

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 11, 1926.

I.—GENERAL; PLANT; MACHINERY.

Practical applications of the Lewis filtration equation. H. C. WEBER and R. L. HERSHEY (Ind. Eng. Chem., 1926, 18, 341—344).—The Lewis filtration equations are of the form, $V=KP^{1-s}$ and $V=K'\theta^{(1+t)/(2+t)}$ for constant rate and constant pressure conditions respectively, where V =volume of filtrate, P =pressure, θ =time, and s and t are dependent on the nature of the cake and the operating conditions. Observation of these practical conditions in the filtration of defecated sugar syrups by Sweetland presses shows that these equations can be most usefully applied to decide whether the requisite amount of filter aid is being used. The value of s obtained by plotting the logarithm of V against the logarithm of P should be zero if sufficient filter aid is being used to prevent the formation of a compressible cake. The value of t will show whether scouring or plugging of the cake is taking place. The complexity of the filtering process, however, makes exact measurements very difficult, since it is almost impossible to keep the operating conditions constant throughout a run.

B. W. CLARKE.

Absorption of gases by a liquid drop. W. G. WHITMAN, L. LONG, JUN., and H. Y. WANG (Ind. Eng. Chem., 1926, 18, 363—367).—The absorption of carbon dioxide by a drop of water falling freely through a column of the gas, treated as a case of liquid film diffusion, gives the coefficient for the average rate of absorption per c.c. of solution, k_L , as 260 g. of carbon dioxide per hour through 1 sq. cm. of interface with unit pressure. The absorption of ammonia by the drop considered as an example of gas film diffusion gives the coefficient, k_g , as 22 g. of ammonia under unit driving potential expressed as partial pressure of ammonia in atmospheres, this figure being confirmed by the value 18 determined from the humidification of air by the drop. Although these rates are higher than those obtained by other methods, e.g., absorption through flat liquid surfaces, from gas bubbles, or from columns with wetted walls, the ratio k_L/k_g is about the same as that obtained by the other methods; this shows that the spray type of equipment is as suitable for slightly soluble gases as the bubble type.

B. W. CLARKE.

Foam meter. H. E. WILLIAMS (Ind. Eng. Chem., 1926, 18, 361—362).—A mechanically operated egg beater is fixed in a bowl-shaped receptacle with an outlet at the lowest level, and the liquid under examination is poured in to cover the beater blades. After stirring for a definite period (3 min.) the foaming liquid is immediately run out so as completely to fill a weighed flask under the

outlet. The volume of the flask is termed the foam meter volume (F.M.V.), and the foaming tendency of paints, varnishes, etc., may be numerically expressed as the ratio of weight of material placed in the bowl at first to the weight of the F.M.V. of material after whipping. The apparatus may also be used to test the froth-reducing powers of anti-foaming oils.

D. G. HEWER.

Increasing the internal volume of silica gels. HOLMES, SULLIVAN, and METCALF. See VII.

PATENTS.

[Fritted quartz] filters. JENAER GLASWERK SCHOTT & GEN. (E.P. 226,182, 22.11.24. Conv., 11.12.23. Addn. to 218,286, B., 1925, 230).—A filter of fritted particles of either natural quartz or broken fused quartz is constructed in a similar manner to the glass filter described in the original patent.

B. M. VENABLES.

Edge filtration. H. S. HELE-SHAW and J. A. PICKARD (E.P. 249,202, 17.12.24).—Paper for use in the edge filters described in E.P. 210,101, 210,376, and 227,890 (B., 1924, 239; 1925, 232) is treated with rubber, chlorinated rubber, celluloid, casein, cellulose esters, synthetic or natural resins, paraffin, hardened gelatin, or asphaltum, with the object of preserving the effectiveness of the filter medium even when this is wiped or scraped to discharge the residue. The substance need only be applied to the filtering edges of the paper and is applied after cutting the sheets to shape, and either before or after assembling into packs.

B. M. VENABLES.

Filter. D. M. GOETSCHINS and L. F. VOGT, ASSRS. to STANDARD CHEMICAL Co. (U.S.P. 1,569,627-8, 12.1.26. Appl., 25.8.22).—(A). A filter designed especially for the filtration of corrosive liquids without the use of filter-cloth comprises a number of units, each of which consists of a fine-grain porous stone mounted in a frame. The units are arranged one above the other, a filter chamber being formed between adjacent filter-stones. Each stone has a bevelled corner portion which serves as a filling port. Outlet passages for the filtrate are disposed vertically in the side portion of each unit. (B). Each unit of the filter consists of a frame and a series of filter-stones arranged edge to edge in the plane of the frame. The filter-stones of adjacent units form a filter chamber between their opposed side faces. Means are provided for feeding mixed liquids and solids to the chamber. The chambers discharge through outlets in the inner edge portions of the stones into outlet passages for the filtrate disposed between the inner opposed edges of adjacent filter-stones.

R. B. CLARKE.

Filter. G. J. PEZOLD (U.S.P. 1,574,409, 23.2.26. Appl., 19.12.23).—A horizontal cylinder contains a filtering screen which is attached to the cylinder where a horizontal plane through the axis cuts the sides, and is dished downwards almost to touch the bottom. A conveyor and scrapers to remove the solid from the screen and means for treating the solid with water, air, or steam are provided. The cylinder has a removable head at one end to allow of easy access to the screen. T. S. WHEELER.

Filtering and thickening process and apparatus. A. L. GENTER (U.S.P. 1,575,907, 9.3.26. Appl., 11.4.21).—A mixture passes through a filtering medium, and the flow of the filtrate is periodically reversed by changing the respective levels of the mixture and the filtrate. The solids accumulated on the medium are thus discharged into the mixture and thicken it. H. MOORE.

Filter unit. J. J. PREBLE, Assr. to SPRAY ENGINEERING Co. (U.S.P. 1,576,121, 9.3.26. Appl., 30.4.25).—A filter cell is formed of a densely-compacted stack of variously-shaped expanded metal sheets disposed between inlet and outlet openings. H. HOLMES.

Filtration and treatment of liquids. C. V. ZOUL, Assr. to CELITE Co. (U.S.P. 1,579,171, 30.3.26. Appl., 13.2.23).—Before using a filtering apparatus for filtration proper a clear liquid is repeatedly circulated through it, and to the liquid a filter aid is added gradually so as to form a bed of filter aid upon the filtering surfaces. B. M. VENABLES.

Ovens or furnaces. H. B. CANNON (E.P. 234,049, 18.12.24. Conv., 14.5.24).—The hearth of the furnace is made of a material with high heat-radiating qualities, e.g., silicon carbide. Above the hearth is a heating chamber containing combustion chambers also made of material with high heat-radiating qualities and communicating with a flue situated below the hearth, the bottom of the flue possessing high heat-absorbing qualities. The heat from the combustion chambers is radiated to the arched roof of the heating chamber and thence to the hearth, which is protected by a screen from direct radiant heat from the combustion chambers. The furnace is specially suitable for firing enamelled ware. B. W. CLARKE.

[Roasting] furnace. H. H. BUBAR (U.S.P. 1,576,033, 9.3.26. Appl., 10.8.23).—Material to be treated in a roasting furnace is delivered through a preheater on to one end of an extended stationary hearth and is moved along the hearth by a rabble. Means are provided for passing the gases from the material around the preheater out of contact with the material in the preheater. H. HOLMES.

Furnace. F. SEEBER (U.S.P. 1,576,371, 9.3.26. Appl., 30.3.25).—An annular hearth is mounted for rotation in a closed annular chamber. Partitions extending from the roof of the chamber into proximity to the hearth define with the roof and side walls of the chamber a closed chamber for charging material to and discharging it from the hearth. A combustion

chamber connected with the furnace chamber is arranged at one side of the charging chamber, a flue communicates with the furnace chamber at the opposite side of the charging chamber, and a gas-tight seal is provided between the hearth and the walls of the furnace chamber. H. HOLMES.

Vertical cone mills for grinding dry granular products. J. H. GENTRUP (E.P. 242,614, 21.10.25. Appl., 5.11.24).—The apparatus comprises an outer rotating vertical cone, and an inner fixed cone, the large ends of the cones being downwards and the space between the two being wider at the top than the bottom, and being adjustable by altering the vertical position of the inner cone. B. M. VENABLES.

Pulverising or grinding mills. J. B. C. SCHERBAUM (E.P. 245,097, 15.12.25. Conv., 24.12.24).—A disc grinding mill is constructed so that the periphery only of the grinding discs is enclosed in a casing which may be maintained under reduced pressure, the larger part of the surface of the discs being left exposed to the air for cooling. The casing bears resiliently but tightly against the rotating disc, and the grinding surfaces of the discs consist of intercalating ridges and grooves which decrease in depth towards the periphery. B. M. VENABLES.

Grinding and the like mills. C. V. GREENWOOD (E.P. 249,631, 28.1.25).—Grinding is effected by the action of a number of discs (driven by a spider) against the interior surface of a tyre or ring. The discs are constructed of laminae preferably disposed at a slight inclination to the plane of rotation, and the outer grinding ring is formed of a number of detachable and renewable bars arranged like the staves of a tub, alternate bars being preferably of hard and soft metal. B. M. VENABLES.

Comminuting solids and forming colloidal solutions. O. AUSPITZER (U.S.P. 1,577,052, 16.3.26. Appl., 23.7.25).—To prevent waste of power in internal friction in a colloid mill, the material issues from the mill at a point directly behind that at which the beaters exert their action and is then conveyed outside the mill back to the point of disintegration through an external pump. Preferably the material is caused to flow in a straight confined tangential course through the mill. E. S. KREIS.

Mill. F. SILVA (U.S.P. 1,577,376, 16.3.26. Appl., 21.11.25).—A rotary casing is provided with external trackways and supporting rollers. Spaced supports project through feed and discharge openings at opposite ends of the casing. Crushing rollers are carried by the supports within the casing, and means are provided for forcing the supports downwards, these means yielding if undue resistance is met, and for maintaining a constant fluid level within the casing. H. HOLMES.

Softening of hard calcareous materials such as water-laid deposits and incrustations [scale]. A. A. COZENS, and GAS LIGHT AND COKE Co. (E.P. 248,886, 28.1.25).—Hard calcareous deposits from water are broken down by treatment with a solution

of free citric acid until a soft mass or sludge is formed which can be removed by flushing. The disintegration may be effected with application of heat. The citric acid may have a concentration of 10–30% or more and may contain 2–3% of an alkali chloride.
S. BINNING.

Heating of vacuum pans. BLAIR, CAMPBELL, AND MCLEAN, LTD., and A. BLAIR (E.P. 248,897, 9.2.25).—Vacuum pans are heated by two or more pipes in the form of a coil the convolutions of which are in the same plane, each of the pipes being independently connected with the steam supply pipe and being provided with a discharge outlet for condensed water. Alternatively the pipe coils may be in pairs, the outer and inner ends of each pair being coupled together and connected to the steam supply pipe and discharge pipe respectively.
S. BINNING.

Drying furnaces.—G. G. BROCKWAY (E.P. 249,381, 1.7.25).—A drying furnace for powdered material, such as fuller's earth, boneblack or other filtering material is constructed of a number of vertical shafts down which the material trickles against hot gases issuing from burners inserted through the walls at the bottom of the shafts. The material is made to take a zig-zag path by blocks of refractory material projecting from the walls and extending about halfway across the shafts, the treated material finally emerging through nozzles of refractory material at the bottom.
B. M. VENABLES.

Drying or calcining. J. A. BARR, Assr. to INTERNAT. AGRICULTURAL CORP. (U.S.P. 1,573,533, 16.2.26. Appl., 9.4.24).—An apparatus for drying or calcining consists of a shaft down which the material under treatment is passed. Hot flue-gases are introduced at the bottom and are withdrawn by suction at the top of an intermediate zone in the shaft. Air drawn in at the bottom of the shaft by the suction in the exit flue cools material which has passed out of the heating zone, and air similarly drawn in at the top of the shaft prevents any moisture passing up from the heating zone and softening untreated material so that it chokes the shaft.
T. S. WHEELER.

Apparatus for concentrating solutions [by freezing]. M. O. JOHNSON (U.S.P. 1,576,136—7, 9.3.26. Appl., 19.9.21).—(A) A dilute solution to be concentrated is cooled by heat exchange with the concentrated juice leaving the apparatus, and is then partially frozen in a refrigerator which is cooled at the upper part only by circulating brine. The remaining liquor or concentrated juice collects in the lower part of the refrigerator and leaves through the heat exchanger (cf. *supra*). The brine is cooled in a brine tank by the expansion of a refrigerant vapour, the latter is then compressed, partially cooled by ordinary means (e.g., water sprays), further cooled by the ice obtained from the juice, and expanded again. All the heat exchanges are effected without mixing the various fluids. (B) In a scheme similar to that described in (A) the refrigera-

tor is provided with means such as a worm conveyor for continuously scraping off and removing the ice formed, which is then centrifuged and the separated liquor returned to the refrigerator. The brine after leaving the refrigerator proper is used to cool the liquor in the feed tank.
B. M. VENABLES.

Absorption apparatus. C. L. VORESS, V. C. CANTER, and R. W. SKOOG, Assrs. to GASOLINE RECOVERY CORP. (U.S.P. 1,577,200, 16.3.26. Appl., 3.8.25).—A perforated distributing pipe extends the entire length of a horizontal drum in its lower portion. A mechanical support is secured to the drum slightly above the distributing pipe, and carries stationary solid absorbent material with the interposition of a second mechanical support provided with openings sufficiently small to prevent the passage of particles of the absorbent material. A second perforated pipe extends along the upper portion of the drum above the absorbent material. H. HOLMES.

Charcoal absorption apparatus. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of R. TIEDTKE and J. BENZ (G.P. 421,027, 29.8.24).—The patent relates to improvements on the apparatus described in G.P. 377,520 and 378,608 (cf. E.P. 187,223 and 191,005; B., 1923, 386A; 1924, 237). An annular chamber containing charcoal rotates above a second annular chamber divided into two circular compartments, the planes of contact of the chambers being so perforated that the perforations can be brought into or out of alinement. Gas passes into the outer compartment of the lower chamber, then through perforations into the upper chamber; any unabsorbed gas is led out through the inner compartment.
R. B. CLARKE.

Air-cooled still. E. C. BALLMAN, Assr. to J. H. CASSIDY (U.S.P. 1,577,675, 23.3.26. Appl., 19.1.20).—The still comprises a lower boiling vessel open at the top, surmounted by an upper condensing vessel closed at the top, but open at the bottom. A small gutter is provided around the interior of the condenser to catch the condensate.
B. M. VENABLES.

Regulating the temperature of superheated steam. FARBENFABR. VORM F. BAYER & Co., Assees. of K. HENCKY (U.S.P. 1,578,313, 30.3.26. Appl., 7.11.24).—The superheated steam is passed downwards at high velocity over a thin film of water also flowing downwards and spread over an extensive area of surface.
B. M. VENABLES.

Multiple-deck pulp thickener. C. W. ANKENY, Assr. to DORE Co. (U.S.P. 1,578,625, 30.3.26. Appl., 26.4.22).—The apparatus comprises a number of superposed settling compartments having central openings for the downward discharge of settled solids and openings connecting adjacent compartments for the upflow of supernatant liquid. Scraping devices pass over the bottom of each compartment and immediately adjacent thereto in order to deliver settled solids towards the discharge opening.
J. S. G. THOMAS.

Apparatus for effecting the transfer of heat between gases. [Recuperators.] T. TEISEN (E.P. 249,919, 31.12.24).

Powdered fuel [boiler] furnaces. C. HOLD (E.P. 250,103, 18.9.25).

Dryers, mixers, screeners and the like. E. E. WELCH (E.P. 249,998, 18.3.25).

Separation of liquids of different specific gravities. B. D. COMYN and W. A. WHITE (E.P. 250,360, 10.2.25).

Silicious alkaline - earth product (U.S.P. 1,574,363).—See VII.

Calcium silicate derivative (U.S.P. 1,574,380).—See VII.

Electric gas purifier plants (E.P. 240,844).—See XI.

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

Limits of inflammability of firedamp in atmospheres which contain blackdamp. H. F. COWARD and F. J. HARTWELL (Safety in Mines Res. Bd., Paper No. 19, 1926, 11 pp.).—The limits of inflammability of methane in atmospheres composed of air with additions of nitrogen and carbon dioxide have been determined at atmospheric temperatures and pressures. Two series of experiments were carried out, with tubes of 2 in. and 10 in diam. respectively, and upward propagation of flame. The differences observed with the two tubes were almost inappreciable. The upper and lower limits of inflammability approach one another and finally coincide as the proportion of diluent is increased. Flame propagation is not possible with methane mixed with air containing more than 25% of carbon dioxide or 38.5% of nitrogen, or intermediate percentages of blackdamp, depending on the proportions of nitrogen and carbon dioxide in the latter. The upper limit of inflammability of methane falls rapidly as a diluent gas is added to the air. The lower limit rises comparatively slowly in atmospheres containing up to 20% of diluent gas, showing that the addition of moderate amounts of either nitrogen or carbon dioxide to the air would not appreciably diminish the danger of a firedamp explosion underground. A. B. MANNING.

Electric ignition of firedamp: alternating and continuous currents compared. R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 20, 1926, 18 pp.).—The minimum current required in order that the spark or "momentary arc" produced when the circuit is broken should cause ignition of a mixture of methane and air has been determined under a variety of conditions. Three types of apparatus were used in which the contact between two metallic poles could be broken automatically every 5 sec., the poles remaining in contact for 0.5 sec. The break of circuit in one apparatus was similar to that occurring during underground sig-

nalling, and in the others to the sudden break of a wire under tension. The poles were enclosed in a glass vessel which could be evacuated and filled with the gas mixture under investigation, which in most of the experiments contained about 8% of methane. The igniting current, for a given mixture of methane and air, varies with the volatility of the metal at the spark-gap, being lower the lower the b.p. of the metal. Other conditions being the same, the igniting current is lower the more rapid the break of circuit and the smaller the area of contact at the moment of break of circuit. The relation between igniting current and inductance of the circuit varies with the mechanical conditions under which the sparks are produced. Under one set of conditions the relationship could be expressed by the equation $Li^{1.4}=k$. In general, for low voltages, particularly with high self-inductive circuits, the current is of far greater importance than the impressed voltage as regards the igniting power of the spark. With higher voltages, however, the igniting current increases. The character of the current supply, whether continuous or alternating, does not materially affect the igniting power of the spark, the results obtained in this series of experiments differing from those recorded by Thornton (Trans. Roy. Soc., 1914, A 90, 272). A. B. MANNING.

Simplified method of evaluation of coals. F. LANDSBERG (Glückauf, 1925, 61, 1427—1439; Chem. Zentr., 1926, I, 2271—2272).—The method, which is used by the German State Railways, depends on the relation between calorific value and content of volatile constituents. The cal. val. rather than the quantity of the volatile hydrocarbons is determined, and by plotting the cal. val. of the clean coal against its volatile content (or the volatile content reckoned as percentage against the cal. val. per 1% of volatile matter) curves are obtained which enable coals of various origins to be classified. W. T. K. BRAUNHOLTZ.

Aspects of coke-oven heating in the light of modern tendencies in design. R. W. DAVIES (Gas World, 1926, 84, Coking Sect., 38—42).—A heat balance for a 50-oven battery of standard design taking a charge of 8½ tons of dry coal is given. The possible economies by the adoption of the most modern designs are estimated as follows. With an oven 40 ft. by 16 in. by 12 ft. 10 in. (charge 14½ tons) the radiation losses would be reduced from 160 B.Th.U. per lb. of coal to 92 B.Th.U. The use of insulating brick would bring this latter figure down to 61 B.Th.U. The chimney loss could be reduced by one-third by restricting the air supply, a part of the waste gases being returned to the ovens so that the total bulk of gases circulating and the flame conditions are not altered. The heat loss through open doors is reduced by reducing the pushing time. The total effect of these improvements would be the raising of the thermal efficiency from 46.0% to 66.2%, or a saving of fuel gas equivalent to 350 B.Th.U. per lb. of coal. C. IRWIN.

Behaviour of coke-oven gas at low temperatures. W. H. NAYLOR (*Gas World*, 1926, 84, Coking Sect., 42—45).—Coke-oven gas stripped of carbon dioxide, hydrogen sulphide, and moisture was passed at the rate of 0.5—1.0 cb. ft. per hr. through tubes cooled with liquid air. 10 c.c. of condensate per cub. ft. of gas were obtained, containing on an average 79% of methane and 16% of ethylene. By fractional evaporation these constituents can be readily separated.

C. IRWIN.

Origin of carbon disulphide in the carbonisation of coal. W. J. HUFF (*Ind. Eng. Chem.*, 1926, 18, 357—361).—Coal of high sulphur content can be carbonised in the laboratory without producing carbon disulphide if it is heated slowly and gasification proceeds freely. Carbon disulphide apparently obtained in laboratory experiments by the interaction of coal gas and coke has been shown to be derived from the reheated coke alone; carbon disulphide presumably is not produced as the result of a secondary reaction between carbon and hydrogen sulphide. When coal is heated rapidly so that a dense coherent coke is produced, *i.e.*, under conditions approximating to gas-works' practice, so that the tarry and gaseous vapours are only in contact with the heated coke for a relatively short time, considerable quantities of carbon disulphide are produced. Carbon disulphide is therefore a primary product of carbonisation, formed by rapid heating of the coal under conditions where local deficiencies of carbon and hydrogen exist, and the disulphide formed is rapidly removed from the hot coke. This offers an explanation for the low proportion of carbon disulphide produced in processes with a low rate of heat transmission, *e.g.*, low temperature carbonisation, vertical retorts, and coke-ovens, compared with horizontal gas retorts.

B. W. CLARKE.

Solution of common salt as a confining liquid for gas analysis. H. TROPSCH (*Z. angew. Chem.*, 1926, 39, 401; cf. Hoffmann, B., 1926, 146).—The addition of a small amount of sulphuric acid to solutions of salt used as confining liquid for gas analysis makes no appreciable difference to the solubility of carbon dioxide therein, but serves to neutralise traces of alkaline absorption liquids with which it becomes contaminated, and which otherwise would cause appreciable absorption of carbon dioxide. If the acidified solution is saturated with carbon dioxide and then left for some time exposed to the air, the amount of carbon dioxide it will subsequently absorb from gases containing a relatively high proportion of that constituent is inappreciable. To detect readily any change of reaction of the solution addition of a few drops of phenolphthalein is recommended.

A. B. MANNING.

Experiences in benzol recovery by the vacuum method. GAREIS (*Gas- u. Wasserf.*, 1926, 69, 269—272).—The Raschig system of benzol recovery (cf. B., 1923, 390 A) is characterised by the following features:—(1) The enriched oil is preheated only by the stripped oil leaving the still; (2) distillation is effected by indirect steam under high vacuum;

(3) motor benzol is produced continuously without redistillation. The enriched oil is brought to 130° in the preheater (a tall narrow tube of wrought iron) and only requires a further heating to about 155° in the still. The evacuation of the still also tends to steam economy, the consumption being 3 kg. of steam per kg. of benzol against 6—10 kg. in older installations. The use of indirect steam produces a great saving in cooling water, the consumption being 35—60 litres per kg. of benzol. The deterioration of wash oil is very slow, one batch in the plant described having served for 21 months. This also appears to be due to the exclusion of direct steam. The cost of steam, cooling water, wash oil, and power for pumps per metric ton of benzol is estimated at M.47 against M.107 in the old process. The rotary benzol washer used is described and it is pointed out that the capacity of a washer depends not only on efficient contact of the gas and oil but on the efficient stripping of the latter, its temperature, and its mol. wt. Lignite oil was found unsatisfactory and coal-tar creosote low in naphthalene was used. With this oil naphthalene was by no means completely extracted from the gas. The stripped wash oil passes from the heat exchanger to the cooler at 35°, this low temperature facilitating separation of oil and water. A continuous plant for the treatment of benzol with acid and alkali was unsuccessful and was abandoned.

C. IRWIN.

Autoxidation of mineral oils and determination of the tar value. J. MARUSSON and W. BAUERSCHÄFER (*Chem.-Ztg.*, 1926, 50, 263—264).—The acids produced in the sludge test of mineral oils have been examined. Those from a normal, unrefined transformer oil (d_{20}^{20} 0.891, open flash pt. 154°, acid value nil, tar value 0.88, ash 0.015%, viscosity at 20° 3.09), had a density greater than 1 and the copper salts were completely soluble in benzene. Treatment with benzene caused a separation into 60% of soluble, oily acids and 40% of insoluble, asphaltic acids. The soluble acids had acid value 66, saponif. value 130, acetyl value 58, iodine value 16, mean mol. wt. (Rast) 285. No reaction was given in the formolite test. The insoluble acids had acid value 67, saponif. value 269, acetyl value 127, iodine value 18. It is concluded that the soluble acids consist of hydroxynaphthenic acids formed from saturated naphthenes and that the insoluble acids are produced from unsaturated hydrocarbons by union of two molecules and the addition of oxygen. Of the acids from a refined white oil (d_{20}^{20} 0.842, open flash pt. 181°, free acid nil, sulphur 0.015%, tar value 14.5, viscosity at 20° 3.98), 90% were soluble in benzene and this fraction had d 1.014, acid value 121, saponif. value 240, acetyl value 44, iodine value 16, mean mol. wt. 357, and gave a negative formolite reaction. The copper salts were soluble in benzene and the peroxide content was very slight, the amount of active oxygen present being 0.06% (cf. Brauen, B., 1914, 951). A modified sludge test is described, wherein, by the use of sodium hydroxide as catalyst, a saving in time and oxygen is effected. 50 g. of the oil are mixed with 10 g. of

pumice saturated with sodium hydroxide (prepared by addition of the pumice to 0.7 g. of sodium hydroxide in 10 c.c. of water and drying at 105°) in a 200-c.c. conical flask and heated at 120° for 24 hrs. The mixture is cooled, 50 c.c. of 50% alcoholic sodium hydroxide (containing 7.5% NaOH) are added, and the liquid is heated under a reflux condenser for 20 min. The soap solution is separated, acidified, and extracted with benzene. From the extract, after removal of mineral acid by washing, the tarry matter is obtained by evaporation. This method, with refined transformer oils, gives results differing by only a few hundredths of 1% from those obtained by the standard German method. E. H. SHARPLES.

Decomposition of ozokerite by cracking. E. TOPORESCU (Bull. Acad. Sci. Roumaine, 1926, 10, [2], 1—3).—See B., 1925, 980.

Chlorine in the petroleum industry. L. BURGESS (Trans. Amer. Electrochem. Soc., 1926, 49, 157—160. Advance copy).

PATENTS.

Drying of coal and other solid bituminous substances of the character of coal. KOHLENVEREDLUNG GES. M. B. H., and C. GEISSEN (E.P. 249,774, 17.11.25).—The moist material is brought into contact with the steam-heated walls of a dryer, hot inert gases being simultaneously passed through the material for the purpose of carrying away the vapours produced.

W. T. K. BRAUNHOLTZ.

Manufacture of fuel. E. R. SUTCLIFFE (E.P. 249,170, 22.9.24).—Hard, homogeneous and stone-like briquettes are produced by compressing coal and lime which have been ground to pass a 200-mesh screen and have been intimately mixed in either the dry or the wet state. No binding agent is added to the mixture before briquetting, nor is the mixing carried out in such a way as to develop hydration of the lime or to render the mass plastic or more easily consolidated. The briquettes are carbonised and, after cooling and quenching, are broken into pieces of suitable size. The fuel so obtained is hard, dense, and uniformly porous, and it may be used as a substitute for charcoal. The sulphur which it contains is fixed by the lime and remains in the ash after burning.

A. W. HOTHERSALL.

Block or briquette of lignite. E. FERNHOLTZ, Assr. to FERNHOLTZ MACHINERY Co. (U.S.P. 1,577,902, 23.3.26. Appl., 22.12.24).—The lignite is crushed, aged by storage for a limited time, dried by indirect heat derived from steam, cooled and simultaneously mixed and fed as a uniform mass into a mould of the extrusion type. The high pressure attained in the constricted mould causes the bitumen in the material to melt forming a strong waterproof binder. The briquettes are delivered from the mould without shock, and are fed gently along a channel until they have set and cooled.

A. W. HOTHERSALL.

Utilising coke contaminated with vegetable matter derived from the treatment of house

refuse. G. H. HADFIELD (E.P. 249,247, 6.1 and 24.3.25).—The mixture of coke, coal, and vegetable matter obtained after treating house refuse in a liquid separating medium is dried, crushed, and briquetted, with or without the addition of small coal, coke, or anthracite duff. A. W. HOTHERSALL.

Carbonisation of coal. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (E.P. 242,622, 26.10.25. Conv., 6.11.24).—Finely-pulverised coal is introduced into the top of a vertical retort, and is carbonised in suspension, at a temperature of about 600—800° by an upward stream of hot gases produced by the partial combustion of the material at the bottom of the retort by means of a limited supply of air. The volatile products of carbonisation are removed at the top of the retort and the semi-coke at the bottom.

W. T. K. BRAUNHOLTZ.

Apparatus for carbonisation of fuel. S. R. ILLINGWORTH, ILLINGWORTH CARBONIZATION CO., R. DEMPSTER AND SONS, and H. J. TOOGOOD (E.P. 244,837, 25.9.24. Addn. to 245,190; B., 1926, 228).—The apparatus previously described, in which a system of vertical metal retorts is heated by radiant heat from flues in the walls of the setting surrounding the retorts, is modified by the addition of horizontal cross-over flues at right angles to the wall flues, and passing between the retorts. The hot gases pass through ports in the cross-over flues and come into direct contact with the retorts, which are thus heated by conduction as well as by radiant heat. The passage of the hot gases from the wall flues into the cross-over flues is controlled by dampers. Supplementary burners are provided in the wall flues directly over the cross-over flues. The upper ends of the retorts communicate with a header and are held by rings and preferably trough plates above the rings, and the lower ends have flanges of such a size resting on a base-plate that the retorts are free to be withdrawn through the header.

A. B. MANNING.

Producing combustible gases and coke from bituminous fuel. POWER GAS CORP., LTD., and N. E. RAMBUSH (E.P. 248,518, 30.12.24. Addn. to 235,958).—A modification of the main patent (B., 1925, 661) in which a portion of the mixed gas leaving the top of the shaft generator during the cold-blowing period is, after being scrubbed, returned to the bottom of the generator to replace part or whole of the steam used during that period, thereby producing gas of increased heating value.

A. B. MANNING.

[Prevention of corrosion in the] manufacture of fuel gases. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (E.P. 248,841, 11.12.24).—Fuel gases are partially dried by being brought into intimate contact with a water-absorbing material in liquid form, e.g., a solution of calcium chloride. The solution passes from the absorbing chamber to a heater, where the absorbed water is expelled, and the regenerated solution is returned to the chamber.

By this means the moisture content of a gas may be sufficiently reduced to prevent deposition of water in the mains, and the corrosive action of certain of the gas constituents is minimised or inhibited.

A. B. MANNING.

Carbonisation of fuel by vertical retorts and the like. J. RUDE (E.P. 248,967, 21.5.25).—A number of vertical retorts, arranged in concentric circular rows, are surrounded by cylindrical shell walls forming flues of which the inner wall accompanies the retorts in an intermittent rotary movement around a hollow axis, through which the distillation gases are withdrawn, whilst the outer shell is stationary and contains openings for the entry and exit of the heating gas. The waste gases pass through heat exchangers where they give up their heat to a gaseous medium (*e.g.* the fuel gas, or air) which goes to preheat the fuel to be carbonised. The rotary movement of the retorts serves to bring them in succession to the place where the discharge of the coke into a common cooling or quenching chamber takes place and where the recharging from a common hopper is effected. The connexions between the rotary and stationary parts of the apparatus are rendered gastight by means of liquid seals.

W. T. K. BRAUNHOLTZ.

Fuel distillation. MERZ & MCLELLAN, and W. A. RILEY (E.P. 249,236, 30.12.24).—The distillation of fuel is accomplished by injecting into the retort a mixture of hot gases produced by the combustion of producer or other gas in a special chamber, low-pressure steam which is superheated by contact with these combustion gases, and air which is admitted in regulated quantity to the combustion chamber in which the producer gas is burnt. The amount of admixed air is such that the required limited degree of combustion of fuel in the retort is attained (*cf.* E.P. 223,387, B., 1924, 1004). The steam is introduced by an injector nozzle either into the combustion chamber or into the conduit leading from the latter to the adjacent or superimposed retort, and it supplies the energy necessary to force the gases into the retort.

A. W. HOTHERSALL.

Carbonising apparatus. O. H. HERTEL (E.P. 249,710, 15.6.25).—The retort may be heated in stages progressively by separately controlled burners. It is provided with a central hollow shaft, which may carry a screw conveyor. The shaft is heated internally and progressively in stages by separately controlled burners. The pipes carrying the fuel gases to the internal burners are cooled, *e.g.*, by jackets in which air, steam, or water circulates. Firebrick may be interposed to prevent the play of the flame on the wall of the hollow member.

R. A. A. TAYLOR.

Water-gas generator with regenerative heat-storing device. M. FRANKL (E.P. 240,466, 23.9.25. Conv., 26.9.24).—In the walls of a water-gas producer, on opposite sides of the central chamber, are two heat accumulators consisting of sets of vertical tubes open at the lower ends and connected by a manifold at the upper ends. During one gas

generating and blowing period the tubes of one accumulator are heated internally by the "run" gases, and externally by the "blow" gases, and during the succeeding period the second accumulator is so heated. The heat stored is utilised for the generation of the steam necessary for gas production, by injecting water alternately into the tubes of the two heat accumulators. The steam and air are blown alternately in opposite directions horizontally through the fuel bed.

A. B. MANNING.

Production of water-gas. HUMPHREYS AND GLASGOW, LTD., Asses. of C. S. CHRISMAN (E.P. 242,237, 19.8.25. Conv., 31.10.24).—Approximately one-half of the total water-gas is produced by supplying low-temperature steam to the generator fuel bed in a direction opposite to the flow of the air blast, removing the comparatively cool blue water-gas (*e.g.* at about 180°) from an outlet below the fuel bed, and passing it directly to the wash-box or to the holder. The remainder of the gas is produced by an up-steaming operation and is passed directly to the carburettor, which has been previously heated by combustion of the blast gases assisted by secondary air, and thence through the superheater to the wash-box or to the holder, where it may be mixed with the down-run gas to produce illuminating gas. In this way the carburettor may be kept at a high and comparatively uniform temperature, and economy is effected by having an unobstructed connexion between generator and carburettor without the usual hot-valve and reverse steam provisions. Where low-temperature steam is used in the generator, and blast products of low temperature are produced, the secondary air supplied to the carburettor may be preheated in order to accelerate the ignition of the blast gases. The heat carried away by the blast gases and by the carburetted up-run gas may be recovered by passing these gases through a recuperator, which serves to preheat the air or steam supply, and/or through a waste-heat boiler.

A. W. HOTHERSALL.

Apparatus for determining temperatures by carbon dioxide content of combustion gases. F. D. POTTER, Assr. to COMBUSTION CONTROL CO. (U.S.P. 1,577,853, 23.3.26. Appl., 24.3.19).—The furnace gases pass through a short-circuit passage in the furnace wall to a carbon dioxide recorder. The usual record sheet is replaced by a chart which directly indicates temperatures and corresponding carbon dioxide contents of the gases. Provided the air admitted to the furnace is equal to, or in excess of, that required for complete combustion, the percentage of carbon dioxide in the gases varies inversely as the quantity of air supplied and directly as the temperature reached.

R. B. CLARKE.

Quantitative determination of selected gases in atmospheres. L. B. ATKINSON (E.P. 248,807, 13.10.24).—Gases are estimated quantitatively by the use of a standardised test paper impregnated with a reagent and indicator. The paper is prepared by being dipped into a solution of the reagent of suitable concentration, leaving part unmoistened, and so obtaining an approximately uniform concentration

of reagent over the moistened area. A paper impregnated with alkali and phenolphthalein may be used for estimating small amounts of carbon dioxide in air by determining the time taken for the colour change to occur, the paper having been calibrated in standard gas mixtures. A. B. MANNING.

Quantitative determination of gases [carbon monoxide]. S. H. KATZ (U.S.P. 1,578,666, 30.3.26. Appl., 12.3.25).—Carbon monoxide is determined in gas mixtures by passing the latter over hopcalite (cf. Lamb, Bray, and Frazer, B., 1920, 424 A) and measuring the rise in temperature produced. Before entering the constant-temperature chamber containing the catalytic cell, the gases are filtered from suspended material which might poison the catalyst, and are preheated to the temperature of the chamber. The rise in temperature is measured electrically.

A. B. MANNING.

Scrubber for separating oil and other liquid particles from gas. SMITH SEPARATOR Co., Asses. of M. F. WATERS (E.P. 234,436, 17.11.24. Conv., 23.5.24).—The scrubber consists of a vertical cylindrical vessel, the "wet" gas entering about half-way up, and being deflected downwards by a baffle. The gas on rising to the upper part of the vessel encounters a conical baffle (apex downwards) filling the whole cross section of the vessel; staggered ports are provided in this baffle, permitting the passage of the gas, the ports each having the shape of a truncated cone (apex upwards). At a still higher level is a non-perforated conical baffle (apex downwards), fixed so as to leave an annular space between the edge of the baffle and the sides of the vessel. By this system of baffles the gas is progressively dried, and passes away from the top of the vessel. The separated oil is conveyed by drain pipes to the lower part, and is drawn off as required.

W. N. HOYTE.

Purifying coal or other gases. J. N. REESON and W. L. MOSS (E.P. 249,312, 11.3.25).—Hydrogen sulphide, carbon disulphide, and thiohydrocarbons may be removed from coal gas or other gases by washing the gas in a solution prepared by adding to a 2% solution of ferrous sulphate (or chloride), 1–5% of an organic substance which will prevent the precipitation of iron hydroxide when the solution is neutralised, e.g., salicylic acid, *p*-hydroxybenzoic acid, phthalic acid, polyhydric alcohols and their derivatives including the glycols and glycerol, and citric acid. The solution is then neutralised with soda ash and oxidised by aeration. It can be regenerated when necessary by aerating with air or oxygen at a suitable temperature and under pressure, and afterwards neutralising with alkali and adding iron salts to replace loss. The sulphur formed during the regeneration process can be removed and the accumulated sulphur compounds can be ultimately recovered as sodium thiosulphate and sulphate.

A. W. HOTHERSALL.

Purifying gas-house liquor. E. W. KEITH, Assr. to GEN. METALS RECOVERY Co. (U.S.P.

1,573,169, 16.2.26. Appl., 30.9.24).—Gas-house liquor is made suitable for hydro-metallurgical use by treating it with zinc oxide in quantity equivalent to the ammonium sulphide present. The precipitated zinc sulphide is removed and calcined to give the oxide for further use. R. B. CLARKE.

Removal of naphthalene and analogous hydrocarbons from fuel gases. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,578,687, 30.3.26. Appl., 26.1.25).—In a two-stage process for removing naphthalene the gas is washed first with oil which is continuously circulated in contact with the gas, and then with fresh oil. S. PEXTON.

Utilisation of liquid slag from gas producers and pulverised fuel furnaces. G. POLYSIUS EISENGIESSEREI & MASCHINENFABR. (G.P. 423,793, 5.5.22. Addn. to 421,427).—The process described in the chief patent (B., 1926, 363), is carried out in rotating kilns, thereby obtaining a more even distribution of heat and a better admixture of the slag and the added constituents. B. W. CLARKE.

[Motor] fuel. A. WOOSNAM. From CANADIAN ELECTRO PRODUCTS Co. (E.P. 249,348, 29.4.25).—Between 2% and 20% of an aldehyde (in particular paraldehyde) saturated with acetylene, is dissolved in motor spirit. It is claimed that the deposition of carbon in the cylinder of the engine is reduced to negligible quantities. About 12 vols. of acetylene are dissolved in 1 vol. of the aldehyde. The heavier the motor spirit the more aldehyde is required; ordinary grades of petrol require 3.5–10%.

W. N. HOYTE.

[Motor] fuel. T. MIDGELEY, jun., Assr. to GEN. MOTORS CORP. (U.S.P. 1,578,201, 23.3.26. Appl., 28.2.20).—The fuel consists of 50% of commercial ethyl alcohol and 50% of a cracked gasoline, the latter containing 84% of saturated hydrocarbons and 16% of unsaturated (olefines). W. N. HOYTE.

Agent for raising the calorific power of motor fuels. D. COSTAGUTA (F.P. 599,271, 9.6.25).—The agent consists of a nitrophenol, e.g., trinitrophenol, one or more ethers, and one or more liquid hydrocarbons. It is added to the motor fuel in the proportion of 4.5%. B. FULLMAN.

Decomposing oils. C. M. ALEXANDER (U.S.P. 1,573,532, 16.2.26. Appl., 9.10.19).—A heavy oil is maintained at a temperature of 370–425° and preheated light oil to be cracked is introduced in a spray below its surface so that cracking takes place before vaporisation. The vapours from the cracking still are fractionated and any uncracked oil is returned with fresh light oil. The heavy oil is continuously circulated from the still through a chamber in which any carbon it contains is removed.

T. S. WHEELER.

Apparatus for treating [cracking] oil. F. C. VAN DE WATER and F. R. SUNDERMAN, Assrs. to PETROLEUM LABORATORIES, INC. (U.S.P. 1,575,663, 9.3.26. Appl., 16.1.23).—A cracking still is provided

with a dome, and a supplementary chamber within the dome has a series of diametrically opposed valved openings, one set of which is connected with a supply of gas, e.g., hydrogen, and another set with the still body. H. MOORE.

Apparatus for distilling hydrocarbons. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE CO. (U.S.P. 1,576,564, 16.3.26. Appl., 3.6.22).—A cracking still is connected with a fractionator, and this with a supplementary still connected with a second fractionator. Both fractionators are connected with a condenser. A return pipe leads from the supplementary still to the cracking still. A feed pipe supplies fresh oil through the first fractionator and supplementary still. H. MOORE.

Apparatus for refining [cracking hydrocarbon] oils. W. T. HANCOCK and M. BOYLE (U.S.P. 1,576,742, 16.3.26. Appl., 8.10.23).—A still for the conversion of heavy hydrocarbon oils into lighter oils has perforated pipes running longitudinally, their ends being supported by sheets spaced from the ends of the still to provide steam spaces. There are steam inlets at the ends of the still and a separator chamber connected to its upper portion, a condenser being connected with the top of this chamber. H. MOORE.

Process for separating hydrocarbons. W. C. AVERILL, JUN. (U.S.P. 1,577,440, 23.3.26. Appl., 10.12.23).—Hydrocarbon oil containing water is vaporised and the water vapour is condensed completely together with part of the oil. The remaining oil vapours are scrubbed with oil of high boiling point obtained from a previous distillation and are then subjected to further condensation. The scrubbing oil is that obtained in the first partial condensation. W. N. HOYTE.

Continuous catalytic distillation [of hydrocarbons]. G. L. PRICHARD and H. HENDERSON, Assrs. to GULF REFINING CO. (U.S.P. 1,577,871, 23.3.26. Appl., 14.2.22).—Hydrocarbons of high boiling point are distilled in the presence of aluminium chloride as catalyst. From the vapours the fractions of higher boiling point and the catalyst are first separated, and the vapours then pass on for final condensation. The mixture of catalyst and oil of high boiling point is returned to a second batch of cracking stock and catalyst, which in its turn is distilled. W. N. HOYTE.

Cracking hydrocarbon oil. E. W. ISOM, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,578,035, 23.3.26. Appl., 19.6.25).—Oil is passed through a pipe still heated by furnace gases, and is brought up to cracking temperature. The cracked oil vapours pass through a reflux condenser and the reflux is returned to the main flow of oil. Fresh oil is added as required. The heating gases are circulated repeatedly over the pipes, fresh gases being added as required. W. N. HOYTE.

Distilling oils with volatile catalysts. A. McD. McAFEE, Assr. to GULF REFINING CO. (U.S.P. 1,578,049, 23.3.26. Appl., 20.1.22).—A mixture of the oil with a metal halide as catalyst is distilled, and

the resulting vapours of products of lower boiling point are condensed after having been brought into intimate contact with a petroleum hydrocarbon.

W. N. HOYTE.

Distilling [petroleum oil] with aluminium chloride. A. McD. McAFEE, Assr. to GULF REFINING CO. (U.S.P. 1,578,050, 23.3.26. Appl., 20.1.22).—The vapours resulting from the distillation of the oil with aluminium chloride as catalyst are partially condensed to separate the catalyst and kerosene, and then passed to a final condenser. The catalyst is separated by settling from the kerosene, and is returned to the still. W. N. HOYTE.

Process for improving oils. A. McD. McAFEE, Assr. to GULF REFINING CO. (U.S.P. 1,578,051, 23.3.26. Appl., 20.1.22).—A flow of oil is maintained through a series of stills operated in succession and is distilled in presence of aluminium chloride as catalyst. The volatilised chloride is condensed, collected, and returned towards the first still of the series. W. N. HOYTE.

Removing aluminium chloride residues from [oil] stills. A. McD. McAFEE, Assr. to GULF REFINING CO. (U.S.P. 1,578,053, 23.3.26. Appl., 13.12.23).—The caked mass of chloride is disintegrated by wetting with water, is then flushed out of the still with water. W. N. HOYTE.

Lubricating greases. RÜTGERSWERKE-A.-G., and H. TEICHMANN (G.P., 382,184, 22.7.20).—The lubricants contain resinous matters obtained by the action of high-boiling coal-tar oils on coal, wood, or other substances of organic structure; they are sometimes admixed with other lubricants. They have a high drop-point and are insensitive to temperature changes. B. FULLMAN.

Combustion of solid fuel. M. DE CONINCK (U.S.P. 1,580,656, 13.4.26. Appl., 24.1.24).—See E.P. 210,419; B., 1924, 932.

Eliminating the sulphuretted hydrogen in fuel gas. F. W. SPERR, JUN., Assr. to KOPPERS CO. (U.S.P. 1,578,560, 30.3.26. Appl., 6.6.24).—See E.P. 241,248; B., 1926, 5.

Removing aluminium chloride residues from vessels used for the catalytic treatment of petroleum hydrocarbons. A. J. H. HADDAN. From GULF REFINING CO. (E.P. 250,102, 11.9.25).—See U.S.P. 1,578,053; preceding.

Dehydrating [oil] emulsions. W. CAMERON, Assr. to SIMPLEX REFINING CO. (U.S.P. 1,580,956, 13.4.26. Appl., 15.11.20).—See E.P. 173,644; B., 1922, 131 A.

Maintaining solid [coal] particles uniformly suspended in liquids, more especially liquid fuels. G. E. HEYL (E.P. 250,490, 17.11.25).

Refining of hydrocarbons (E.P. 249,309).—See III.

Extracting hydrogen from industrial gases (E.P. 242,583).—See VII.

Impregnating wood (E.P. 244,449).—See IX.

Distillation of hydrocarbons etc. (G.P. 424,678).—See XII.

III.—TAR AND TAR PRODUCTS.

Benzol recovery by vacuum method. GAREIS.—See II.

PATENTS.

Refining of hydrocarbons. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 249,309, 9.3.25).—Tar from ordinary or low-temperature carbonisation of coal etc., petrol, cracked oil, or the like is distilled over caustic soda or lime using non-superheated steam. The distillate may be further refined by passing the vapours together with hydrogen over finely-divided nickel, copper, iron, etc. at 100–110°. W. N. HOYTE.

Separation of constituents of low-temperature coal-tar without distillation. ZECHE M. STINNES, and A. WEINDEL (G.P. 420,394, 5.9.23).—The tar is washed several times on the counter-current principle with sodium hydroxide of lower strength than 5%. The acid constituents are removed, and the neutral oil is separated unchanged in properties (e.g., viscosity). For example, the tar is intimately stirred with 3% sodium hydroxide at normal or slightly raised temperature, and left for a short time in a separator. At first a large part of the sodium hydroxide is absorbed, and then separation occurs. Four washings are usually sufficient. The phenols are precipitated from the alkaline liquid floating on the neutral oil by acid treatment. B. FULLMAN.

Lubricating greases (G.P. 382,184).—See II.

Distillation of hydrocarbons etc. (G.P. 424,678).—See XII.

IV.—DYESTUFFS AND INTERMEDIATES.

Use of chlorine in the organic chemical industry. J. T. CONROY (Trans. Amer. Electrochem. Soc., 1926, 49, 47–64. Advance copy).

Synthetic colouring matters used in lake making. ROWE.—See XIII.

PATENTS.

Azo dyestuffs. BADISCHE ANILIN- & SODA-FABR., Assees. of F. GÜNTHER (U.S.P. 1,573,605, 16.2.26. Appl., 13.7.25).—Compounds containing the grouping $R \cdot N : N \cdot Ar(p) \cdot X(p) \cdot Ar \cdot N : N \cdot R$ in which Ar stands for an aryl group, R is a salicylic acid derivative, and X is an azo, azoxy, or $\cdot NH \cdot CO \cdot NH \cdot$ group, and also sulphonic acid groups, dye animal, vegetable, or mixed fibres very fast shades. To prepare them a *p*-nitroarylaminesulphonic acid is diazotised and coupled with salicylic acid and the product reduced to the azo- or azoxy-derivative to yield a compound which gives directly orange to red-brown shades on fibres. Alternatively the azo dyes from *p*-nitroarylamines and salicylic acid are sulphonated and

reduced to the corresponding azo- or azoxy-compounds, or sulphonation is performed after reduction. For the carbamide derivatives the unsulphonated dyestuffs from diazotised diaminodiarylcaramides and 2 mols. of salicylic acid or its homologues or derivatives are sulphonated or a sulphonated diaminodiarylcaramide is diazotised and coupled with 2 mols. of salicylic acid, or a *p*-nitroarylaminesulphonic acid is diazotised and coupled with salicylic acid, the nitro-groups are reduced to amino-groups, and the product is treated with phosgene. The carbamide derivatives give light yellow shades. For example *p*-nitroaniline-*o*-sulphonic acid is diazotised and coupled with salicylic acid and the azo-compound is reduced with sodium sulphide to give an azo- or azoxy-derivative which dyes cotton directly. Treated with chromium salts a chromium compound of greatly increased fastness yielding copper shades on cotton is obtained. T. S. WHEELER.

Azo dye. FARBENFABR. VORM. F. BAYER & CO., Assees. of J. HALLER (U.S.P. 1,576,322, 9.3.26. Appl., 7.11.25).—A diazotised *as*-*m*-xylydine (1:3-dimethyl-4-aminobenzene) or its 6-nitro-derivative is coupled with a 2:3-hydroxynaphthoic acid *as*-*m*-xylylide to yield azo dyes which give fuller and clearer shades on vegetable fibres than do the analogous compounds obtained with 2:3-hydroxynaphthoic acid anilide. The shades are very fast to light and kier-boiling. For example, cotton is treated first with 2:3-hydroxynaphthoic acid *as*-*m*-xylylide in caustic soda solution and Turkey-red oil and then with diazotised *as*-*m*-xylydine, when a bluish-red shade is developed. The diazotised 6-nitroxylydine coupled with the 6-nitro-xylylide gives yellow-red shades. T. S. WHEELER.

Substantive green azo dyes. A.-G. FÜR ANILIN-FABR., Assees. of W. LANGE and L. NEUMANN (G.P. 419,585, 22.2.24).—The dyes are obtained by treating in aqueous solution with phosgene a mixture of equivalent quantities of a blue trisazo dye of the type $A \cdot N_2 \cdot M^1 \cdot N_2 \cdot M^{11} \cdot N_2 \cdot M^{11}$ (where A is an aromatic residue, M^1 and M^{11} are aromatic residues having no hydroxyl groups, and M^{11} is a hydroxy-amino-residue of the naphthalene series giving no ring formation with phosgene) and a yellow aminoazo-compound. A dye which produces green shades fast to light on cotton is thus obtained from 1-aminobenzene-2:5-disulphonic acid-azo-1-aminonaphthalene-7-sulphonic acid-azo-1-aminonaphthalene-azo-2-amino-5-hydroxynaphthalene-7-sulphonic acid and 1-aminobenzene-3-carboxylic acid-azo-2-amino-1-methoxybenzene. L. M. CLARK.

Manufacture of new azo dyes and intermediate products. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 248,946, 15.4.25).—New derivatives of the arylides of 2:3-hydroxynaphthoic acid of the general formula,

$C_{10}H_6(OH) \cdot CO \cdot NH \cdot aryl \cdot X \cdot aryl \cdot NH \cdot OC \cdot C_{10}H_6(OH)$ (where X=an azo or azoxy group) are obtained either by condensing the chloride of 2:3-hydroxynaphthoic acid or an *O*-acyl derivative with an aromatic diamino-azoxy or diamino-azo compound

(afterwards removing the acyl group if present), or by treating a nitroarylide of 2:3-hydroxynaphthoic acid with reducing agents, such as dextrose, arsenious acid, zinc dust, or formaldehyde in alkaline solution. These compounds couple with diazo compounds to form azo dyes. The above arylide derivatives have an affinity for vegetable fibre equal to that of direct cotton dyes. By treating the fibre with these derivatives and then with a diazo compound dyeings specially fast to rubbing are obtained, the intensity of which depends on the introduction of a further chromophore into the molecule on the side of the arylide of 2:3-hydroxynaphthoic acid. The new dyes are evenly absorbed from a hot bath and are specially adapted for the dyeing of heavy goods. For example, 52 pts. (by weight) of the chloride of 2:3-hydroxynaphthoic acid and 21 pts. of *p*:*p'*-diaminoazobenzene, each dissolved in acetone, are stirred together for 1 hr. and the mixture is made feebly alkaline with sodium hydroxide. The precipitated *di*-2:3-hydroxynaphthoyl-*p*:*p'*-diaminoazobenzene, a yellowish-brown powder, has m.p. above 360°. *Di*-2:3-hydroxynaphthoyl-*m*:*m'*-diaminoazoxybenzene, a brownish crystalline powder, decomp. above 285° without melting, is obtained by dissolving 30.8 pts. of the *m*-nitroanilide of 2:3-hydroxynaphthoic acid in 22 pts. of aqueous sodium hydroxide (*d* 1.383) and 500 pts. of water at 65° and reducing with a solution of 12 pts. of dextrose in 40 pts. of water at 60–70° for 1–2 hrs. 29.8 pts. of the azoxy-compound, so obtained, are dissolved in 12 pts. of aqueous sodium hydroxide (*d* 1.383), together with some Turkey-red oil and sufficient sodium acetate or carbonate for neutralising the diazo-solution, and treated with 12.9 pts. of diazotised *p*-chloroaniline. The dye separates as bluish-red flakes. As an example of development on the fibre, 0.4 pt. of the azoxy-compound obtained by reduction of the *m*-nitroanilide of 2:3-hydroxynaphthoic acid in 0.8 pt. of aqueous sodium hydroxide (*d* 1.383) is mixed with a little Turkey-red oil and brought up to 200 pts. with hot water. 10 pts. of cotton are introduced at the boil and dyed for 1 hr. while cooling. Development with diazotised *m*-nitro-*p*-toluidine gives a clear bluish Turkey-red shade of good fastness to rubbing and washing. The tints (chiefly yellowish-red to brown) of about 80 dyes are given.

A. COULTHARD.

[Perylene] vat dyes. H. SCHÖPFER, Assr. to F. BENSÄ (U.S.P. 1,574,116, 23.2.26. Appl., 9.4.25).—By heating dinitro- or tetranitro-*p*-erylene with 5 pts. of anhydrous aluminium chloride for 4 hrs. at 160–200°, condensed nitroperylene are obtained. That from the dinitro-compound dyes cotton in the vat fast olive-brown shades; that from the tetranitro-derivative gives olive-green shades.

T. S. WHEELER.

Manufacture of ω -aminoalkylaminonaphthalene compounds and substitution products thereof. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 249,717, 29.6.25).—The compounds are prepared by treating naphtholcarboxylic acids containing the hydroxyl and carboxyl

groups in the *ortho* position to each other, with aliphatic diamines, in the presence of sodium bisulphite. For example, a mixture of 40 pts. of ethylenediamine hydrate, 300–400 pts. of sodium bisulphite solution, *d* 1.357, and a solution of 38 pts. of 2-naphthol-3-carboxylic acid in the requisite quantity of sodium hydroxide solution is heated to 90–100° with stirring until no more carbon dioxide is evolved. After cooling, the pale yellow crystalline precipitate is removed, washed with salt solution and water, stirred with hot, dilute sodium hydroxide solution to remove residual β -naphthol, and again filtered and washed with water. The residue consists of β -naphthyl- β -aminoethylamine. The preparation of β -naphthyl- δ -aminobutylamine and β -naphthyl- β -aminoethylaminoethylamine is also described.

L. A. COLES.

Dyestuff intermediates. M. T. BOGERT (U.S.P. 1,574,337, 23.2.26. Appl., 15.10.24; cf. B., 1926, 354).—2:4-Dinitrodiaryl ethers or 2:4-nitroamino-diaryl ethers are readily reduced to the corresponding 2:4-diaminodiaryl ethers by treatment with iron, water, and a little hydrochloric acid at 100°. These compounds yield useful dyestuffs by reactions similar to those used to obtain analogous dyestuffs from *m*-phenylenediamine. 2:4-Diaminodiphenyl ether has m.p. 67°; *diacetyl* derivative, m.p. 171° *hydrochloride* softens at 190°, m.p. about 210°.

T. S. WHEELER.

Hydrogenation of naphthalene. "PRAX" CHEM. VERSUCHS- U. VERWERTUNGS-G.M.B.H. (F.P. 533,033, 30.3.21. Conv., 5.10.16).—Naphthalene is mixed with nickel carbonate and treated with hydrogen at 200–280°. A colloidal solution of nickel results, followed by reduction.

B. FULLMAN.

Separation of *o*- and *p*-chloronitrobenzene. VEREIN FÜR CHEM. U. METALLURGISCHE PRODUKTION (G.P. 420,129, 29.4.22).—The eutectic mixture of 66.9% of *o*- and 33.1% of *p*-chloronitrobenzene, m.p. 14.65°, is melted with *p*-dichlorobenzene, and the melt cooled to 11°. A mixture of *p*-dichlorobenzene with the greater part of the *p*-chloronitrobenzene crystallises out, and the two compounds are separated by steam distillation. The liquid residue from the eutectic mixture is steam-distilled, and cooled to 15°, when *o*-chloronitrobenzene separates, and may be freed from residual eutectic mixture by centrifuging.

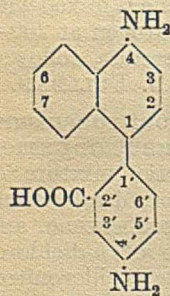
B. FULLMAN.

Preparation of phenols. BADISCHE ANILIN- & SODA-FABR., Assees. of O. SCHMIDT and K. SEYDEL (G.P. 420,393, 28.7.23).—Phenol homologues may be converted into lower members by treating them at high temperatures with hydrogen in the presence of activated catalysts (*e.g.*, Ni, Cu, Ag, Pt, using as activators the substances mentioned in G.P. 307,580 and 408,811; cf. B., 1925, 475) under such conditions that ring-hydrogenation does not occur. For example, if *o*-cresol vapour be led at 400° over a catalyst formed by reduction of nickel chromate on pumice stone, a good yield of phenol is obtained, methane being evolved. With a similar catalyst the vapour of mixed xylenols yields a mixture of phenol and cresol.

B. FULLMAN.

Oxidation of organic substances and the preparation of salt mixtures containing chromate and dichromate. C. F. BOEHRINGER & SOEHNE G.M.B.H. (G.P. 420,444, 24.10.23).—Oxidation is carried out with alkali chromate or dichromate in nitric acid solution, the exhausted liquor concentrated with recovery of the nitric acid which distils, and the residue fused, the resulting nitrous fumes being converted into nitric acid; or chromium nitrate or products containing it are heated to fusion with alkali nitrates, the nitrous fumes being regenerated. For example, anthracene suspended in 10% nitric acid is quantitatively oxidised to anthraquinone by addition of solid sodium dichromate at 75–80°. The residual liquor is treated with an excess of equal parts of sodium and potassium nitrates, evaporated, and the residual solid fused at 280–300°. Aniline is oxidised in 12% nitric acid by addition of sodium dichromate solution, at 5–10°. After extraction of the quinone, the liquor is evaporated, fusion of the residue yielding sodium dichromate. B. FULLMAN.

Production of benzanthrone derivatives. L. CASSELLA UND CO., Assees. of G. KALISCHER, R. MÜLLER, and F. FRISTER (U.S.P. 1,565,229, 8.12.25. Appl., 19.3.25).— α -Naphthaleneazobenzene-*m*-carboxylic acids are reduced in acid solution and the diamino-substituted phenylnaphthalenes thus obtained, of the annexed formula in



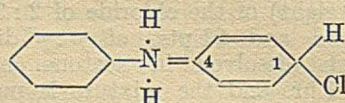
which the C-atoms, 2, 3, 5, 6, 7, 3', 5', 6' may be substituted by Me, halogens, or alkoxy-groups, are condensed to the corresponding diaminobenzantrones by treatment with acid-condensing agents. For example, the sodium salt of *a*-naphthaleneazobenzene-*m*-carboxylic acid (the free acid, m.p. 207°, is obtained by boiling the diazo-compound of the dyestuff from *m*-diazobenzoic acid and α -naphthylamine with alcohol) is

reduced with stannous chloride and the resulting dihydrochloride of 1-(4-amino-2-carboxyphenyl)-4-aminonaphthalene is condensed with chlorosulphonic acid to give 4:4'-diaminobenzanthrone, m.p. 225°. Diazo-tised *m*-amino-*p*-methylbenzenecarboxylic acid and α -naphthylamine when coupled and the product diazotised and boiled with alcohol give the corresponding *a*-naphthaleneazo-6'-methyl-3'-benzenecarboxylic acid m.p. 226°, and this treated as above gives the corresponding 4:4'-diamino-5'-methylbenzantrone. T. S. WHEELER.

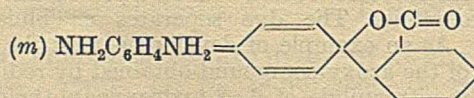
Preparation of 5-hydroxy-*N*-methyloxindole [and *p*-hydroxyphenylmethylglycine]. E. MERCK, CHEM. FABR., Assees. of A. DÜTZMANN (G.P. 421,386, 23.8.24).—*N*-Methyl-*p*-aminophenol sulphate is treated in aqueous solution with 25% ammonia and a little aqueous sulphur dioxide. The free base is washed with aqueous sulphur dioxide, boiled for

several hours with glyoxal sodium sulphite, filtered, and concentrated to crystallisation. The product is recrystallised from 20% sodium acetate solution, and 5-hydroxy-*N*-methyloxindole, m.p. 186–187°, obtained from the purified product (its sodium sulphite) by treatment with hydrochloric acid. The filtrate from the sodium sulphite derivative yields, on treatment with acetic acid, *N*-*p*-hydroxyphenylmethylglycine. B. FULLMAN.

Dyestuffs. W. P. BATES (U.S.P. 1,572,417, 9.2.26. Appl., 25.8.24).—By the interaction of an aromatic diazonium chloride with an aromatic amine or diamine in presence of water at about 90°, a quinonoid compound with the formula



is obtained. Compounds of this type are either dyes for feathers, furs, hair, wool, and the like, or may be used as intermediates for the manufacture of dyes for such purposes. The hydrogen and chlorine in position 1 are readily replaced by various atoms or groups, and the NH_2 group in 4 is also reactive. The product from benzenediazonium chloride and *m*-phenylenediamine dyes furs, feathers, and hair brown. The substitution of the chlorine atom by methoxyl by treatment with sodium methoxide improves the shade. Sodium salicylate reacts with removal of sodium chloride and formation of an ester derivative which when treated with fused zinc chloride loses water and gives a compound with the formula



which gives orange shades on wool in the cold. Replacement of chlorine by a *p*-hydroxyphenylglycine residue yields a product which gives yellow-orange shades, and substitution by carboxyl through the nitrile yields a product giving deep yellow shades. Sulphonation gives products which dye silk from an acid bath rich golden brown to yellow shades. T. S. WHEELER.

Preparation of quinol. A. ERLACH (Austr. P. 98,418, 21.4.23).—Benzoquinone, prepared by the oxidation of aniline by means of chromium trioxide in sulphuric acid solution, is separated from the acid liquor by distillation *in vacuo* in a current of steam in the presence of boric acid, and is subsequently reduced to quinol by treatment with sulphur dioxide. A yield of quinol of about 65–80% of the weight of the aniline is obtained.

L. A. COLES.

Process of manufacturing [benzanthrone] vat dyes. BADISCHE ANILIN- & SODA-FABR., Assees. of A. LÜTRINGHAUS, H. NERESHEIMER, and H. EMMER (U.S.P. 1,580,062, 6.4.26. Appl., 11.11.24).—See F.P. 589,323; B., 1926, 148.

o-Benzylphenol. FARBENFABR. VORM. F. BAYER & Co., Assees. of W. KROPP, W. SCHRANZ, and W. SCHULEMANN (U.S.P. 1,580,053, 6.4.26. Appl., 1.2.24).—See G.P. 406,532; B., 1925, 474.

Diacylacyldiamino-compounds of the aramyl series. CHEM. FABR. GRIESHEIM-ELEKTRO, Assees. of A. ZITSCHER and R. SCHMITT (U.S.P. 1,580,709, 13.4.26. Appl., 15.5.23).—See E.P. 211,772; B., 1924, 413.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Textile] piece [goods] carbonising. M. L. WILSON (J. Soc. Dyers and Col., 1926, 42, 109—110).—Burrs and vegetable impurities are satisfactorily removed from woollen fabrics by carbonising for 20—30 min. at 71° with 7.5% sulphuric acid, and the resulting fabric can be dyed evenly provided that the fabric before carbonising is thoroughly freed from grease and soap and not allowed to lie more than 20—30 min. after removal of the excess acid by mangling or hydro-extraction.

A. J. HALL.

[Pulverulent cellulose and chlorinated hydrocellulose.] C. DE NEYMAN (Chim. et. Ind., 1926, 15, 349—352).—Powdered cellulose is prepared by treating paper with sulphuric or hydrochloric acid or with ferric chloride and disintegrating the mass mechanically. The quantity of acid used and the temperature govern the nature of the product. Much acid and high temperature result in a coloured powder. By treating cellulose with chlorine gas a chlorinated hydrocellulose is produced, characterised by the ease with which it can be acetylated by means of a mixture of acetic acid and acetic anhydride. In this process acetic acid—used merely as a diluent—may be replaced by other neutral liquids in which cellulose acetate is insoluble. Benzene, petroleum spirit, or paraffin oil may be used for the purpose but the proportion of acetic anhydride must be slightly increased. A better grade of cellulose acetate is obtained by the use of cotton and the reaction is accelerated by the addition of small quantities of sulphuric acid. Previous moistening of the cellulose with acetic acid aids the admixture with the acetylating agents.

A. G. POLLARD.

Chemical processes in the ripening of viscose. LEUCHS (Kunstseide, 1925, 7, 286; Chem. Zentr., 1926, I., 2415).—Sodium hydroxide and carbon disulphide react according to the equation $5CS_2 + 12NaOH = Na_2S + 3CS(SNa)_2 + 2Na_2CO_3 + 6H_2O$. By the action of acid the 1 mol. of sodium sulphide yields 1 mol. of hydrogen sulphide, and the 3 mols. of sodium trithiocarbonate yield 3 mols. of hydrogen sulphide and of carbon disulphide. If by treating viscose with acid a % of hydrogen sulphide and b % of carbon disulphide are obtained, $\frac{1}{4}a$ arises from sodium sulphide and $\frac{3}{4}a$ from sodium trithiocarbonate, which latter also gives an equal molecular amount of carbon disulphide which was combined with cellulose. During the ripening of viscose the xanthate is slowly hydrolysed and the

dithiocarbonic acid residues thus formed react with sodium hydroxide. The differences in the amounts of carbon disulphide liberated by acid at different stages of ripening are only 2/5 of the amounts produced by the decomposition of the xanthate. The differences are so small that an illusory constancy of the ratio of carbon disulphide to cellulose is obtained.

A. GEAKE.

Absorption of sodium hydroxide from its solutions and from "black liquor" by sulphate-cellulose. C. KULLGREN (Papier-Fabr., 1926, 24, 153—155, 185—189, 206—208).—The absorption of sodium hydroxide from its solutions by sulphate-cellulose, as kraft cellulose and as bleachable pulp, was determined by the indirect method. 2 g. of cellulose were shaken with 100 c.c. of the solution and 50 c.c. were then titrated with acid. When the concentration of sodium hydroxide was 3.5 g. per 100 c.c. absorption was complete in 2—60 min., according to the conditions of shaking, and it diminished with rising temperature between 18° and 50°. At 75° acidic substances were formed by oxidation of the cellulose by atmospheric oxygen. With increasing concentration of sodium hydroxide from 0 to 7% the absorption curve was bent first towards the concentration axis and then away from it. It is probable that a strongly hydrolysed compound of sodium hydroxide and cellulose is formed. The absorption is reversible, and the equilibrium may be reached from either side. No absorption of sodium chloride or carbonate could be detected but the addition of the former to sodium hydroxide markedly increases the absorption of this latter. The absorption of sodium hydroxide from solutions of sodium sulphide, which is nearly completely hydrolysed in dilute solution, and from black liquor from sulphate-cellulose manufacture was determined by comparing the change in electrical conductivity when kraft cellulose was added with that produced by the addition of small amounts of sodium hydroxide. The method is not very accurate on account of the large temperature coefficient of the conductivity. The absorption in both cases is greater than from pure sodium hydroxide solutions of the same alkalinity, and this is ascribed to the presence of other sodium salts. It is computed that the loss due to absorption from black liquor amounts to 4—8 kg. of sodium sulphate per ton of cellulose.

A. GEAKE.

Determination of lignin in wood and wood cellulose. W. J. MÜLLER and W. HERMANN (Papier-Fabr., 1926, 24, 185).—After dissolution of the cellulose by any of the known methods, the solution, containing the lignin in suspension, is filtered through a layer of finely-divided naphthalene in a glass or porcelain filter-crucible. To prepare the filter a suitable volume (e.g., 10—20 c.c.) of a 5% solution of naphthalene in alcohol is poured into twice its volume of water, and the paste thus obtained introduced into the filter, pressed down, and washed. After use the naphthalene is sublimed over a water-bath and the lignin is thus obtained pure and loose.

A. GEAKE.

PATENTS.

Manufacture of cellulosic material for paper, textiles, and the like. A. L. BURLIN, S. LEICESTER, and L. B. HOLMAN (E.P. 248,118, 10.12.24).—Cellulosic material containing lignone, or of a silicious character, is treated with bleaching powder (0.5 lb. per gall.) and sodium bicarbonate (2–4 lb. per 100 lb. of material) at a relatively low temperature, *e.g.*, 28°. When the cellulose is lignified it may have a preliminary treatment with an organic acid, *e.g.*, benzoic acid. A final treatment with a fixing agent, *e.g.*, alum, may be given to prevent subsequent discoloration. The process may be applied to mechanical wood pulp, or to the manufacture of paper pulp from waste jute material. A. GEAKE.

Manufacture of cellulosic products [esters]. H. DREYFUS (E.P. 249,173, 25.9.24).—Wood pulp or similar material is converted into cellulose sufficiently pure for esterification, by repeated or prolonged treatment with hot or boiling solutions containing $\frac{1}{4}$ –3% (preferably not more than 2%) of an alkali hydroxide, or with solutions at the ordinary or at slightly raised temperatures, containing about 5–10% of an alkali hydroxide, followed by boiling with dilute or, preferably, with glacial acetic acid, or with other lower aliphatic acids. The alkali hydroxides may be replaced by other basic materials, such as sodium carbonate, potassium carbonate, barium hydroxide, sodium zincate, sodium aluminate, ammonia, or organic bases, in which case higher concentrations are used, and, if necessary, the treatment is effected under increased pressure. In treating mechanical wood pulp or other material from which lignocellulose, resins, and similar constituents have not been removed in a preliminary process, the basic material is used in sufficient quantity to ensure elimination of these constituents and to bring the cellulose into the desired condition. L. A. COLES.

Manufacture of filaments and films from viscose. W. MENDEL (U.S.P. 1,576,529–31, 16.3.26. Appl., 29.4.25).—Undesirable sulphur-containing constituents are removed from cellulose products by treatment with trisodium phosphate; thus viscose is so treated after precipitating (A) in an acid bath, (B) in an aqueous solution containing sodium hydrogen sulphite, or (C) by heat. A. GEAKE.

[Lamp] wicks. F. D. MILES, and NOBEL'S EXPLOSIVES CO., LTD. (E.P. 249,571, 26.9.24).—Wick is tendered by heating with hydrochloric or nitric acid and the surface then reinforced by treatment with a nitrocellulose solution. Wick so treated is easily cut level and is not distorted in use. A. GEAKE.

Treating viscose silk in a vacuum. A. KÄMPF (U.S.P. 1,579,711, 6.4.26. Appl., 26.3.25).—See E.P. 232,200; B., 1925, 914.

Artificial horn (E.P. 230,025).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cotton: affinity of derivatives of dehydrothiitoluidine and primuline. P. RUGGLI and

S. M. PESTALOZZI (Helv. Chim. Acta, 1926, 9, 364–378).—A cotton skein weighing 5 g. was boiled for 1 hr. with 0.15 g. of dye and 100 c.c. of distilled water and the amount of dye absorbed from solution was determined. The skein was then rinsed and treated with 100 c.c. of boiling water and the dye stripped out estimated after 24 hrs. The difference between these two values, expressed in % of the amount of dye originally taken is proposed as an empirical measure of the affinity of a dye for cotton. The affinity is estimated for a range of substances in the dehydrothiitoluidine and primuline classes. The affinity of the sodium salt of dehydrothiitoluidine-3-sulphonic acid is almost negligible but it can be developed by introduction into the molecule of suitable substituents such as nitro- or amino-groups. The affinity is markedly increased by introduction of a second thiazole nucleus to give the corresponding primuline-sulphonic acid. In the azo-derivatives of these compounds, however, the lower homologue shows, in the cases considered, the higher affinity, although the primuline dyes are less readily stripped from the cotton. The choice of coupling components in the preparation of these derivatives has considerable influence on the affinity. The dehydrothiitoluidine and primuline azo-dyes, as a class, show small affinity compared with that of the bisazobenzidine dyes. When the dyeing process is carried out in the presence of sodium sulphate, an increased affinity is obtained, notwithstanding the greater ease with which the dye is stripped from the cotton afterwards. The degree of dispersity of the dye in solution is considered to be an important factor in determining the affinity.

M. CLARK.

Behaviour of different starches towards dye-stuffs and iodine. I. J. HUEBNER and K. VENKATARAMAN (J. Soc. Dyers and Col., 1926, 42, 110–121).—An investigation, using titration and colorimetric methods, of the amount and rate of absorption of direct, acid, and basic dyes from cold aqueous solutions by ungelatinised granules of maize, potato, rice, sago, tapioca, and wheat starches. Basic dyes are absorbed to a greater extent than acid and direct dyes by starch, the amount of absorption varying with different starches and the resulting shades of the dyed starch being (in several instances) unrelated to the amount of dye absorbed. Starch granules may be uniformly dyed throughout by means of Prussian blue, but they have no affinity for picric acid. Starch dyed with most dyes is fairly fast to washing with water. The absorption by starches of a basic or acid dye from solutions of increasing concentration approaches a maximum but the corresponding absorption of a direct dye is irregular. The maximum amounts of Magenta and Methylene Blue absorbed by potato starch and of Crystal Scarlet by wheat starch are 0.923%, 0.473%, and 0.195% respectively. The power of various starches to absorb basic dyes decreases in the order, rice, maize, potato, tapioca, wheat, and sago, but maize and potato, and tapioca and wheat starches respectively frequently interchange places. The rate of absorption of Methylene Blue from aqueous solutions

is nearly complete within a few minutes. The absorption of Benzopurpurine 4B is slightly and considerably increased by the addition of sodium phosphate and sodium chloride respectively. The affinity of all starches except sago for basic dyes is decreased after gelatinisation and drying. Starched and ordinary bleached cotton fabric, absorb Methylene Blue equally, but after dyeing the latter has the deeper shade. Absorption of Methylene Blue by starch is considerably decreased by the presence of alcohol. Starch absorbs copper but not iron salts from aqueous solutions; potato starch absorbs 0.37% of copper sulphate from a 0.2% solution. Starches absorb 0.85—2.66% of tannic acid.

A. J. HALL.

[Printing] white and red discharges on indigo grounds. J. POKORNY (Sealed Notes 2318 and 2328, 24.4 and 22.6.14. Bull. Soc. Ind. Mulhouse, 1925, 91, 759—761. Report by P. SEYDER, *ibid.*, 762—764).—During investigations on a printing paste containing lead chromate, manganese dioxide, and sodium chlorate for obtaining white or coloured discharges on indigo-dyed fabric, Pokorny found that the manganese dioxide and sodium chlorate were not essential for coloured discharges and that white discharges could be obtained without sodium chlorate, but with a smaller proportion of manganese dioxide. For example, in obtaining red discharges, cotton dyed with indigo is mercerised, prepared with β -naphthol, printed with a paste containing 4800 g. of lead chromate, 9325 g. of gumtragacanth + starch thickening, 6575 g. of diazotised *p*-nitroaniline, and 3000 g. of water, then passed during 3—5 sec. through hydrochloric acid of 17° B. (d 1.13) at 37°, steamed for 35—40 sec. at 35°, and washed. White discharges are obtained by means of a similar discharge paste containing 4800 g. of lead chromate, 7000 g. of gum tragacanth + starch thickening, 3000 g. of water, and 600 g. of manganese dioxide, and are cleared by soaping. Seyder confirms the excellent results obtained by the processes but indicates their similarity to that mentioned by the B.A.S.F. ("Indigo pur," 179). The discharged portions of the fabric absorb Methylene Blue strongly and appear to contain oxycellulose.

A. J. HALL.

Exposure tests with the Osram Pointolite lamp. E. HOCHHEIM and E. KNEBEL (Melliands Textilber., 1925, 6, 914—916; Chem. Zentr., 1926, I, 1720).—An arc lamp filled with an inert gas and containing two hemispherical tungsten electrodes which are separated with formation of an arc (when the electric current is first passed through the lamp) by the expansive force produced by the heating of a bimetallic strip, gives light, particularly when the red rays are reduced by passage through a screen of water, which is especially suitable for carrying out fading tests and the comparison of colour tones. The light obtained from the lamp is uniform over considerable periods and is suitable for carrying out quantitative measurements.

A. J. HALL.

PATENTS.

Treating [mercerising] textiles. F. B. VOEGELI (U.S.P. 1,577,393, 16.3.26. Appl., 9.2.25).

—Textile goods are squeezed and mangled during treatment with hot caustic liquors, then cooled and stretched.

A. J. HALL.

Imparting a linen-like character to cotton. H. MATT (E.P. 244,485, 11.12.25. Conv., 11.12.24).—Cotton yarns of less than 60's count and fabrics made therefrom are given a permanent linen-like appearance by treatment for not less than 4 min. at 0—5° with sulphuric acid of 49—50° B. (d 1.516—1.532), being subsequently well washed and mercerised under tension by means of caustic soda of 15° B (d 1.116) at ordinary temperatures exceeding 0°.

A. J. HALL.

Production of colours or lustre on natural or artificial fibrous material. N. V. NEDERLANDSCHE KUNSTZIJDEFABRIEK (F.P. 597,231, 25.4.25. Conv. 30.12.24).—A precipitate of sulphur is produced locally on parts of the material, and dyeing is then carried out. The material may be dipped in a solution of alkali polysulphides and treated with acid, or soaked in a solution of sulphur in an organic solvent with subsequent evaporation of the latter. The presence or absence of sulphur on the fibre results in different tints on dyeing or treatment with metal salts. For viscose silk the desulphurising process is omitted. If viscose is treated with desulphurising agents after dyeing a non-lustrous fibre is obtained. For the production of patterns the sulphur is removed only in parts.

B. FULLMAN.

Bleaching organic materials. R. HAMBURGER and S. KAESZ (U.S.P. 1,580,136, 13.4.26. Appl., 16.11.23).—See E.P. 209,073; B., 1924, 508.

Means for facilitating the mercerisation or like treatment of yarn samples. P. CALDWELL, and BRIT. COTTON and WOOL DYERS' ASSOC., LTD. (E.P. 250,392, 6.4.25).

Azo-dye (U.S.P. 1,576,322).—See IV.

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

Absorption of chemical fogs. Determination of sulphur trioxide in moist burner gases. H. GILLE (Z. angew. Chem., 1926, 39, 401—402; cf. Remy, B., 1926, 255).—The sulphur trioxide, together with the sulphur dioxide, produced by roasting pyrites, can be absorbed by passing the dry gases through caustic potash solution, and so estimated. After the gases have passed the washer, the sulphur trioxide appears in the form of a moist fog, which is not completely absorbed by passage through several wash-bottles. It is removed, however, by passing the gases through a 10 cm. length of (glass) wool, from which it can subsequently be recovered by washing, and estimated. The latter procedure, even with moistened wool, fails to remove all the sulphur trioxide from the dry gases. A. B. MANNING.

Treatment of crude potassium salts. M. PAPPÉE (Kali, 1925, 19, 174—179, 217—223, 235—241, 260—265, 275—281, 300—305, 321—324, 356—364,

402—406; Chem. Zentr., 1926, I, 1872).—A review of processes for the extraction of potassium salts from potash deposits is given, including the treatment of the silicious residues. A detailed description is given of large-scale tests on the elutriation process in the extraction of carnallite, using agitating tables, magnesium sulphate, calcium sulphate, and insoluble salts being separated at various stages in the process. The concentration and nature of the washing solutions are important, and the size of the crystals must be considered. Results obtained from various plants are tabulated. Potassium chloride and kieserite can be separated on the agitating table by the use of suitable extracting and washing liquors.

B. W. CLARKE.

Colour reaction of saponin with nitrates. C. A. MITCHELL (Analyst, 1926, 51, 181).—In the test for nitrates pure commercial saponin (B.D.H.) was successfully used to replace brucine or strychnine. If 1 drop of concentrated sulphuric acid is added to minute quantities of saponin and a nitrate, the blood-red coloration produced appears to be proportional to the amount of nitrate, and apparently to that of the saponin present.

D. G. HEWER.

Chemistry of bleaching powder. S. OCHI (Trans. Amer. Electrochem. Soc., 1926, 49, 137—146. Advance copy; cf. B., 1924, 54, 130, 131, 253).—The development of crystals of $\text{Ca}(\text{OCl})\text{Cl}\cdot\text{H}_2\text{O}$ (cf. B., 1923, 603 A) depends on the conditions of manufacture and the nature of the materials; it is favoured by slow chlorination at low temperature in absence of water. In the manufacture it is considered that the hypochlorous and hydrochloric acids, formed by solution of chlorine in the water adhering to the slaked lime, neutralise the lime with the formation of a solid solution of oxychloride, chloride, hypochlorite, and basic oxychloride of calcium from which crystals of $\text{Ca}(\text{OCl})\text{Cl}\cdot\text{H}_2\text{O}$ are deposited. All oxygen-forming decompositions of bleaching powder are exothermic; all chlorine-forming decompositions are endothermic. The equilibrium pressures of oxygen and chlorine over $\text{Ca}(\text{OCl})\text{Cl}\cdot\text{H}_2\text{O}$ were determined by Nernst's heat theorem. A powder with 42—45% of available chlorine was prepared by chlorination of very finely divided slaked lime. S. K. TWEEDY.

Increasing the internal volume of silica gels by moist heat treatment. H. N. HOLMES, R. W. SULLIVAN, and N. W. METCALF (Ind. Eng. Chem., 1926, 18, 386—388).—Silica gel prepared by adding ferric chloride to a solution of sodium silicate, and boiling the gelatinous precipitate with hydrochloric acid, has a high absorptive capacity, absorbing 62% of benzene vapour against 32% for a standard silica gel. This is owing to the porous nature of the gel produced by the extraction of ferric hydroxide during the boiling with acid. The absorption can be enormously increased by slow drying of the gel; further, if the partially-dried gel with a water content of about 60%, is submitted to syneresis in a closed vessel for 2—3 weeks, before boiling with acid, a gel which absorbs up to 133% of benzene vapour can be obtained.

B. W. CLARKE.

Economics of chlorine. D. A. PRITCHARD (Trans. Amer. Electrochem. Soc., 1926, 49, 73—85. Advance copy).

PATENTS.

Stabilising liquid hydrocyanic acid. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,577,057, 16.3.26. Appl., 16.9.25).—Stabilisation of liquid hydrocyanic acid is effected and corrosion of the iron container prevented by adding 0.1—0.2% of a halogen derivative of the acid, e.g., cyanogen chloride, or its polymer, cyanuric chloride.

R. B. CLARKE.

Distillation of persulphuric acid and of solutions of persulphuric-acid salts. G. BAUM (U.S.P. 1,577,201, 16.3.26. Appl., 5.5.24).—Heat is supplied directly to the liquid by an alternating current of a frequency below 500.

H. HOLMES.

Production of barium hydroxide from barium sulphide. A. JAHL (E.P. 249,402, 29.8.25. Conv., 18.4.25).—1000 kg. of porous, crude barium sulphide, containing 65% BaS , is slowly stirred with 250 litres of a solution of barium polysulphide, containing 36% BaS_4 . Mixing is effected in such a way that the mass remains dry. After 20—30 min. the mass is leached with boiling water. A concentrated solution of 160 kg. of sodium hydroxide is added to the liquor leaving the leaching vats at 60—65° and on cooling nearly all the barium is deposited as barium hydroxide. If the liquor is cooled without addition of alkali only half the amount of hydroxide is precipitated, but, on passing carbon dioxide into the mother liquor a mixture of barium carbonate and sulphur is obtained. This yields barium sulphide when calcined with carbon.

R. B. CLARKE.

Distillation of crude ammoniacal liquor in a column apparatus. C. STILL (G.P. 423,849, 26.9.22).—The distillation is carried out without the use of foreign vapours for heating. The liquor freed in the first column from the greater part of the volatile ammonium compounds, is led into a boiler before treatment with lime. The steam containing carbon dioxide from the boiler is passed into the lower part of the second column, and the hot liquor together with lime is introduced into the upper part. The method offers heat economies. B. FULLMAN.

Evaporation and concentration of [salt] solutions. C. COLLARD (E.P. 227,843, 14.1.25. Conv., 14.1.24).—The process described in E.P. 166,896, may be applied to the concentration and evaporation of salt solutions by using an elongated cylindrical drum divided into compartments by steam-tight partitions. Each compartment has at the bottom an inlet nozzle for the hot solution and steam coming from the heater and sprayer and an outlet pipe to convey the unevaporated solution to the next stage, and at the top a vapour outlet pipe opening into a common main. S. BINNING.

Recovering sodium chloride from saline liquors. A. W. GAUGER and H. H. STORCH, Assrs. to BURNHAM CHEMICAL Co. (U.S.P. 1,576,739, 16.3.26. Appl., 16.2.25).—Brine is evaporated until

sodium chloride, with its impurities, crystallises. The crystals are separated, ground, and purified by washing with the original dilute brine.

C. IRWIN.

Preparation of sodium sulphide or similar inorganic fusible chemicals in the form of small lumps. CHEM. FABR. KUNHEIM & Co. (E.P. 233,316, 17.3.25. Conv., 1.5.24).—Sodium sulphide or similar compounds are obtained in tablet form by allowing drops of the fused salt of the desired size to fall vertically on to a cold hard surface of metal or ebonite, covered if necessary with vaseline. The shape of the tablets is determined by the temperature of the fused salt and the height through which the drops fall.

C. IRWIN.

Manufacture of alkali hydrosulphites [hyposulphites]. FARBENFABR. VORM. F. BAYER & Co. (E.P. 247,523, 12.12.25. Conv., 13.2.25).—Considerably increased yields of hyposulphite are obtained from alkali amalgam and bisulphite solution if a portion of the mother liquors be removed periodically or continuously, and replaced by water or a salt solution.

R. B. CLARKE.

Manufacture of barium nitrate. D. TYRER (E.P. 248,593, 6.5.25).—Witherite is dissolved in hydrochloric acid and the saturated solution of barium chloride obtained treated with nitric acid (*d* 1.40). Crystalline barium nitrate is precipitated and the mother liquor is returned for use in a further extraction. Much larger yields are obtained in proportion to the materials handled than in the direct treatment of witherite with nitric acid, on account of the low solubility of barium nitrate.

C. IRWIN.

Making bleaching earth from slag. ERDWERKE MÜNCHEN O. LIETZENMAYER (E.P. 248,639, 17.8.25. Conv., 25.3.25).—Undried clay is ground to extreme fineness and mixed with sufficient acid to convert impurities into soluble salts. It is then dried to a paste which still contains free acid, lixiviated with water to remove soluble salts, and the sludge dried and ground.

C. IRWIN.

Manufacture and manipulation of colloidal or semi-colloidal substances, precipitates or sediments and the recovery of the liquid or solid components. SPENCER CHAPMAN & MESSEL, LTD., and J. B. LIEBERT (E.P. 249,647, 19.2.25; cf. E.P. 236,087; B., 1925, 671).—A turbid solution of a salt or oxide of titanium, thorium, iron, etc., can be quickly clarified, without settling, by adding a small amount of glue of animal origin, stirring in a light, inert substance which presents a large surface to the solution, *e.g.*, asbestos, and finally filtering.

R. B. CLARKE.

Article of manufacture. [Calcium silicate derivative.] H. A. ENDRES, Assr. to CELITE Co. (U.S.P. 1,574,380, 23.2.26. Appl., 23.6.25).—60 pts. of finely divided diatomaceous earth are heated at 100° with 56 pts. of lime and 1000 pts. of water for 3 hrs. A vigorous reaction takes place with for-

mation of hydrated calcium silicate, which is calcined at 550–900° preferably in presence of carbon dioxide to remove free lime. The product is a very finely divided powder, the average particle size being 1 micron. It is of value as a filler, a filter aid, and a decolorising agent, and as an ingredient of cements.

T. S. WHEELER.

Purifying aluminium nitrate. T. MEJDELL and O. RAVNER, Assrs. to NORSK HYDRO-ELEKTRISK KVÆLSTOFAKTIESELSKAB (U.S.P. 1,575,634, 9.3.26. Appl., 27.2.22).—Hot and cold solutions of aluminium nitrate are mixed, the conditions of temperature and concentration being so regulated that crystallisation occurs. Iron compounds remain in solution.

C. IRWIN.

Method of making aluminium chloride. A. McD. McAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,578,052, 23.3.26. Appl., 1.3.23).—Chlorine is passed over a mixture of carbon and aluminium oxide. The period of induction in the reaction is shortened by mixing an oxide of carbon with the gas until the reaction starts.

W. N. HOYTE.

Manufacturing arsenates. D. LOPEZ (U.S.P. 1,578,150, 23.3.26. Appl., 2.5.24).—A slurry consisting of calcium hydroxide (5% of which is freshly precipitated) and water at 30–50° is continuously circulated, by means of a centrifugal pump, from the bottom of an open tank, with a conical bottom, to a tubular oxidising chamber extending from above the top to the bottom of the tank. Arsenious oxide is added to the slurry in the tank, which is provided with a stirrer, and chlorine gas is passed into the oxidising chamber. Tri-calcium arsenate and scarcely any water-soluble arsenate is formed. Milk of lime is added after completion of the oxidation and a light, fluffy product suitable for use as an insecticide is obtained.

R. B. CLARKE.

Making oxidised leaden powder [litharge]. C. A. HALL (E.P. 248,602, 26.5.25).—The known process for the preparation of litharge by blowing air through a rotating drum containing lead balls is modified by cooling the drum with water. This enables the speed of the drum and the quantity of lead under treatment to be increased. The temperature within the drum is not allowed to rise above 182°. In a drum 6 ft. diam. and 10 ft. long, making 14 revs. per min., 480 lb. of lead per hr. can be oxidised.

C. IRWIN.

Manufacture of iron carbonyl. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 248,683, 14.12.25. Addn. to 244,895; B., 1926, 191).—In the preparation of iron carbonyl by the action of carbon monoxide on iron, pressures below 50 atm., even down to 1 atm., may be used if there is sufficient speed in the gas current to avoid deposition of liquid iron carbonyl on the mass. Reaction gases containing 4–6% of iron carbonyl are obtained. Small amounts of ammonia, methyl alcohol, or formaldehyde accelerate the reaction, but even traces of oxygen or carbon dioxide may render the surface of the iron inactive.

C. IRWIN.

[Making] zirconium carbide. M. L. HARTMANN, Assr. to CARBORUNDUM Co. (U.S.P. 1,576,275, 9.3.26. Appl., 13.3.24).—A zirconium silicate ore is fused in an electric furnace under reducing conditions.

C. IRWIN.

Extraction of hydrogen contained in industrial gases and chiefly in coke-oven gas. AMMONIA (E.P. 242,583, 23.12.24. Conv., 6.11.24).—Hydrogen is separated from coke-oven or other industrial gas, after the preliminary removal of water vapour, carbon dioxide, and heavy hydrocarbons, by condensing the remaining components from the compressed gas, utilising, once the apparatus is in operation, the cold produced by the expansion of the residual hydrogen, and using liquid nitrogen as an additional external source of cold. The gas mixture is cooled successively in two separators, in one of which the cooling agent is liquid methane, and in the other mainly liquid nitrogen and finally expanded hydrogen. The hydrogen so separated is passed through liquid nitrogen, the expansion of the hydrogen being so adjusted that the two gases issue from the apparatus in the correct proportion for the synthesis of ammonia.

A. B. MANNING.

Making graphite. V. M. WEAVER (U.S.P. 1,576,883, 16.3.26. Appl., 2.3.23).—At the bottom of a trough-shaped furnace having an insulating lining of powdered lampblack is placed a slab of carbon, surrounded by a vertically movable frame. The space inside the frame is charged with a mixture of lime and petroleum coke, a layer of the latter covering the main charge. A number of electrodes are arranged above the slab. Fluid calcium carbide is formed at first at the bottom of the furnace but is later converted into graphite and calcium; the latter volatilises and rises forming fresh carbide. The electrodes and frame are raised to keep pace with the reaction which takes place in zones progressively upward. The graphite finally obtained may be compressed while hot by transferring the whole furnace to a press.

R. B. CLARKE.

Lime burning process. I. WARNER (E.P. 250,045, 15.5.25).—See U.S.P. 1,542,195; B., 1925, 631.

Drying calcium hypochlorite compounds. CHEM. FABR. GRIESHEIM-ELEKTRON, Asses. of G. PISTOR (U.S.P. 1,572,952, 16.2.26. Appl., 26.3.25).—See E.P. 232,560; B., 1925, 714.

Treating crude oxide and carbonate of zinc. W. T. CHIDDEN and W. G. RAGG, Assrs. to CHANCE AND HUNT, LTD. (U.S.P. 1,579,302, 6.4.26. Appl., 28.11.23).—See E.P. 228,244; B., 1925, 241.

Production of ammonia from gases containing hydrogen cyanide. B. F. HALVORSEN, Assr. to NORSK HYDROELEKTRISK KVAELSTOFAKTIESELSKAB (U.S.P. 1,580,038, 6.4.26. Appl., 16.2.25).—See E.P. 231,134; B., 1925, 957.

Manufacture of hydrogen sulphide. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,580,451, 13.4.26. Appl., 4.4.21. Renewed 3.7.25).—See E.P. 209,171; B., 1924, 173.

Recovery of hydrogen sulphide. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,580,452, 13.4.26. Appl., 8.12.21. Renewed 3.7.25).—See E.P. 190,118; B., 1924, 173.

Separating hafnium and zirconium. D. COSTER and G. VON HEVESY, Assrs. to NAAML. VENNOOTS. PHILIPS' GLOEILAMPEN-FABR. (U.S.P. 1,580,650, 13.4.26. Appl., 13.3.24).—See E.P. 220,359; B., 1924, 869.

Determination of carbon monoxide (U.S.P. 1,578,666).—See II.

Preparation of mixtures of chromates and dichromates (G.P. 420,444).—See IV.

VIII.—GLASS; CERAMICS.

Baroni reaction for neutral glass for pharmaceutical purposes. L. TIRELLI (Boll. chim. farm., 1926, 65, 65—72).—For glass to be used for pharmaceutical purposes and particularly for holding material to be injected, Baroni (Boll. chim. farm., Nov. 30, 1925) has suggested the following test. A vessel of the glass, filled with distilled water and sealed in the flame, is immersed in steam at 2 atm. pressure for 1 hr., and, when cold, re-opened. When treated with 3—4% of its volume of a fresh alcoholic 1% solution of crystallised hæmatoxylin, the water should then exhibit no pink or violet-blue coloration within 24 hrs. The author finds that great care is necessary to ensure that the reagents are pure and to avoid contamination with dust, vapours, etc., from the air. With glass having a basis of silicates, the test gives a sensitive indication of removal of bases or metals, especially copper, from the glass. When, however, the glass contains an excess of borates, negative results leave the issue in doubt. The test should be used in conjunction with, and not instead of, that in which phenolphthalein is employed. It gives no indication of the loss by the glass of neutral salts, such as sodium sulphate, or of acidic residues, such as silica, borosilicic acid, etc., which might affect the keeping properties of special injections like colloidal metals, gelatinous sera, alkaloids, alkaline liquids, etc.

T. H. POPE.

Testing for refractoriness and after-contraction, and experiences with refractories in vertical [gas] retorts. T. F. E. RHEAD and R. E. JEFFERSON (Trans. Ceram. Soc., 1925—26, 25, 6—38).—See B., 1925, 501.

PATENTS.

Heating metal, glass or other material to render it workable. J. W. VAN METER (U.S.P. 1,572,975, 16.2.26. Appl., 5.6.22; cf. U.S.P. 1,573,784 p. 444).—Sawdust is mixed with zinc, iron, or the like and packed into a glass tube which it is required to bend. On passing chlorine through the tube the heat evolved by the action of the gas on the metal softens the glass. The sawdust is carbonised and forms a packing which prevents distortion of the tube. The latter is cooled and cleaned by

passing compressed air through it. Articles may be heated externally by surrounding them with a mixture of iron and sawdust and treating the latter with chlorine.
T. S. WHEELER.

Tunnel kilns. W. L. HANLEY, JUN. (E.P. 249,265, 26.1.25).—The firing chamber of a tunnel kiln consists of two firing zones separated by a longitudinal partition and heated by furnaces on either side of the chamber. Goods to be fired travel through the kiln on cars on parallel tracks in opposite directions, deflecting walls adjacent to the outer walls protecting the cars from direct heat. The products of combustion are removed through eduction flues, in which dampers are placed so that the temperatures of individual furnaces may be regulated. The firing zones terminate at either end in tunnel sections carrying two parallel tracks, forming combined preheating and cooling zones, in which unfired goods introduced into the kiln are heated by heat from the fired goods from the firing chamber travelling in the opposite direction. This method of kiln construction results in a considerable saving in first cost and operating charges and is specially applicable to the production of large quantities of cheap ware.

B. W. CLARKE.

Unburned refractory brick. R. H. YOUNGMAN (E.P. 250,480, 24.10.25).—See U.S.P. 1,564,394; B., 1926, 192.

Fritted quartz filters (E.P. 226,182).—See I.

Furnaces (E.P. 234,049).—See I.

IX.—BUILDING MATERIALS.

Corrosion of concrete. J. R. BAYLIS (Proc. Amer. Soc. Civ. Eng., 1926, 52, 549—579).—The corrosion of concrete under ordinary conditions appears to depend upon the solubility of calcium carbonate, which is determined by the alkalinity, hydrogen-ion concentration, and salt content of the surrounding water. The alkalinity of a concrete structure, *i.e.*, parts of calcium carbonate dissolved per million, is a good guide as to the state of the concrete; a value below 200 is an indication that disintegration is liable to occur rapidly. The porosity of the mortar binding the larger aggregate in the concrete is the most important factor in determining the actual rate of disintegration; a high porosity enables the disintegrating solution to penetrate more rapidly, and also accentuates the destructive influence of freezing.
B. W. CLARKE.

Durability of cement drain pipe and concrete in alkali soils: Fourth Progress Report (1923). G. M. WILLIAMS and I. FURLONG (Tech. Papers U.S. Bur. Standards, 1926, 20, [307], 191—240; cf. Wig and others, B., 1917, 649).—Concrete drain pipes and concrete blocks even of the best materials and manufacture, are severely attacked by prolonged exposure in soils containing considerable quantities of salts of the sulphate type, but less seriously where salts of the chloride or carbonate type predominate. The durability of cement drain pipe varies with the

richness and consistency of the mix, machine-made pipes giving the best results. Wet-mix pipes in general are more resistant to attack than dry-mix pipes, probably owing to decreased permeability. Owing to variations in salt concentration and moisture content from place to place and from season to season in the soil, it is impossible to estimate the maximum concentration to which the test-pieces may have been exposed. Adequate drainage reduces the average concentration of salts in the soil and lessens the rate of attack, and is essential for all concrete work in alkali soils. The presence of reinforcement appears to accelerate disintegration where the permeability of the concrete is high owing to the corrosion and expansion of the metal. The action of the sulphate waters appears to be on the lime set free in the setting of the concrete, producing a compound which occupies a greater volume than that of the original.
B. W. CLARKE.

PATENTS.

Cement objects having polished and translucent surfaces. SOC. "LAP," Assees. of S. SEAILLES (E.P. 234,846, 29.5.25. Conv., 30.5.24).—Special aluminous cements, containing less than 1.5% of iron, *e.g.*, composed essentially of monocalcium aluminate, are used in contact with polished moulds to form articles having a polished and somewhat translucent surface, which may be coloured or decorated.
B. W. CLARKE.

Production of lasting coloured stains in and upon the surface of cement or other building materials. J. KOEBIG (U.S.P. 1,577,729, 23.3.26. Appl., 1.11.24).—Two substances capable of interacting with formation of a coloured substance insoluble in water are dissolved in a solvent which will prevent the formation of the insoluble substance. The cement, the surface of which has been rendered porous by treatment with acid and subsequent washing, is impregnated with this solution. On penetrating the cement the solvent evaporates and the coloured insoluble substance is deposited. For instance, the greenish-yellow basic precipitate obtained by mixing aqueous solutions of cupric sulphate and sodium chromate is dissolved in ammonia solution to form the impregnating solution.
R. B. CLARKE.

Impregnating wood. J. PINTSCH A.-G. (E.P. 244,449, 28.11.25. Conv., 12.12.24).—Crude tar from gas producers is a suitable impregnating material for timber, having a low viscosity and high penetrating power at 80°, and a high viscosity at low temperatures, with a relatively high proportion of high-boiling constituents.
B. W. CLARKE.

Producing homogeneous, mouldable powders from fibrous [asbestos] cements. I. E. and O. E. LANHOFFER, Assrs. to S. J. DANNENBERG (U.S.P. 1,580,787, 13.4.26. Appl., 18.3.25).—See E.P. 199,350; B., 1924, 383.

Production of artificial stone, stoneware, and the like from boiler slag, dust slag, or ashes.

F. KÖRBER and W. HESSEL (U.S.P. 1,580,906, 13.4.26. Appl., 7.6.24).—See E.P. 218,275; B., 1925, 319.

Utilisation of liquid slag from gas producers etc. (G.P. 423,793).—See II.

Basic carbonate of magnesium (U.S.P. 1,573,603-4).—See VII.

Calcium silicate derivative (U.S.P. 1,574,380).—See VII.

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

Qualitative and economic importance of acid electric steel. A. MÜLLER-HAUFF (Stahl u. Eisen, 1926, 46, 213—218, 289—294).—The acid-lined electric furnace resembles a large crucible and the high quality of the steel produced is due to the action of nascent silicon and the deoxidation and degasification which occur. As carbon is only slowly reduced the scrap used should be as soft as possible. During melting 50—80% of the manganese present is burned away. The materials balance of an acid electric heat is given. The slag plays a less important rôle than in the basic electric furnace. The melting slag amounts to about 4% of the charge. In the deoxidising slag the silica increased up to 75%. The reduction of silica is an important reaction in the acid electric furnace; it is affected by the fluidity and temperature of the slag. A diagram shows the amount of silicon taken up in relation to the extent of the reacting surfaces. The acid electric furnace lining has a good resistance to oxides of manganese and iron, and the life of the roof is 3 to 4 times that of the basic furnace. The silica in the hearth decreases with service. Phosphorus is not reduced and sulphur only slightly during the oxidation period. Mechanical tests on steels with increasing phosphorus content show that not until 0.066% P is reached does deterioration occur in the reduction of area and impact test, the favourable structure compensating for high phosphorus content. A number of acid and basic carbon tool steels were hardened at 750—800°. The acid steel though not superior in mechanical strength showed greater regularity and was less sensitive to hardening temperature. Static and dynamic tensile tests and impact and repeated impact tests were made on medium carbon and nickel-chromium steels made in the acid electric and basic Siemens-Martin and electric furnaces. The electric steels were superior to the open-hearth steels and acid steel was better than basic as regards yield point, maximum strength, and repeated impact test, but slightly inferior in reduction of area. The acid electric process is expensive since pure scrap must be used for ingots, and is principally used for castings where purity need not be so high. The time required for melting and alloying high-carbon and nickel-chromium steels is half that for the basic furnace, and there is less loss of alloy elements in high-chromium and tungsten steels. The combination of a refining

furnace with an acid electric furnace is recommended as a quick method of working for high final quality. The cost for lining and upkeep of the acid electric furnace is less than for the basic. As the silicon content of acid steel tends to increase after deoxidation little time is available for the addition of ferro-alloys.

T. H. BURNHAM.

Present position of the basic hearth refining process in comparison with the Thomas process. F. BERNHARDT (Stahl u. Eisen, 1926, 46, 1—7, 39—44, 73—78, 137—142).—The pig iron-ore process is divided into two periods—refining and finishing. It may be carried out continuously or by heats, in a stationary or tipping furnace, and in one or more furnaces. The relative merits of these features are considered with reference to the Hoesch, Dortmund Union, Witkowitz-Talbot, and Königshütte processes. In the last-named refining is effected continuously in a tipping furnace and the finishing process is performed in a separate furnace. The metallurgical details of the process are given in a table including slag analyses at the various stages of operation. Dephosphorisation and decarbonisation take place in the finishing furnace without danger of rephosphorisation. For the same output the Witkowitz and Königshütte processes seem equally economical, but the latter possesses the advantage of better metallurgical control. Under all circumstances the use of tipping furnaces is advantageous. A comparison is made of Siemens-Martin with electric furnace and crucible steel. For certain purposes where the highest degree of deoxidation and degasification is required, crucible steel is unequalled, but steel can be made in the Martin furnace of a high degree of purity, and this furnace is now metallurgically equal to and more economical than the electric furnace. From the heat economy point of view, when the blast furnace and steel works are considered as a unit, the converter process does not excel the Martin furnace and as regards materials it is economically inferior. The ore and coke consumption and slag production are compared for the Thomas and the pig iron-ore and pig iron-scrap processes. Based on pre-war figures a simple formula has been deduced which shows when the Thomas and when the Siemens-Martin process is more advantageous, depending on local conditions. The cost of the construction of a Siemens-Martin installation is higher than that for a Thomas works of the same output, but in the first case the blast-furnace installation is smaller so that the total cost is approximately the same.

T. H. BURNHAM.

Heat treatment and testing of chromium magnet steel. E. H. SCHULZ and W. JENGE (Stahl u. Eisen, 1926, 46, 11—13).—The magnetic properties of chromium steel depend on the hardening temperature, the time the metal is held thereat, the rate of heating, and the size of the specimen. Tests on a steel containing 1% C and 2% Cr showed that the product of remanence and coercive force was a maximum for 10 min. heating at a hardening temperature of 800°. The theories of Gumlich (cf. B., 1922, 143 A) and Evershed (J. Inst. Elect. Eng., 1920, 58, 780) for the deterioration of magnetic properties

with continued heating at the hardening temperature are discussed. The authors consider that owing to the slow diffusion of the carbon in these steels the solid solution is not homogeneous, and on quenching the change starts at numerous centres in the interior of the crystals. The longer the temperature is maintained the less are the differences in concentration and the fewer the number of nuclei at which the change can start. The optimum time of heating should be determined in each case and for greater certainty should be checked by electrical resistance determinations. The finer the grain and distribution of the carbide before hardening the better are the magnetic properties, so that unnecessary annealing should be avoided. Simultaneously with the carbide going into solution, the deleterious processes of diffusion and grain growth set in. Undissolved carbide inclusions are not necessarily deleterious unless above a certain size. Although a high hardening temperature increases the rapidity of solution it must not be above a certain limit, as there is a greater risk of hardening cracks and of the formation of austenite. The finest distribution of carbides also gives the optimum magnetic properties to cobalt steel. For a steel containing 1.09% C, 16.0% Co, 8.8% Cr, and 2.4% Mo, the following treatment gave satisfactory results:—heated to 1200° and cooled in air, tempered at 750°, and subsequently hardened at 1000° and cooled in air. No definite conclusions on magnetic properties can be drawn from chemical composition. Maximum values can only be obtained in the absence of flaws in the steel and after the optimum heat treatment. Characteristic curves should be determined statistically, not from a single series of tests.

T. H. BURNHAM.

Pickling iron with hydrochloric and sulphuric acids. H. BABLIK (Stahl u. Eisen, 1926, 46, 218—222).—To determine which acid is the more efficient, discs of mild steel were immersed in 5% hydrochloric and sulphuric acids. Hydrogen produced by attack of the subjacent iron caused mechanical flaking of the scale, this action being more effective in the case of sulphuric than of hydrochloric acid. The specific pickling effect is greater for sulphuric acid and less acid is required, but the pickling period is longer. The pickling velocity of hydrochloric acid rises continuously with increasing concentration; for sulphuric acid the maximum is at 25% strength. At 60° the pickling velocity of both acids increased tenfold. For practical purposes a warm bath of sulphuric acid is required. The pickling velocity was decreased by the presence of reaction products. The amount of chlorides left on the surfaces, which have to be removed by washing is less than in the case of sulphates. Boiling water should be used for complete removal. Pickling with hydrochloric acid is handier and simpler but more expensive. The solution velocity of iron wire and sheet in acids of different concentrations was determined with and without the addition of 0.1 to 0.3% of Vogel's "VBZ" reagent. As only those reactions which liberate hydrogen were retarded by the VBZ reagent, it is thought that its action is not

due to adsorption but to its effects on the surface tension and viscosity of the pickling reagents which produce a mechanical hindrance to hydrogen evolution. By the use of Vogel's reagent a saving in iron and acid is effected and a smoother surface imparted to the iron.

T. H. BURNHAM.

Iron-nickel equilibrium diagram. L. GRENET (Rev. Mét., 1926, 23, 143—154; cf. B., 1925, 808).—The nature of transformations is discussed and the α - γ transformations in the iron-carbon, iron-nickel, and iron-cobalt series are considered in the light of the existence of a two-phase region between the α and γ phases.

M. COOK.

Cementation of ferrous alloys by chromium. J. LAISSUS (Rev. Mét., 1926, 23, 155—178; cf. B., 1925, 635).—On heating polished steel specimens containing 0.15% C in powdered ferrochrome the outside bright layer of carbide increases in thickness as the cementation temperature is raised, the intermediate zone of eutectic increases, and the interior zone of solid solution diminishes until after heating, for 5 hrs. at 1200° it entirely disappears. As the duration of heating is increased the solid solution formed reaches a maximum at 1100° after 5 hrs. and the carbide layer increases linearly. The process of cementation of electrolytic iron is similar to that of 0.15% C steel but the absence of carbon favours the formation of the carbide layer. Raising the carbon content to 0.4% and 0.85% causes a diminution of the solid solution zone, which is practically nil at 0.4% C, and lowers the temperature of formation of the carbide layer which decreases in thickness. The thickness of the layer formed in grey iron is greater than that formed in 0.4 and 0.85% C steels. With steel containing 13% Cr and 0.15% C the bright outside layer contains particles of carbide disseminated through it. Increasing the carbon content of the ferrochrome causes a decrease in the outer carbide layer until it reaches an approximately constant value, the eutectic gradually disappears, and the solid solution increases. No zone of solid solution exists after cementing with powdered electrolytic chromium, which produces the greatest thickness of carbide layer at 1200°. The addition of 45% of aluminium to the powdered chromium results in a thinner layer being formed.

M. COOK.

Heat treatment of aluminium-copper alloys. L. GUILLET and J. GALIBOURG (Rev. Mét., 1926, 23, 179—190; cf. B., 1926, 92).—The change in hardness, by quenching from 525° and tempering at 100°, 200°, and 300°, of a series of alloys containing from 5 to 50% Cu, has been studied. Owing to partial fusion at 525° alloys containing 45 and 50% Cu were quenched from 450°. The hardness immediately after quenching, for the whole series of alloys, was greater than that of alloys heated to 450° and slowly cooled, and in the case of the 20% Cu alloy the value was doubled. After annealing for 1 hr. at 100° immediately after quenching, the hardness was slightly less than that of quenched alloys, and annealing at 200° for 1 hr. caused further softening. The hardness was increased by prolonging the annealing

at 100°, the maximum hardness for this tempering temperature being attained in 48 hrs. The electrical resistivity reached a maximum in the quenched alloys after 48 hrs. at ordinary temperature, and fell as the tempering temperature was raised. In general the maximum hardness was attained at tempering temperatures which were lower as the copper content increased. The dimensions of a piston of 13% Cu alloy showed no change on heating for periods of 2 hrs. at 250° after water-quenching from 475°. The hardness became constant at about 84 (Brinell) after the fourth reheating. The addition of 1% Mg still further increased the hardness of the alloys after heat treatment. M. COOK.

Measuring polarisation and resistance of electrolytes. HARING.—See XI.

PATENTS.

Electrolytically treating ferrous metal objects. E. POTTER. From MADSENELL CORP. (U.S.P. 248,797, 11.9.24).—Prior to coating iron or steel with another metal, preferably nickel, either by electro-deposition or otherwise, it is made the anode in a cell containing sulphuric acid of at least 84.5% strength and a lead cathode. The initial voltage is 6–8 volts and the flow of current 20–50 amp. per sq. ft. The treatment is continued for about 3 min. until the flow of current drops to $\frac{1}{5}$ to $\frac{1}{10}$ of its initial value. After washing, the anode is coated with nickel by electrodeposition preferably within the critical period of about 1 min. required for maximum adherence. M. COOK.

Steel-like alloy for church bells. F. LANGE and P. STEINEL (G.P. 421,576, 10.2.22).—A steel-like alloy for church bells consists of iron with 2.8% C, 0.31% Mn, 0.64% P, 0.08% S, and 0.24% Si. A. R. POWELL.

“Tempering” metals and other materials. J. W. VAN METER (U.S.P. 1,573,784, 16.2.26. Appl., 9.6.22).—The method described in U.S.P. 1,572,975 (p. 440) is applied to “tempering” steel articles by the action of cyanogen. A cyanide is added to a mixture of sawdust and iron, and the heat generated by the action of chlorine liberates cyanogen from the cyanide. Alternatively steel heated by the method described may be treated with cyanogen gas under pressure. T. S. WHEELER.

Conversion of cuprous materials. M. H. MERRISS, Assr. to NICHOLS COPPER CO. (U.S.P. 1,576,776, 16.3.26. Appl., 1.8.25).—Blister copper is obtained from black copper by heating the latter until at least partially melted and blowing air in regulated quantity and under controlled pressure through the mass to effect rapid oxidation of the impurities. M. COOK.

Extracting metallic values from ore concentrates and the like. H. FLECK and W. G. HALDANE (U.S.P. 1,577,217, 16.3.26. Appl., 8.8.22).—Ore concentrates or slimes are mixed with a reagent

which will convert the silica present into a compound that will volatilise readily and this compound is then allowed to evaporate. A. R. POWELL.

Ore concentration. C. P. LEWIS, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,577,328, 16.3.26. Appl., 26.7.20).—Ore pulp is mixed with a flotation agent containing a heavy hydrocarbon carried in a hydrocarbon of the benzene series, and gaseous bubbles are brought into contact with the solid particles in the pulp so as to produce a mineral-bearing froth which is separated. M. COOK.

Recovering rare metals from their ores. K. B. THEWS, Assr. to W. A. J. BELL (U.S.P. 1,577,411 16.3.26. Appl., 12.7.21).—Carnotite ore is heated in the presence of a reducing agent and afterwards treated with an acid solvent to dissolve the vanadium, uranium, and radium present. M. COOK.

Electrolytic [metal] separation apparatus. A. DASBACH, Assr. to IRVINGTON SMELTING and REFINING WORKS (U.S.P. 1,577,898, 23.3.26. Appl., 11.7.24).—An apparatus for electrolytic separation of metals comprises a number of dissolving vats in which anodes and cathodes are supported. Scrapers interposed between the cathodes and anodes are supported on detachable bars mounted upon separate movable carriages. Detachable connexions are provided between the carriages, and means to permit the removal of each carriage as a unit. J. S. G. THOMAS.

White-gold alloy. E. M. WISE, Assr. to WADSWORTH WATCH CASE CO. (U.S.P. 1,577,995, 23.3.26. Appl., 28.10.25).—The alloy contains 30–80% Au, and nickel, copper, and zinc in the relative proportions of 33.0–53.1 pts. Ni, 26.4–46.5 pts. Cu, and 10.6–30.7 pts. Zn. A. W. HOTHERSALL.

Aluminium alloy. T. S. FULLER and D. BASCH, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,578,979, 30.3.26. Appl., 18.12.24).—The alloy contains zinc, 3–8%, magnesium, 0.25–1.5%, and the remainder aluminium. L. M. CLARK.

Purifying molten metals. H. G. LAPSLEY, Assr. to METALS REFINING CORP. (U.S.P. 1,578,044, 23.3.26. Appl., 4.8.25).—A chemical compound of aluminium and boron is added to the molten metal. A. W. HOTHERSALL.

Recovery of metals [copper, gold, silver] from their ores. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,578,618, 30.3.26. Appl., 12.11.23).—Ores containing gold, silver, and copper are treated with a cyanide solution, and the metals precipitated by a suitable precipitant. A soluble cyanide is regenerated in the solution, which is then used cyclically, the strength of the solution being such that during precipitation of the copper the solution contains more than 4 mols. of cyanide, calculated as sodium cyanide, per 1 mol. of copper in the solution. J. S. G. THOMAS.

Separating metal from metalliferous material. L. E. WEMPLE (U.S.P. 1,578,694, 30.3.26. Appl.

16.11.23).—A mixture of metalliferous material, solid carbonaceous fuel, and flux, in proportions sufficient wholly to reduce the material, is blown into a heated furnace chamber, the temperature of which is maintained high enough to volatilise the metal produced. The vaporised metal passes through a tortuous passage, and during its travel is oxidised, the oxidised metal finally being collected.

A. B. MANNING.

Reduction of metal oxides. E. B. PRATT (Can. P. 250,234, 12.6.24).—Mixtures of hydrocarbons with metal oxides, or material containing them, especially iron ore, are heated in tubes of refractory material, to a temperature at which the hydrocarbons are decomposed with formation of carbon, and reduction is completed by heating the material in an electrically heated crucible. L. A. COLES.

Apparatus for production of electrolytic iron. E. DUHME, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,570,212, 19.1.26. Appl., 17.6.24).—See G.P. 418,139; B., 1926, 163.

Apparatus for the manufacture of earth metals. H. DOLTER (U.S.P. 1,580,469, 13.4.26. Appl., 27.10.24).—See E.P. 240,163; B., 1925, 996.

Alloy. P. GIRIN, Assr. to SOC. ANON. DE COM-MENTRY, FOURCHAMBAULT & DECAZEVILLE (U.S.P. 1,580,662, 13.4.26. Appl., 16.4.19).—See E.P. 127,243; B., 1920, 412 A.

Apparatus for galvanising iron plates by the lead-zinc process. R. PASSEKER (E.P. 241,226, 12.10.25. Conv., 11.10.24).

Annealing and similar furnaces. H. GRÜNE-WALD (E.P. 250,373, 6.3.25).

Roasting furnace (U.S.P. 1,576,033).—See I.

Purifying gas-house liquor (U.S.P. 1,573,169).—See II.

Heating metal (U.S.P. 1,572,975).—See VIII.

XI.—ELECTROTECHNICS.

Dependence of electromotive force of [lead] accumulators upon acid strength. K. SCHREBER (Z. Elektrochem., 1926, 32, 143—149).—Two accumulators opposed to each other and containing unequal strengths of sulphuric acid tend to equalise their acid concentration during discharge. The E.M.F. difference is calculated by considering the work done in a reversible cycle in which the richer acid is diluted by condensation of water vapour, and the weaker acid is concentrated by evaporation. The theoretical values are different, depending on whether it is assumed, with Faraday, that the vapour arising from the acid solution has the same temperature as that of the vapour from the pure solvent at the same pressure, or whether, with Gay Lussac, it is supposed that the vapour has the same temperature

as the solution. The work in the latter case is greater. Comparison is made between theory and experiment, using results of Dolezalek and Thibaut. It is found that Faraday's assumption leads to satisfactory agreement within the experimental error, while, with Gay Lussac's assumption, this is not the case.

C. H. D. CLARK.

Small mercury-vapour lamp for laboratory use. W. CLARK (Phot. J., 1926, 66, 185—186).—The lamp consists of a quartz or water-jacketed glass test tube, with about 1 cm. of clean mercury at the bottom, and a carbon rod down the tube with its square-cut end 2 mm. or so above the mercury surface. Contact with the mercury is made by a wire down the inside of the tube, and the carbon rod is held in position by an asbestos string wad. The arc burns with a current of from 0.3 to 3 amp. Carbon should be negative to avoid a continuous background in spectrum work. The lamp is used for calibrating spectrographs and preparing wave-length scales. W. CLARK.

Laboratory [electric] furnace for high temperatures. J. and W. B. MITCHELL (Trans. Ceram. Soc., 1925—26, 25, 39—50).—See B., 1925, 511.

See also pages 426, **Electric ignition of firedamp** (WHEELER). 427, **Autoxidation of mineral oils** (MARCUSON and BAUERSCHÄFER). 452, **Test for rubber insulation** (HIPPENSTEEL).

PATENTS.

Non-sagging spiral filaments of highly refractory metal [for incandescence lamps]. N.V. PHILIPS' GLOEIILAMPENFABR. (E.P. 234,489, 20.5.25. Conv., 24.5.24).—Tungsten wire is wound into a spiral and heated to a temperature exceeding 1500°, which causes recrystallisation and prevents the filament from subsequently sagging in use, the sagging of helical wires in incandescence lamps being due to recrystallisation, which occurs principally during the first few hours of burning. The wire may be wound on a refractory core such as molybdenum or tantalum, which is removed by chemical treatment after the annealing operation. M. COOK.

Gas-filled electric lamps. GEN. ELECTRIC CO., Assees. of PATENT-TREUHAND-GES. FÜR ELEKT. GLÜHLAMPEN M.B.H. (E.P. 248,680, 28.11.25. Conv., 18.3.25).—The incandescent body or electrodes of an electric lamp may be prevented from volatilising to any appreciable extent by adding small quantities of hydrogen and hydrogen halide to the inert gas used for filling the lamp. The hydrogen and hydrogen halide mixture may be added direct to the filling, or suitable substances such as a mixture of hydrogen and tungsten chloride may be introduced, whereby hydrogen halide is produced when the lamp is running, tungsten being deposited on the filament. Alternatively, hydrogen halide alone may be introduced; this attacks the filament, hydrogen being liberated. This filling raises the average life of the

lamp and makes it more uniform; also, the metal sputtered from the filament is transformed into halides which are volatile at the temperature of the bulb and, if deposited on the bulb, produce only a translucent layer. The danger of short-circuiting is also diminished.
M. E. NOTTAGE.

Incandescence [electric] lamp. R. E. MYERS, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,572,670, 9.2.26. Appl., 25.6.21).—If glass free from lead, e.g., lime glass, be used for the manufacture of hydrogen-filled incandescence lamps, no blackening of the glass occurs during use. T. S. WHEELER.

Increasing the ultra-violet radiation from mercury vapour lamps made of quartz. H. GEORGE (E.P. 234,834, 29.5.25. Conv., 30.5.24).—The emission in the ultra-violet region is much increased when the lamp is supplied with alternating current at high frequency and high tension.

M. E. NOTTAGE.

Mercury vapour lamps. J. NISBET (E.P. 248,796, 11.9.24).—The patent relates to improvements in the design of mercury vapour lamps, especially for projecting pictures from transparencies or opaque objects on to a screen. M. E. NOTTAGE.

Electric gas purifier plants. SIEMENS-SCHUCKERTWERKE GES. M.B.H. (E.P. 240,844, 30.9.25. Conv., 1.10.24).—Dust from the electrodes of gas purifying plants is collected in pockets provided with inclined floors and leading to a common receptacle, arranged under each collecting electrode, spaces being left between them through which the gas to be purified can pass up to the electrodes.

M. E. NOTTAGE.

Devices for the chemical treatment of gases. METROPOLITAN-VICKERS ELECTRICAL CO., LTD., Asses. of L. H. HILL (E.P. 241,547, 9.10.25. Conv., 14.10.24).—A device for the chemical treatment of gases, to be used more especially in removing water or other deleterious substances from the air or gas drawn into oil-insulated transformers during "in-breathing," comprises a movable container for the active chemical, supported within a casing through which the gas passes, the relative position of container and casing in use indicating the condition or degree of exhaustion of the active chemical.

J. S. G. THOMAS.

Positive electrodes for electric batteries. SOC. ANON. LE CARBONE (E.P. 243,300, 15.12.24. Conv., 19.11.24; cf. E.P. 226,769; B., 1925, 555).—The efficiency of the depolarising medium of positive electrodes may be maintained by protecting it with an external covering of pectisable colloid, impermeable to liquids but permeable to gases. The depolarising medium, which may consist of a mixture of manganese dioxide, plumbago, and wood charcoal, moistened with a solution of ammonium chloride, is compressed on to the carbon positive electrodes forming channels, which permit of the access of the outer air to the interior of the composition. The liquid contained in the pores is then evaporated

either by stoving at 150° or by prolonged exposure to air, after which the external surface of the depolariser is covered with a colloidal pectised coating by immersing in or coating with a colloidal solution. The depolarising composition may also be contained either in a bag or in a porous pot open at the top, the colloidal covering being applied to the inner face of the vessel; or the grains of powder may be individually covered with a colloidal coating.
M. E. NOTTAGE.

Electric cells. C. GAISER (E.P. 248,942, 8.4.25).—The patent relates to various modifications of cells of the type in which the electrodes have the form of bands or threads which are gradually brought into action by contact of part with the electrolyte while the remaining part is kept outside, preferably wound up and is unwound according to requirements. A layer of absorbent material which may be impregnated with an electrolyte in dry form is placed between the electrodes. The positive electrodes preferably consist of magnesium or of a metal more electro-positive than magnesium, the negative electrodes of oxides such as copper oxide or lead peroxide, and the electrolyte of acid or alkaline compounds of magnesium.
M. E. NOTTAGE.

Electrolytes for lead accumulators. A. J. A. DE LA PORTE (E.P. 249,016, 28.8.25).—An electrolyte for lead accumulators which has a great regenerative capacity and can be used even in deteriorated accumulators consists of a solution of ammonium alum in dilute sulphuric acid in the proportions: 16 kg. of sulphuric acid, *d* 1.835, 30 litres of distilled water, and 2.5 kg. of ammonium alum; or 25 kg. of sulphuric acid, *d* 1.835—1.837, 80 litres of water, and 5 kg. of alum.

M. E. NOTTAGE.

Electric-furnace system. J. W. HARSCH, Assr. to LEEDS & NORTHRUP CO. (U.S.P. 1,578,027, 23.3.26. Appl., 20.2.25).—The temperature of the chamber of an electric furnace is raised rapidly, and air simultaneously circulated within the chamber, the direction of circulation being reversed at intervals which are short compared with the time taken by the chamber to attain the desired temperature.

J. S. G. THOMAS.

Resistance element. W. OTTO, Assr. to NEDERLANDSCHE TECHNISCHE HANDEL MAATS. "GIRO" (U.S.P. 1,577,981, 23.3.26. Appl., 14.2.24).—An electric resistance comprises two portions composed of a mixture of finely divided graphite and at least partially vulcanised rubber in different proportions, the graphite in at least one of the portions being embedded in the rubber.
J. S. G. THOMAS.

Preservation of oil used in electrical apparatus. MASCHINENFABR. OERLIKON (G.P. 425,051, 6.5.24. Conv., 14.4.24).—Acids formed in oil contained in electrical apparatus are neutralised without the oil being attacked, by maintaining the oil in contact with "octahedral" borax, Na₂B₄O₇·5H₂O. For example, textile material which has been steeped

in hot, saturated borax solution and dried is suspended in the oil.

L. A. COLES.

Electrical treatment of liquids. E. ANDERSON, ASSR. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,578,624, 30.3.26. Appl., 20.11.23).—A colloidal suspension of a material dispersed in a poorly conducting liquid is subjected to the action of a silent electric discharge produced in a gas in contact with the liquid, whereby the suspended material is separated from the liquid.

J. S. G. THOMAS.

Insulating bodies and method of producing them. C. L. DAWES and W. A. BOUGHTON, ASSRS. to NEW ENGLAND MICA Co. (U.S.P. 1,578,812-3, 30.3.26. Appl., 26.10.22).—(A) Mica fragments are mixed with an inorganic binding material, which at atmospheric pressure becomes fluid at a temperature substantially as high as the disintegration temperature of mica, and the mixture is heated to a temperature below that at which mica disintegrates, pressure being applied to cause the binding material to flow at that temperature. (B) A mixture of mica fragments and an alkali silicate containing substantially no water is subjected to pressure.

J. S. G. THOMAS.

Electric furnaces. W. A. LOKE (U.S.P. 1,580,060, 6.4.26. Appl., 2.6.25).—See E.P. 239,249; B., 1925, 886.

Electrode connexions for electric furnaces. M. MCGEE (E.P. 250,002, 21.3.25).

See also pages 438, Distillation of persulphuric acid (U.S.P. 1,577,201). 440, Graphite (U.S.P. 1,576,883). 444, Electrolytically treating ferrous metals (E.P. 248,797); Electrolytic apparatus (U.S.P. 1,577,898). 456, Purification of glue and gelatin (U.S.P. 1,577,660).

XII.—FATS; OILS; WAXES.

Bromometric examination of fats. H. P. KAUFMANN (Z. Unters. Lebensm., 1926, 51, 3—14; cf. B., 1925, 302).—The activity of bromine in solution in various solvents is discussed and a 0.1 *N*-solution in absolute methyl alcohol saturated with sodium bromide is recommended for the determination of the iodine value of fats. Such a solution, probably due to the formation of sodium tribromide, is very stable and loses only 1% of its active bromine in 56 days. It is almost odourless and can be measured with a pipette without danger. The determination is simply and quickly performed with an accuracy comparing favourably with the Hanus method.

E. H. SHARPLES.

Use of thiocyanogen in fat analysis. H. P. KAUFMANN (Z. Unters. Lebensm., 1926, 51, 15—27; cf. B., 1926, 165).—The "thiocyanometric" iodine values of mixtures of fats have been determined. From a knowledge of this value and the usual bromometric iodine value of a mixture of three fats and its constituents the composition of the mixture may be calculated. For a mixture of two fats, the "thiocyanometric" iodine value alone

offers no advantage over the usual iodine value owing to the smaller difference in the values of the single components.

E. H. SHARPLES.

Rapid method of determining the iodine value with iodine and alcohol. W. AUSTEN (Pharm. Zentr., 1926, 67, 209—210).—In carrying out the method of Margosches, Hinner, and Friedmann (B., 1924, 639) it is of importance that 200 c.c. of water be added to the fat solution, and that the minimum time of contact be 6 min. Observing these precautions, lard, beef tallow, and linseed oil gave iodine values of 58, 35.5, and 172.8 against the Hübl values of 58, 36.4, and 173.4 respectively.

A. RAYNER.

New value for milk-fat. J. KUHLMANN and J. GROSSFELD (Z. Unters. Lebensm., 1926, 51, 31—42).—The "butyric acid value" is a modification of the Reichert-Meissl value and is a measure of the water-soluble volatile fatty acids of lower molecular weight than caprylic acid (coconut oil fatty acids). 5 g. of fat are saponified with 2 c.c. of potassium hydroxide solution (75%) and 10 c.c. of glycerol, and the soap solution is diluted with 100 c.c. of water at 100°. The mixture is cooled to 20° and 50 c.c. of dilute sulphuric acid (2.5%), 15 g. of anhydrous sodium sulphate, 10 c.c. of coconut oil soap solution (100 g. of coconut oil saponified with 100 g. of glycerol and 40 c.c. of 75% potassium hydroxide solution and the solution diluted to 1 litre with water), and about 0.1 g. of kieselguhr, are added. After shaking for 10 min. the liquid is filtered and 125 c.c. of the filtrate are diluted with 50 c.c. of water and distilled with the usual precautions. The first 110 c.c. of the distillate, which contain about 96% of the total butyric acid, are directly titrated with 0.1 *N*-alkali and from the result is subtracted that given by a blank without fat. The difference, multiplied by 1.4, is the butyric acid value. The value for coconut-oil is 0.9 and for butter-fat 20, this latter being directly proportional to the Reichert-Meissl value. By the application of small corrections for coconut oil, the amount of milk-fat in admixture with other fats can be calculated from the butyric acid value and the saponification value to within an accuracy of 1%.

E. H. SHARPLES.

Simplified determination of milk fat in fatty mixtures. J. GROSSFELD (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 49—51, 65—66, 84—85, 97—99, 114—115, 130—132; cf. Kuhlmann and Grossfeld, B., 1926, 165).—The results obtained by the method of Bertram, Bos, and Verhagen (*loc. cit.*), and the distillation method of Gilmour (B., 1925, 564), are compared and the principle of these methods extended and made more sensitive by the determination of saponification value and butyric acid value. The ratio between the factors for milk fat and coconut oil is 3.8 for the Reichert-Meissl value, 12 for the "B" value, 20 for the Gilmour distillation value, and 81 for the butyric acid value (cf. preceding abstract). The probable content of milk fat, *M*, for a mixture having the butyric acid

value, b , is given by the formula $M=5.115b-0.085v$, where v equals the saponification value minus 200. A table is given for reading off this value. The method gave butyric acid values from 0.0 to 0.6, with saponification values of 194 to 223, from which may be calculated a milk fat content of 0—1.1%, or an average of 0.5%, when applied to several sorts of commercial margarine. On the contrary, butyric acid values of 18.6 to 23.0 (corresponding to butyric acid contents of 3.28 to 4.05%), or an average of 20.3, were obtained with various samples of commercial butter. This corresponds to an average Reichert-Meissl value of 27.3. The average conversion factor for the calculation of the butyric acid value from the Reichert-Meissl value was found to be 0.745. A. RAYNER.

China wood [tung] oil. A. EIBNER and J. SCHWAIGER (Chem. Umschau, 1926, 33, 77—85).—Mainly a summary and critical review of work published during the past year or two. Reference is made to the isomerisation of α -elæostearic acid on bromination and a few experimental results are quoted. H. M. LANGTON.

Data used in graph for beeswax. A. A. WEIR (Analyst, 1926, 51, 181; cf. B., 1925, 813).—The limits taken for pure beeswax in the graph were:— d 0.976—0.941; m.p., 60.5—66.5°; n_D , 43.2—45.7; iodine value, 5.42—17.1; saponif. value, 87—106; acid value, 16.7—23.6; ester value, 65.9—85; ratio value, 2.8—4.5; and unsaponif. matter, 48—53%. D. G. HEWER.

Alcohol, acetic acid, and other by-products from arachis nut [pea-nut] shells. G. DE BELSUNCE (Bull. Mat. Grasses, 1926, 1—3).—The Meunier process (B., 1922, 212 A; 1923, 25 A) for the hydrolysis of cellulosic materials with dilute sulphuric acid and high-pressure steam is described. Two experiments with pea-nut shells, one lasting for 30 min. and the other for 60 min., gave the following respective amounts of products calculated on the dry material:—Sugar, 21.8%, 15.8%; acetic acid 2.26%, 4.38%; furfuraldehyde 2.42%, 2.66%; acetone 0.155%, 0.47%; other substances soluble in ether 0.755%, 1.68%. The sugar is entirely fermentable and the residue, consisting principally of lignin, has sufficient heating value to fulfil all the fuel requirements of the process.

E. H. SHARPLES.

Activated fuller's earth. O. ECKART (Z. angew. Chem., 1926, 39, 332—334).—During the bleaching of fatty oils by means of activated fuller's earth the acidity of the oil is increased due to hydrolysis of the glycerides by traces of acid in the earth; this increase is more marked if the earth is moist than after drying. Neutralisation of the acidity of the bleaching agent by treatment with alkaline solutions results in the deposition of thin films of ferric and aluminium hydroxides on the surface of the particles of earth with a consequent diminution, or even total loss, of bleaching power. Addition of calcium carbonate to fuller's earth, forming the

so-called "neutral earth," does not prevent the increase in the acidity of the oil after treatment, owing to the sluggish action of the carbonate in an oily medium. A marked diminution in the increase of acidity is, however, produced by the addition of calcium hydroxide equivalent to ten times the acidity of the earth; this addition must be made when the earth is mixed with the oil and not previously. A short time of contact is essential, otherwise the colouring matter again becomes dispersed in the oil; in most cases treatment for 10 min. suffices. Fuller's earth is improved if heated to 400—450° before use and then allowed to adsorb moisture from the air; without the moisture its bleaching power is reduced 10%. Heating above 450° entirely destroys the ability of fuller's earth to remove the colour from oils.

A. R. POWELL.

Colour of oils etc. FONROBERT and PALLAUF.—See XIII.

Drying of fatty oils. AUER. WOLFF.—See XIII.

"Voltol-factice." HOCK.—See XIV.

PATENTS.

Extracting oil, gelatinous water and solid material from whale blubber, whale flesh, fish products, and similar raw materials. K. HOLTER and S. THUNE (E.P. 232,601, 15.4.25. Conv., 16.4.24).—The raw material is fed under pressure down a shaft on to the surface of a revolving grindstone enclosed in a chamber, the temperature of which may be raised by the admission of water or steam. The moist disintegrated material is removed from the base of the chamber by a helical conveyor and passed into a horizontal screening chamber where the main part of the oil and gelatinous water is removed. The screening chamber is provided with a tapered portion near its outlet and, aided by the conveyor, the material is compressed, thus expelling the rest of the liquid. The temperature of the screening chamber may be controlled by the introduction of steam through the perforated hollow shaft of the conveyor. E. H. SHARPLES.

Neutralising and bleaching oils under heat and vacuum conditions. E. BATAILLE (E.P. 233,345, 29.4.25. Conv., 30.4.24).—The oil is accurately and automatically measured, treated with a measured quantity of neutralising and/or bleaching agent, and drawn by suction into a "beater," i.e., a steam-jacketed cylinder provided with a rotating helical stirrer. The steam supplied to the jacket of the "beater" operates a steam ejector by which a vacuum is produced in the "beater."

H. M. LANGTON.

Distilling fatty acids and other volatile substances from oils and fats, acid oils, and crude fatty acids. J. J. V. ARMSTRONG. From NAAML. VENNOOTS. A. JURGENS' MARGARINEFABR. (E.P. 248,828, 10.12.24).—Pure odourless neutral or almost neutral undecomposed fats or oils and pure fatty acids are obtained by a distillation process charac-

terised by the following features. A very short period of heating of the liquid phase is obtained by continuously supplying the raw material and continuously and completely removing the whole residue of the distillation. A large surface of contact between the liquid and the steam or inert gases used to distil the fatty acids is obtained by causing the oil to flow in a thin layer or film over a perforated plate placed in a drum, the steam or inert gases being introduced underneath. A reduced pressure is applied in order to keep the consumption of steam or inert gases (in spite of the relatively low temperature) within economical limits. The temperature of the liquid phase measured anywhere within the liquid-flow system is not allowed to exceed 250°.

H. M. LANGTON.

Rendering fat- and oil-containing solids of animal origin. W. LAABS, Assr. to ALLBRIGHT-NELL Co. (U.S.P. 1,578,245, 23.3.26. Appl., 22.6.25).—The moisture content of the material is reduced to 60% by agitating and heating at temperatures and pressures not sufficiently high to cause substantial disintegration and separation of oils and fats. The temperature is then raised above 117° and by agitation and the action of high-pressure steam developed from the natural moisture content, the material is disintegrated and the oils and fats are liberated. When the moisture content has fallen to 17% the pressure is reduced to less than 1 atm. and agitation continued at temperatures below 117° until the moisture content is substantially under 15%, when the solid residue is pressed.

E. H. SHARPLES.

Distillation of oils, liquid fats, hydrocarbon mixtures, and similar liquids. C. MELHARDT (G.P. 424,678, 18.1.25).—Low-boiling constituents are distilled off by forcing the liquids through water or dilute salt solution, which is kept boiling vigorously in a space between members of porous material.

L. A. COLES.

Purifying and deodorising oils and fats. NAAML. VENNOOTS. A. JURGENS' VEREENIGDE FABR. (Austr. P. 96,154, 7.4.21).—Oils and fats are treated with steam or inert gases other than hydrogen, if necessary *in vacuo*, in the presence of inert material upon which are deposited finely-divided oxides, basic carbonates, or silicates of heavy metals, preferably with the addition of finely-divided iron or fuller's earth.

L. A. COLES.

Preparation of sulphur soaps. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 424,499, 6.2.25).—Fatty acids, oils, or fats, alone or mixed with resins, resin acids and other substances imparting smell, or colour, or acting medicinally, are saponified with polysulphides. Soaps are formed from which the sulphur can be dissolved out by water in an active colloidal form.

W. CLARK.

Decomposing fats, oils, and waxes. W. SCHRAUTH (U.S.P. 1,576,005, 9.3.26. Appl., 12.4.24).—See Can. P. 245,373; B., 1926, 287.

Cooling of liquids to form powdered or granular solids, particularly soap powder. H. GUTTIN (E.P. 250,441, 17.7.25).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Particle size of pigments and its influence on paints and paint films. C. P. VAN HOEK (Farben-Ztg., 1926, 1237—1239, 1295—1297, 1350—1353, 1399—1401, 1455—1457, 1513—1516).—A general review including the influence of particle size on the protective power, the adhesion of the particles to the medium, the colloidal character of paint suspensions, flocculation, the determination of particle size, and its dependence on the method of preparation of the material and the grinding of the paint, and the relation of particle size to the oil content, colour and covering power of paint. A bibliography is given.

D. F. TWISS.

Some synthetic organic colouring matters used in lake making. F. M. ROWE (J. Oil Colour Chem. Assoc., 1926, 9, 56—80).—A review of the typical synthetic colouring matters used for lakes, mainly with reference to their constitution, and to their physical properties that largely depend upon the constitution. The colours are considered under the headings:—Azo-, pyrazolone-, nitroso-, nitro-, quinoline-, ketonimine-, triphenylmethane-, xanthene-, thiazine- and anthraquinone-derivatives.

S. S. WOOLF.

Antimony sulphides. L. M. CURRIE (J. Physical Chem., 1926, 30, 205—238).—A study of the nature and number of phases in the so-called antimony pentasulphides, of the colours of the antimony sulphides, and of the factors affecting these. In agreement with Bosek (J.C.S., 1895, 67, 519), pure antimony pentasulphide can be prepared by following the procedure of Bunsen (Annalen, 1878, 192, 305). Cold sulphur solvents have no effect on the pure pentasulphide, but with hot solvents, especially carbon disulphide, some decomposition occurs. The passage of a slow stream of hydrogen sulphide through solutions of quinquevalent antimony reduces some of the antimony, forming the trisulphide and sulphur. Long-continued passage of the gas, high temperatures, and low acid concentration favour the formation of the trisulphide. The decomposition of sodium thioantimonate by dilute acids yields a mixed pigment consisting of sulphur and a solid solution of sulphur in antimony tetrasulphide, the amount of which depends on the conditions of decomposition. The composition of this solid solution can be varied between the limits corresponding to $Sb_2S_4 \cdot 66S$ and Sb_2S_4 . Rise in the temperature of reaction or of drying, the passage of a current of air or hydrogen sulphide, or precipitation in presence of free alkali, are all accompanied by an increase in the amount of free sulphur formed, as a separate phase, at the expense of that in the solid solution. Contrary to Hahn (Kolloid-Z., 1922, 31, 200) and others, sodium thiosulphate and tervalent antimony in dilute acid give antimony trisulphide and sulphur, no pentasulphide, oxysulphide, or solid solution

being formed. The sulphur content, in this case, is increased by raising the temperature of mixing, the ratio of thiosulphate to antimony, the concentration of thiosulphate, and the acidity of the final mixture up to a limit (approx. 30%). All attempts, other than mechanical, to add sulphur to the trisulphides failed. The pure pentasulphide is more or less completely decomposed at 70–75° yielding the tetrasulphide. This, in turn, is slowly decomposed at 105° and rapidly at 120–155°. At 65° and below decomposition is slight and very slow. Intimately mixed sulphur appears to retard the decomposition of the tetrasulphide, the solid solutions of Sb_2S_4 and S being stable above 100°. At 105–108°, the rate of decomposition is slow, becoming rapid at 112–115°; at 150° decomposition is complete in a few hours. Above 150° oxidation or sublimation of the sulphur chiefly occurs, with an accompanying change in the physical properties of the trisulphide formed. X-Ray examination shows that the precipitated sulphides of antimony are all amorphous solids. Prolonged heating near 200° converts them into the black crystalline variety of the trisulphide. The amorphous trisulphide in a finely divided state is pale yellow; larger particles are crimson. Heating the suspensions favours coagulation and darkens the colour of any precipitated sulphide. Addition of gelatin, up to a limit, prevents agglomeration and the suspensions and precipitates become lighter in colour as a result. Acids tend to flocculate, and alkalis to peptise it, with the expected colour changes. Maroon-coloured antimony sulphides are mixtures of crimson and black sulphides. Electrical disintegration of stibnite by Svedberg's method gave a yellow crystalline trisulphide (finely-divided form) which had an X-ray pattern identical with that of the natural sulphide, and with that of the artificially prepared crystalline sulphides of antimony. Antimony pigments precipitated in the presence of S or SH ions give various shades ranging from gold to brown and, finally, to black. The difference between the maroon and brown modifications is due to adsorption of hydrogen sulphide by the latter, which, by preventing intimate contact of the particles, prevents the formation of the larger crimson particles. The effect of heat on the colour of the penta- and tetrasulphides is but slight, a general darkening only being noticeable.

L. S. THEOBALD.

U.S. Government master specification for shellac, flake orange. (U.S. Bureau of Standards, Circ. No. 302, Feb. 9, 1926, 6 pp.)—Four types, A, B, C, and D, of the manufactured product of stick lac, freed from most of the lac dye and prepared in "free" flake form are defined (seed, garnet, and button lac being excluded). Types A, B, and C must show not more than 1.75, 2.50, and 3.00%, respectively, of matter insoluble in hot 95% alcohol, other requirements being the same for the three types, viz.:—maximum for iodine value 18.0, moisture and volatile matter 2.0%, water-soluble matter 0.5%, wax 5.5%, and ash 1.0%. Type D agrees with type C except in iodine value, where the maxi-

mum is raised to 24.5%. Colour is not specified except in the use of comparison samples mutually agreed on by buyer and seller. Methods of sampling and testing are detailed.

S. S. WOOLF.

Method for estimating the colour depth or brightness of resins, varnishes, oils, etc. E. FONROBERT and F. PALLAUF (Farben-Ztg., 1926, 31, 1354; cf. B., 1926, 201).—In connexion with the method described earlier, the use of colorimeters consisting of glass prisms filled with iodine solutions of known strength is recommended; these show very little alteration on storage.

D. F. TWISS.

Drying of fatty oils. L. AUER (Farben-Ztg., 1926, 31, 1240–1241).—Linseed oil films in an atmosphere of carbon dioxide or in a vacuum (10 mm. pressure) dry in the course of a few days and show an increase in weight. This result is attributed to a primary coagulation of the oil by gas similar to that of a lyophobic sol by an electrolyte; in the high vacuum produced by a mercury pump no drying was observed. The increase in weight arises during the transfer from the containing vessel to the balance. When drying occurs in the open atmosphere, the coagulation and adsorption are concurrent; chemical combination succeeds the adsorption.

D. F. TWISS.

Drying process of the "drying oils." H. WOLFF (Farben-Ztg., 1926, 31, 1239–1240, 1457; cf. B., 1925, 45).—Linseed oil varnishes will dry slowly in an atmosphere containing so little oxygen that the drying process must be essentially of colloidal type, and is possibly independent of the oxidation reaction.

D. F. TWISS.

German oil of turpentine. I. BISCHOFF (Farben-Ztg., 1926, 31, 1517).—Analytical and practical tests with a sample of German oil of turpentine indicate this to be as good as the imported article for many applications.

D. F. TWISS.

New methods of varnishing with cellulose esters. H. RASQUIN (Farben-Ztg., 1926, 31, 1293–1294).—The advantages and disadvantages of the method of varnishing by spraying nitrocellulose are discussed and modified processes indicated enabling the cheaper application of such varnishes by means of a brush.

D. F. TWISS.

Foam meter. WILLIAMS.—See I.

Apparent viscosity of solutions of nitro-cotton. MCBAIN, HARVEY, and SMITH.—See V.

Exposure tests with pointolite lamp. HOCHHEIM and KNEBEL.—See VI.

High and low stiffening carbon blacks. SPEAR and MOORE.—See XIV.

PATENTS.

Process of making carbon black. O. G. MESSENGER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,577,481, 23.3.26. Appl., 18.10.23).—Hydrocarbon gases are made to react with successive

small proportions of oxygen so as to effect the liberation of carbon.

D. F. TWISS.

Carriers for dyes etc. MAISON BRETON, J. FICHOT & Co. SUCCESEURS, and G. L. A. CRUT (F.P. 599,868, 23.9.24).—As carriers there are used the precipitates obtained on treating solutions of aluminium sulphate with quantities of alkali carbonates or ammonium carbonate insufficient for complete precipitation. These products are less transparent than those obtained with larger quantities of soluble carbonate.

B. FULLMAN.

Production of red antimony sulphide pigments. P. J. F. SOUVIRON (F.P. 600,894, 18.7.25).—By treating antimony solutions with thiosulphate in the presence of an aluminium salt, antimony sulphide pigments of strong colour and great stability towards light and heat are obtained.

D. F. TWISS.

Manufacture of polymerised styrene and its homologues. NAUGATUCK CHEMICAL Co., Assees. of I. OSTROMISLENSKY (E.P. 233,649, 15.9.24. Conv., 7.5.24).—The patent relates to the preparation from styrene and its homologues of vitreous polymerisation products, which are tough, transparent, practically colourless if necessary, of relatively high melting point, and capable of being cut in thin films or parings, and which differ in several respects from the resinous polymerisation products of similar chemical composition. The styrene and its derivatives are obtained from the products of pyrogenetic decomposition described in E.P. 232,909 (B., 1926, 299), *e.g.*, styrene, *o*- and *p*-methylstyrene, *s*-dimethylstyrene, and ethylstyrene. In every case the compound $\text{Ar}\cdot\text{CH}:\text{CH}_2$, mixed with at least one other substance, is polymerised to the "resinous" polymer by heating at 140–220° (temperatures differing in different cases), the resinous polymer purified, depolymerised by heating at 350–600°, the $\text{Ar}\cdot\text{CH}:\text{CH}_2$ purified, and polymerised to the vitreous polymer by heating at 135–140°. For example, crude styrene (from the pyrogenetic decomposition of ethylbenzene) is heated at 140–180°, for 16–48 hrs., in an autoclave or under reflux. The resinous styrene polymer (90–100% of the original styrene) is separated by steam or heat distillation, and dry-distilled at 350–500°, the product yielding on purification 45–55% of styrene (containing a small quantity of low-boiling impurity). A 50–65% solution of this substance in xylene is heated at 135–140° under reflux, when a solution is obtained from which "vitreous" polymerised styrene, in 70% yield, may be obtained by evaporation, precipitation with alcohol, etc. In many cases the final polymerisation is carried out in the absence of a solvent.

B. FULLMAN.

Manufacture of polymerised styrene. NAUGATUCK CHEMICAL Co., Assees. of I. OSTROMISLENSKY and W. A. GIBBONS (E.P. 243,768, 15.9.24. Conv., 7.5.24).—"Vitreous" polymerised styrene (*cf.* preceding abstract) is prepared by heating a mixture containing at least 40% of styrene (such as a solution of styrene in an organic solvent) at 135–200°, the temperature depending on the relative concentrations

of styrene and the other substances. For example, a mixture of styrene (84%) and ethylbenzene is heated under reflux for 25 hrs. at 175°, and the product then purified by heating *in vacuo* (to ensure absence of air) with the temperature rising slowly to about 130°, to remove volatile residues. If high-boiling impurities be present the substance is dissolved in a low-boiling solvent (*e.g.* benzene), which is then evaporated; or the polymerised styrene in the crude product may be precipitated by addition of alcohol, solution in benzene, re-precipitation, and final evaporation as usual. Details are given of the preparation of suitable styrene mixtures, for instance, those obtained by distillation of the product obtained from the tube furnace in the pyrogenetic decomposition of ethylbenzene as described in E.P. 232,909 (B., 1926, 299).

B. FULLMAN.

Artificial resins. BRIT. CYANIDES Co., LTD., and E. C. ROSSITER (E.P. 248,477, 5.12.24).—The almost universal presence of formic acid in commercial formaldehyde imparts an acid reaction to thiocarbamide-formaldehyde condensation products with possible harmful action. This is obviated without the introduction of alkaline products, by passing a current of dry air or an inert gas through the solution while it is being evaporated at a temperature below 100°.

S. S. WOOLF.

Dispersion of unbleached shellac. SIEMENS & HALSKE A.-G., Assees. of C. HARRIES and W. NAGEL (G.P. 421,337, 15.2.23).—Peptising agents such as organic acids yield disperse phases, which may be again completely taken up by alcohol. Small quantities of mineral acids exert a coagulating effect.

B. FULLMAN.

Linoleum cement. P. SLANSKY, and DEUTSCHE LINOLEUM-WERKE HANSA (E.P. 248,974, 5.6.25).—In the production of linoleum cement by melting solid oxidised oils with resins in an open pan at temperatures below 150–160°, oils of a limited state of oxidation only may be used, the higher temperatures required to melt more strongly oxidised oils causing ignition and crumbling. If, however, the melting is carried out in an autoclave in the absence of oxygen, the more highly oxidised oils may safely be used. The melting is accelerated by the addition of not more than 10% of water.

S. S. WOOLF.

Heat-plastic materials (E.P. 249,172).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

"Cyclopentadiene rubber." A new cyclic synthetic polymerisation product. H. A. BRUSON and H. STAUDINGER (Ind. Eng. Chem., 1926, 18, 381–383).—*cyclo*Pentadiene as a methylene derivative of butadiene should be capable of polymerisation to a "rubber." Several halogen compounds, particularly stannic chloride, act as catalysts, polymerisation being effected in chloroform at 0°. A quantitative yield is obtained of a product closely resembling freshly coagulated rubber; it readily

undergoes atmospheric oxidation to $(C_5H_6O)_x$, forms a bromine additive compound $(C_5H_6Br_2)_x$, a nitrosite, and a highly coloured hydrochloride and hydrobromide; vulcanisation with sulphur chloride gave products ranging from a soft jelly to a vulcanite-like material.
D. F. TWISS.

High and low stiffening carbon blacks [in rubber or oils]. E. B. SPEAR and R. L. MOORE (Ind. Eng. Chem., 1926, 18, 418—420).—No simple relation exists between the stiffening power of carbon blacks and their adsorptive power for organic dyes or hexamethylenetetramine. High tensile strength in a rubber mixing containing a carbon black is always associated with the colloidal dispersion of at least a portion of the carbon in the rubber matrix, the colloidal particles being too small to be resolved by the microscope; the stiffening effect, however, appears to be independent of this feature. Mere particle size is undoubtedly a factor but not to the exclusion of other influencing conditions.
D. F. TWISS.

Internal mixers for rubber stocks. P. S. SHOAFF (Ind. Eng. Chem., 1926, 18, 345—347).—A review of the advantages and disadvantages of internal mixing machines relative to roll mills. Recommendations are made as to the order of procedure for mixing various types of rubber "compounds."
D. F. TWISS.

Vulcanisation in solution. W. GREINERT and J. BEHRE (Kautschuk, March, 1926, 63—64).—The quality of unworked rubber is indicated by the period necessary for gelation when 10 c.c. of a solution of the dry rubber in dry benzol is treated with 1 c.c. of an 11% solution of sulphur chloride in benzol (cf. Le Blanc and Kröger, B., 1921, 667 A; 1924, 141). The method not only distinguishes between low-grade and high-class forms of rubber but reveals marked differences between various samples of pale crêpe rubber. The adverse effect of mastication is evident not only in the prolongation of the gelation period, but also in the behaviour on hot vulcanisation with sulphur; in this case the resistance to load is decreased with consequent modification of the stress-strain curve.
D. F. TWISS.

Accelerated ageing tests [for rubber]. C. R. PARK (Rubber Age, London, 1926, 7, 64—68; Kautschuk, March, 1926, 57—61).—The requirements for an accelerated ageing test for rubber are that a given period shall be equal to a definite period of natural ageing and that the physical properties shall change in the same direction and to a comparable degree in accelerated and in natural ageing. The behaviour of vulcanised samples containing rubber and sulphur only, or rubber, sulphur, and zinc oxide with mercaptobenzothiazole, hexamethylenetetramine, diphenylguanidine, and litharge respectively reveals no simple relation between the alteration in the stiffness index and in the tensile product on ageing naturally, at a higher temperature, and at a higher temperature by oxygen under pressure,

respectively. Natural ageing is probably a more complex phenomenon than has been assumed hitherto and the inadequacy of accelerated ageing tests is attributable to over-emphasis or neglect of certain factors.
D. F. TWISS.

Oxidation of rubber exposed to light. I. WILLIAMS (Ind. Eng. Chem., 1926, 18, 367—369).—Natural oxidation of vulcanised rubber may result in general deterioration throughout, in formation of a surface film, or in the development of cracks. Exposure to light aids surface oxidation with formation of an almost inelastic film, light of shorter wave length being more active. Surface cracking or "checking" under the influence of ultra-violet light will not occur in the absence of air and is attributable to the formation of ozone; unstrained rubber is not "checked" by ozone. Surface oxidation hinders the development of "checking," which is consequently retarded by the surface application of a copper salt.
D. F. TWISS.

Addition of light to accelerated ageing [of rubber]. F. P. JECUSCO (Ind. Eng. Chem., 1926, 18, 420—424).—Exposure to light during accelerated ageing of vulcanised rubber at 71° expedites the decrease in strength, and the increase in the acetone extract; the rubber undergoes a smaller loss in weight than when not illuminated and becomes covered with a red insoluble coating. This activity is not restricted to ultra-violet light; green and infra-red radiation also have a marked effect, especially on rubber activated by previous ageing (cf. Asano, B., 1925, 816).
D. F. TWISS.

New mechanical test for rubber insulation. C. L. HIPPENSTEEL (Ind. Eng. Chem., 1926, 18, 409—411).—A 2-inch length of insulated wire, without braid, is compressed between two parallel steel surfaces which approach one another at a uniform rate. A small self-recording machine is described, the record of the compression of the insulation forming a sort of stress-strain curve.
D. F. TWISS.

Economical use of reclaimed rubber as a substitute for new rubber. J. M. BIERER and C. C. DAVIS (Indiarubber J., 1926, 71, 565—571; Ind. Eng. Chem., 1926, 18, 348—353; cf. Holt and Wormeley, U.S. Bur. of Standards Tech. Paper 294).—It is possible to replace part of the new rubber, even in a tyre tread, by a correspondingly greater amount of suitable reclaimed rubber without loss of abrasion resistance or depreciation of the stress-strain curve; the procedure roughly is a replacement of new rubber by approximately the same volume of rubber component in the reclaimed rubber, with a simultaneous adjustment of the quality, necessitated by the lower value of the old rubber component, by alteration of the other compounding ingredients. It is desirable to maintain a high proportion of carbon black. Depreciation of ageing qualities due to the use of reclaimed rubber from inner tubes can be effectively obviated by the introduction of suitable anti-oxidants. Selenium greatly increases the abrasion-resistance of rubber.
D. F. TWISS.

PATENTS.

Heat-plastic materials [from rubber]. E. A. WHITE. From B. F. GOODRICH Co. (E.P. 249,172, 24.9.24).—Well-masticated rubber (100 pts.), if necessary with the addition of pine tar, is mixed with an isomerising agent (up to 15 pts.), containing the grouping $R\cdot SO_2X$, e.g., sulphuric acid, an organic sulphonic acid or a sulphonyl chloride, or with a mixture of such agents. The mixture is then heated between 110° and 175° , and reworked after cooling. According to details of the treatment, the product may be tough and heat-plastic like balata, or hard and friable, but yielding on fusion a mass resembling, and capable of replacing, shellac. D. F. TWISS.

Accelerator for vulcanisation of rubber. P. M. PAULSON, Assr. to ROESSLER AND HASSLACHER CHEMICAL Co. (U.S.P. 1,575,865, 9.3.26. Appl., 17.6.25).—Vulcanisation of rubber is expedited by the addition of a compound of the formula $NH_2\cdot C(:NH)\cdot NRR^1$, where R and R^1 are dissimilar organic radicals. D. F. TWISS.

Accelerator for vulcanisation of rubber. C. S. WILLIAMS, Assr. to ROESSLER AND HASSLACHER CHEMICAL Co. (U.S.P. 1,575,884, 9.3.26. Appl., 17.6.25).—Rubber is vulcanised with the aid of an accelerator of the constitution $NH_2\cdot CH(:NH)\cdot NHR$, where R is a hydrocarbon radical. D. F. TWISS.

Vulcanising rubber. N. A. SHEPARD and S. KRALL, Assrs. to FIRESTONE TIRE AND RUBBER Co. (U.S.P. 1,576,072, 9.3.26. Appl., 20.6.23. Renewed 4.8.25).—A mixture of rubber with a small amount of a reaction product of an aldehyde-ammonia and a mustard oil is heated with a vulcanising agent. D. F. TWISS.

Vulcanising caoutchouc. D. N. SHAW, Assr. to GOODYEAR TIRE AND RUBBER Co. (U.S.P. 1,580,089, 6.4.26. Appl., 18.11.24).—Rubber is heated with a vulcanising agent, a nitrosocarbazole, and an activator. D. F. TWISS.

Vulcanisation of rubber. ATLANTIC DYESTUFF Co., Asses. of A. C. BURRAGE, JUN. (Can. P. [A] 245,929, 27.2.24, [B] 245,930, 27.2.24, and [C] 247,108, 19.2.23).—(A) Diphenyl-*o*-tolylguanidine is used to accelerate vulcanisation. (B) Compounds of the type of diphenyl-*o*-tolylguanidine for use as vulcanisation accelerators are produced by the action of litharge and an aromatic amine on a diarylthiocarbamide. (C) In vulcanising rubber, a mixture of accelerators, e.g., of arylthiocarbamides with other accelerators or of methylene-*p*-toluidine with tri-*o*-tolylguanidine, is used, the constituents becoming active at different temperatures. D. F. TWISS.

Separation of textile from rubber in rubber goods. P. MARMIER and F. DE GAALON (F.P. 598,722, 14.5.25).—The materials (tyres) are heated in a closed vessel under pressure with a mixture of 3 pts. of benzine and 1 pt. of vaseline oil, in the presence of turnings of copper, tin, or lead.

B. FULLMAN.

Regeneration of vulcanised rubber. L. GALLOIS (F.P. 599,869, 23.9.24).—Vulcanised rubber is heated in a closed vessel under pressure with rubber solvents, e.g., benzine, benzol, petroleum, xylene, or turpentine oil, to about 150 – 200° . On evaporation of the solvent, a vulcanisable regenerate is obtained. B. FULLMAN.

Hydrogenation of artificial and natural rubber. SIEMENS U. HALSKE A.-G. (G.P. 424,281, 24.2.23).—The rubber, after being made sufficiently plastic, is dissolved in an organic solvent and treated with hydrogen, in the presence of metallic catalysts. B. FULLMAN.

Production of rubber articles. R. DITMAR (Austr. P. 99,664, 27.11.23).—Rubber latex is mixed with 0.1–10% of glycerol as an anti-coagulant, and with compounding ingredients, including sulphur; it is then dried, moulded, and vulcanised. Fibrous or porous materials, e.g., fabrics or bricks can be impregnated with the compounded latex by spraying. D. F. TWISS.

Rubber vulcanisation. DOVAN CHEMICAL CORP. (E.P. 226,836, 29.12.24. Conv., 29.12.23).—See U.S.P. 1,546,876; B., 1925, 1000.

XV.—LEATHER; GLUE.

Chemico-histological study of leather manufacture. I. Structure and properties of freshly flayed steer skin. H. G. TURLEY (J. Amer. Leather Chem. Assoc., 1926, 21, 117–156).—The routine histological procedure used is described. By the use of simple stains, the skin proteins are classified into acidic and basic. The following elements are described in detail:—*stratum corneum*, *stratum granulosum*, *stratum germinativum*, hyaline layer, *corium minor*, hair and hair root sheaths, sebaceous glands, sudoriferous glands, muscle, wandering cells, elastic fibres, blood vessels and lymphatics, grain membrane, *corium major*, connective tissue fibre bundles, aerolar tissue sheaths, and the interfibrous material. On staining certain sections with thionin or toluidine-blue, and Bielschowsky stain respectively, the hyaline layer was shown to be different from collagen. It resembles the epithelial tissue of the epidermis and seems to be identical with the basement membrane of histologists. The elastic fibres are present mainly in the grain layer, and only to a small extent elsewhere. The elastic fibres in the grain layer are $2\ \mu$ in diameter and $1\ \mu$ or less in the grain network. They are basic, i.e., stain with acid dyes. The connective tissue fibres in the grain layer are $5\text{--}20\ \mu$ wide. They are studded with nuclei of collagen-forming cells. The nuclei are large, flat, oval discs about $16\ \mu \times 5\ \mu$, and since they are seen on edge and at various angles it is inferred that there is no general symmetrical arrangement of the collagen fibres. The large bundles of connective tissue fibres are $180 \times 100\ \mu$, and somewhat elliptical in cross section, and the individual fibres $55 \times 23\ \mu$. The connective tissue fibrillae are $3\text{--}6\ \mu$

in diameter. The findings are illustrated by photomicrographs and a coloured plate made from stained sections.

D. WOODROFFE.

Action of lime on enzymes. R. L. COLLETT (J. Soc. Leather Trades Chem., 1926, 10, 100—112).—Pancreatic trypsin, bacterial proteases from *Staphylococcus albus*, pancreatic lipase, "thrombase," urease, and yeast amidase respectively were mixed with milk of lime and at definite intervals, the activity of the enzyme was measured. The proteolytic and fat-splitting enzymes were rapidly inactivated by the lime. "Thrombase" was inactivated, and did not function at the alkalinity of tannery lime liquors. There was no evidence that the thrombase system had a secondary proteolytic stage which might suggest that it affected unhairing. These results and those described previously (B., 1924, 25) show that neither bacteria nor enzymes, by acting in lime liquors, can produce the ammonia which is always found in old or mellow limes. Lime itself, however, acting on digested proteins produces large quantities of ammonia. D. WOODROFFE.

Official method [of Society of Leather Trades Chemists] of quantitative tannin analyses (J. Soc. Leather Trades Chem., 1926, 10, 30—42).—The method is essentially the same as that outlined in the Leather Chemists' Pocket Book (Spon, 1914). Evaporating basins must be shallow, flat-bottomed, without sharp angles, and not less than 6.5 cm. in diameter. Porcelain basins may be used, but silver ones are preferable. Residues shall be dried in a water, steam, vacuum steam, or electric oven at a uniform and constant temperature of 98.5—100°. Berkefeld filter candles shall be used for the filtration of the tannin infusions. Distilled water must be free from sulphates and chlorides, have p_H 5.0—6.0, and leave a residue < 0.002 g. per 100 c.c. Kaolin should be washed with hydrochloric acid and then with distilled water; 1 g. suspended in 100 c.c. of water should show p_H 4.0—6.0. The residue from 100 c.c. of 0.01N-acetic acid shaken with 1 g. of kaolin should be less than 1 mg. Tanning materials must be ground to pass through a sieve of 5 wires per cm. Fibrous materials are to be ground, separated into fine and coarse portions, each weighed, and the same proportions of each used together for analysis. Samples of ground sumach must be thoroughly mixed in a rotating churn before weighing out for extraction. Solid extracts shall be ground in a mortar before weighing. Pasty extracts or extracts of uneven moisture content are to be weighed in a flat-bottomed basin, dried in an air oven at 70°, exposed overnight to the laboratory atmosphere, weighed, and loss of water calculated. The extract is then powdered and a weighed portion dried in the oven at 98.5—100°. The total loss in moisture is calculated. Liquid extracts should be mixed in a shaking machine. Viscous extracts should be heated to 45° on the water bath, well mixed, cooled, and then weighed out at once. The tannin infusion must contain 3.75—4.25 g. of tannin per litre. Liquid extracts must be washed with boiling distilled water

into a litre flask. Sumach, myrobalans, and synthetic tanning extracts must be diluted with water at 70°. Solid and pasty extracts shall be heated on the water bath with ten times their weight of water until no solid fragments remain, except fibrous matter. Solid tanning materials shall be extracted in a Procter extractor. The material must be soaked overnight in cold distilled water, the cold infusion (< 100 c.c.) siphoned off, the water bath heated to 45—50°, 100 c.c. of distilled water at 45—50° poured on to the material, left for 20 min., and then siphoned off rapidly. This is to be followed by three similar macerations, after which the temperature of the bath is raised to boiling, and four macerations with 125 c.c. of boiling water carried through. The infusion must be well mixed and cooled to 17—18° by immersing the flask in a large vessel containing water. The cool infusion must be made up to 1 litre, well mixed, and filtered at once. The moisture in solid tanning materials and solid and pasty extracts shall be determined directly on the material, using a squat-shaped, wide-mouthed weighing bottle. Infusions must be filtered, repeatedly if necessary, until "optically clear," both by reflected and transmitted light. The non-tannins are determined by shaking 100 c.c. of the infusion with a known weight of chromed hide powder for 15 min. Specific gravity must be determined by the pycnometer or bottle at 15°. Four samples of materials should be taken, packed in glass bottles, and sealed. In a consignment containing x packages, $0.7 \times \sqrt{x}$ packages shall be sampled, the samples taken shall be mixed together, and the bulk reduced by quartering to the desired size. Detailed instructions are given for sampling the different kinds of materials. The analysis of used liquors and spent tanning materials is performed in the same way. Used liquors must be diluted to d 1.007—1.008. For weaker liquors the amount of dry hide powder used in the detannisation shall be 1 g. for every 0.001 the d is greater than 1.000. The amount of spent tanning materials taken for analysis shall be sufficient to yield an infusion containing 3.5—4.5 g. of tannin per litre if possible, but in no case must the total solids exceed 10 g./litre. Analytical results for spent tan must be calculated on the dry material and for spent or used tan liquors be reported in g./100 c.c. The density of used liquors must be reported. D. WOODROFFE.

Sampling tanning materials, leather, etc. for analysis. H. G. BENNETT (J. Soc. Leather Trades Chem., 1926, 10, 57—58).—By a slight modification of the formula $n=0.7\sqrt{x}$ for sampling (cf. preceding abstract) it becomes $x=2n^2$, where x is the number of packages and n the number to be selected for sampling. D. WOODROFFE.

Report of a committee of the French section of the Society of Leather Trades Chemists on quantitative tannin analysis. G. HUGONIN (J. Soc. Leather Trades Chem., 1926, 10, 89—100).—Comparative analyses by different chemists have been made on various liquid tanning extracts using the official chromed hide powder and powders

chromed with other solutions. A powder chromed with a 10% or 12.5% solution of normal chromium chloride gave lower figures for non-tannin than the others. Normal chrome alum solutions gave high non-tannin figures, due to less dissociation by the chromium sulphate. Determinations of the p_H value by electrometric and colorimetric methods showed a lower value by the latter method. The electrometric method yielded more concordant results by different observers. Solutions detanned with powder chromed with normal chromium chloride yield non-tan liquors with a lower p_H value than those detanned with the other powders. Comparison of the Berkefeld filter candle with centrifuging and sedimentation shows that it gives a higher content of insoluble matter. Particles of 1μ or larger should be classed as "insoluble matter." The chroming of hide powder with normal chromium chloride is favoured.

D. WOODROFFE.

Behaviour of synthetic tanning materials towards hide substance. E. WOLESENSKY (Tech. Papers, U.S. Bur. Standards, 1926, 20, [309], 275—287).—A study has been made of the hydrolytic action of synthetic tannins on hide substance during tanning, the rate of fixation of the tanning material by the hide, and the maximum amount of tanning material which may be fixed under various conditions of concentration and length of treatment. The hydrolytic action on the hide varied with the tanning materials used, being nil for one derived from cresol; 3—6% of hide substance was dissolved by using synthetic tannins derived from phenol-sulphonic acid, and from cresol and sulphuric acid, also a commercial product of unknown composition. Synthetic tanning materials produced by condensing a sulphonated aromatic compound with formaldehyde combine rapidly with pelt but not in large quantity. Those produced by sulphonating the product of condensation of an aromatic compound with formaldehyde, possess greater filling power, and require longer time to reach maximum absorption by the pelt; their absorption depends on the concentration. Synthetic tanning materials of the former group appear to combine chemically only. Both chemical and colloidal combination enter into the absorption of the latter group of synthetic tannins by pelt. High concentration and long treatment with synthetic tannins are unnecessary, especially with the former group, and may be distinctly harmful.

D. WOODROFFE.

Polluted atmosphere a factor in the deterioration of bookbinding leather. F. P. VEITCH, R. W. FREY, and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1926, 21, 156—176; cf. B., 1901, 819).—Leather from the back and sides of various old leather-bound volumes has been analysed. The leather in the binding most exposed to air and light was most deteriorated, and contained in the water extract the highest amounts of free mineral acid, of sulphates, and of nitrogen as determined by distillation with magnesia. The direct correlation between deterioration, chemical composition, and degree of exposure has been confirmed by comparative

analyses of leather from the sides, backs, top edges, and bottom edges of books, portions covered with skiver and uncovered portions, embossed and unembossed leathers. The increase in sulphur or sulphate content of leather during exposure must result from an absorption of those components from the air, and since bindings that had not been exposed to gas lighting were not exceptional, the deterioration resulting from exposure is attributed to the absorption of sulphurous and acidic impurities from the air of large towns. The nitrogen content shows that the deterioration of the leather is partly the result of a slow hydrolysis of the hide substance by the acid originally present and that taken up from the air. Short-life bindings had a high acidity (4.80—9.10%; 0.68—3.67% in the sides; determined by the Procter-Searle method), indicating the presence originally in the leather of excessive quantities of acid. Several very old bindings (91—161 yrs.) had a low acidity in the sides (1.6 to 2.8%) and some had only 0.1—0.5% of free acid in the back. Slight alkalinity or very low original acidity promotes longer life in leather bindings. Coatings, finishes, oils, greases, and waxes are beneficial as they prevent the absorption of atmospheric impurities.

D. WOODROFFE.

Acetylation of tannin. FERNÁNDEZ and FERNÁNDEZ.—See XX.

PATENTS.

Preparation of tanning materials. BADISCHE ANILIN- & SODA-FABR. (G.P. 420,593, 8.2.24).—The materials are prepared from substances of a coaly nature, fossilised or of recent origin, by the action first of dilute and then of stronger nitric acid. For example, finely powdered wood charcoal is boiled under reflux with 40% nitric acid for 6 hrs., and the paste obtained on filtration is slowly brought to boiling in 96% acid and boiled for two days. The solution yields on evaporation an orange-yellow soluble tanning material, applicable at once or after partial neutralisation. Humus coal, peat, and lignite also yield tanning materials on similar treatment.

B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of A. MITTASCH and O. BALZ (G.P. 420,645, 4.12.21).—The tanning is carried out by means of aqueous solutions of products obtained by the action of nitric acid on humic acid or substances rich in it, alone, or in the presence of other natural or synthetic tanning materials. The products are obtained by the action of nitric acid on substances obtained from humus coal freed from iron and bitumen. For example, humic acid or ammonium humate from humus coal is added to 65% nitric acid, and heated for some time at 70—80° with powerful stirring. The excess acid is distilled off, and a reddish-brown amorphous powder is obtained on purification by solution in water and evaporation.

B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of K. H. MEYER and H. SCHÜTTE (G.P. 420,646, 12.5.23).—Tanning may be

effected by means of the soluble products obtained by the action of concentrated sulphuric acid or its monohydrate on crude or purified mineral oil or mineral oil fractions. For example, machine oil is stirred with sulphuric acid monohydrate for a long time at a moderately high temperature. The sulphonated product is used in moderately acid dilute solution. Similar products may be obtained from petroleum, even when free from unsaturated hydrocarbons.

B. FULLMAN.

Tanning materials. BADISCHE ANILIN- & SODA-FABR., Assees. of M. EGNER (G.P. 420,647, 5.2.24).—Tanning materials are formed by mixing the formaldehyde condensation products of naphthalene-sulphonic acids or their salts with the substances obtained by the oxidation, with nitric acid or nitrous fumes, of fossilised vegetable matter, *e.g.*, lignite, peat, or coal. The formaldehyde-naphthalene-sulphonic acid condensation products serve the purpose of rendering the colour of the leather lighter. For example, the products obtained by the action of 40% nitric acid on humic acid or peat, or of 65% acid on lignite, are dried, and mixed with the formaldehyde-naphthalenesulphonic acid condensation product obtained in the presence of sodium hydroxide.

B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of R. ALLES (G.P. 420,648, 14.5.24).—Pelts are treated simultaneously or successively with solutions of tanning materials obtained by the oxidation (with nitric acid or nitrous fumes) of fossilised plant substances, *e.g.*, lignite, peat, or coal, and with quebracho extract or other pyrocatechol tannins, such as pine bark extract. The use of pyrocatechol tannins prevents the production of dark-coloured leather. The leathers may afterwards be tanned by pyrogallol tannins. For example, "Casseler Braun" (a lignite product) gives on oxidation with nitric acid a soluble tannin, which is mixed with sulphited quebracho extract or gambier extract and diluted, yielding a solution which tans a delimed pelt in 6–8 days, yielding a brown, full leather. The latter, tanned moist with oak wood extract of d 1.03 for 2 days in a fulling cylinder, yields a brown, somewhat lighter coloured leather.

B. FULLMAN.

Tanning materials from sulphite-cellulose waste liquors. M. HÖNG and W. FUCHS (Austr. P. 88,650, 10.4.20; G.P. 420,802, 17.8.21).—The waste liquor is neutralised with calcium hydroxide, and boiled with 1% of the hydroxide for 1 hr. The filtered liquid is concentrated to d 1.16–1.20, and treated with an acid forming an insoluble calcium salt. A substance having the properties of a tannin is obtained. By treating the aqueous solution of the calcium salts with sulphuric acid of d 1.84 to expel volatile organic acids and then with aluminium or chromic sulphate or chromic fluoride soluble aluminium or chromium salts of the ligninsulphonic acids are formed. The filtered liquid is concentrated *in vacuo* to d 1.26–1.285. These salts do not swell pelts as much as the free ligninsulphonic acids, and give a more compact leather.

B. FULLMAN.

Manufacture of gelatin. A. H. TOD (E.P. 249,736, 8.8.25).—Discoloration and deterioration of bone gelatin are due to the presence in the bone material of porous fragments which absorb and retain blood, dirt, etc. Such porous fragments are separated mechanically by means of a frame consisting of a ring, which fits closely within a vat, and is connected with a central stem by a series of radial rods on each of which is hinged a finely perforated flap adapted to close by gravity but held open by a catch rod. The bone material is immersed in a bleaching and disinfecting fluid of $d < 1.116$ in the vat, the frame with the flaps open is submerged in the liquid, the catch rods are then released so that the flaps close, and the frame is raised. The porous fragments float to the surface of the liquid and are removed by the separator.

D. WOODROFFE.

Electro-osmotic purification of glue and gelatin. W. RUPPEL and K. WOLF, Assrs. to AMER. ELECTRO-OSMOSIS CORP. (U.S.P. 1,577,660, 23.3.26. Appl., 13.3.24).—Glue, gelatin, and similar materials are purified by submission, in aqueous solution, to an electric current; the solution is separated from the electrodes by an anodic diaphragm of wool fabric impregnated with chromated gelatin and a cathodic diaphragm of parchment.

D. F. TWISS.

Production of artificial horn. F. SCHMIDT (E.P. 230,025, 26.1.25. Conv., 27.2.24).—Mixtures containing albuminous material are gelatinised by kneading with water admixed with a volatile, water-soluble solvent, such as alcohol, acetone, or ethyl lactate, capable of dissolving cellulose derivatives and not capable of acting alone as a gelatinising agent for albumins but capable of acting as such when admixed with water. Cellulose derivatives are separately kneaded with gelatinising agents containing the same volatile solvent, or a mixture of it with water, or water alone, and the two masses are then incorporated by kneading. For example, two mixtures, one containing 30 kg. of nitrocellulose, or a mixture of it with camphor or with camphor substitute, 10 kg. of glycol diacetate, and 20 kg. of alcohol, and the other containing 60 kg. of casein, 5 kg. of glycol diacetate, 5 kg. of alcohol, and 10 kg. of water are kneaded together to a homogeneous mass in an open kneading machine, followed, if necessary by compression or rolling.

L. A. COLES.

Extracting oil etc. from whale, whale blubber and flesh, fish products, etc. (E.P. 232,601).—See XII.

Pyrrrole etc. from animal waste (U.S.P. 1,572,552).—See XX.

XVI.—AGRICULTURE.

Determination of available phosphoric acid of calcareous soils. S. DAS (Mem. Dep. Agric. India, 1926, 8, 69–104).—The use of 1% citric acid as a solvent for available phosphoric acid in

soils, when applied to calcareous soils, amounts, in effect, to extraction with solutions which vary in composition according to the amount of calcium carbonate present. Addition of more citric acid to neutralise the carbonate gives unreliable results. Experiments with ammonia and various salt solutions (1% ammonium citrate, sodium sulphate, ammonium sulphate, ammonium carbonate, and others) as solvents for available phosphoric acid are recorded. It was eventually found that the use of a 1% solution of potassium carbonate gave reliable results, agreeing well with expectation in the case of soils of known history and treatment. It is recommended that, for the determination of available phosphoric acid in calcareous soils, 100 g. of soil should be shaken for 24 hrs. with 1 litre of 1% potassium carbonate solution and the phosphoric acid in the extract determined by the ammonium molybdate method.

C. T. GIMMINGHAM.

Significance of soil reaction in practical agriculture. M. TRENEL (*Z. Pflanz. Düng.*, 1926, B5, 169—181; cf. B., 1925, 858).—Examples are given of the practical value of detailed "reaction maps" of agricultural land, which should include information as to the reaction of the subsoils. Determinations of the p_H values by the electrometric method on soil suspensions in potassium chloride solution give better correlation with crop growth than measurements in water suspensions. The soil reaction limits of various crops are given.

C. T. GIMMINGHAM.

Colloidal behaviour of soils and soil fertility. II. The soil complex capable of base exchange and soil acidity. J. S. JOFFE and H. C. MCLEAN (*Soil Sci.*, 1926, 21, 181—195; cf. B., 1925, 858).—The problem of soil acidity in relation to base exchange is discussed. It is considered to be a question of the state of saturation or unsaturation of the soil complex capable of base exchange. The titratable acid liberated by *N*-barium chloride and potassium chloride respectively was determined for a number of soils of different origin and manorial history. The speed of cation replacement is affected by the colloidal nature of the soil and by the concentration of replaceable cations. Replacement reactions may be slowed down by temperature changes which produce coagulation. The time-rate curves are typical of adsorption reactions.

G. W. ROBINSON.

Residual effects of neutral salt treatments on the soil reaction. C. H. SPURWAY and R. H. AUSTIN (*Soil Sci.*, 1926, 21, 71—74).—Soils from varying depths were treated with neutral solutions of metal chlorides and after leaching out soluble products, the residual soil reaction was determined. The p_H values of the treated soils were increased, the order of effectiveness of the cations being calcium, magnesium, potassium, and sodium. Calcium chloride caused only very slight changes in reaction. Increased p_H values are ascribed to the increased solubility and hydrolysis of the soil material containing the fixed cation.

A. G. POLLARD.

Use of the quinhydrone electrode for measuring the hydrogen-ion concentration of soils. L. D. BAVER (*Soil Sci.*, 1926, 21, 167—179).—The saturated calomel cell is the most convenient for use with the quinhydrone electrode. The quinhydrone electrode gives results which agree closely with those obtained by the hydrogen electrode; with soils below p_H 8.0, the potential rapidly attains constancy. For accurate results 0.05 g. of quinhydrone per 15 c.c. of solution is a suitable concentration. The most desirable soil to water ratio is 1:1.

G. W. ROBINSON.

Production and use of sulphate in humid and arid soils as affected by cropping and sulphur treatments. W. W. JOHNSTON (*Soil Sci.*, 1926, 21, 233—244).—The formation of sulphate from sulphur applied to the soil is retarded in arid soils by growing crops which respond to sulphur treatment. In the case of humid soils crop growth increases the production of sulphate. The sulphate supply is generally sufficient under humid conditions owing to the greater ability of crops to obtain sulphur consequent on the higher hydrogen-ion concentration of humid soils. Such soils may recuperate more readily than arid soils in their sulphate content. This may explain the marked effect sometimes obtained by application of sulphur to semi-arid soils which have grown lucerne continuously for a long period.

G. W. ROBINSON.

Influence of available nitrogen on the fermentation of cellulose in the soil. J. A. ANDERSON (*Soil Sci.*, 1926, 21, 115—126).—The decomposition of cellulose in the soil increases with the supply of available nitrogen up to the amount of available nitrogen necessary for maximum growth of micro-organisms, which, in the case of the experiments described, corresponds with a ratio of nitrogen to cellulose equal to 1:35. The ratio of cellulose fermented to nitrogen utilised decreases with increasing supply of nitrogen. Although nitrification can proceed in the presence of cellulose the nitrate produced is immediately utilised unless present in amounts more than sufficient for the requirements of bacteria. Ammonia is available for cellulose bacteria without being first converted into nitrates. Addition of carbohydrates to soil does not result in an inhibition of the soil processes. Crop failures are due to the competition of cellulose bacteria with ordinary crops for the supply of available nitrogen. Urea, aspartic acid, asparagine, alanine, tyrosine, peptone, casein, and the nitrogenous compounds of stable manure can also serve as sources of nitrogen for cellulose bacteria. In the case of the more complex compounds their availability would appear to depend on the rate at which they are hydrolysed to the amino-acid stage.

G. W. ROBINSON.

Present position and previous experience in regard to fermented manure preparations. J. WEIGERT (*Z. Pflanz. Düng.*, 1926, B5, 145—161). **So-called hot fermentation of farmyard manure.** O. LEMMERMANN (*ibid.*, 162—168).—The results of

a number of experiments with fermented manure preparations and the changes taking place during the fermentation are discussed, with special reference to the material known as "*Edelmist*." The agricultural value of this manure has not yet been accurately determined on account of the difficulty of obtaining a satisfactory basis of comparison with ordinary farmyard manure, but it is concluded that such preparations are well worth further investigation.

C. T. GIMMINGHAM.

Availability of nitrogen in garbage tankage and urea in comparison with standard materials.

A. L. PRINCE and H. W. WINSOR (Soil Sci., 1926, 21, 59—68).—Pot experiments with barley, rape, and sorghum indicated that the fertiliser value of garbage tankage was very low. The whole of the nitrogen present, although small, becomes very slowly available. The chief value of the material is probably as a filler. In all cases urea was found to give better crop yields than ammonium sulphate and very nearly equalled sodium nitrate. The relative availability of these fertilisers was sodium nitrate, 100; ammonium sulphate, 88.2; standard tankage, 53.4; fish, 49.2; and garbage tankage, 14.2. Chemical processes for comparing the availability of nitrogenous fertilisers proved of little value. The rate of conversion of urea to ammonia was investigated in soil and sand mixtures. In soil 90% conversion occurred in 5 days. Acid soils retarded the rate of conversion, and in these cases applications of lime brought about an acceleration, proportional to the amount of lime added.

A. G. POLLARD.

Potassium ferrocyanide and ferric ferrocyanide as sources of iron for plants. C. G. DEUBER (Soil Sci., 1926, 21, 23—26).—*Spirodela polyrhiza* and soya bean plants made fair growth in culture solutions containing 0.033 and 0.066 pt. per million of iron supplied as potassium ferrocyanide. Toxic effects were noted when larger concentrations of this salt were used. Ferric ferrocyanide was used satisfactorily as a source of iron for soya beans in solutions having a reaction of p_H 5.0. Growth in solutions of less acidity was unsatisfactory.

A. G. POLLARD.

Deleterious action of smoke gases on vegetation in the light of metabolism pathology. K. NOACK (Z. angew. Chem., 1926, 39, 302—304).—A series of experiments on the action of sulphur dioxide, with and without the assistance of light, on the growth of the moss, *Fontinalis*, gave results which appear to indicate that sulphur dioxide arrests the assimilation of carbon dioxide by the plant, and the photo-oxidative energy of the chlorophyll, which finds its normal acceptor in carbon dioxide, is diverted to other acceptors present in the protoplasm and in the chlorophyll itself, so that the cells are rapidly killed and the chlorophyll is bleached.

A. R. POWELL.

PATENTS.

Arsenic preparations. [Insecticides and fungicides.] J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 248,975, 10.6.25).—Solutions of the yellow modification of arsenic are prepared

by passing the vapours of a suitable solvent (*e.g.*, carbon disulphide or benzene) over arsenic heated in a tube. The condensate contains yellow arsenic in the form of a solution which is stable in daylight and at ordinary temperatures. Insecticides and fungicides are prepared by emulsifying the arsenic solution with solutions of soap or saponin or Turkey-red oil.

A. G. POLLARD.

Preparation of insecticides. W. K. SCHWEITZER, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,574,118, 23.2.26. Appl., 29.9.24).—Dimanganous arsenate, $MnAsO_4$, prepared by heating a mixture of manganese dioxide and arsenious oxide with water at 100°, contains about 3% of soluble arsenic oxide. This can be reduced by heating the product for 18 hrs. with water and about 5% of a compound which forms with dimanganous arsenate a trimanganous arsenate. Suitable compounds are the neutral or slightly alkaline carbonates of metals which form insoluble arsenates, *e.g.* magnesium, calcium, or manganese. When manganese carbonate is used the product has the formula $Mn_3(AsO_4)_2$ and the content of soluble arsenic oxide is reduced to 1—1.5%. Trimanganous arsenate is a white powder which is of value as an insecticide.

T. S. WHEELER.

Making a calcium nitrate fertiliser. BADISCHE ANILIN- & SODA-FABR., Assees. of W. WILD and K. EYER (U.S.P. 1,579,989, 6.4.26, Appl., 29.6.25).—See E.P. 246,377; B., 1926, 293.

Arsenates (U.S.P. 1,578,150).—See VII.

Di- and poly-piperidyls (G.P. 420,445).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Small-range refractometer. H. SCHULTZ (Chem.-Ztg., 1926, 50, 265—266).—The optical arrangement is similar to that of the Goerz sugar-refractometer and the range is from 1.33299 to 1.39214 corresponding with the refractive indices of pure water and a 36% sugar solution respectively. With suitable temperature control, results having an accuracy ten times greater than those given by the ordinary sugar-refractometer are obtained. E. H. SHARPLES.

Application of Lewis filtration equation. WEBER and HERSHEY.—See I.

Behaviour of starches towards dyes. HUEBNER and VENKATARAMAN.—See VI.

PATENTS.

Production of maltose from starchy materials. H. C. GORE, and FLEISCHMANN Co. (E.P. 249,428, 22.12.24. Addn. to 226,812; B., 1925, 733).—An aqueous starchy suspension, adjusted to a hydrogen-ion concentration corresponding to p_H 4.6—6, is saccharified by the addition of about 5% of malt or other diastatic agent, and maintained at 45—55° for 5—10 days in the presence of toluene as preservative. After filtering, the liquid is concentrated to a syrup, inoculated with maltose crystals, and allowed to solidify to a fondant-like mass, which is subdivided, cured by subjecting it to a suitable temperature, *e.g.*,

50°, until the particles become dry, and finally comminuted to a dry powder. C. RANKEN.

Clarifying saccharine liquors. I. H. MORSE (U.S.P. 1,573,733, 16.2.26. Appl., 29.6.25).—Cane sugar juice is treated with sulphur dioxide, and then caused to flow slowly upwards through a cylindrical tank in which it is heated. By means of a rotating cone near the top of the tank it is forced in the form of a thin whirling film into an annular collecting tank, while milk of lime fed to the upper side of a rotating disc above the cone forms a fine spray which falls on the thin film and is mixed intimately with it. The juice flows successively through other similar tanks in which it is treated with further lime and subsequently with phosphoric acid, the precipitate formed in each tank being separated. By adding the bulk of the lime in the first tank and accurately measured small quantities in the others the juice can readily be brought to exact neutrality. T. S. WHEELER.

Multichamber apparatus for conducting leaching, washing, or like operations on solid or semi-solid material [e.g., removing sugar from plant cuttings]. W. RAABE (U.S.P. 1,575,936, 9.3.26. Appl., 7.2.25).—The material is subjected to counter-current treatment in a drum provided with partitions to form leaching chambers. Members are provided for lifting the non-liquid material over the partitions, and over surface members, pervious to liquids, arranged adjacent to the partitions. H. HOLMES.

Manufacture of edible sugar from wood. L. PINK (G.P. 425,023, 7.6.22).—Wood pulp mixed with hydrogen peroxide is saturated with chlorine, and, after thorough washing, the product is saccharified by means of dilute acid. L. A. COLES.

Lactose from whey (U.S.P. 1,571,626).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Comparative effects of pressing and draining yeast. D. H. F. FULLER and F. E. B. MORITZ (J. Inst. Brew., 1926, 32, 171—176).—Drainings or pressings from yeast have a higher content of phosphoric anhydride, nitrogen, and alcohol, than the beer from which the yeast was obtained, the excess representing the amount of these substances lost (or formed) by the yeasts when drained, pressed, or filtered. Pressed yeast loses substantially less nitrogen and phosphorus than does drained or filtered yeast, and less alcohol is found in pressings than in drainings, although the latter are, in view of the temperature, more subject to loss by evaporation. The main factor determining the amount lost by the yeast when it is separated from its surrounding beer is not pressure but the time taken in separation. The temperature, however, also has considerable influence. C. RANKEN.

Industrial manufacture of absolute alcohol. H. GUINOT (Chim. et Ind., 1926, 15, 323—330).—A process is described for the rectification of alcohol based upon that previously recorded (B., 1925, 821).

The presence of impurities in the alcohol does not in many cases affect the purity of the final product, and slight modifications are introduced to deal with particular difficulties. In a few instances, notably that of isopropyl alcohol, complete separation is impossible by this process. Methyl alcohol as an impurity can be completely removed, and in view of this, the substitution of isopropyl for methyl alcohol in denaturants is suggested. The cost of production of absolute alcohol by this means is quite as low, if not lower than that of the ordinary 96% rectified spirit. A. G. POLLARD.

Detection of ethyl phthalate in spirits. R. SCHMITT (Z. Unters. Lebensm., 1926, 51, 56—57).—The test for the detection of ethyl phthalate depending upon the formation of a violet-red colour when it is mixed with pyrogallol and sulphuric acid, is not conclusive. The colour only differs from that produced by pyrogallol and sulphuric acid alone by its redder shade and it is recommended, that, for the detection of ethyl phthalate in spirits and liqueurs, the colour produced by the above test should be compared with that given by a mixture of ethyl phthalate and a pure spirit, adulteration only being proved when the shades of the two colours are identical. E. H. SHARPLES.

Alcohol and other by-products from pea-nut shells. DE BELSUNCE.—See XII.

Action of lime on enzymes. COLLETT.—See XV.

Fermentation of cellulose. ANDERSON.—See XVI.

PATENTS.

Fermenting cellulosic materials. H. LANGWELL (E.P. 248,795, 10.9.24 and 23.10.24).—Improvements in the process described in E.P. 134,265 (B., 1920, 38 A) are claimed. The fermenting mash is so treated as to maintain the hydrogen-ion concentration between the limits of 10^{-9} and 10^{-5} , by the use of suitable phosphates, carbonates, or oxides of alkali or alkaline-earth metals] or of magnesium or ammonium. The cellulosic material may be in the form of naturally occurring products, e.g., fresh grass, bamboo, etc., alone or mixed with sulphite pulp, dry maize cobs, etc. Where green material is used artificial inoculation may be unnecessary. The mass should be stirred or agitated. Dried materials should be pulverised and green material crushed or pulped. A. G. POLLARD.

Process of manufacturing yeast. H. W. DAHLBERG (U.S.P. 1,580,999 and 1,581,000, 13.4.26. Appl., 8.6.22 and 5.3.25).—See E.P. 246,002; B., 1926, 295.

Cheese with increased vitamin content (E.P. 226,549).—See XIX.

XIX.—FOODS.

Storage of eggs. T. MORAN and J. PIQUÉ (Food Investigation Special Rep., 1926, [26], 1—61, 62—80; cf. B., 1925, 897).—Success in the cold storage of eggs is dependent on the following factors: the

eggs must be fresh and free from cracks either of the shell or of the membrane, these points being determined by "candling"; previous to the storage period the eggs should not be exposed to a temperature exceeding 15°. Cleanliness both of the eggs themselves and of the packing material is essential. The temperature of the store should be maintained at 0—0.6° and the air should be in constant circulation or at least frequently renewed, and the optimum humidity is 80% saturation; a drier atmosphere leads to loss of weight of the eggs by evaporation, a more humid one encourages the growth of moulds. On removal from the store the temperature of the eggs should not be allowed to rise rapidly.

C. R. HARRINGTON.

Rapid sorting test for small quantities of tartaric acid in self-raising flour. A. F. LERRIGO (*Analyst*, 1926, 51, 180—181).—The flour (about 4 g.) is shaken with 20 c.c. of water, left for 2 min., filtered, and a few drops of dilute ammonia are added, with shaking, to 10 c.c. of the filtrate. About 0.05 g. of powdered silver nitrate is dropped in, and the test-tube placed in water at 70° without shaking. After 2 min. the tube is shaken, when a flour free from tartrate gives a pale yellow turbid liquid, while if tartrate is present the colour produced varies from pale grey to black according to the amount present. Even with 0.1—0.5% of tartrate a mirror is not produced under these conditions.

D. G. HEWER.

Examination of fruit juices. H. ECKART and A. DIEM (*Z. Unters. Lebensm.*, 1926, 51, 48—56).—For the accurate evaluation of fruit juices, in addition to the results of analysis, a series of standards are necessary which must be systematically determined for each harvest period. From the results of numerous investigations of representative samples of raspberry juice, correlating the refractive index, density, sugar content, and acidity, is derived a normal type of raspberry juice (n_D 1.3467, d 1.0374, sugar 9.2% on dry substance, total acid 1.47%) for the 1925 harvest. Abnormal juices and syrups have been examined and the influence of fermentation and dilution with water on the physical and chemical characteristics is fully discussed. The refractometer is recommended for rapid and accurate practical control of fruit juice manufacture. E. H. SHARPLES.

Determination of pectin. C. F. AHMANN and H. D. HOOKER (*Ind. Eng. Chem.*, 1926, 18, 412—414).—To a solution containing 0.25—1.0 g. of pectin in 200 c.c. a known quantity (50 c.c.) of alkali is added so that the concentration of the alkali will be about 0.1*N*. The solution is made up to 250 c.c. and kept at 55° for 12 hrs. in a vessel sealed to prevent entrance of carbon dioxide. An aliquot portion is then pipetted off and titrated with hydrochloric acid which should be of about a quarter the strength of the alkali. Taking the neutralisation equivalent of pectic acid at 55° as 194.9 (equivalent to 208.9 g. of pectin) the amount of pectin (x) may be calculated from the amount of alkali used, since $\text{NaOH} : \text{pectin} = \text{weight of alkali combined} : x$. Results obtained by this method

compare well for pure pectin with those obtained by precipitation, but for plant material the titration method is better as it avoids interference of occluded material. The neutralisation equivalent of pectic acid should be determined for the conditions adopted in the determination.

D. G. HEWER.

Legumin. M. A. RAKUSIN and G. PEKARSKAJA (*Z. Unters. Lebensm.*, 1926, 51, 43—45).—The preparation and properties of legumin from legumes are described. Its chief reactions compared with those of casein show complete analogy. Like casein, it contains phosphorus, is lævo-rotatory (in pepsin-hydrochloric acid solution), gives the same protein and carbohydrate reactions, is soluble in ammonia and alkalis forming water-soluble leguminates similar in optical properties to the alkali salts of casein and is precipitated from these solutions by dilute acids. The titer, obtained by the determination of silver in silver leguminate, approximates closely to that of casein.

E. H. SHARPLES.

Glass for pharmaceutical purposes. TIRELLI.—See VIII.

Determination of milk fat in mixtures. GROSSFELD.—See XII.

Determination of coconut oil and milk fat. BAUMANN, KUHLMANN, and GROSSFELD.—See XII.

New value for milk fat. KUHLMANN and GROSSFELD.—See XII.

PATENTS.

Preparation of cheese having an increased vitamin content. MELLEMEUROPEISK PATENT-FINANCIERINGS-SELSKAB A./S., Assecs. of HAMBURGER & Co., and H. LIEBERS (E.P. 226,549, 18.12.24. Conv., 21.12.23).—The vitamin-containing addition consists of yeast which is incorporated in the fresh or liquefied state with the finished cheese. The yeast is pasteurised prior to or after its incorporation so that the proteolytic enzymes are rendered inactive while the other enzymes and vitamins are only slightly detrimentally influenced. Bacteria in the cheese may be destroyed by heat prior to incorporation with the yeast, or proteolytic enzymes of the yeast and bacteria of the cheese may be rendered ineffective simultaneously by kneading the yeast and cheese together under the influence of heat. A cheese of the rindless Emmenthal type is produced by heating the cheese pulp until of a doughy consistence, then cooling to 60—70°, and intimately kneading with fresh or liquefied yeast.

C. RANKEN.

Artificial drying of crops. B. J. OWEN (E.P. 248,935, 31.3.25. Addn. to 235,273, cf. B., 1925, 648).—The improvement on the prior patent described consists in the arrangement of the supply duct conveying air to the central distributing space within the stack. The duct is bell-mouthed and carried to the distributing space through a channel excavated in the ground beneath the stack.

A. G. POLLARD.

Process of obtaining milk sugar [lactose from whey]. H. V. DUNHAM, Assr. to ROSEMARY CREAMERY Co. (U.S.P. 1,571,626, 2.2.26. Appl., 30.1.23).—In the preliminary treatment of whey for the preparation of lactose, the bulk of the albumin is precipitated by boiling the whey at atmospheric pressure by direct fire heat, currents of air being blown against the surface of the liquid to prevent excessive foaming. The boiling is continued until the liquid reaches at least d_{4}^{20} 1.16. At this stage the precipitated albumin has been converted into a readily filterable condition and after separating it the whey is further concentrated by boiling at atmospheric pressure.

D. G. HEWER.

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

Choice of indicators for alkaloidal titrations.

H. WALES (Ind. Eng. Chem., 1926, 18, 390—392).—The range of colour change for a given indicator was found by dissolving 100 mg. of the alkaloid in a slight excess of acid, diluting to 50 c.c., and titrating the excess acid with alkali, using quinhydrone and saturated calomel electrodes and recording voltages at definite intervals. The variation in voltage was plotted against the volume of alkali used, the end-point (centre of break) of the titration determined, and the hydrogen-ion values computed. An indicator is most suitable for a given titration when its mid-point of colour range lies in the region of rapid change in hydrogen-ion value. The following indicators or others of similar range are recommended for the alkaloids named. Morphine, codeine, and nicotine, methyl-red, narcotine, hyoscyne, and delcosine, bromophenol-blue, cotarnine, propyl-red or bromocresol-purple, but the yellow colour of the alkaloid may mask the colour of the latter; papaverine is a very weak base and, while no indicator was satisfactory, bromophenol-blue has its colour change at the right point; narceine is apparently completely dissociated in aqueous solution; hydrastine, no indicator satisfactory as the break (in the bromophenol-blue range) is poorly defined; quinine hydrochloride and quinidine, bromocresol-purple and propyl-red; cinchonine, bromocresol-purple to the first change of colour, or propyl-red; strychnine, methyl-red or propyl-red and bromocresol-purple; sparteine, bromothymol-blue for indication of the total amount of acid radical present. D. G. HEWER.

Ether. III. Determination of aldehyde as a contaminant. E. P. PHELPS and A. W. ROWE (J. Amer. Chem. Soc., 1926, 48, 1049—1053; cf. A., 1924, ii, 876).—A standard Schiff's reagent solution is prepared by mixing 30 c.c. of 0.1% aqueous fuchsin solution with 200 c.c. of saturated sulphur dioxide solution, shaking, adding 3 c.c. of concentrated sulphuric acid, keeping the mixture for 24 hrs., and filtering if necessary. Pure acetaldehyde is prepared by depolymerising paraldehyde with dilute sulphuric acid and distilling through a 90-cm. Hempel column. To each of two cylindrical containers 10 c.c. of pure alcohol are added. To one, 1 c.c. of a standard solution

of the pure aldehyde in pure ether and 4 c.c. of ether are added, and to the other are added 5 c.c. of the ether sample under examination. The mixtures are kept for 15 min., and if the colours do not nearly match the procedure is repeated using different quantities of the standard aldehyde solution until approximately equal colorations are obtained in the two tubes, which are finally compared in a colorimeter. The depth of colour is not proportional to the concentration of aldehyde present, but by the above matching process accurate determinations can be made, and positive results are given with an aldehyde content of 0.003%. F. G. WILLSON.

Reagent for the recognition of neosalvarsan unfit for [therapeutical] application. A. KIRCHER and F. VON RUPPERT (Pharm. Ztg., 1926, 71, 264—265).—Strontiuran (strontium chloride-carbamide solution) is described by Hirsch (Mönc. Med. Woch., 1925, 1549) as giving a turbidity with impure (oxidised) neosalvarsan. The appearance or non-appearance of the turbidity, however, depends on the manner in which the neosalvarsan and strontiuran are mixed, different results being obtained according to whether the neosalvarsan is added to the strontiuran solid or in solution, or the strontiuran solution is added to the solid neosalvarsan. Hirsch's further statement that neosalvarsan condemned by his test gives iodine values (c.c. of 0.1N-iodine equivalent to 1 g. of neosalvarsan) considerably below 149 is also incorrect, since normal values may be obtained.

B. FULLMAN.

Acetylation of tannin and analysis of commercial acetyltannins. O. and J. M. FERNÁNDEZ (Anal. Fis. Quím., 1926, 24, 62—67).—Tannin (pentadigalloylglucose) and digallic anhydride give almost identical products on acetylation. Complete acetylation of tannin, involving the entrance of 25 acetyl groups into the molecule, was effected by the action of excess of acetic anhydride in the presence of small quantities of sulphuric acid. A less completely acetylated product containing 19—20 acetyl groups in the molecule was obtained by acetylating in the presence of a quantity of ethyl acetate equal to the acetic anhydride used. The completely acetylated product is too highly astringent for pharmaceutical purposes. G. W. ROBINSON.

Examination of commercial tannic acid by comparative methods. W. B. FORBES (Pharm. J., 1926, 116, 225—229).—Hooper's cinchonine method (B., 1925, 369) and a modification of Crouzel's phenazone method (A., 1903, ii, 113) appear to be the most satisfactory for the determination of gallo-tannic acid in astringent drugs. The modification of Crouzel's method consists in the addition of the tannic acid to excess of phenazone solution without any sodium bicarbonate; on filtration through a Jena glass filter a clear filtrate is obtained. The precipitate after drying at 100° contains 58% of tannic acid. Four samples of tannic acid B.P. gave by the above methods:—tannic acid 81.7—85.9%, gallic acid 6.4—6.9%, water 8.0—9.38%. Gallic acid was determined by Dreaper's volumetric copper

method (A., 1904, ii, 793), a factor being obtained from experiments with the pure acid, viz., gallic acid = $\text{CuO} \times 0.92$.

E. H. SHARPLES.

Aldehydes of peppermint oil. R. E. KREMERS (Amer. J. Pharm., 1926, 98, 86—89).—The aldehydes were separated from crude peppermint oil (d 0.869, aldehydes 6%, ester value of aldehyde-free oil 8.2) by means of the bisulphite compounds and fractionated. The fractions had the physical characters of aldehydes of the aliphatic series and the predominating fractions, which were slightly optically active, were within the distilling range of the valeraldehydes. *iso*Valeraldehyde, characterised by conversion into *isoleucine*, was the most important constituent quantitatively. The presence of *d*-methyl-2-butanal (*a*-methyl-*n*-butaldehyde) was not confirmed, and the substance responsible for the optical activity could not be identified. Contrary to the observation of Curtius and Franze (B., 1912, 706), the presence of hexen-2-al-1 ($\alpha\beta$ -hexylenealdehyde) could not be detected. E. H. SHARPLES.

Essential oils from Irish-grown plants.

I. Oil of lavender. J. REILLY, P. J. DRUMM, and C. BOYLE (Econ. Proc. Roy. Dublin Soc., 1926, 2, 273—284).—Preliminary investigations on the cultivation of lavender in Ireland. A sample of the mixed oils from *Lavandula vera*, vars. *angustifolia* and *delphinensis* had d 0.8970, ester value 15.8, acid value 0.9, a (100 mm.)—6.3, and n_D 1.4678. These characteristics compare favourably with those of English lavender oil. E. H. SHARPLES.

Determination of ascaridole in chenopodium oil. H. PAGET (Analyst, 1926, 51, 170—176).—

In view of the ready conversion by heat of ascaridole into ascaridole glycol anhydride which is easily hydrated (by steam) to ascaridole glycol, these last two substances must be present in chenopodium oil, and since like ascaridole they are miscible with 60% acetic acid, the U.S.P. method of determination of ascaridole must be considered untrustworthy. A reduction method by means of titanous chloride has been worked out whereby 1:4-terpin is formed by addition of 4 atoms of hydrogen to ascaridole. 1 g. of chenopodium oil is diluted to 200 c.c. with 96% alcohol, and to 10 c.c. of this, titanous chloride (66 c.c. of the commercial 15% solution made up to 2250 c.c. and standardised) is added in excess (about 50 c.c.) in a flask through which a current of carbon dioxide is passing. The flask is then closed with a Bunsen valve and the contents are heated almost to boiling point for 1—2 min.; if the pale violet colour disappears more titanous chloride must be added. About 1 c.c. of 5% potassium thiocyanate is then added, the solution titrated with a standard solution of iron alum, and the quantity of titanous chloride oxidised found. Until the products of reduction have been studied 1 g. of ascaridole is taken to be reduced by 1.2770 g. of titanous chloride, which is the mean of several determinations. D. G. HEWER.

Colour reaction of saponin with nitrates. MITCHELL.—See VII.

PATENTS.

Production of unsymmetrical arsenobenzene compounds. DEUTSCHE GOLD- & SILBERSCHEIDEANSTALT VORM. ROESSLER, and A. ALBERT (E.P. 249,584, 24.11.24).—Readily soluble, unsymmetrical arsenobenzene compounds of therapeutic value, are prepared by reducing, e.g., with sodium hyposulphite, mixtures containing: (i) aromatic aldehyde-arsinic acids, aromatic keto-arsinic acids, or mixed aliphatic-aromatic keto-arsinic acids, or the corresponding arsenoxides, prepared as described in E.P. 199,091 and 220,668 (cf. B., 1923, 862 A; 1924, 966); (ii) condensation products prepared by the action of hydrazine or its derivatives upon the arsinic acids or arsenoxides mentioned above; (iii) condensation products of the arsinic acids or arsenoxides mentioned above, with organic compounds containing reactive amino-groups, other than hydrazine or its derivatives, (cf. E.P. 249,588, following abstract); and (iv) ter- or quinquivalent organic arsenic compounds such as halogen-, nitro-, hydroxy-, or amino-aryl arsenoxides, or carboxy- or sulpho-arsinic acids, or glycine-arsinic acids. The mixtures to be reduced contain approximately equimolecular proportions of two different compounds from groups i, ii, or iii, or of one of the compounds in any of these groups and one compound in group iv.

L. A. COLES.

Production of derivatives of organic arseno-compounds. DEUTSCHE GOLD- & SILBERSCHEIDEANSTALT VORM. ROESSLER, and A. ALBERT (E.P. 249,588, 28.11.24. Addn. to 199,092; cf. B., 1923, 862 A).—The compounds are prepared by the action of organic compounds containing reactive amino-groups, such as hydroxylamine and its derivatives, aniline, carbamide, glycine, *p*-aminoacetophenone, *o*-aminobenzaldehyde, and sulphanilic acid, but not including hydrazine and its derivatives, on aromatic aldehyde-arsinic acids or mixed aliphatic-aromatic keto-arsinic acids, or the corresponding arsenoxides. The compounds may subsequently be treated with reducing agents for conversion into the corresponding arsenoxides or arsenobenzene derivatives, without affecting the C:N linkage.

L. A. COLES.

Production of alkyl esters [formates]. BADISCHE ANILIN- & SODA-FABR., Assees. of R. WIETZEL (U.S.P. 1,572,698, 9.2.26. Appl., 20.5.25).—A solution of sodium methoxide in methyl alcohol containing about 12% Na is treated at 30—150° with carbon monoxide free from iron carbonyl, sulphur, and water, at pressures of 60—1000 atm. At 80° and 60 atm. 75% of the methyl alcohol is converted into methyl formate in 3 hrs.; at 1000 atm. conversion is complete in a few minutes. The product is withdrawn or distilled from the autoclave, and the sodium methoxide, which is insoluble in methyl formate, remains in the autoclave and is used as catalyst for the next batch. Ethyl formate is prepared in a similar manner.

T. S. WHEELER.

Condensation products of compound aliphatic-aromatic ketones with polyalcohols. J. ALTWEGG

and E. F. CHERMETTE, Assrs. to SOC. CHIM. USINES DU RHÖNE (U.S.P. 1,572,176, 9.2.26. Appl., 19.3.24).

—Products similar to acetoneglycerol are obtained by allowing alkyl-aryl ketones, e.g., acetophenone, to react with polyhydroxy-compounds in presence of a primary alcohol containing a small quantity of a mineral acid at about 0° for periods of from 1 hr. to 2 days. With excess of ketone practically quantitative yields are obtained. Suitable polyhydroxy-compounds include glycol, glycerin, chlorohydrin, monoacetin, ethyl tartrate and citrate. The products are colourless oils or solids soluble in the usual organic solvents but not in water. They can be distilled under reduced pressure and are of value as solvents, as plasticising agents for cellulose esters, and therapeutically, possessing sedative and slightly hypnotic properties. For example, 450 pts. of ethyl alcohol, 200 pts. of glycerol, and 5 pts. of sulphuric acid are treated at -10° with 500 pts. of acetophenone, and the cold mixture is neutralised with sodium carbonate after 24 hrs. and filtered. The alcohol and excess of ketone are removed by distillation, the latter under reduced pressure. *Acetophenoneglycerol* then distils at 134°/5 mm. It forms a viscous oil d^{20}_4 1.159. *Propiophenoneglycerol* has b.p. 140°/5 mm. and d^{20}_4 1.1255, *acetophenoneglycol* has m.p. 62°, b.p. 130°/40 mm., and *acetophenone-monochlorohydrin* has b.p. 153°/40 mm.

T. S. WHEELER.

Obtaining pyrrole, pyrrole derivatives, and pyrocoll from animal waste. J. MICHELMAN (U.S.P. 1,572,522, 9.2.26. Appl., 11.11.24).—Collagenous tissue such as glue stock from slaughter-houses or leather stock from tanneries is freed from glycerin and glycerides by treatment with lime and heated out of contact with air. The distillate separates into two layers, an aqueous and an oily layer. The oily layer is diluted with toluene or the like, when pyrocoll separates. The liquid is distilled and the fractions to 200° and 200—300° are collected. The first fraction, which contains toluene and pyrrole and substituted pyrroles, is treated with solid potassium hydroxide, when the potassium salts of pyrrole and the substituted pyrroles separate. The higher fraction contains pyrrole derivatives and sometimes hydrocarbon oils, which if present separate as an upper layer.

T. S. WHEELER.

Phenolphthalein. E. B. PUTT (U.S.P. 1,574,934, 2.3.26. Appl., 10.7.25).—100 pts. of pure crystalline phenolphthalein are dissolved in 100 pts. of water, containing half its volume of 50% sodium hydroxide solution, at the ordinary temperature, and the solution is diluted with 1800 pts. of water. Dilute acetic acid and then dilute hydrochloric or other mineral acid are added until the phenolphthalein is completely precipitated, the temperature being maintained below 40°. After keeping for 1 hr. the precipitate is filtered off, washed, and dried at 100° for 8 hrs. Pure phenolphthalein is thus obtained as a white, bulky, amorphous powder, more soluble in alcohol and alkali than crystalline phenolphthalein

and possessing on this account greater laxative properties.

T. S. WHEELER.

Preparation of ethyl chloride. CHEM. FABR. VORM. WEILER-TER MEER, Assees. of H. SUIDA (G.P. 420,441, 5.2.24).—Ethyl chloride is prepared by passing a mixture of ethylene, or gases containing it, with hydrogen chloride, under atmospheric or increased pressure, over porous material impregnated with the liquid double compound of aluminium chloride and ethylene, (C₂H₄.AlCl₃)_x, obtained by passing ethyl chloride over powdered aluminium chloride at 100—120°, until it is converted completely into a dark brown liquid. On passing a mixture of hydrogen chloride with 95% ethylene through a tube containing the catalytic material at 90°, removing excess hydrogen chloride by washing with water, and recovering the ethyl chloride by cooling and compression, a yield of about 66% is obtained; the yield is higher, however, if increased pressure is used and the temperature may be considerably lower.

L. A. COLES.

Preparation of formaldehyde from methyl alcohol. BADISCHE ANILIN- & SODA-FABR., Assees. of A. BERTRAM and E. KUSS (G.P. 420,442, 29.2.24).—Synthetic methyl alcohol is freed from volatile iron compounds (iron carbonyl) before it is oxidised catalytically to formaldehyde, by passing it, before or after admixture with the air for oxidation, through a tube, 20—80 cm. long, containing inert material, such as glass, quartz, or porcelain, at 150—300°. The iron carbonyl is decomposed, ferric oxide being deposited. The yield of formaldehyde from methyl alcohol vapour containing 19 mg. of iron as iron carbonyl per litre, is increased from 55.8% to 83—85%, and the life of the catalyst is prolonged considerably, by removal of the iron carbonyl.

L. A. COLES.

Production of butyric aldehyde [butaldehyde]. C. E. PIGG, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,576,544, 16.3.26. Appl., 25.7.24).—A mixture of *n*-butyl alcohol vapour with water vapour is passed over a dehydrogenating catalyst at 200—350° and the product is condensed.

H. HOLMES.

Preparation of butyric acid. C. W. HANCOCK, Assr. to COMMERCIAL SOLVENTS CORP. (Can. P. 249,868, 30.5.24).—A colloidal solution of manganese butyrate in butaldehyde, spread over material so as to expose a large surface, is treated with a current of oxygen or of gases containing it, until the greater part of the aldehyde is oxidised to butyric acid.

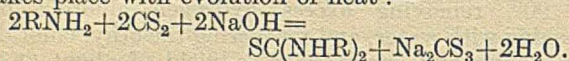
L. A. COLES.

Oxidising aromatic side-chain compounds. P. SEYDEL, Assr. to SEYDEL CHEM. Co. (U.S.P. 1,576,999, 16.3.26. Appl., 16.7.21).—850 pts. of 67% nitric acid, 800 pts. of water, 500 pts. of toluene, and 5 pts. of manganese dioxide are heated to 85° under a pressure of 35—40 lb. per sq. in., and oxygen or a gas rich in oxygen is introduced under pressure at intervals. After 6 hrs. the spent oxides of nitrogen are blown off and fresh oxygen is introduced, this process being repeated at similar intervals until 24

hrs. have elapsed, when the temperature is raised to 110° and the pressure to 75 lb. per sq. in. An 80% yield of benzoic acid is obtained. It contains some nitrobenzoic acids from which it can be separated by sublimation. In place of manganese dioxide, vanadium oxide may be used as a catalyst. The nitrotoluenes can be similarly oxidised.

T. S. WHEELER.

Manufacturing aromatically - disubstituted thioureas [thiocarbamides] of symmetric constitution. W. FLEMMING (U.S.P. 1,577,797, 23.3.26. Appl., 19.3.25).—An aromatic amine is treated with carbon disulphide and an aqueous solution of a strong base in a closed vessel when the following reaction takes place with evolution of heat:—



For example, by agitating 100 pts. of aniline, 50 pts. of sodium hydroxide in 150 pts. of water and 82 pts. of carbon disulphide in a closed vessel a yield of over 90% of thiocarbanilide is obtained in 10 min.

T. S. WHEELER.

Preparation of *n*-butyl salicylate. FARBENFABR. VORM. F. BAYER & Co. (F.P. 595,871, 27.3.25. Conv., 11.4.24).—*n*-Butyl salicylate, an oil of b.p. 128°, with a pleasant odour, and of value in the manufacture of perfumes, is obtained by the usual esterification processes.

L. A. COLES.

Perfume. FARBENFABR. VORM. F. BAYER & Co. (F.P. 598,002, 12.5.25. Conv., 14.5.24).—The perfume consists of 2:6-dimethylhepten-2-ol-6 or its esters.

W. CLARK.

Preparation of di- and poly-piperidyls. K. RÜLKE and F. CLOTOWSKI (G.P. 420,445, 20.4.24).—Pyridine and its homologues are reduced electrolytically, with lead cathodes, in 2—3 equivalents of sulphuric acid, of concentration under 30%, preferably 15—25%. For dipiperidyls with little poly-piperidyls, the current density should be 8—14 amp./dm.², and for a high total yield of di- and polypiperidyls the current density should be 20 amp./dm.² of cathode surface. For example, technical pyridine bases thus reduced with a current density of 13.6 amp./dm.² at not above 18° yield 32.9% of dipiperidyls, distilling mainly at 155—162°/17 mm. with solidification, and 11.4% of resin. Using a current density of 27 amp./dm.² at not above 30°, the yields are 25.5% of dipiperidyls and 38.7% of resin (polypiperidyls). These products are used for destroying pests.

B. FULLMAN.

Obtaining halogenated hydrocarbons. BADISCHE ANILIN- & SODA-FABR., Assecs. of G. WIETZEL and J. DIERKSEN (G.P. 420,500, 29.3.23. Addn. to 409,181).—In modification of the process described in the chief patent (B., 1925, 448), adsorbents which have taken up hydrocarbons are treated with chlorine or bromine. For example, active charcoal in a series of towers (cf. U.S.P. 1,412,954; B., 1922, 454 A) is charged respectively with benzol, sulphur, and ethylene. The sulphur-containing charcoal is treated with chlorine as described in the

chief patent (*loc. cit.*). Chlorine is led through the other adsorption-vessels giving respectively chlorinated benzene hydrocarbons, and dichloroethane and its homologues. The latter may, if required, be hydrolysed to, *e.g.*, ethylene glycol, and this converted into ethylene chlorohydrin by means of the sulphur monochloride obtained in the process. The charcoal may be used again after removal of the chlorinated products.

B. FULLMAN.

Preparation of double compounds from aliphatic amino-acids and inorganic alkali salts [phosphates]. C. F. BOEHRINGER & SOEHNE G.M.B.H. (G.P. 420,910, 24.4.24).—Double compounds of, *e.g.*, glycine, leucine, or alanine with disodium phosphate, or of glycine with dipotassium phosphate or trisodium phosphate, are prepared by combining stoichiometric proportions of the constituents in aqueous solution, with subsequent isolation in the solid form by evaporation, salting out, or precipitation with alcohol. Solutions of the compounds, after sterilisation, are suitable for injection in cases of fractured bones, being better than sodium phosphate for this purpose, as they do not cause rise in temperature, headache, or sickness.

L. A. COLES.

Preparation of pure α -sparteine methiodide. E. MERCK, CHEM. FABR., Assecs. of A. DÜTZMANN (G.P. 421,387, 23.8.24).—The methiodide prepared from the crude sparteine base is dissolved in twice its volume of alcohol, and treated warm with an equal volume of ethyl acetate. After keeping for 1 day the crystalline product is filtered off, and freed from chloroform by solution in water and boiling. On cooling pure α -sparteine methiodide crystallises out (compound with $\frac{1}{2}$ CHCl₃, m.p. 236—238°). The mother liquor yields α' -sparteine methiodide.

B. FULLMAN.

Preparation of a complex thorium compound of the reaction product of tyrosine and formaldehyde. CHEM. FABR. FLORA (Swiss P. 113,053, 13.11.24).—The product obtained from the reaction between tyrosine and 40% formaldehyde in the presence of 30% sodium hydroxide is treated in aqueous solution with acetic acid and excess of freshly precipitated thorium hydroxide. The solution, after warming, filtering, and concentrating, is treated with ether, whereby a yellow amorphous complex compound, containing 10% Th, is precipitated. It has therapeutic application.

B. FULLMAN.

Pharmaceutical product. J. CALLSEN, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 1,572,768, 9.2.26. Appl., 26.6.25).—Aminoalcohols are treated with chloroquinoline derivatives in presence of sodium to yield basic ethers of quinoline which form mono- and di-hydrochlorides and are of therapeutic value in the treatment of diseases of the heart. For example, 178 pts. of dimethylaminoethyl alcohol are dissolved in 800 pts. of xylene, 46 pts. of sodium and 355 pts. of α -chloro- γ -methylquinoline are added, the mixture is heated to 100°, filtered from sodium

chloride, and the filtrate treated with dilute hydrochloric acid. The precipitated hydrochloride is treated with alkali and the free base distilled under reduced pressure. *a*-Hydroxy- γ -methylquinoline dimethylaminoethyl ether thus obtained has b.p. 176°/10 mm., is soluble in the usual organic solvents and insoluble in water; the dihydrochloride has m.p. 163° and the monohydrochloride m.p. 190°. *a*-Hydroxy- γ -methylquinoline piperidoethyl ether from *a*-chloro- γ -methylquinoline and piperidoethyl alcohol has b.p. 233°/17 mm., dihydrochloride m.p. 187°. γ -Hydroxy-*a*-methylquinoline dimethylaminoethyl ether from γ -chloro-*a*-methylquinoline and dimethylaminoethyl alcohol has b.p. 190°/10 mm.

T. S. WHEELER.

Organic derivatives of tin. C. OECHSLIN, Assr. to ETABL. POULENC FRÈRES (U.S.P. 1,573,738, 16.2.26. Appl., 4.9.24).—225 pts. of stannous chloride dissolved in 380 pts. of water are mixed with about 700 pts. of sodium hydroxide solution, *d* 1.36, and 300 pts. of ice, and treated with 80.5 pts. of ethylene chlorohydrin. The sodium salt of hydroxyethyl stannonic acid $\text{OH}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{SnO}_2\cdot\text{Na}$ is formed. It is unstable and is preferably isolated as its thio-derivative, by adding barium chloride and saturating with hydrogen sulphide, neutralising, keeping overnight, filtering, and adding acetic acid. The hydroxyethylthiostannonic acid $\text{HO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{SnS}\cdot\text{S}\cdot\text{SnS}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{OH}$, so obtained, decomposes on heating to 80° yielding tin sulphide and oxysulphide, or on treatment with acids. Glycerol monobromohydrin gives dihydroxypropylstannonic acid which is also isolated as the thio-derivative. T. S. WHEELER.

Polymerisation of butaldehyde. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,576,503, 16.3.23. Appl., 4.6.24).—*n*-Butaldehyde treated with 0.5–2% of a mineral acid or zinc chloride or the like at 15–50° gives an 85–90% yield of parabutaldehyde, b.p. 210–220° (decomp.) at ordinary pressure, 129–130°/35 mm., *d*²¹ 0.917. Zinc chloride is particularly suitable as condensing agent as no cooling is then required. The condensation is complete in 4 days and after washing out the catalyst the product is distilled under reduced pressure. T. S. WHEELER.

Preparation of a derivative of 1-phenyl-2:3-dimethyl-5-pyrazolonyliminopyrine. KNOLL & Co., and H. BOIE (G.P. 420,446, 28.12.23).—The compound is obtained by fusing together, or by bringing together in solution, equimolecular proportions of the iminopyrine and salicylic acid. For example, the constituents are heated with alcohol until dissolved, and, after cooling, the salicylate is precipitated as crystals containing 3 mols. of water, m.p. 70–71°, by the addition of ether. The compound is only slightly soluble in water, but by the addition of carbamide, urethane, sodium salicylate, or glycerin, concentrated solutions can be prepared suitable, e.g., for intravenous injection in treating *arthritis deformans*. L. A. COLES.

Preparation of ethers of aliphatic or aromatic dialkylaminoethanol esters and their deriva-

tives. W. SCHOELLER (G.P. 420,447, 20.4.23).—Allyl esters of aliphatic or aromatic acids are treated with mercury salts of oxygen acids in alcoholic media, the resulting complex mercury compounds being converted, preferably in non-aqueous media, via their halogenated mercury derivatives into the corresponding halogen derivatives, and these, by the action of secondary aliphatic amines, into dialkylaminoethanols or their derivatives. For example, allyl acetate on treatment with mercury acetate in methyl alcoholic solution yields the corresponding mercuriacetate, which on treatment with potassium bromide yields *a*-aceto- β -methoxypropane γ -mercuribromide (the corresponding β -ethoxy-derivative being obtained in ethyl alcoholic solution). Action of iodine gives *a*-aceto- β -methoxy- γ -iodopropane, b.p. 82–83°/1.5 mm., leading to *a*-aceto- β -methoxy- γ -diethylaminopropane, b.p. 99–102°/14 mm. The corresponding dimethylamino-compound forms a methiodide, m.p. 157°. The following were also prepared:—*a*-benzoyl- β -methoxypropane γ -mercuri-iodide, the corresponding γ -iodopropane derivative, b.p. 95–98° *in vacuo*, and the diethylamino-derivative, b.p. 100° *in vacuo*; *a*-*p*-nitrobenzoyl- β -methoxypropane γ -mercuribromide, m.p. 100–102°, the corresponding syrupy iodopropane derivative, and the hydrochloride of the diethylamino-derivative, m.p. 143–144°, reduction of the latter yielding *a*-*p*-aminobenzoyl- β -methoxy- γ -diethylaminopropane hydrochloride, m.p. 158–159°. The ethers of diaminoethanol esters are effective in local anæsthesia and in increasing intestinal peristalsis.

B. FULLMAN.

Removing nicotine from tobacco. T. SCHLOESING (E.P. 234,845, 29.5.25. Conv., 30.5.24).—Tobacco is freed from nicotine by subjecting it at about 100° to the action of a current of a mixture of steam and ammonia, in which the ammonia is maintained at a constant pressure. The vapour leaving the apparatus is condensed, nicotine is extracted from the condensed liquid by treating it with a nicotine solvent, e.g., kerosene, and the residual liquor, which contains ammonia and small quantities of the aromatic constituents of the tobacco, is boiled, and the steam generated is returned to the process, further quantities of ammonia being added, if necessary. Nicotine is recovered from the kerosene solution, e.g., by extraction with sulphuric acid, and the kerosene is used again. L. A. COLES.

Tobacco denicotinisation. H. W. SMITH (U.S.P. 1,577,768, 23.3.26. Appl., 7.6.22).—Tobacco is treated first with steam at 100–150° and a pressure of up to 55 lb. per sq. in., and then with a volatile base such as ammonia or methylamine. The nicotine thus liberated is extracted with an organic solvent, from which it is recovered by treatment with aqueous acid. The solvent freed from nicotine but saturated with other substances extracted from the tobacco is used for treating succeeding batches. The denicotinised tobacco is treated with steam to remove the volatile base and traces of the solvent. The nicotine content of tobacco can in this way be reduced to 0.4–0.2%. T. S. WHEELER.

Production of acridinium compounds. L. CASSELLA & Co., Assees. of L. BENDA and O. SIEVERS (U.S.P. 1,565,891, 15.12.25, and 1,577,415, 16.3.26. Appl., 22.12.23 and 18.4.25).—See E.P. 217,715 and 218,542; B., 1924, 768, 808.

Manufacture of acetic anhydride and aldehyde. G. J. ROY, Assr. to Soc. CHIM. USINES DU RHÔNE (U.S.P. 1,579,248, 6.4.26. Appl., 10.7.25).—See E.P. 238,825; B., 1925, 900.

Heart-affecting pure glucoside from *Bulbus scillæ*. CHEM. WORKS FORMERLY SANDOZ, Assees. of E. SUTER (U.S.P. 1,579,338, 6.4.26. Appl., 26.5.24).—See E.P. 217,247; B., 1924, 997.

Producing a preparation for treating arteriosclerosis. V. MLADEJOVSKY (U.S.P. 1,579,828, 6.4.26. Appl., 12.12.24).—See E.P. 237,087; B., 1925, 784.

Manufacture of 1-phenyl-2:3-dimethyl-4-dimethylamino-5-pyrazolone. G. LOCKEMANN (U.S.P. 1,580,059, 6.4.26. Appl., 31.3.24).—See E.P. 214,261; B., 1925, 378.

Injectable, homogeneous, oily preparation of bismuth [naphthenate]. W. EICHHOLZ and O. DALMER (U.S.P. 1,580,592, 13.4.26. Appl., 26.11.24).—See E.P. 226,226; B., 1925, 737.

Oxidation of organic substances (G.P. 420,444).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitizers of gelatin. A. AND L. LUMÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1925, iii., 12, 338—341).—In 1906 the authors removed by repeated extraction with cold water the sensitising properties from a gelatin which gave very fast emulsions. The residues remaining after evaporation of the extracts were very active, and sensitised inactive gelatins, especially those which had been rendered insensitive by washing. The residue contained inorganic matter and organic substances containing sulphur and nitrogen. The nitrogen content of the initial gelatin decreased on washing. In 1910, 900 substances were examined for their effect on the sensitivity of X-ray emulsions. Among those which were selected as having practical value in increasing or decreasing sensitivity or modifying gradation were: æsculine, salts of codeine and thebaine, diethylenediamine, thiocarbamide, guanidine thiocyanate, salts of vanadic acid, and salts of copper. W. CLARK.

Silver iodide in photographic emulsions. W. D. BALDSIEFEN, V. B. SEASE, and F. F. RENWICK (Phot. J., 1926, 66, 163—184).—A formula is given for the preparation of a silver iodide emulsion having a wide range of grain-sizes and free from clumps. The emulsion was separated by sedimentation into a number of fairly uniform emulsions of different grain sizes, and the effect of its addition to silver bromide emulsions at various stages of

manufacture was studied. It is established that silver iodide emulsion can be substituted, before mixing, for the equivalent amount of potassium iodide in an iodobromide emulsion without producing any essential change in photographic properties, turbidity, or distribution of silver iodide in the bromide. This holds for both double neutral emulsification and single ammonia emulsification processes, and for 2% and 3.75% of iodide. The size of particle of the silver iodide emulsion used is without influence, the quantity only being important. Emulsions containing 2% of iodide are slower and more "contrasty" than those containing 3.75%. If silver iodide emulsion is added immediately after the first emulsification in the case of a double emulsification emulsion or after mixing in the case of a single emulsification emulsion or a single ammonia emulsification emulsion, slow emulsions are obtained, and the iodide is fairly uniformly distributed in all layers of the sedimented emulsion. It is concluded that when silver iodide is added to a freshly precipitated silver bromide emulsion the latter is able to peptise the silver iodide grains and form mixed iodobromide grains with them. A fully ripened silver bromide is unable to peptise silver iodide. The silver iodide added just after emulsification exerts a true desensitising effect rather than an anti-ripening influence. The substitution of silver iodide prepared with a slight excess of silver ions for that prepared with excess iodide ions gave the same results. As the time of addition of silver iodide is delayed in the process of manufacture of a double emulsification emulsion, there is a progressive speed decrease for constant time of ripening. The most sensitive emulsion is obtained if the iodide is added just before the first mixing; the lowest speed if the iodide addition is made just after the second mixing. The desensitising action of potassium iodide added after mixing to both single and double emulsification emulsions is no greater than that of silver iodide. The size of grain of the silver iodide emulsion used has no influence on the photographic characteristics of the resulting silver iodobromide emulsion. If coarse, dried silver iodide prepared in absence of gelatin is used, the resulting emulsions are much less sensitive than those made with potassium iodide. If freshly precipitated, wet silver iodide made in absence of gelatin is employed, results similar to those found with silver iodide emulsion or potassium iodide are obtained. The presence of gelatin in the silver iodide is not necessary for it to behave like potassium iodide, although the physical condition is of vital importance. Ripening is assumed to be accompanied by a change of distribution tending to bury the iodide within the bromide. W. CLARK.

Characteristics and anomalies of emulsions on development. I. S. E. SHEPPARD (Phot. J., 1926, 66, 190—200).—The paper is mainly a plea for a definite classification of photographic emulsions of reproducible type, capable of specification, and for the elimination as far as possible of emulsions not meeting the specifications. The variability of

behaviour of emulsions is considered, and data in this connexion are given. Emulsions are classified, according to their behaviour on development for a series of different times, as orthophotic and anorthophotic. Orthophotic emulsions are those in which the straight-line portions of the family of characteristic curves meet at a definite single convergence point either below, upon, or above the log E axis. Variations of emulsions within the limits of orthophotic types are discussed, and it is shown that all industrially employed emulsions, including those of portrait type, can be produced within this restriction. Anomalous kinds of emulsions have a lower degree of complete reproducibility, *i.e.*, of giving an identical family of characteristic curves for different times of development. These emulsions are less amenable to application of the laws of tone reproduction, to automatic processing, and to application in science and technology. Anorthophotic emulsions are not necessarily consequences of the complexity of emulsion making.

W. CLARK.

Supposed connexion between sensitisation of silver halide emulsions and the bleaching of dyes. S. E. SHEPPARD (Phot. Ind., 1926, 301).—There is no connexion between the sensitisation of the bleaching of dyes by thiocarbamides and the sensitisation of photographic emulsions by such compounds. In the latter case sensitising occurs only after decomposition of the thiocarbamide-silver bromide complex. Salts and esters of thiocarbonic acid and dithiocarbonic acid are all photographic sensitisers, but have little or no action on the bleaching of dyes. Numerous criticisms of the silver sulphide theory of sensitivity are dealt with.

W. CLARK.

Chemical [photographic] sensitisers. G. KÖGEL and A. STEIGMANN (Phot. Ind., 1926, 355).—Further evidence dealing with the hydrogenation theory of sensitisation is given.

W. CLARK.

Sensitivity of photographic papers.—MAUGE (Bull. Soc. Franç. Phot., 1925, iii., 12, 366—381).—Data are given of the relative speeds of 27 commercial papers, measured by the threshold value, using 5 different metol-quinol development formulæ. The speed of any one paper varies in an irregular manner, according to the composition of the developer, some papers showing considerable variation and others practically none. Using a developer of composition based on the mean proportions of the five formulæ, speeds of practically the mean values were obtained. Variations permissible in a formula without appreciably affecting the results are discussed. Except for special baths, sodium carbonate should be the only alkali used in a typical developer. Addition of desensitiser to prevent aerial fog is advised. The paper speeds are also given for a diaminophenol developer, and lie in general above those found with metol-quinol. Results with iconyl (*p*-hydroxyphenylglycine) agree practically with those obtained with metol-quinol. Actual and "useful" sensitivities are discussed. For a constant light source and constant development, a variation of exposure time of

$\pm 15\%$ is permissible for obtaining a correct print. Variations of sensitivity among papers of one type are of most importance in the case of "contrasty" papers. Speed and contrast characteristics of 31 French papers are tabulated.

W. CLARK.

Arrest of development in [photographic] plate testing. S. O. RAWLING (Phot. J., 1926, 66, 187—189).—Neither acid fixing baths (thiosulphate, sodium sulphite, acetic acid, potash alum) nor acetic acid-alum baths exert any noticeable solvent action on the developed (pyrogallol-soda) image on a photographic plate. Comparative tests of the two baths and a water spray for their efficiency in arresting development showed that the two acid baths are equally efficient, while the water spray permits considerable development to go on after removal of the plate from the developer. After long times of development the differences between the methods are negligible.

W. CLARK.

Photographic photometry with intermittent exposure from the standpoint of a new blackening law. H. M. KELLNER (Z. wiss. Phot., 1926, 24, 41—62).—A formula for the blackening of a plate when a rotating sector is used is evolved and tested. The Schwarzschild exponent, p , varies with i, t according to the relation $p = a + b e^{-c \log i t}$, where a, b , and c are constants. An explanation of the decrease of p to a constant minimum with increasing $i t$ is advanced. The following "general blackening law" is deduced: $S = m e^{-g \sqrt{i t}}$ where g and m are constants, and determine the contrast and maximum density respectively. Latent image formation is ascribed to liberation of electrons from bromine ions having deformed electron orbits in the silver bromide lattice, the degree of "excitation" being distributed among the bromine ions according to Maxwell's law. Ripening consists in the formation of deformed electron shells. In optical sensitisation the sensitiser is probably arranged on the grain surfaces at the points occupied by the highly-excited bromine ions, and on exposure the dye complex and not the bromine yields electrons to the silver ions. A knowledge of the variation of p with $i t$ is necessary for accurate photographic photometry, and ignorance of this invalidates much earlier work.

W. CLARK.

Small mercury vapour lamp. CLARK.—See XI.

PATENT.

Preparation of stripping films. A. VEITINGER (G.P. 422,913, 25.12.23).—Gelatin or similar colloid films are provided with a substratum of gelatin or other similar substance prepared by treating the gelatin etc. with oxidising agents, metal salts (with the exception of light-sensitive chromium salts), hardening or tanning agents, or dehydrating agents, or with ultra-violet or other chemically active rays, with subsequent exposure, washing, and drying. The gelatin under-film can be prepared without the use of glass, *e.g.*, on a paper base.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Apparent viscosity of solutions of nitro-cotton. MCBAIN, HARVEY, and SMITH.—See V.

PATENTS.

Removing colouring matter from smokeless powder. E. C. PITMAN and G. F. HUNTER, ASSRS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,577,075, 16.3.26. Appl., 20.11.22).—Colouring matter is removed from "pyro" smokeless powder by decomposing the insoluble colouring matter by a dilute acid with formation of soluble products, which are then extracted by a solvent.

S. BINNING.

Manufacture of detonating compositions. H. RATHSBURG (U.S.P. 1,580,572, 13.4.26. Appl., 3.8.22).—See E.P. 195,344; B., 1923, 804 A.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of water and action of various waters on lead and copper pipes. J. C. THRESH and J. F. BEALE (Water and Water Eng., 1925, 27, 475—478).—Data derived from operation of a 25,000-gall. experimental plant for treatment of water from the Rivers Chelmer and Blackwater are given. The purification process consisted of excess lime treatment, recarbonation, and rapid sand filtration. Sufficient lime was added to maintain an excess of 1 pt. per 100,000 after 24 hrs., the process being controlled by reaction to brilliant-cresyl-blue. With this excess the total number of bacteria was reduced from thousands to units, *B. coli* was invariably absent in 100 c.c., and organic matter was reduced 50%. To obtain rapid precipitation and decolorisation it was necessary to add, prior to the lime, 1 grain per gall. of aluminium sulphate. Lime water was more efficient and economical than milk of lime. Laboratory experiments have shown that 1 pt. of phenol in 5000 million parts of water will give rise to taste. Potassium permanganate is ineffective for destroying this taste, but an extremely small quantity of ammonia added with the chlorine appears to prevent its formation. *B. coli* was found to flourish in the presence of the weed *Enteromorpha intestinalis*, which is usually found in brackish water but which in this case was growing abundantly in the reservoir and filter beds. Similar multiplication occurred in presence of *Oscillatoria nigra*. Decaying weeds give rise also to an enormous increase of *B. aerogenes*. The presence of *B. coli* is thus not always evidence of manurial pollution. The metals zinc, iron, lead, and copper are slowly oxidised by dissolved oxygen in water. Lead oxide is fairly soluble, and as the action of lead on the human system is cumulative, its presence in drinking water even in minute amounts is dangerous. Copper oxide is much less soluble and there is no evidence of cumulative effect. The action of water on lead is influenced by the saline content of the water, but this does not appear to be a factor in the case of copper. Addition of sodium silicate to water prevents the oxidation of lead, and addition of a trace

of carbonate converts any oxide formed into oxycarbonate, which coats the metal and prevents further action. Metal used for tinning copper pipe for use in acid waters should contain 99% of tin; pipe lined with 50:50 lead-tin alloy is attacked by acid water nearly as vigorously as lead alone.

R. E. THOMPSON.

Biology of Jersey water works. W. RUSHTON and P. A. AUBIN (Water and Water Eng., 1925, 27, 487—488; cf. B., 1925, 260).—Microscopical examination of the water showed that the dominant diatom present was *Asterionella*. Contrary to reports of most observers that this organism in numbers exceeding 2000 per c.c. gives rise to a geranium or fishy odour, numbers as high as 31,184 per c.c. were recorded without perceptible taste or odour. It is suggested that absence of taste and odour may be due to the high chloride content of the water, which is normally 7.5 pts. per 100,000. During one period the number of *Asterionella* present interfered with filtration and it was necessary to apply copper sulphate. Continued employment of the spraying method of applying copper sulphate has demonstrated its efficacy over other methods. Temperature does not appear to be the determining factor in the increase of diatoms, which frequently follows an increase in inflow of soil water. R. E. THOMPSON.

[Coagulation in water purification.] A. V. DE LA PORTE (43rd Annual Rept., 1924, Provincial Bd. of Health, Ontario, 1925, 112—114).—Laboratory experiments on the use of sodium aluminate as a coagulant were fairly successful with highly acid waters, but few waters were found which were sufficiently acid to precipitate completely the amount required for efficient filtration. When solutions of alum and soda ash of certain strengths were added together, the first precipitate of aluminium hydroxide dissolved, and when the resulting solution was added to water an aluminium hydroxide floc was obtained immediately. Laboratory experiments on coagulation of a number of different types of water by this method have given excellent results.

R. E. THOMPSON.

Zeolite water softening. F. B. BEECH (Eng. and Cont., Water Works Issue, 1925, 64, 1051—1054).—Experiences with a zeolite water-softening plant for treating boiler feed water are outlined. The saving in fuel consumption and boiler maintenance effected during the first 10 months of working was sufficient to pay the cost of the softening plant. The base-exchange compound used is glauconite (greensand), a hydrous silicate of iron, aluminium, and potassium, and after 20 months' use the only important change detectable in the material was an increase in the manganese content from 0 to 0.28%. Approximately 1/3 lb. of sodium chloride is required for regeneration per 1000 grains of hardness removed. The plant will remove manganese and *Crenothrix*, rendering the water suitable for laundry purposes etc. There is little difference in the cost of softening by this method and by the lime-soda process.

R. E. THOMPSON.

Chlorine as a sterilising agent for water, especially for flood-water. R. SCHWARZBACH (Gas- u. Wasserf., 1926, 69, 272—275).—Sterilisation is especially necessary in the case of surface water supplies in times of flood when the operation of filter-beds may be deranged. The use of bleaching powder has several disadvantages which led to a chlorine installation being substituted for it. In this apparatus chlorine derived from a cylinder is dissolved in water and this water added to the main water supply in the proportion desired. Bacterial counts are given showing a superior result for chlorine as against bleaching powder, attributed to the greater facility of control of the former. C. IRWIN.

Significance of bacteriophage in surface water. L. ARNOLD (Amer. J. Pub. Health, 1925, 15, 950—952).—Bacteriophagic substances in variable amounts were found in the water of the Chicago River, the concentration being in direct proportion to the amount of pollution by domestic sewage. The bacteriological examination of water may be influenced by the content of bacteriophagic substances in the water. Impregnation of water filters with bacteriophagic substances active against certain pathogenic bacteria offers a new field of investigation in water purification. R. E. THOMPSON.

Hydrogen-ion concentration—its meaning and its application [in water purification and water bacteriology]. W. A. TAYLOR (Eng. and Cont., Water Works Issue, 1926, 65, 15—20).

Electrical conductivity and hydrogen-ion control of waste [sewage] disposal. H. C. PARKER (Water and Water Eng., 1925, 27, 445—448).—Recent improvements and developments in apparatus for determining p_H (cf. B., 1925, 555, 696) are discussed, and their practical application in the control of the direct oxidation process of sewage treatment is described. R. E. THOMPSON.

Treatment of sewage at Manchester. E. ARDERN (Surveyor, 1925, 68, 465).—Data on the operation of activated sludge units and contact beds at Manchester are given. Dewatering of sludge produced in the treatment of tank effluent by the activated sludge process yielded disappointing results, but preliminary treatment with alum of sludge derived from the treatment of raw screened and detritus-free sewage by this method was more encouraging. Sufficient alum was added to allow of using the maximum rate of filtration, as determined by laboratory tests with a Büchner funnel. Quantities of alum in excess of this amount produced a drier product but did not materially alter the rate of filtration. Adjustment of p_H by acid alone yielded unsatisfactory results, but economy was effected by use of acid with the alum when the sludge leaving the aeration tanks was in good condition. Trade wastes continued to cause difficulty, the greatest disturbance being caused by presence of oily and tarry matter, which retards the activity of the sludge and results in the production of turbid effluents.

R. E. THOMPSON.

Activated [sewage] sludge experiments at Bradford. J. A. REDDIE (Surveyor, 1925, 68, 451—453).—Tests carried out during two 30-day periods in an experimental activated sludge tank treating Bradford sewage, which has an abnormally high nitrogen content, to determine nitrogen losses, gave the following results: as free and saline ammonia, 71.3 and 100% loss; as albuminoid ammonia, 2.07 and 14.1% gain; as nitrate, 100 and 100% loss; in other forms 34.3 and 34.2% gain. The total nitrogen loss was 8.6 and 18.6%, respectively. Experiments were also carried out to determine the value of the activated sludge system as a partial process, either as final treatment after preliminary tank treatment, or as a preliminary to a high-rate filtration process. It was found that the effluent from an experimental activated sludge tank could be passed on to filters at 4 times the rate that ordinary tank effluent can be satisfactorily treated, whereas tank effluent of similar strength, produced by secondary precipitation, could not be successfully treated at even double the rate. During one period of the operation of the experimental unit, analyses of the influent and effluent indicated a reduction of 91.2% of the solids entering the tank and almost complete destruction of grease, a constituent which amounts to 40—50% of the dry solids of the Bradford sewage. This destruction is attributed to the presence in the sewage, during the period, of a very large proportion of liquefying bacteria, which varied from 30 to 80% of the total count, compared with 15—20% under normal conditions. R. E. THOMPSON.

Heat-drying of [sewage] sludge. C. E. KEEFER (Eng. News-Rec., 1926, 96, 238—240).—A review of the experience during 6½ years at Baltimore, U.S.A., of heat-drying (rotary dryer) and marketing of sludge as a means of disposal. The results obtained indicate the method to be an expensive one to use under the conditions now existing at Baltimore, and when dealing with sludge of low nitrogen content. The material treated was air-dried sludge containing 68% of water. The product contained 10—20% of water and approx. 2% of nitrogen as available ammonia. If the sludge had contained about 4% of nitrogen as available ammonia, the plant could possibly have been operated at a slight profit. Agglomeration of the sludge into balls was guarded against by using care in feeding, and the warping of the end of the dryer nearest the furnace was prevented by increasing the draught of air.

W. T. LOCKETT.

Sewage tank gases. A. M. BUSWELL and S. I. STRICKHOUSER (Ind. Eng. Chem., 1926, 18, 407—409).—Gases evolved from foaming Imhoff tanks were found to differ from those from non-foaming tanks in having a higher content of carbon dioxide and a lower content of nitrogen. The presence or absence of hydrogen in gases from septic tanks may be influenced by the proportion of cellulose in the tank, the type of anaerobic decomposition occurring and the using or giving up of any hydrogen so formed, and bacterial formation of methane and water from

hydrogen and carbon dioxide. The gas evolved from Imhoff tanks was found to have sufficient heating power (about 700 B.Th.U. per cub. ft.) to warrant its collection for fuel purposes. D. G. HEWER.

Report on sewage sludge. L. PEARSE and others (Amer. J. Pub. Health, 1926, 16, 39–42).—The report deals mainly with the utilisation of activated sludge. As a temporary measure activated sludge is being digested at Indianapolis in tanks with primary sludge produced by fine screening and rapid settling. Digestion of activated sludge with primary sludge in Imhoff tanks is being carried out in the Ruhr district, Germany, and in the Sanitary District of Chicago. Imhoff proposes to dispose of sludge in primary digestion tanks and to utilise the gases formed as power for compressing air required for the purification process. As reagents for the conditioning of sludge for filtration or pressing, aluminium sulphate is used at Chicago, sulphur by Houston, and sulphuric acid at Milwaukee. Recent experiments at Chicago indicate that ferric chloride is more efficient but more expensive. Artificial heating of sludge prior to dewatering has not been found necessary at Chicago.

R. E. THOMPSON.

Determination of the activity of commercial disinfectants. H. LÜERS and F. WEINFURTNER (Woch. Brau., 1926, 43, 25–29, 35–39, 45–49).—A modification of the Rideal-Walker test was used, with brewers' yeast as substrate. The figures given below represent the reciprocals of the percentage concentrations at which the various disinfectants had a toxic action on yeast suspensions equal to that of a 1% solution of phenol. Ammonium bifluoride 0.4, hydrofluosilicic acid 0.4, "Pyricit" (chiefly sodium borofluoride with acid sodium sulphate) 0.6, formaldehyde (36%) 0.9, formic acid 1.6, "Antiformin" and "Radoform" (solutions containing 5–7% each of sodium hydroxide and hypochlorite) 33, "Mianin" (sodium *p*-toluenesulphochloramide with 10% active Cl) 5, benzoic acid 5, "Activin" (similar to "Mianin") 8, "Chloramin" (similar to "Mianin" but purer, with 12.6% active Cl) 9, "Pantosept" (sodium salt of *N*-dichloro-*p*-sulphamidobenzoic acid, with 16.8% active Cl) 11–13, "Novocit" 12–13, sulphurous acid 25, "Magnocid" (basic magnesium hypochlorite, with 32% active Cl) 30, salicylic acid 35, bleaching powder 35, active chlorine 65, *cyclotellurodimethylpentan-3:5*-dione (impure) 80, mercuric chloride 110, "Caporit" (crystalline calcium hypochlorite with 47% active Cl) 120. J. H. LANE.

Bactericidal properties of the quinone group. HILPERT.—See A., Mar., 321.

PATENTS.

Sewage purification plant. M. PRÜSS (E.P. 247,066, 13.6.25. Addn. to 235,872; cf. B., 1926, 302).—A settling basin and a digestion chamber comprising a sewage purification plant, are hydraulically separated by a thin wall of heat-permeable material, the walls of the digestion chamber, which extend upwards, being well insulated.

The settling basin is divided by partitions into three compartments, and in order to prevent floating up of the sewage, the liquid level in the middle part is kept at a different height from that in the outer parts by a system of gutters and off-flows. The sludge is raised from the bottom of the settling chamber to the digestion chamber by an air lift operated by a supply of compressed air, and serves to keep the contents of the digestion chamber agitated by flowing along guiding walls fixed in the chamber.

B. W. CLARKE.

Method of killing micro-organisms. W. RUPPEL, Assr. to AMER. ELECTRO-OSMOSIS CORP. (U.S.P. 1,577,659, 23.3.26. Appl., 4.8.21).—See E.P. 150,318; B., 1921, 673 A.

Base-exchanging materials for softening water, from clay. PERMUTIT A.-G. (G.P. 423,224, 18.11.16).—Clay in the form of granules of 2–4 mm. diameter, is ignited at 500–550°. E. S. KREIS.

Sterilising water and other liquids. SIEMENS & HALSKE A.-G., Assees. of G. ERLWEIN (G.P. 422,902, 10.5.24).—Liquefied ozone is used. The liquid ozone may be led direct from the ozone condenser to the mixing chamber, where it is warmed by the water and converted into gaseous ozone, which dissolves more readily and is more active than ozonised air. E. S. KREIS.

Apparatus for the purification of sewage with activated sludge. K. IMHOFF (G.P. 418,319, 21.8.23).—The floors of an aerating chamber and of a settling chamber in the upper part of it, are sharply inclined and so constructed that the solid material from both chambers settles continuously into the lowest part of the aerating chamber, where there are devices for aerating the sewage and circulating the sludge. L. A. COLES.

Further decomposition of partially decomposed sewage sludge. H. BACH (G.P. 421,160, 10.2.24).—Further decomposition of sewage sludge, in which decomposition of the organic material has practically ceased, is aided by addition to the sludge of material containing sugars, such as dairy waste or liquor from beet-slicing machines. L. A. COLES.

Clarification and decolorisation of waste water from dyeworks. G. ULLMANN (Austr. P. 100,735, 10.10.24).—The water is subjected to vigorous atmospheric oxidation, dyes are precipitated by the addition of alkalis or by salting out, and, after filtration through a rapid filter, the last traces of dyes are absorbed from the water by treatment with finely-divided material such as wood or straw. L. A. COLES.

Base exchange materials. H. J. C. FORRESTER. From INTERNAT. FILTER CO. (E.P. 248,070, 26.11.24).—See U.S.P. 1,515,007; B., 1925, 24.

Methods of, and means for, exterminating vermin. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER UND CO. (E.P. 247,249, 10.10.24).—See G.P. 387,988; B., 1924, 804.