 85—87).—In sand culture with sugar cane sodium chloride was added in varying proportions to the nutrient. Maximum yields were obtained with 0.006% of chloride. With 0.018% of chloride yields had fallen below those from chloride-free nutrients. The chlorine content of the cane was but little affected by variations in that of the nutrient. Plants growing in light soils were more sensitive to the effects of chlorides than those in heavy soils. In practice, drainage and leaching are necessary for the removal of excessive chlorides. A. G. POLLARD.

Has manuring with ammonium sulphate a noxious effect on sugar cane? O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 37, 939—941; Proc. Internat. Soc. Soil Sci., 1929, 4, 85).—Ammonium sulphate in amounts commonly used can only affect the yield of sugar cane on very acid soils. In Java soils no difference in manurial effect was observed on acid, alkaline, or neutral soils. A. G. POLLARD.

Noxious effect of molasses on soil. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 31, 791—798; Proc. Internat. Soc. Soil Sci., 1929, 4, 90—91).—

The reduced crop yields following the application of molasses to soil result from the withdrawal of nitrate from the soil by organisms decomposing the molasses. The more severe effects noted in heavy soils are solely the outcome of the longer period of decomposition of molasses, compared with sandy soils, wherein decomposition is complete in about 4 weeks. A. G. POLLARD.

Manurial effects of phosphorites on podsol soils. A. T. TULIN and WOSBUTSKAJA (Agric. Exp. Stat. Perm, No. 1, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 76—77).—The principal factors affecting the availability of rock phosphates in soils are the acidity and base-saturation of the soil, nitrification, and the properties of the soil minerals. In pot experiments, acidification of soils with nitric and sulphuric acids, with the sulphates of iron and aluminium, and also treatment with sodium bicarbonate increased the availability of rock phosphate over that in neutral soils. Availability was, however, decreased by acidification with gypsum and with aluminium sulphate in the presence of lime. The latter effect is ascribed to the greatly increased concentration of calcium in the soil solution and the consequent depression of the solubility of calcium phosphate. After the application of soluble phosphates, the greater assimilation and crop yields in neutral soils are due to the more favourable conditions for nitrification. Analysis of drainage waters from the treated soils showed the greater concentration of calcium in acid soils, and the increased nitrate in neutral soils. In alkaline soils the enhanced concentration of phosphorus appears to result from the formation of soluble organic phosphorus compounds. A. G. POLLARD.

Liming of soil and the use of heavy dressings of potash salts. VINCENT and HERVIAUX (Ann. Sci. Agric., 1928, 45, 335—357; Proc. Internat. Soc. Soil Sci., 1929, 4, 72—73).—Pot experiments and field trials show that preliminary saturation of the soil colloids leads to a better assimilation of potash fertilisers by plants. Heavy applications of potash for this purpose are expensive, and may involve the necessity of frequent liming. By the use of sufficient lime to control soil acidity, and bring about a satisfactory condition of the soil humus, practically complete assimilation of potash fertilisers may be attained. A. G. POLLARD.

Value of by-products of carbonate filters as a fertiliser. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1928, No. 5, 100—108; Proc. Internat. Soc. Soil Sci., 1929, 4, 91—92).—The by-product contains finely divided chalk and appreciable amounts of nitrogen and phosphorus, and is valuable as a sugar cane fertiliser. A. G. POLLARD.

Importance of nitrogen nutrition of crops. O. ARRHENIUS (Med. No. 313, Centralanst. försöksv. jordbruks., 1927, 1—17; Proc. Internat. Soc. Soil Sci., 1929, 4, 92—93).—The intake of nitrate by cereals and sugar beet takes place in two definite periods, and it is essential for maximum crops that optimum quantities of nitrate should be present at these times. The period of nitrogen intake varies with the nature of the crop. High concentrations of nitrate induced root rot in sugar beet and intended to produce sterile grain in maize. A. G. POLLARD.

Importance of potash and phosphate nutrition of crops. O. ARRHENIUS (Med. Centralanst. försöksv. jordbruks., Avdeln. lantbruksbot., 1927, 1—12; Proc. Internat. Soc. Soil Sci., 1929, 4, 74).—In sand culture experiments increasing concentrations of phosphate in the nutrient yielded increasing crops up to a maximum of 9 mg. PO_4 /litre. Higher concentrations in some cases reduced the yields, except in the case of clover, where the optimum concentration was 30 mg./litre. Similar experiments with increasing concentrations of potassium indicated an optimum concentration of 6 mg./litre for cereals and about 20 mg./litre for clover and sugar beet. Comparison of the total intake of potash and phosphate by plants with the concentration of these nutrients in soil solutions showed that, in general, the concentration of potassium in soils approached the optimum, but that of phosphate averaged about one half of the optimum value. The ability of plants to take up nutrients by other means than direct absorption of soil solutions is suggested. Manuring should be directed to increasing the concentration of the soil solution to the optimum, rather than to supplying the total nutrient required by the plant. A. G. POLLARD.

Root rot and soil properties. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, 129—143; Proc. Internat. Soc. Soil Sci., 1929, 4, 59).—The prevalence of root rot could not be correlated with the reaction or phosphate content of soils, but was related to the nitrifying power and certain physical properties. It is not clear whether these last two factors are interdependent or are separately effective. A. G. POLLARD.

Passage of ash-substance into the plant. I. Reaction of the medium as a factor in the mineral nutrition of plants. D. A. SABININ and G. S. KOLOTORA (Agric. Exp. Stat. Perm., 1926, 1, 91—113; Proc. Internat. Soc. Soil Sci., 1929, 4, 81—82).—Maize grown in water cultures showed maximum growth with nutrients at p_{H} 4 and minimum growth at p_{H} 8. The reaction of the plant sap was not influenced by that of the nutrient, but the latter greatly affected the relative proportions of anions and cations entering the plant system. Acid media favour the intake of a preponderance of anions, and *vice versa*. The rate of absorption of mineral matter varies irregularly during the growth of the plant, the maximum in the case of phosphates occurring during the first 3—4 weeks of development. A. G. POLLARD.

Chlorate method for determination of nitrate, nitrogen, total nitrogen, and other elements in soils and plant tissues. E. M. EMMERT (J. Assoc. Off. Agric. Chem., 1929, 12, 240—247).—*Nitrate nitrogen*: The ground, dried tissue (0.2—0.5 g.) or green tissue or soil (1—3 g.) is heated in a Kjeldahl flask with 25 c.c. of 50 vol.-% sulphuric acid, and the expelled gases are led through a condenser into an absorption tower containing 150 c.c. of freshly prepared chlorine dioxide solution made by dropping concentrated hydrochloric acid on to 15 g. of sodium chlorate and passing the gas evolved into water until a deep yellowish-green coloration develops. The exit from the tower is fitted with a Kjeldahl (Hopkins) trap adjusted so that some of the solution is forced into it. The heating of the flask is continued until white fumes begin to be evolved. The

combined washings from condenser, trap, and connections are boiled until colourless, and an aliquot portion containing at least 0.25 mg. of nitrogen is shaken first with 0.05—0.1 g. of silver sulphate and later with 0.5—1 g. of calcium oxide; an aliquot portion of the clear filtered liquor, containing at least 0.2 mg. of nitrogen, is then evaporated to dryness, and 2—3 c.c. of phenoldisulphonic acid solution are added, followed after 5—10 min. by 20—30 c.c. of water so as to dissolve most of the salts. Sodium hydroxide solution (25%) is now added, a few c.c. in excess of that required to develop a yellow colour. The solution is made up to 100 c.c., shaken with 0.5 g. of calcium hydroxide, and sufficient filtered to compare colorimetrically with a standard solution containing 0.0025 mg. of nitrogen per c.c. The volume of the unknown is adjusted until its strength is nearly equal to that of the standard, and the nitrate nitrogen in the sample is calculated from the colorimetric readings. In some samples from which gaseous evolution is violent, e.g., clay loam soils, much of the nitrate nitrogen was lost. *Reduced nitrogen*: The residue from the distillation of nitrate nitrogen is cooled, diluted with 10 c.c. of water, and again cooled. Then 1 g. of sodium chlorate is added for each 0.1 g. of dry tissue or each 0.5 g. of green tissue or soil, and the mixture is shaken and heated rapidly in an open Kjeldahl flask until the green fumes formed decompose and only white fumes are present. When the temperature reaches 100° the flame is lowered, the flask connected to the distillation and absorption apparatus, and, after the violent reaction ceases, the nitric acid is distilled rapidly into water or dilute chlorine peroxide solution. When white fumes appear and the solution remains colourless, the flask is disconnected before turning out the flame. The method is continued as in that for nitrate nitrogen. *Total nitrogen*: An appropriate quantity of the sample is placed in a Kjeldahl flask with sodium chlorate in the same proportion as for reduced nitrogen and 25 c.c. of 50 vol.-% sulphuric acid are added. The method is carried out as for reduced nitrogen, care being taken to prevent accumulations of chlorate and sample near the flame. *Non-volatile elements*: The flask is now tilted so that the neck slopes downward without the contents running out, and the sulphuric acid is distilled off and collected. The residue is cooled, and made up to a known volume or used as a whole. When sodium is to be determined, potassium chlorate may be substituted for the sodium salt. *Carbon*: The gases evolved in the oxidation are collected and measured, and an aliquot part is passed through a suitable absorption train, the carbon dioxide being absorbed and weighed in a soda-lime-phosphorus pentoxide bulb. Preliminary tests gave good results. W. J. BOYD.

XVIII.—FERMENTATION INDUSTRIES.

Proteins of different barleys and pasteurisation turbidities. W. HESSELBURGER (Woch. Brau., 1929, 46, 285—288).—By treatment with suitable adsorbents beers of normal flavour and head retention can be obtained which remain bright when submitted to the most adverse conditions, independently of age, temperature, treatment, malt quality, and mashing

conditions. Examination of the heat-coagulum from untreated and treated beers after hydrolysis by Van Slyke's method shows a marked difference in the adsorption of the different nitrogen compounds. Thus, arginine- and histidine-nitrogen is mostly adsorbed, but not cystine- and lysine-nitrogen. Applications of the method to barleys, malts, and beers are quoted from which it appears that these qualitative differences may relatively greatly exceed the difference in total nitrogen between two barleys, and may prove to be a determining factor in the brewing quality of barley and malt.

F. E. DAY.

Determination of the volatile acids in beer. W. WINDISCH, P. KOLBACH, and E. SCHILD (Woch. Brau., 1929, 46, 245—248, 255—259).—Acetic acid is not volatilised by passing a stream of air through 370 c.c. of a solution requiring 3.77 c.c. of 0.1*N*-caustic soda for neutralisation, nor is formic acid lost under similar conditions. Added acetic acid cannot be quantitatively recovered from beer by steam distillation at ordinary pressure, or by evaporating to dryness *in vacuo*. On adding successive quantities of 50 c.c. of water to the dry residue of beer distilled *in vacuo* and again distilling, a point is reached after about two such additions at which the amount of additional volatile acid obtained is small, and the total yields from duplicate tests agree satisfactorily. If the acidity of the beer is increased by adding a non-volatile acid, *e.g.*, phosphoric acid, the yield of volatile acid changes only slightly between p_H 3 and 2.5, but is markedly increased at p_H 2.0, and at p_H 3.0 the increased volatile acidity obtained after the second addition of water and redistillation is at a minimum. Tests with beers buffered to definite reactions show that above p_H 3 acetic and formic acids are not completely removed, and that lactic acid volatilises from more acid beers. No appreciable quantity of volatile acid is formed under the conditions of distillation. Though in a few cases a third addition of water and redistillation gave a distinctly higher result, exceeding 0.1 c.c. of 0.1*N*-caustic soda per 100 c.c. of beer, this is regarded as abnormal, possibly due to butyric acid. For the determination the beer is aspirated with air freed from ammonia and carbon dioxide till free from the latter. To 100 c.c. 0.1*N*-phosphoric acid is added to bring the p_H to 3.0 (1.3 c.c. for each 1% of original extract), and 30 c.c. of tartaric acid-sodium tartrate buffer solution, p_H 3.0, 0.5*M* in respect to total tartaric acid. The mixture is distilled from a Claisen flask of which the exit is bent first upwards, then down, through a 100-cm. Liebig condenser. A few drops of octyl alcohol, renewed with the water additions, and a little paraffin, m.p. 40—42°, prevent frothing. The distillate is collected in 100 c.c. of water in a 500-c.c. cylinder cooled in ice. The flask is heated on a water-bath, heat being applied after vacuum is established. The bath temperature should be 50° at the syrupy stage and 60° at dryness, at which it is kept till not more than one drop of distillate per minute falls at the adapter. 50 c.c. of water are added to the flask, the residue is dissolved and the distillation repeated, and this yet again. The distillate is freed from carbon dioxide by aeration and titrated with 0.1*N*-caustic soda, using 4 drops of 1% phenolphthalein as indicator. Results are quoted showing the amount of volatile acid in various

types of beer and in beer during fermentation. Steam distillation of beers which have been brought to p_H 3.0 give results approximating to those of the method described if 600 c.c. of distillate are collected from 100 c.c., but differences occur in some cases and the results of duplicate determinations are less consistent.

F. E. DAY.

Divinylethylene glycol as cause of the bitter taste in the disease of bitterness of wines. E. VOISENET (Compt. rend., 1929, 188, 1271—1273).—The compound isolated from bitter wine (B., 1929, 413) has now been identified with divinylethylene glycol, $\text{CH}_2\cdot\text{CH}\cdot\text{CH}(\text{OH})\cdot\text{CH}(\text{OH})\cdot\text{CH}\cdot\text{CH}_2$ (Griner, A., 1893, i, 237). It would appear to be formed in the wine by the action of reductases on acraldehyde.

R. K. CALLOW.

XIX.—FOODS.

Effect of dry skim milk on the water absorption of doughs and the plasticity of flour suspensions. J. L. ST. JOHN and C. H. BAILEY (Cereal Chem., 1929, 6, 140—150).—Addition of dry skim milk increases the water-imbibing capacity of dough so that it is necessary to add an equal weight of water to maintain the same degree of plasticity as measured by means of the power-driven dough mixer and wattmeter. Extensibility of dough measured with the Chopin extensimeter is not substantially affected by the inclusion of 10% or less of dry skim milk. The mobility of flour-water suspensions decreases with addition of dry skim milk. In order to maintain the same mobility, water must be added equal in weight to that of the milk added. The mobility of flour-water or flour-water-skim milk suspensions measured by Bingham and Murray's method increases with time during several hours. W. J. BOYD.

Granulation of flour and its relation to baking quality. C. B. KRESS (Cereal Chem., 1929, 6, 202—214).—Baking tests have been carried out on portions of flour separated on various sieves, No. 10—16. It is concluded that, on the whole, granulation of the flour makes little difference to its baking quality, the medium granulation giving a more even grain. The best loaf (volume, colour, texture, and grain) is made from the 30—40% of flour remaining after removing the coarsest and the finest flour. The fineness does not affect appreciably the rate of fermentation or development of acidity. E. B. HUGHES.

Correlation between diastatic power of flour and crust colour in the test loaf, and its significance. M. J. BLISH, R. M. SANDSTEDT, and H. PLATENIUS (Cereal Chem., 1929, 6, 121—127).—When flours are baked by a fixed procedure, all factors being precisely controlled, depth of crust colour is positively correlated with residual sugar in the baked loaf on the one hand and with relative diastatic power and fermentation tolerance of the flour on the other. W. J. BOYD.

Quick viscosimetric method for measuring the staleness of bread. L. P. KARACSONYI (Cereal Chem., 1929, 6, 241—243).—The viscosity at 20° of a 10% suspension in water of finely-sieved crumb is measured in an Ostwald viscosimeter with slightly modified capillary. Examples quoted show a reduction of viscosity of about 35% when bread is stored for 48 hrs.

E. B. HUGHES.