

B.—APPLIED CHEMISTRY

NOV. 21 and 28, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Scientific thermal principles of industrial furnaces. G. ZOROS (Rev. Mét., 1930, 27, 352—361).—An exposition of the principles of heat production and exchange in relation to the construction of furnaces.

A. R. POWELL.

Effect of surface conditions on heat transmission. S. J. KOHLI (Inst. Chem. Eng., Oct., 1930. Advance copy. 19 pp.).—The overall and film coefficients of heat transfer through a copper tube within which a current of hot air passed, and which was cooled with water outside, were studied with especial reference to variations in the character of the surface. The water flow was maintained constant, and the gas flow varied as required, being kept uniform in each case until all temperatures were constant. Temperatures were measured by thermocouples and porcelain stuffing-boxes were used to minimise conduction. Using a polished copper tube, results were obtained for the over-all coefficient in fair agreement with Joule and Ser's formula, $k = 16\sqrt{v}$ to $19\sqrt{v}$, where v is the gas velocity in metres/sec. The author's results are summarised by the equation k (over-all) $= 16.912v^{0.625}$ and k (air-copper) $= 16.116(w/a)^{0.625}$, where w/a is the mass-velocity. Other formulæ are discussed and criticised. Tests with a silvered copper surface showed lower coefficients at high velocities, but higher coefficients at velocities below 2.2 kg./m.²/sec. Below the critical velocity the heat transfer falls rapidly. Generally, similar results are reported for a surface covered with carbon, dulled with a sand-blast, or having a screw thread. Coatings with red, green, and violet enamels increased the heat transfer throughout. It is suggested that radiations of short wave-lengths may be absorbed and converted into heat energy. At high velocities convection plays an overwhelming part, and therefore the thickness of the air film is important. At lower velocities radiation and absorption are of more importance. The metal-water film coefficients calculated in a few cases show an increase with the logarithmic mean temperature difference, due no doubt to increased convection. These coefficients are markedly increased by black surfaces on the gas side.

C. IRWIN.

Trends in heat transfer. D. F. OTHMER (Ind. Eng. Chem., 1930, 22, 988—993).—Reference is made to a mercury boiler which has been in operation for several years at a U.S.A. power station, and to the use of diphenyl vapour in the distillation of lubricating oils. The Swenson forced-circulation evaporator has had its greatest success in the concentration of caustic soda. It operates by minimising the effect of the stagnant

surface film. Other recent forms of evaporator include the "vapour recompression system," in which the vapours from a single effect are recompressed to the pressure of the heating steam by a turbo-blower, and the "multiple-effect flashing system" in which the liquid is heated under pressure in a series of heaters, the last of which is supplied with boiler steam. The liquid is evaporated by flashing into chambers at progressively lower pressures, and heats the incoming liquid. The Ruths steam accumulator and the Merkel process, obtaining the same effect by passing low-pressure steam into caustic soda solution, are mentioned, together with several types of heat interchanges. C. IRWIN.

Regenerative chambers. II. Determination of the heat exchange and pressure losses with various grate arrangements. H. KISTNER (Arch.-Eisenhüttenw., 1929—1930, 3, 751—768; Stahl u. Eisen, 1930, 50, 1027—1028).—Mathematical expressions are derived for calculating the heat lost by radiation and the reduction in gas pressure in the heat-exchange chambers of various types of regenerators.

A. R. POWELL.

Pan grinding. J. W. MELLOR (Trans. Ceram. Soc., 1930, 29, 271—279).—Mathematical. The relationship between the best speed of revolution and the diameter of the runner of the pan (for grinding colours, flint, etc.) has been worked out theoretically.

R. J. CARLIDGE.

Relation between mass transfer (absorption) and fluid friction [in gas mixtures]. A. P. COLBURN (Ind. Eng. Chem., 1930, 22, 967—970).—The absorption of soluble components from gas mixtures and the condensation of vapours are analogous to the processes of heat or momentum transference. They depend on the rate of diffusion of gas molecules through a film on the liquid surface and on eddy currents in the turbulent portion of the gas stream. The mass-transfer coefficient $k = FM_v/u_m p_{gf} M_m \phi$, where F is the frictional resistance of fluid per unit surface area, M_v the mol. wt. of diffusing material, u_m the mean velocity in the turbulent portion, p_{gf} the logarithmic mean partial pressure of the noncondensing gas in the film, M_m the mean mol. wt. of gas mixture, and ϕ is a function of gas velocities, viscosities, and densities which approximates to unity. Data obtained on the drying of moist air are in good agreement with this theoretical result. The conditions assumed are also met with in the absorption of soluble gases in scrubbing towers where the gas velocity is high compared with that of the liquid and entrance turbulence is small. One investigation of such a case showed fairly good agreement with the equation.

C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Steam and electric power plant of Imperial Chemical Industries, Ltd., at Billingham. H. A. HUMPHREY, D. M. BUIST, and J. W. BANSALL (J. Inst. Elect. Eng., 1930, 68, 1233—1290).

Rubber mills and Banbury mixers. SCHNUCK.—See XIV. **Purification of boiler feed-water.** MOUSCADET.—See XXIII.

PATENTS.

Heat-treatment kiln. H. G. LYKKEN (U.S.P. 1,760,244, 27.5.30. Appl., 2.1.25).—The kiln has a stationary, annular hearth with provision for the application of heating, reducing, or oxidising gases upon it, and of heating gases below. A rotating cover makes a gastight joint with the hearth by means of sand seals, and carries rabbles; it may also have dependant radial division plates so that the hearth may be divided into zones for different treatments. The cover is preferably driven by a rope wrapped round its circumference.

B. M. VENABLES.

Drying of materials [e.g., timber]. F. A. SECORD (U.S.P. 1,760,444, 27.5.30. Appl., 25.1.22. Renewed 19.10.29).—The material, e.g., a stack of timber, is first subjected to a high temperature by means of saturated air, and then to localised currents of cooler air, while a blanket of heat is maintained above.

B. M. VENABLES.

Disintegrating mill. A. M. MARSH, Assr. to ALLIS-CHALMERS MANUF. CO. (U.S.P. 1,762,122, 3.6.30. Appl., 12.3.28).—A disc grinder has horizontal discs, the upper one of which is fixed and provided with a feed aperture, the lower disc is firmly coupled to the shaft of a motor, the whole being adjustable vertically to adjust the grinding size; the frame of the motor is flexibly supported in the horizontal direction.

B. M. VENABLES.

Grinder and disintegrator. E. W. SCHULTZ and F. D. BANHAM (U.S.P. 1,759,245, 20.5.30. Appl., 4.5.29).—A power-driven device for operating a pestle in a manner similar to the action of a human hand is described.

B. M. VENABLES.

Pulverising mill. G. GALAN (U.S.P. 1,762,056, 3.6.30. Appl., 27.8.27).—A pulveriser is provided with two feeding devices either of which is sufficient to deliver the full quantity required by the mill. When the feeder in operation chokes, the other one automatically comes into operation.

B. M. VENABLES.

Pulverising apparatus. W. K. LIGGETT, Assr. to JEFFREY MANUF. CO. (U.S.P. 1,761,083, 3.6.30. Appl., 11.5.25. Renewed 25.4.29).—The hammers of a disintegrator are held in the operative position by fragile means so that when an uncrushable piece is struck the hammers can swing back to an inoperative position.

B. M. VENABLES.

Apparatus for pulverising materials. F. H. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,759,100, 20.5.30. Appl., 21.8.26).—The casing is divided into two pulverising zones (preferably by the disc of the rotor) which communicate only at the circumference. The primary zone has a pocket for catching tramp iron, and a beater with wide radial blades which act also as a fan; the secondary zone has means such as pins for finer grinding.

B. M. VENABLES.

Device for (A) reducing and feeding reduced materials, (B) reducing materials. H. G. LYKKEN (U.S.P. [A] 1,760,245, 27.5.30, and [B] 1,761,138, 3.6.30. Appl., [A] 22.3.26, [B] 23.2.26).—The devices are disintegrators working substantially by cyclonic action, there being considerable clearance between the rotor and casing. In (A) the shaft is horizontal and the feed is supplied to the lower part of the casing; in (B) the shaft is vertical and the feed central.

B. M. VENABLES.

[Apparatus for] material agitation. R. J. PEPPER, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,760,374, 27.5.30. Appl., 19.2.26).—A vessel is provided with a helical blade which runs close to the wall and tends to lift the material, and any material that sticks to the blade is removed by a fixed scraper in the upper part of the vessel.

B. M. VENABLES.

Bow-tube film-type evaporator. R. C. JONES, Assr. to GRISCOM RUSSELL CO. (U.S.P. 1,760,907, 3.6.30. Appl., 26.6.23).—A bundle of steam-heated tubes is substantially horizontal and the liquid is sprayed or dribbled over the tubes to form films. Scale is removed by expansion and contraction, causing changes in curvature of the tubes which are intentionally bowed and rigidly supported at their ends.

B. M. VENABLES.

Centrifugal separator. H. G. KOEPKE, Assr. to STANDARD BRANDS, INC. (U.S.P. 1,760,071, 27.5.30. Appl., 17.9.26).—A centrifuge suitable for the separation of yeast from wort, having continuous discharge of both products, is described.

B. M. VENABLES.

Rotary or spinning [centrifugal] extractor. W. G. PETERS (U.S.P. 1,760,775, 27.5.30. Appl., 17.8.28).—A self-balancing centrifugal extractor for laundry or other work is described. The rotor is adapted to retain a layer of water on the interior of the outer wall, and an inner container for the goods is free to slide on the bottom to a certain extent and displace water from a position where the goods happen to be charged more heavily.

B. M. VENABLES.

Centrifugal scrubber. P. M. KUEHN, Assr. to BARTLETT HAYWARD CO. (U.S.P. 1,759,315, 20.5.30. Appl., 31.5.27).—An apparatus of the type in which the rotors have a combined lifting and spraying action on the washing liquid is described.

B. M. VENABLES.

Carrying out reactions with gases, vapours, or liquids, in which hydrogen, oxygen, or sulphur is present in the free or combined form. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,926, 15.4.29. Addn. to B.P. 293,077; B., 1928, 644).—The vessels used for carrying out the reactions described in the prior patent are made of iron or iron alloys which have been rendered insensitive to ageing by heating above Ac₃, quenching, and tempering below 721°.

A. R. POWELL.

Apparatus for contacting vapours with solids. W. MILLER, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,761,270, 3.6.30. Appl., 8.11.26).—The apparatus is suitable for the polymerisation of unstable hydrocarbons produced by cracking. The solid-treating material is supported on a perforated, conical, false

bottom within a gastight shell which is heated by products of combustion from the cracking still passing spirally round a jacket; the hot gases may also be passed through the solid material to regenerate it.

B. M. VENABLES.

Gas and liquid contact apparatus. M. H. KOTZEBUE (U.S.P. 1,759,750, 20.5.30. Appl., 26.5.27).—A bubbling tower is provided with tubular heat-exchangers at some of the trays to regulate the temperature of the reflux liquid.

B. M. VENABLES.

Apparatus for treating gases. M. AURIG (U.S.P. 1,760,623, 27.5.30. Appl., 29.4.29. Ger., 26.2.29).—A method of attaching sinusoidal plates in spaced relation on a wheel is described.

B. M. VENABLES.

Manufacture of liquids suitable for hydraulic machines or apparatus. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 333,200, 26.4.29).—In place of water or compressor oils there is used a glycol (or its highly concentrated aqueous solution) to which a high-molecular sulphonic acid is added to increase the lubricating power. A little butyl alcohol will suppress frothing. Examples are: ethylene glycol (63 pts.), a polyglycol ether (7 pts.), water (30 pts.), with 10–20% of Turkey-red oil or sulphoricinoleic acid; ethylene glycol (30 pts.), glycerol (30 pts.), water (20 pts.), 50% aqueous sulphonated olein (30 pts.). The f.p. of such mixtures is below -45° , and the viscosity at low temperatures is much less than that of compressor oil.

C. HOLLINS.

Balance. W. C. SEYTER, Assr. to TORSION BALANCE Co. (U.S.P. 1,759,139, 20.5.30. Appl., 21.5.29).—In a chemical balance a lens for reading the position of the rider is mounted on the rider arm in such way that it will slide longitudinally, but will always remain in focus with the beam.

B. M. VENABLES.

[Tunnel] kiln. H. M. ROBERTSON (U.S.P. 1,773,011, 12.8.30. Appl., 9.5.28).—See B.P. 331,224; B., 1930, 863.

Production of cooling [refrigerating] liquids. N. DAHL (U.S.P. 1,777,913, 7.10.30. Appl., 5.4.26. Nor., 7.2.21).—See B.P. 275,849; B., 1927, 801.

Crushing machine. E. B. SYMONS, Assr. to NORDBERG MANUF. Co. (U.S.P. 1,776,454, 23.9.30. Appl., 8.10.28).—See B.P. 327,216; B., 1930, 537.

Production of uniform pulverulent mixtures. H. BOLLMANN (U.S.P. 1,776,721, 23.9.30. Appl., 21.5.29. Ger., 5.7.28).—See B.P. 314,941; B., 1930, 170.

Separation of materials of different physical characters. H. M. CHANCE (B.P. 334,877, 7.5.29).—See U.S.P. 1,730,123 and 1,730,189; B., 1929, 964.

Manufacture of absorbent materials. J. BADDILEY and E. CHAPMAN, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,459, 7.10.30. Appl., 14.7.27. U.K., 15.7.26).—See B.P. 280,262; B., 1928, 41.

Apparatus for treating and mixing comminuted or finely-divided materials. A. B. and C. R. SMITH (U.S.P. 1,772,640, 12.8.30. Appl., 7.9.29. U.K., 2.8.29).—See B.P. 333,051; B., 1930, 932.

Apparatus for circulation of liquids. R. F. McKAY, H. WILLSHAW, W. G. GORHAM, and R. F. LEE,

Assrs. to AMER. ANODE, INC. (U.S.P. 1,777,648, 7.10.30. Appl., 27.2.28. U.K., 2.3.27).—See B.P. 293,072; B., 1928, 658.

Pasteuriser. F. LASSEN, Assr. to A./S. DE FORENEDE BRYGGERIER (U.S.P. 1,777,208, 30.9.30. Appl., 4.9.28. Denm., 19.2.25).—See Austr.P. 105,348; B., 1927, 801.

Gas-purifying apparatus. K. BÖMCKE, Assr. to DEUTS. ERDÖL A.-G. (U.S.P. 1,777,957, 7.10.30. Appl., 25.3.27. Ger., 23.12.25).—See B.P. 271,018; B., 1927, 516.

Liquefaction of gases. C. W. P. HEYLANDT, Assr. to FLÜGA A.-G. (U.S.P. 1,777,040, 30.9.30. Appl., 29.11.27. Ger., 29.12.26).—See B.P. 282,813; B., 1929, 501.

Apparatus for determining refraction and dispersion. H. VOELLMY (U.S.P. 1,776,712, 23.9.30. Appl., 12.1.28. Ger., 17.1.27).—See B.P. 283,859; B., 1928, 698.

Furnace walls. O. NYGAARD (B.P. 318,513, 19.8.29. U.S., 4.9.28).

[Fluid-cooled] furnace side walls. AMER. ENG. Co. (B.P. 335,067, 27.9.29. U.S., 8.8.29).

Refrigerator plant. J. STONE & Co., LTD., and E. G. ROWLEDGE (B.P. 335,208, 19.6.29).

Refrigerating apparatus of the absorption type. G. MAIURI and R. F. BOSSINI (B.P. 335,199, 17.4.29).

Continuous absorption refrigerating apparatus. J. O. BOVING (B.P. 335,039, 2.9.29).

Vaporising nozzles. J. FETTER (B.P. 335,222, 19.6.29).

Atomising nozzles more particularly for emulsifying purposes. A. BERGSVIK, and VITACREAM, LTD. (B.P. 335,159, 8.6.29).

Heating of materials in moving containers (B.P. 333,227). **Distillation apparatus** (B.P. 313,876).—See II. **Grinding balls** (B.P. 319,350).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Washing of coal on the Hoyois washer. A. GROUNDS (Fuel Econ., 1930, 5, 561–564).—The Hoyois washer is based on a combination of the trough washer and the upward-current classifier. Its advantages are: automatic running irrespective of variations in the shale content of the raw coal, large capacity for small ground space, high efficiency, close screening not required, and easy starting up and shutting down. Salient points in the design of the washer and the two main types of apparatus are described. Details are given of the unit installed at Gilly (Houilleres Unies du Bassin de Charleroi) for the washing of fine coal up to $\frac{1}{2}$ -in. size.

C. B. MARSON.

Properties of coal which influence carbonisation. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 518–523).—A discussion of the factors affecting the coking power of coal.

S. I. LEVY.

Analysis of [solid] fuels. E. TERRES and H. K. KRONACHER (Gas- u. Wasserfach, 1930, 73, 645–651, 673–677, 707–711).—Determinations carried out on four bituminous coals have shown that the loss of weight

amounts only to 55–60% of the weight of water actually evolved on heating in a current of air, nitrogen, or carbon dioxide. In the presence of air, moreover, some oxidation of the coal occurs. Similar but smaller differences between the loss of weight on heating and the true moisture content are exhibited also by cokes. Accurate values can be obtained only by direct determination of the water evolved, *e.g.*, by absorption in a calcium chloride tube, the fuel being heated at 100° in a current of inert gas. At higher temperatures, and even at 100° with some coals, appreciable decomposition of the sample may occur. The influence of the temperature on the yield of coke obtained in the crucible test has been determined over the range 450–1100°, and with times of heating of 15, 30, and 60 min., respectively. The coke yield-temperature curves run almost parallel for the four gas and coking coals examined. A combustion method for determining carbon, hydrogen, and nitrogen in solid fuels in one operation has been developed. The sample is burned in a current of pure electrolytic oxygen in a combustion tube provided with an especially long column of copper oxide. After absorption of the water and carbon dioxide in the usual manner the nitrogen and excess of oxygen are collected over alkaline pyrogallol, and the volume of the residual nitrogen is measured.

A. B. MANNING.

Marshall-Bird test for determining the agglutinating value of coal. K. A. JOHNSON and H. F. YANCEY (Fuel Econ., 1930, 5, 553–555).—This test consists in carbonising an intimate mixture of 10 pts. by wt. of sand (40–50-mesh) and 1 pt. of coal (to pass 100-mesh) in a porcelain crucible at a uniform temperature of 950° in a vertical, cylindrical, electrically heated furnace; after carbonisation the resultant buttons are crushed in a testing machine and the agglutinating value reported is the average breaking strength of 10 buttons. The variables involved in the test on investigation gave the following results: (1) The agglutinating values of the coals examined decreased with increase in temperature of carbonisation up to about 950°, at which point the values of most coals increased slightly; a temperature of 950° is recommended. (2) The agglutinating values decreased with increase in the period of carbonisation; a period of 20 min. is suggested. (3) As the ratio of sand to coal increased, the agglutinating value decreased; the best ratio is that of 10 to 1. (4) The addition of one drop of glycerin to the sand-coal mixture is recommended; segregation of the coal or inert material is thereby avoided. (5) Tests carried out on the same coals in different laboratories give fairly satisfactory agreement, considering the empirical character of the test.

C. B. MARSON.

Determination of coking power of coal. G. DE Vos (Natuurwetensch. Tijds., 1930, 12, 163–170).—The various methods in use in coke-oven laboratories in Belgium and Northern France are briefly described and adversely criticised. It is concluded that the content of resinous and volatile matter is of less importance than are the proportion and chemical nature of the mineral constituents, which determine the quantity and m.p. of the ash. Examples are given of contradictory and unreliable results obtained by the various methods

in use, and it is proposed that where reliable tests are necessary the material should be submitted to actual carbonisation on the large scale, following determinations of humic acid and of oxidisability, and X-ray examination.

S. I. LEVY.

Method of determination of activity of coke, and results obtained. D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 514–518).—An apparatus is described by means of which dry air freed from carbon dioxide is caused to pass at a constant rate over a definite volume of the coke of definite particle size, contained in a platinum boat in a tube heated in an electric resistance furnace, the products being passed for a definite time through suitable absorption vessels containing solid absorbents. The activity is measured by the weight of carbon dioxide formed per unit of surface per unit of time. Very great differences are observed with different cokes at moderate temperatures (500° and 600°), but these diminish as temperature rises, and at 800° the activities are the same within the limits of experimental error. The activity of graphite is much below that of coke, but is increased by intimate admixture with coke, and by presence of catalysts (iron oxide). Diamond gave anomalous results.

S. I. LEVY.

Impact hardness, abrasion hardness, and reactivity of coke. R. A. MOTT (Gas World, 1930, 93, Coking Sect., 97–101).—The shatter test, used for determining coke hardness, may be employed also to assess abrasability. If a coke is abrasable, its use in a blast furnace is decreased, and a coke with a $\frac{1}{2}$ -in. shatter index of less than 97 is undesirable for blast-furnace purposes. The specific reactivities of cokes towards oxygen and carbon dioxide at temperatures above 1500° do not differ very much. Non-caking materials, such as anthracite and low-temperature breeze, when blended with coking coals give the most reactive, and good caking coals the least reactive, cokes; most cokes which are highly reactive with oxygen are also highly reactive with carbon dioxide. It is usually found that cokes of high shatter index, high real sp. gr., high porosity, and low reactivity give the highest combustion temperatures. The size of a coke is of more importance than its specific reactivity in influencing combustion phenomena under conditions comparable with those of a blast furnace; decrease in coke size increases reactivity and decreases the average combustion temperature.

C. B. MARSON.

Evaluation of active charcoal. E. BERL and W. HERBERT (Z. angew. Chem., 1930, 43, 904–908).—Existing methods for the evaluation of active charcoal are found on examination to be untrustworthy, depending in many cases on the nature and amount of the inorganic constituents. A method based on the adsorption of methylene-blue from a 0.15% aqueous solution (60 c.c. per 0.1 g. of carbon) is described, the change in concentration of the solution being measured colorimetrically. The active surface of the sample is defined as 1 cm.² per 0.001 g. of dye adsorbed. In the case of highly active charcoals the values obtained vary with the final concentration of dye, although the variation may be decreased by increasing the initial concentration of methylene-blue; a modified method is therefore de-

scribed, using a 0.3% solution (50 c.c. per 0.1 g. of carbon). An end-point based on the complete disappearance is impracticable, and a standard final concentration of 0.1% is suggested. The variation of the observed adsorption with final concentration is determined for a number of technical varieties of active charcoal; the logarithms of these values give a straight-line graph, from which the desired results for a final concentration of 0.1% may be obtained by interpolation. A graph and a scale for the correction of results obtained by the above two methods to this final concentration are also given. H. A. PIGGOTT.

Recovery of organic nitrogen by dry distillation of agricultural waste. V. DIEGO (*Affinidad*, 1930, 10, 97—101; *Chem. Zentr.*, 1930, i, 2790).—Ammonia was obtained in 2.7% yield, together with tar, charcoal, acetic acid, and methyl alcohol. A. A. ELDRIDGE.

Hydrogenation of fuels. M. G. LEVI, C. PADOVANI, and A. MARIOTTI (*Annali Chim. Appl.*, 1930, 20, 361—404).—A description is given of the working of the Leuna plant for the hydrogenation of coal, lignite, heavy oils, and primary tars by the Bergius process. The results of a large number of experiments on Arsa coal (exceptionally rich in organic sulphur) and on a bituminous coal from Heraclea (Asia Minor), either alone or in presence of various catalysts, show that the hydrogenation of coal exhibits three phases and may take place in three ways: (1) At temperatures below 300°, at which decomposition or transformation of the complex molecules constituting the coal just begins, without generation of gases or vapours. The absorption of hydrogen is slight and a solid or semi-solid mass of high bitumen (substances extractable by solvents) content and marked agglomerating properties is obtainable. (2) At 350—400°, when destructive distillation of the coal begins, the hydrogenation is mainly in the liquid phases and, in intermittent operations, leads to the highest yields of total liquid products. (3) At 400—500° under 200—250 atm. pressure, cracking of the more resistant molecules takes place; hydrogenation occurs principally in the gas and light vapour phases, large amounts of hydrogen being absorbed. In general, addition of nickel oxide as catalyst does not appear to displace sensibly the temperature of hydrogenation, but the rapidity and intensity of the hydrogenation are greatly increased. With both coals the best results were obtained in presence of ferric or nickel oxide. Thus hydrogenation of the Arsa coal alone gave 18% of total oil (b.p. below 300°) and about 7% of benzine, whereas in presence of nickel (ferric) oxide 30 (28) and 14 (12)% were the respective yields. Sulphur is more abundant in the oils obtained by hydrogenation in presence of a catalyst, but this sulphur is mostly present in the medium and heavy oils. Hydrogenation benzine contains at most 1.6% S, whereas distillation benzine contains an average of 8% and resists all attempts to desulphurise it. Ferric oxide seems to be a better desulphuring agent than nickel oxide, but the loss on treatment with concentrated sulphuric acid and the phenol coefficient are lower in the liquid products obtained in presence of nickel oxide. T. H. POPE.

Determination of the calorific value of coke-oven

gas with the Junker calorimeter. H. GREWE (*Arch. Eisenhüttenw.*, 1930—1, 4, 75—85; *Stahl u. Eisen*, 1930, 50, 1269—1270).—Accurate results for the calorific value of a gas are obtained by the Junker calorimeter only when the incoming gas and air and the outgoing burnt gas are completely saturated with water vapour, the burnt gas is cooled to approximately the same temperature as the original gas mixture, and an accurate measurement is made of the volume of the burnt gas and residual air. Automatic recording apparatus for ensuring the observance of these precautions and for standardising the calorimeter are described in the original article. A. R. POWELL.

Volumetric relationships of the carbon monoxide process. R. NITZSCHMANN (*Metallbörse*, 1929, 19, 2413—2415, 2525—2526, 2581—2583; *Chem. Zentr.*, 1930, i, 2497).—Tabulated data relating to the production of producer gas between 477° and 1127° for reaction pressures of 0.5, 1.0, and 2.0 atm. and for oxygen-nitrogen mixtures containing 80—0% N are given.

A. A. ELDRIDGE.

Recovery of sulphur from coal gas. The Petit process. T. P. L. PETIT (*Natuurwetensch. Tijds.*, 1930, 12, 155—160).—An account of the process (cf. B.P. 195,061; B., 1923, 929 A) and of the reactions on which it is based is given. S. I. LEVY.

Velocity of ignition of gaseous mixtures. K. BUNTE and W. LITTERSCHEIDT (*Gas- u. Wasserfach*, 1930, 73, 837—842, 871—878, 890—896).—Experiments have been made with the object of investigating the influence of the several components of technical gas mixtures on the velocity of ignition, by the method of Gouy and Michelson. Binary and ternary mixtures of hydrogen, carbon monoxide, and methane were studied, and curves are drawn showing the variation of the velocity of ignition with composition for different proportions of gas and air. There is no simple relation between velocity and composition, and the curves, particularly those for the ternary mixture, have a very complicated form. Methane exercises a relatively large influence in lowering the velocity. The effect of carbon dioxide in lowering the velocity is more pronounced than that of nitrogen, presumably owing to the larger sp. heat of the former gas. A graphic method for determining the maximum velocity of ignition for any given mixture (with respect to the proportion of air required) is worked out on the basis of the measurements, and the values so obtained agree well with observations made on technical gas mixtures. The results obtained by the static method are, in general, higher than those given by the dynamical method used in this investigation, probably on account of the production of turbulence in the former. On the other hand, there is excellent agreement between the values given by the dynamical method and those obtained from the radial propagation of flame in soap bubbles.

F. L. USHER.

New methods in the manufacture of gas by suitable combination of standard arrangements. SCHUMACHER (*Gas- u. Wasserfach*, 1930, 73, 861—867).—A lecture, in which are discussed the methods by which the various operations in a gasworks may be most

economically adapted to meet a varying demand while maintaining a substantially uniform load on the furnaces. Examples of suitable lay-out are given.

F. L. USHER.

Defects in dry gas meters. WEHRMANN (Gas- u. Wasserfach, 1930, 73, 901–902).—The discs of dry meters are frequently found with incrustations which may prevent them from revolving. Examination of the incrustation (about 1 g.) collected from 50 instruments showed it to contain (1) an insoluble dust consisting of carbon, silica, and smaller proportions of metals; and (2) a soluble, dark brown, fluorescent, and viscous oil, evidently formed by the polymerisation of hydrocarbons present in the gas and absorbed by the grease used to lubricate the discs. The high degree of purification of the gas necessary to prevent this effect is impracticable, and alternative mechanical remedies are suggested.

F. L. USHER.

Determination of phenols (acid oils) in gas liquors and carbonisation liquors. E. KRES (Brennstoff-Chem., 1930, 11, 369–371. Cf. Heiduschka and Römisch, Gesundheitsing., 1930, 53, 53).—Fifty c.c. of a 10% solution of copper sulphate are added to 100 c.c. of the filtered liquor, the mixture, after acidification with dilute sulphuric acid, is distilled until about 20 c.c. remain, and the residue is then diluted with 50 c.c. of water and again distilled until only 15 c.c. remain. The phenols in the distillate are determined by means of a standard bromide-bromate solution, or colorimetrically. By using the procedure described the impurities which would otherwise interfere with the determination are either destroyed or rendered non-volatile.

A. B. MANNING.

Preparation of sulphur-containing balsamic mass from acetic acid tar. E. PALLAS (Pharm. Zentr., 1930, 71, 609–611).—The tar remaining after distillation in superheated steam of crude pyrolygneous acid is washed several times with warm or boiling water. The brown milky extract is rendered alkaline with calcium hydroxide, saturated with sulphur dioxide, and the clear liquid evaporated to a syrup. This is soluble to the extent of 95% in water, is a strong, slightly acid reducing agent, contains phenol, pyrogallol, and derivatives of pyridine and pyrrole and considerable amounts of sulphur derivatives, but no calcium salts, and resembles Peru balsam in its antiseptic properties. If the extract is not neutralised a darker product results, whilst if sodium hydroxide is used the product contains sodium sulphite.

H. E. F. NOTTON.

Some sources of error in the determination of the b.p. or of the boiling limits of commercial benzols by the method of Krämer and Spilker. W. MELZER (Brennstoff-Chem., 1930, 11, 371–372).—In using the distillation apparatus designed by Krämer and Spilker errors of 1% or more may arise through variations in the thickness of the wall or in the diameter of the condenser tube, or by judging the rate of distillation from the rate of formation of the drops (2 drops/sec.) instead of adjusting it accurately to 5 c.c./min.

A. B. MANNING.

Must the air content of a pycnometer be taken into account in determining its tare in the de-

termination of the sp. gr. of oils? SCHLÜTER (Chem.-Ztg., 1930, 54, 698).—The error introduced into a determination of the d of an oil by omitting to allow for the weight of air contained in the pycnometer is independent of the volume of the vessel and depends only on the d of the oil, varying from 0.00012 to 0.00036 when $d = 0.9–0.7$; i.e., it is within the limits of experimental error of the method. A. R. POWELL.

Heat transfer. OTHMER.—See I. Fruit-tree sprays. WEICHERZ.—See XVI.

See also A., Oct., 1255, **Speed of flame in a closed tube** (NAGAI). 1268, **Electro-decomposition of hydrocarbons** (AUSTIN). **Decomposition of paraffin hydrocarbons** (EGLOFF and others). 1277, **Determination of metal in [anti-knock] compounds** (DOSIOS and PIERRI).

PATENTS.

Vertical retorts for the carbonisation of coal and the like. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (B.P. 333,381, 1.8.29).—Coke-extracting and -discharge means consisting of a pivotally mounted coke chamber closed at its lower end are described. A curved plate attached to the top of the chamber serves to close the base of the retort when the chamber is tilted.

A. B. MANNING.

Retort and dryer for use in low-temperature carbonising. W. A. COLLIS (B.P. 333,020, 4.7.29).—The apparatus consists of a vertical tapering chamber heated on all sides by hot gases, and surmounted by an inclined chamber within which is a series of steps or terraces increasing in capacity as they descend. Each step is divided laterally into compartments provided with revolving scrapers which propel the charge from step to step and finally discharge it into the vertical chamber below. The inclined upper chamber has heating flues at the bottom and sides only. One or more gas and vapour outlets are provided at suitable positions.

A. B. MANNING.

Heating of materials in moving containers particularly applicable for destructive distillation. WOODALL-DUCKHAM (1920), LTD., and (SIR) A. McD. DUCKHAM (B.P. 333,227, 4.5.29).—The material is charged into containers which are then placed in turn on the platform of an annular kiln in such a manner that an outlet from the lower end of each container makes sealed connexion through an opening in the platform with a collecting main beneath the latter. The containers travel with the platform first through a zone of gradually rising temperature, then through a zone of high temperature, and finally through a zone of gradually falling temperature. They are then removed from the platform and their contents discharged. An annular liquid seal permits the rotation of the platform while maintaining a gastight connexion between the outlets from the containers and the collecting main. The apparatus may be modified to permit a current of gas to pass through the containers while they are travelling through the kiln.

A. B. MANNING.

Hydrogenation of the gases [and vapours] derived from the low-temperature distillation of solid fuels. COMP. INTERNAT. POUR LA FABR. DES

ESSENCES ET PÉTROLES (B.P. 311,280, 7.5.29. Fr., 8.5.28).—Either by predrying the solid fuel or by suitable treatment of the gases the water content of the latter as they enter the catalytic reaction chamber is adjusted to correspond with only the water of constitution of the initial material. Thus the gases leaving the retort may be treated by condensation followed by separation of part of the water and revaporisation of the tar and remaining water.

A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of C. S. CHRISMAN (B.P. 333,072, 6.9.29. U.S., 18.10.28).—The fuel bed of the generator is supported by a rotatable bosh of inverted conical shape, having a central opening through which projects a perforated body or tuyère. Arrangements for removing the ash and clinker and for crushing the latter are claimed.

A. B. MANNING.

Water-gas generators for bituminous fuels and by-product coke recovery therefrom. H. HIDAKA (B.P. 333,021, 4.7.29).—A revolving coke-extraction tube projects centrally up into the generator, and is adapted to remove a part of the coke carbonised at a comparatively low temperature. The remainder of the coke is gasified, yielding the amount of water-gas necessary to maintain a definite desired calorific value of the resulting mixed gas. The extraction tube is adjustable in a vertical direction. Within the tube is an extracting screw of variable speed of revolution and possessing a hollow shaft through which steam can be passed. A variable proportion of the blow gas is passed through the superheater to the waste-heat boiler, while the remainder is passed directly to the boiler, so that any fall in temperature in the generator can be compensated by increasing the degree of superheating of the steam supplied.

A. B. MANNING.

Purification of distillation gases. CHEMINOVA GES. ZUR VERWERTUNG CHEM. VERFAHREN M.B.H. (B.P. 311,404, 6.5.29. Ger., 11.5.28).—The gas is charged at the ordinary pressure with a naphthalene solvent, the concentration of which, however, is below that necessary to bring about saturation of the gas at the temperature of the cooler places of the pipe system. The gas is then compressed so that at these places solvent charged with naphthalene separates out, and the gas finally has its pressure reduced to that at which it is used. This method of purification is suitable for systems using long-distance pressure transmission. No separation of the solvent can then occur at places where it may have an injurious effect, *e.g.*, in dry gas meters.

A. B. MANNING.

Purifying from naphthalene the distillation gases of coal. GEWERKSCHAFT M. STINNES (B.P. 305,545 and 333,297, 6.2.29. Ger., 6.2.28).—(A) The gas is allowed to take up the vapour of a naphthalene solvent at about 100° and is then cooled, whereupon the solvent vapours are precipitated, carrying down with them the whole of the naphthalene in the gas. The solvent may be recirculated after being freed from at least a part of the naphthalene absorbed. (B) When the gas is to be delivered under pressure, *e.g.*, in long-distance transmission, the process is carried out by first compressing the gas, then bubbling the hot compressed gas through

the solvent, and finally cooling the gas charged with solvent in order to precipitate the latter with the naphthalene. [Stat. ref. to (B).] A. B. MANNING.

Extraction of ethylene from ethylene-containing gaseous mixtures. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 308,687, 23.3.29. Fr., 26.3.28).—Ethylene-containing gas such as coke-oven gas is partly liquefied and the liquid so produced is used to scrub the unliquefied portion free from such impurities as acetylene and oxides of nitrogen. The liquid is then distilled, the uncooled gases before the liquefaction being used as a source of heat.

T. A. SMITH.

Removal of organic sulphur compounds from gases. O. W. LUSBY (B.P. 305,027, 25.1.29. U.S., 28.1.28).—The gases are treated with a catalyst containing one or more metals of group VI, *e.g.*, uranium, chromium, and/or molybdenum, with one or more metals of groups I—V, *e.g.*, cerium, copper, lead, etc., the process being carried out at a temperature below the m.p. of any component of the catalyst, and preferably between 200° and 500°. The organic sulphur compounds are thereby converted into hydrogen sulphide, which is then removed from the gases in known manner. [Stat. ref.]

A. B. MANNING.

Method and apparatus for distilling [hydrocarbon oil]. E. B. BADGER & SONS Co., Assees. of W. T. HALL (B.P. 313,876, 7.6.29. U.S., 18.6.28).—Oil is fed on the plates of fractionating columns after passing through preheaters, in which it is heated by means of the hot residual oil from the columns. Since insufficient heat for the vaporisation of the desired fractions is introduced in this way, residue from the columns is passed through a heating tube and then returned to the plates of the fractionating column. The burner of the heating element is thermostatically controlled by the temperature of the oil entering the fractionating column. A cracking unit may be arranged to consist of a fractionating column at the top of which cracking stock is fed so that it comes in contact with the vapours entering from a reaction chamber at a point lower in the column. Oil from the bottom of the column is passed through a tube heater and delivered to the reaction chamber under thermostatic control. Part of the heated oil is returned to the lower portion of the fractionating column to control the evaporation in the column.

T. A. SMITH.

Distillation of hydrocarbon oils. R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,758,590, 13.5.30. Appl., 4.4.25).—Oil is vaporised by superheated steam into a series of evacuated, superposed, frusto-conical conduits fitted with baffle plates which prevent unatomised oil being carried forward. Any spray caught by the baffles and walls of the conduits is reatomised by the steam jet. The oil is preferably preheated and the steam is used at 530—650°. The distillation is carried out at 25—100 mm. T. A. SMITH.

Conversion of hydrocarbon oils. G. F. BENHOFF (U.S.P. 1,748,201, 25.2.30. Appl., 1.4.24).—Crude oil is heated to distillation temperature and the vapours are passed to a dephlegmator. The residual oil is passed

through a cracking tube to an evaporator, the vapours produced being scrubbed by the condensate from the first towers in a second set. Part of the reflux from the dephlegmators is returned to the cracking coil for further cracking. The oil and carbon residues from the cracking evaporator are used as fuel. T. A. SMITH.

Cracking of hydrocarbon oils. E. C. HERTHEL and T. DE C. TIEFT, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,747,437, 18.2.30. Appl., 12.8.27).—Oil, heated to cracking temperature under pressure by being passed once through a heating coil, is transferred to a dephlegmator, where it is maintained under pressure. Vapours containing gasoline are removed and the reflux is added to the raw cracking stock. The unvaporised oil from the first operation is passed to a second dephlegmator under a lower pressure, where part is volatilised, some of which is obtained as reflux. This reflux is heated to cracking temperature in a second heating coil and returned to the first dephlegmator. T. A. SMITH.

Cracking of hydrocarbon oils. J. E. BELL and E. W. ISOM, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,758,818, 13.5.30. Appl., 22.1.25).—A horizontal cracking drum is divided by partitions into a number of sections, arrangements being made for oil to pass from section to section as required. Oil from each section is pumped through a heating coil designed to suit the heat requirements of that part of the circuit. The coils of each section are placed in flues through which the combustion gases pass in succession, such gases passing first through the flue containing the coil through which the least cracked oil is being pumped. The heating surface of the coils is increased and the speed of circulation decreased from section to section. The furnace gases are "tempered" by returning a portion of them from the last flue to the first by an injector device. Raw stock is preheated by bringing it in direct contact with the vapours from the drum in a reflux tower, the reflux and raw oil being delivered into the first section of the cracking drum. A portion of the oil containing tar is continuously removed from the last section of the drum to prevent its accumulation in the system. T. A. SMITH.

Cracking of hydrocarbons. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 334,241, 30.5.29).—Hydrocarbons are cracked by passing them through baths of molten salts which have high b.p., so that little or no salt is vaporised during the operation. An equimolecular mixture of barium and calcium chlorides, m.p. about 500°, is suitable for the cracking of butane at 750°. The products consisted of 18.5% of propylene and 18.7% of ethylene. A number of other suitable salt mixtures the m.p. of which range from 426° to 663° are given. Molten tellurium may also be used and has a beneficial catalytic effect. In working with tellurium it is necessary to use aluminium-lined steel vessels or other material not attacked by tellurium. T. A. SMITH.

[Cracking] treatment of [hydrocarbon] oil. C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,018, 29.4.30. Appl., 10.2.22. Renewed 2.11.25).—A mixture of oil and aluminium chloride is fed into a dephlegmator in which vapours from the cracking opera-

tion are being treated. The reflux from the dephlegmator column is passed through a heating coil and delivered into a drum from which vapours are taken to the dephlegmator. A certain amount of residue is continuously withdrawn from the drum and the aluminium chloride is recovered. The aluminium chloride does not escape from the system with the cracked vapours, and it is not necessary to add it continuously. T. A. SMITH.

Cracking of petroleum and mineral oils and their different fractions. P. NACHTSHEIM (U.S.P. 1,758,618, 13.5.30. Appl., 8.5.26).—Oil is heated in cracking coils and delivered into towers fitted with inclined baffles in which the evolved vapours are repeatedly caused to meet countercurrentwise the unvaporised and reheated fractions. The vapours are then taken through a dephlegmator to a condenser. Separators for carbon are placed in the oil stream and are periodically cleaned. T. A. SMITH.

Cracking of heavy hydrocarbons to produce lighter hydrocarbons and coke. H. L. PELZER and E. C. HERTHEL, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,757,843, 6.5.30. Appl., 16.9.22).—Oil is cracked under pressure by circulating through it superheated gases until the oil has been reduced to a coke residue. Carbon formed in the gas-heating coils is removed by "burning out." External heating of the pressure coke still is avoided. T. A. SMITH.

Production of lower-boiling hydrocarbons. C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,019, 29.4.30. Appl., 14.1.25. Renewed 22.1.29).—Oil is circulated under pressure through a heating coil and a horizontal vaporising chamber, the volume of oil and free space in this chamber being adjusted by the height of the intake to the circulating pump. A portion of the heavy carbon-containing residue is continuously withdrawn from the bottom of the vaporising chamber. The diameter of the heating tubes is 2–4 in. and of the vaporising chamber 20–48 in. T. A. SMITH.

Manufacture of acetylene and other [unsaturated hydrocarbon] products. R. G. WULFF (B.P. 334,178, 25.2.29).—Hydrocarbons such as casing-head gasoline, benzene, and gas oil are heated under a partial pressure of 0.5–75 mm. Hg to above 720° in a carbundum tube. The low partial pressure is attained by mixing the vapour with a diluent such as steam, mercury vapour, nitrogen, etc. The duration of heating is 0.002–5 sec., after which the gases are immediately cooled. Acetylene, ethylene, etc. are then separated and the residual gas is used for heating the reaction tube. Suitable apparatus is described. T. A. SMITH.

Manufacture of viscous oils and hydrocarbon products of low b.p. I. G. FARBERIND, A.-G. (B.P. 313,879, 11.6.29. Ger., 18.6.28).—Hydrogenation of oils etc. is carried out in two stages, the first at the lower temperature so that asphaltic materials are not converted into coke. The lighter constituents are removed after the first treatment and the residue is then cracked or hydrogenated. Catalysts may be employed in both stages. The first stage should be effected in the liquid phase at below 420°. Solid paraffins may be separated

from the distillate after the first hydrogenation. The liquid fractions contain good lubricating oils.

T. A. SMITH.

Refining of mineral oils and like carbonaceous materials. H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 332,944, 27.4. and 21.11.29).—The oils are heated with hydrogen under pressure in the presence of a colloidal molybdenum compound, *e.g.*, the oxide, which is adsorbed on a finely-divided solid such as active carbon. The conditions are so chosen that no considerable cracking of the oil takes place.

A. B. MANNING.

Refining of [hydrocarbon] oils. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,026, 29.4.30. Appl., 6.6.23. Renewed 20.2.28).—In the treatment of oils with refining liquids, streams of oil and liquid are passed at high velocity through tubes containing spiral baffles, whereby intimate contact is effected. The mixture is then allowed to settle and is separated.

T. A. SMITH.

Refining of hydrocarbon oils. E. C. HERTHEL, and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,756,154, 29.4.30. Appl., 16.6.28).—Crude oil is vaporised and the vapours are passed into a dephlegmator. The heaviest portions are removed and the vapours then passed through a supply of hot oil maintained alkaline by addition of caustic soda. The vapours are then passed through a series of fractionating columns, whereby lubricating oil fractions of good colour and stability are obtained.

T. A. SMITH.

Processing of heavy oils. A. J. SLAGTER, Assr. to TRANSCONTINENTAL OIL Co. (U.S.P. 1,757,596, 6.5.30. Appl., 28.3.24).—A pressure still is fitted with a dephlegmator into which the oil to be treated is sprayed and brought in direct contact with the vapours. The oil thus preheated enters the still through the vapour line, and is circulated in the still by means of a pump submerged in the oil. The circulating pump may be connected to an external heating coil so that oil may be circulated from the still through the heating coil and be delivered again into the still.

T. A. SMITH.

Decolorisation of hydrocarbon oils. N.V. MEJNBOW-EN CULTUURMAATS. "BOETON" (B.P. 334,833, 5.4.29. Holl., 30.1.29).—The oil is treated with a solution containing at least 58% of a hygroscopic heavy-metal chloride (*e.g.*, ferric chloride solution, *d* 1.6). Less loss is obtained than with sulphuric acid. The process is suitable for cracked gasoline, kerosene, or lubricating oil. Some oils may require more than one treatment. The chloride solution may be used again after the removal of the polymerised substances.

T. A. SMITH.

Production of hydrocarbons of high b.p. range [suitable for lubricants] from coal, shale, and other bituminous substances. I. G. FARBENIND. A.-G. (B.P. 312,050, 8.4.29. Ger., 18.5.28).—The initial materials are treated with organic solvents at raised temperatures and under pressure, and the products thereby extracted are subjected to treatment with reducing gases, under such conditions of temperature, pressure, and time, that no substantial decomposition

to products of lower b.p. occurs. The extracted oil is preferably freed from paraffin wax prior to the reduction treatment, which, if desired, may be carried out in the presence of a suitable catalyst, *e.g.*, a mixture of molybdc acid and zinc oxide.

A. B. MANNING.

Manufacture of activated carbon. R. THRELFALL (U.S.P. 1,777,943, 7.10.30. Appl., 31.5.27. U.K., 4.6.26).—See B.P. 270,505; B., 1927, 515.

Reactivation of decolorising carbon. F. W. MEYER (U.S.P. 1,771,719, 29.7.30. Appl., 14.9.29. Ger., 8.9.28).—See B.P. 317,017; B., 1930, 936.

Manufacture of water-gas. F. WINKLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,876, 30.9.30. Appl., 7.5.28. Ger., 27.9.22).—See G.P. 437,970; B., 1927, 548.

Purification of liquid hydrocarbons. F. B. THOLE and S. T. CARD, Assrs. to ANGLO-PERSIAN OIL Co., LTD. (U.S.P. 1,776,340, 23.9.30. Appl., 6.11.24. U.K., 11.1.24).—See B.P. 231,944; B., 1925, 436.

Deodorisation of petroleum hydrocarbon distillates. Manufacture of lubricating oils. L. EDELEANU, Assr. to EDELEANU GES.M.B.H. (U.S.P. 1,776,752—3, 23.9.30. Appl., [A] 16.1.28, [B] 28.5.28. Ger., [A] 25.6.27, U.K., [B] 14.4.28).—See B.P. 301,955 and 307,649; B., 1929, 161, 386.

Oxidising paraffins, waxes, and the like. F. HOFMANN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,762,688, 10.6.30. Appl., 14.3.28. Ger., 18.3.27).—See B.P. 298,704; B., 1928, 919.

Fuel burners. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 335,806, 20.11.29).

Burners for pulverised fuel. G. E. K. BLYTHE (B.P. 335,275, 25.6.29).

Contacting vapours with solids (U.S.P. 1,761,270). **Liquids for hydraulic machines etc.** (B.P. 333,200).—See I. Pure naphthalene (B.P. 333,352). **Pure anthracene** (B.P. 319,762).—See III. **Hydrogen** (B.P. 311,737). **Carbon disulphide** (B.P. 314,060 and 334,856).—See VII.

III.—ORGANIC INTERMEDIATES.

Use of calcium chloride in the dehydration of alcohol. K. B. EDWARDS and R. LACEY (J.S.C.I., 1930, 49, 422 T).—The use of fused calcium chloride in recovering concentrated alcohol from residues is recommended. When 20% (wt./vol.) was added to 80% alcohol and rapidly fractionated, a 95% alcohol distillate was obtained. A repeat distillation after a similar addition gave 52% of 98% and 24% of 95.5%. The process is simple and economical, and miscibility tests are given for control.

Measuring and recording p_H . WULFF and KORDATZKI.—See XI.

See also A., Oct., 1270, **Determination of purity of acetic anhydride** (ROSENBAUM and WALTON). 1279, **Methylation of α -naphthylamine** (GOKHLE and MASON). 1280, **Formation of diazo-oxy-compounds** (BUCHERER and TAMA). 1282, **Novel preparation of 3:5-dinitro- p -cresol** (HODGSON and SMITH). **Sulphonation of β -naphthol in presence**

of boric acid (ENGEL). 1292, New synthesis in the 1:2-naphthanthraquinone series. Derivatives of naphthanthraquinone (WALDEMAN), 1303, Rapid determination of furfuraldehyde (NOLL and others).

PATENTS.

Production of acetaldehyde from acetylene. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 334,427, 21.10.29).—In the conversion of acetylene into acetaldehyde by action of steam a catalyst is used comprising zinc oxide activated with a molybdate or molybdic acid. C. HOLLINS.

Manufacture of acetic anhydride. W. W. GROVES. From E. B. BADGER & SONS CO. (B.P. 333,991, 17.6.29).—During addition of sulphur chloride and chlorine (or sulphuryl chloride and chlorine) to sodium acetate pasted with acetic anhydride, the reaction mixture is cooled by means of coils carrying ammonia and brine circulating through a compressor. The temperature is adjusted by means of the expansion valve, and the time of charging is materially shortened. A suitable plant is figured. C. HOLLINS.

Manufacture of acetic acid [from formaldehyde and carbon monoxide]. BRIT. CELANESE, LTD., S. J. GREEN, and R. HANDLEY (B.P. 334,207, 27.5.29).—A mixture of carbon monoxide and formaldehyde vapour is heated at 300–400° and under 100–300 atm. pressure, preferably in presence of phosphoric acid, with or without addition of copper or copper compounds. Aqueous formaldehyde or methylal may be used. C. HOLLINS.

Manufacture of aliphatic compounds [acids from alcohols etc. and carbon monoxide]. BRIT. CELANESE, LTD., S. J. GREEN, and R. HANDLEY (B.P. 334,189, 27.5.29).—The paraffin hydrocarbon by-products produced in the catalytic manufacture of acids from alcohols etc. and carbon monoxide are removed, *e.g.*, by adsorption on charcoal or silica gel, before recirculation of the reaction gases. C. HOLLINS.

Production of compounds from propylene. J. W. WOOLCOCK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 334,228, 30.4.29).—Propylene is absorbed in a mixture of sulphuric acid and acetic acid, or of isopropyl hydrogen sulphate and acetic acid, below 60°; on dilution with water a mixture of isopropyl alcohol and acetate separates, and the aqueous layer is boiled to yield more isopropyl alcohol, or the whole diluted liquor is boiled without separation in order to increase the proportion of alcohol to ester. A mixture of equal volumes of 99.5% sulphuric acid and glacial acetic acid absorbs propylene alone from admixtures with ethylene; higher olefines may be removed by pretreatment with cooled 80% sulphuric acid. C. HOLLINS.

[Production of esters by] catalytic dehydrogenation. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,345, 3.4.29. U.S., 26.5.28).—An aliphatic alcohol is passed in vapour form over a catalyst comprising copper and a difficultly reducible oxide or mixture of oxides, *e.g.*, oxides of magnesium and manganese, or of magnesium and zinc, at 375° or above, and at a pressure of at least 4000 lb./in.² The product from ethyl alcohol contains 20% of ethyl acetate. C. HOLLINS.

Production of esters [of sulphonated higher fatty acids etc.]. H. T. BÖHME A.-G. (B.P. 315,832, 24.6.29. Ger., 19.7.28).—Higher fatty or other suitable acids are esterified with an alcohol of relatively low mol. wt. (other than glycerol), and the esters are sulphonated, preferably below 15°, to give wetting agents. Butyl ricinoleate, *e.g.*, is treated with 1.4 pts. of concentrated sulphuric acid below 15°. C. HOLLINS.

Preparation of alkyl cyanides. IMPERIAL CHEM. INDUSTRIES, LTD., and T. S. WHEELER (B.P. 333,989, 14.6.29).—An alkyl halide and a metal cyanide, *e.g.*, ethylene dichloride and sodium cyanide, are heated in presence of the corresponding nitrile (*e.g.*, succinic nitrile) as diluent. C. HOLLINS.

Manufacture of carbohydrate compounds [soluble hydroxyalkyl ethers]. A. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,282, 4.6.29).—The carbohydrate (potato meal, dextrin, cotton) is treated with an alkylene oxide at 15° in presence of aqueous caustic alkali of 20% or lower concentration without application of pressure. C. HOLLINS.

Preparation of pyridylalkines and piperidylalkines. A. BOEHRINGER (B.P. 312,919, 29.5.29. Ger., 1.6.28).—Pyridines having side-chains, $R \cdot CO \cdot CH_2 \cdot$, in 2- or 2:6-positions are hydrogenated in stages or otherwise in presence of nickel, platinum, etc. 2-Phenacylpyridine yields 2- β -hydroxy- β -phenylethylpyridine, m.p. 107°, and on further hydrogenation the corresponding hexahydrocarbinol, m.p. 87°, b.p. 160°/1 mm. C. HOLLINS.

Separation of mixtures of secondary and tertiary amines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,349, 28.6.29).—The mixture is treated with the calculated amount of phthalic anhydride in absence of a neutral solvent. The phthalamide acid is extracted with alkali, leaving pure tertiary amine; the secondary amine may be recovered from the phthalamide acid. The application to mixtures of mono- and diethylanilines, methylanilines, benzylanilines, of diphenylamine and diphenylethylamine, and of 4-methoxy-3- β -hydroxyethoxytoluene and dimethylaniline, is described. C. HOLLINS.

Production [purification] of 2:4:6-tribromoaniline and its acyl derivatives. BRIT. CELANESE, LTD., and B. E. M. MILLER (B.P. 334,217, 29.5.29).—Crude 2:4:6-tribromoaniline is sublimed at 160–165° and then acylated, especially diacetylated, to give colourless products suitable for incorporation with cellulose acetate etc. for non-inflammable films. C. HOLLINS.

Manufacture of benzyl chloride. IMPERIAL CHEM. INDUSTRIES, LTD., N. BENNETT, H. DODD, W. C. SPRENT, and F. HOLT (B.P. 334,260—1, 1.6.29).—(A) The crude benzyl alcohol recovered from the benzylation of cellulose is freed from iron etc. by addition of alkali (anhydrous sodium carbonate), filtered, and esterified with hydrochloric acid. (B) After esterification the product is treated with chlorine, whereby unesterifiable benzyl compounds are converted into benzyl chloride and benzaldehyde; the latter is removed by means of bisulphite, and benzyl chloride is distilled. C. HOLLINS.

Purification of phenylethyl alcohol. E. C. BRITTON, Assr. to DOW CHEM. CO. (U.S.P. 1,752,365, 1.4.30. Appl. 9.6.23).—The phenylethyl alcohol in a crude product is converted selectively into its hydrogen phthalate by heating with phthalic anhydride in benzene. The hydrogen phthalate is removed as sodium or other soluble salt and hydrolysed. C. HOLLINS.

Manufacture of aromatic [hydr]oxyaldehydes. J. D. RIEDEL—E. DE HAËN A.-G. (B.P. 333,313, 18.5.29. Addn. to B.P. 285,451 and 290,649; B., 1928, 873; 1929, 672).—The mixture of 3-hydroxy-4-methoxy-methoxy-1-propenylbenzene and methoxyvanillin obtained from safrole by the process of the prior patent is separated by fractional acidification. The former compound is converted into vanillin by methylation, hydrolysis with dilute acid, and oxidation; the latter is acylated, hydrolysed, methylated, and de-acylated to give vanillin, or is hydrolysed to protocatechualdehyde with alcoholic acid. C. HOLLINS.

Manufacture of quinaldine. L. P. KYRIDES, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,752,492, 1.4.30. Appl. 16.7.23).—Aniline is added to a mixture of hydrochloric acid in nitrobenzene and at 90–110° aldol or crotonaldehyde is slowly run in. Improved yields are obtained. C. HOLLINS.

Manufacture of monobrominated menthane. SCHERING-KAHLBAUM A.-G. (B.P. 334,474, 24.12.29. Ger., 31.12.28).—Bromomenthane, b.p. 115–120°/15 mm., is obtained by bromination of menthane in presence of iodine at 0°. C. HOLLINS.

Purification of naphthalene. GES. F. TEERVERWERTUNG M.B.H., and A. and G. SPILKER (B.P. 333,352, 1.7.29).—Centrifuged, crude (75–80%) naphthalene is melted and treated with about 0.15 pt. of aqueous sodium hydroxide (*d* 1.1). The separated naphthalene is washed with water and fractionally distilled, whereby 60–65% of the material is obtained pure (m.p. above 79°) and 10% of m.p. 78–79°. C. HOLLINS.

Production of hydrocarbons from naphthalene. J. VARGA B.P. 333,453, 11.10.29. Ger., 13.10.28).—Naphthalene is hydrogenated under pressure of more than 100 atm. in presence of molybdenum or tungsten compounds and hydrogen sulphide (or substances which produce it) which serves to activate the catalyst. The temperature is maintained above or below the critical point (480–500°) at which a sudden absorption of heat occurs, followed by a rise of about 100°. For tetra- and deca-hydronaphthalenes naphthalene is heated for 1 min. at 460° with 4% of sulphur, 2% of molybdc acid, and hydrogen at 120 atm. For benzene the same proportions are used at 540° for 1 hr. C. HOLLINS.

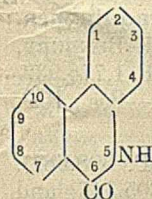
Production of pure anthracene. RÜTGERSWERKE A.-G., and L. KAHL (B.P. 319,762, 30.8.29. Ger., 29.9.28).—Crude anthracene is dissolved in 2 pts. of an organic base (or mixture of bases, *e.g.*, pyridine, quinoline, crude coal-tar bases, etc.) and heated at 160–170° with potassium hydroxide equivalent to the carbazole content. Pure anthracene is filtered off after cooling. Part of the bases used may be replaced by solvents such as benzene, halogenated hydrocarbons, etc. C. HOLLINS.

[Synthesis of] anthraquinone derivatives. H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,166, 21.2.29).—The carbamide made by phosphogenating *o*-(4-chloro-3-aminobenzoyl)benzoic acid is cyclised with 100% sulphuric acid at 120° to give 2:1- and 3:2-chloroaminoanthraquinones, separable by fractional dilution. C. HOLLINS.

Manufacture of anthraquinone intermediates and dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and W. W. TATUM (B.P. 333,992, 18.6.29).—The mixture of 1:5- and 1:8-diaminoanthraquinones obtained by reduction of the product of nitration of anthraquinone is converted into the sulphate, which is heated at 200–220° to yield a mixture of diaminoanthraquinone-disulphonic acids. The product may be brominated, giving bromodiaminoanthraquinonesulphonic acids, from which, by replacement of bromine by a *p*-toluidino-group, a blue wool dye is obtained. C. HOLLINS.

Manufacture of nitrogen-containing derivatives of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 333,882, 13.3.29. Addn. to B.P. 307,926; B., 1930, 810).—Under milder conditions mononitro-compounds are obtained. The nitration of 2:3:7:8-dibenzpyrene-1:6-quinone in nitrobenzene at 15° for 12 hrs., and of 1:2:7:8-dibenzpyrene-3:6-quinone in nitrobenzene at 130–135° for 5 min., is described. C. HOLLINS.

Manufacture of phenanthridone, homologues, derivatives, and substitution products thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,173, 2.5.29).—A fluorenone in sulphuric acid is treated with hydrazoic acid in benzene, whereby the oxime probably formed first undergoes the Beckmann transformation with production of a phenanthridone. Fluorenone gives a 93% yield of phenanthridone (annexed formula). The preparation of 3:8-dibromo- (m.p. 313°), 3-nitro- (m.p. 343°), 3-amino- (m.p. 285–286°), and 3-hydroxy-phenanthridones, phenanthridone-1-carboxylic acid, and 2-hydroxy-1:2-benzophenanthridone, is described. C. HOLLINS.



Manufacture of emulsions. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,587, 15.3.29).—The residue from the distillation of synthetic methyl alcohol (from carbon oxides), *e.g.*, "isobutyl oil," b.p. 150–280°, containing ketones (45%), lactones (19%), acids and esters (15%), alcohols (15%), and phenols (6%), is mixed with an alkynaphthalenesulphonic acid, or with the sulphonation product from palm oil and phenol, to give an emulsifying agent for cresol, carbon disulphide, chlorinated hydrocarbons, etc. C. HOLLINS.

Cleaning of liquid solvents. J. H. BEAUMONT. From R. R. STOLLEY CORP. (B.P. 333,217, 6.5.29).—Solvents after use in dry-cleaning processes are purified by passing them upwards through aqueous caustic alkali, surmounted by a layer of active charcoal if desired, then through water, and finally over calcium chloride. C. HOLLINS.

Preparation of carbocyclic hydrocarbons. O. NICODEMUS and W. BERNDT, Assrs. to GEN. ANILINE

WORKS, INC. (U.S.P. 1,776,924—5, 30.9.30. Appl., [A] 20.4.26, [B] 21.11.27. Ger., [A] 24.4.25, [B] 22.6.25).—See B.P. 251,270 and 253,911; B., 1927, 742, 828.

Aminoalkylation of amines. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,752,617, 1.4.30. Appl., 12.1.28. Ger., 25.1.27).—See B.P. 301,401; B., 1929, 164.

Manufacture of halogen-substituted aromatic tertiary amines. G. KALISCHER and K. KELLER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,777,266, 30.9.30. Appl., 8.7.26. Ger., 15.7.25).—See B.P. 288,665; B., 1928, 441.

Manufacture of aromatic derivatives of formamide. R. WITZEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,777,777, 7.10.30. Appl., 11.3.26. Ger., 13.3.25).—See B.P. 269,302; B., 1927, 458.

Naphthalene derivatives and their applications in dye preparations. W. GIBSON, A. J. HAILWOOD, J. B. PAYMAN, and A. SHEPHERDSON, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,776,970, 30.9.30. Appl., 1.8.28. U.K., 29.8.27).—See B.P. 300,800; B., 1929, 124.

Application of naphthalene derivatives to dye preparations. W. GIBSON, A. J. HAILWOOD, J. B. PAYMAN, and A. SHEPHERDSON, Assrs. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,777,038, 30.9.30. Appl., 21.10.29. U.K., 29.8.27).—See B.P. 300,800; B., 1929, 124.

Manufacture of anthraquinone intermediates. W. W. TATUM, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,869, 30.9.30. Appl., 10.2.26. U.K., 11.1.26).—See B.P. 268,891; B., 1927, 437.

Manufacture of halogenated anthraquinones. A. SHEPHERDSON, W. W. TATUM, and H. M. BUNBURY, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,932, 30.9.30. Appl., 10.2.26. U.K., 11.1.26).—See B.P. 271,023; B., 1927, 518.

Ethylene from gaseous mixtures (B.P. 308,687). **Naphthalene from coal gas** (B.P. 305,545 and 333,297). **Acetylene and other unsaturated hydrocarbons** (B.P. 334,178).—See II. **Soluble leuco-esters** (B.P. 333,146—7).—See IV. **Aluminium alcoholates** (B.P. 334,820).—See VII. **Vulcanisation accelerators** (U.S.P. 1,747,187—8).—See XIV. **2:3 [$\beta\gamma$]-Butylene glycol by fermentation** (B.P. 315,263).—See XVIII.

IV.—DYESTUFFS.

See A., Oct., 1260, Effect of light on dyes (KRAIS). 1280, Mechanism of azo-dye coupling (BUCHERER and TAMA). 1291, Reactions of quinol (EVANS and DEHN). 1303, Combination between dyes and gelatin granules (RAWLINS and SCHMIDT).

PATENTS.

Production of colouring matters and intermediates [soluble leuco-esters]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,146, 25.1.29).—The sodium salt of the disulphuric ester of leuco- β -aminoanthraquinone is oxidised with ferri-cyanide in feebly acid solution to form the disul-

phuric ester of leucoindanthrone, together with a more soluble dark green compound which can be diazotised and coupled with β -naphthol; the azo compound couples with *p*-nitrodiazobenzene in alkaline solution. Similar products are obtained from leuco-esters of β -acetamidoanthraquinone, or of 3-chloro-2-aminoanthraquinone.

C. HOLLINS.

Vat dye derivatives [soluble leuco-esters]. L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,147, 29.1.29).—The leuco-ester of a vat dye is mixed in paste form with a dispersing agent (sulphite-cellulose liquor, aminated or oxidised sulphite-cellulose liquor, soaps, formaldehyde-naphthalenesulphonic acids, molasses, saponin, alkyl-naphthalenesulphonic acids, naphthalene-di- or -tri-sulphonic acid), and dried.

C. HOLLINS.

Manufacture of vat dyes [from isatin and hydroxynaphthacarbazoles]

G. FARBENIND. A.-G. (B.P. 316,099, 22.7.29. Ger., 21.7.28).—Isatin- α -anil is condensed in acetic anhydride with 4-hydroxy-11-methyl- α -naphthacarbazole (annexed formula) to give a deep black vat dye.

C. HOLLINS.

Manufacture of [indigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 318,107, 16.8.29. Ger., 27.8.28).—A 5-halogeno-7-alkoxy-4-methylisatin α -chloride (etc.) is condensed with a 4-halogeno- α -naphthol to give blue vat dyes fast to light. 5-Chloro-7-methoxy-4-methylisatin α -chloride and 4-chloro- α -naphthol give a greenish-blue.

C. HOLLINS.

[Anthraquinone vat] dyes. W. G. WOODCOCK, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,191, 31.1.29).—Vat dyes are purified or improved by treatment with hypochlorite and boric acid, sodium hydrogen carbonate, or similar weakly acidic substances.

C. HOLLINS.

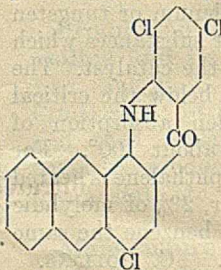
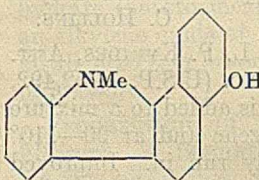
Manufacture of vat dyes of the anthraquinone-acridone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,240, 30.5.29).—Trichloroanthraquinoneacridone (annexed formula) is condensed with

amides or sulphonamides, whereby the α -chlorine is replaced by an acylated amino-group, yielding violet to blue vat dyes which may be hydrolysed to give greenish-blue vat dyes. The latter may be re-acylated. Condensations with toluenesulphonamide and benzamide, hydrolysis, and re-acylation with 1-chloroanthraquinone-2-carboxylic chloride, are described.

C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 319,365, 20.9.29. Switz., 21.9.28).—3:3'-Dibenzanthronyl is nitrated at 10—20° with mixed acid, and the product is fused with alkali to give a blue-grey vat dye. Nitration of this yields a blackish-green vat dye which produces deep black shades on oxidation on the fibre with hypochlorite.

C. HOLLINS.



Manufacture of monoazo dyes [for wool etc.]. CHEM. FABR. VORM. SANDOZ (B.P. 312,582, 27.5.29. Ger., 26.5.28).—*m*-Aminobenzenesulphonamides of the type, $\text{NH}_2\cdot\text{Ar}\cdot\text{SO}_2\text{NRR}'$, in which Ar carries no nitro-group to the amino-group, and no carboxyl in *o*-position, and R and R' are alkyl, aryl, or aralkyl groups, are diazotised and coupled with 1-sulphoaryl-5-pyrazolones or in acid medium with γ -acid. Examples are: aniline-*m*-sulphonethylanilide, m.p. 78°, $\rightarrow \gamma$ -acid (yellowish-red); *p*-toluidine-2-sulphonethylanilide \rightarrow 1-(2:5-dichloro-4-sulphophenyl)-3-methyl-5-pyrazolone (greenish-yellow). C. HOLLINS.

Manufacture of [insoluble] azo dyes dyeing black tints [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 311,400, 22.4.29. Ger., 11.5.28).—2:3-Hydroxynaphthoic *p*-2-cymidine (1-methyl-4-isopropyl-2-anilide) is coupled in substance or on the fibre with diazotised *o*-phenetidine $\rightarrow \alpha$ -naphthylamine or *p*-nitroaniline \rightarrow 2:5-dimethoxyaniline to give black dyes. C. HOLLINS.

Manufacture of polyazo dyes. CHEM. FABR. VORM. SANDOZ (B.P. 313,562, 4.6.29. Ger., 14.6.28).—Tetrazotised benzidine etc. is coupled with 1 mol. of H-acid in acid medium, and then in alkaline medium with 1 mol. of a diazo compound and 1 mol. of an *N*-alkylated (etc.) *m*-aminophenol, to give blacks for cotton, viscose silk, wool, silk, leather, and mixtures. An example is: aniline \rightarrow H-acid \leftarrow benzidine \rightarrow 3-hydroxy-2'-methylidiphenylamine. C. HOLLINS.

Solubilisation of a perylenetetracarboxylic diimide dye and dyes produced thereby. A. J. HALLWOOD, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,971, 30.9.30. Appl., 25.7.27. U.K., 19.8.26).—See B.P. 280,647; B., 1928, 83.

Manufacture of aminoanthraquinone derivatives [dyes for cellulose acetate]. O. BALLY and P. GROSSMANN, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,777,953, 7.10.30. Appl., 16.7.28. Switz., 4.8.27).—See B.P. 295,257; B., 1929, 710.

Primary disazo dyes. J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,801, 30.9.30. Appl., 19.4.28. U.K., 28.4.27).—See B.P. 294,922; B., 1928, 741.

Manufacture of colour lakes. L. RUDOLF and B. ZSCHIMMER, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,776,931, 30.9.30. Appl., 16.7.26. Ger., 22.7.25).—See B.P. 274,627; B., 1927, 742.

Anthraquinone dyes (B.P. 333,992). Mononitrated benzanthrone derivatives (B.P. 333,882).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Down of *Apocynum venetum*. N. L. KARAVAEV and P. N. ODINTZOV (J. Appl. Chem., Russia, 1930, 3, 91—97).—The cellulose content approaches that of straw. The down might serve as a thermal insulator, but is unsuitable for the manufacture of nitrated products. CHEMICAL ABSTRACTS.

Determination of dry weight of wood pulp. B. WIESLER (Papier-Fabr., 1930, 28, 616—618).—A discussion of the difficulties attending the accurate

determination of moisture content in pulp deliveries, and the various commercial customs relating to the expression of "absolute dry" and "air-dry" weights.

T. T. POTTS.

Rosin size manufacture. RIDLEY.—See XIII.

See also A., Oct., 1275, **Lignin and related compounds** (HIBBERT and others). 1303, **Rapid determination of furfuraldehyde** (NOLL and others). 1322, **Sinkage of pulp-wood** (GIBBS). 1325, **Hemicelluloses of jute fibre** (CHOUDHURY and SAHA). **Hemicelluloses of maize cobs** (PREECE).

PATENTS.

Effecting continuous digestion in the extraction of cellulose. J. HOLMES and H. A. KINGCOME (B.P. 333,426, 20.9.29).—A continuous, fractional-digestion process is described, which works on the countercurrent principle and uses, if desired, liquors of different composition in the various pressure stages. The apparatus consists of a number of vertical tubular members, the pressure in each stage being maintained by a balancing column of liquor. The digestion liquor is heated indirectly by steam and the condensate is used in the countercurrent washing of the digested material.

D. J. NORMAN.

Cellulosic compositions. BRIT. CELANESE, LTD. (B.P. 313,134, 7.6.29. U.S., 7.6.28).—Materials containing organic esters of cellulose are rendered less inflammable or non-inflammable by incorporation therein at any convenient stage of one or more aromatic compounds containing bromo-substituted aliphatic side-chains, e.g., benzyl bromide, benzylidene bromide, in the proportion of 2—40 pts. to 100 pts. of the cellulose derivative. F. R. ENNOS.

Manufacture of pulp and paper. L. M. BOOTH (U.S.P. 1,761,069, 3.6.30. Appl., 22.10.27).—After the production of pulp by an alkaline digestion, the usual washing is either dispensed with or is discontinued while an appreciable amount of alkali still remains, and addition is made of an acid coagulating salt (ferrous sulphate, ferric chloride) and of chlorine.

F. R. ENNOS.

Preparation of electrolytic writing and drawing paper and the like. R. HORN (B.P. 313,561, 4.6.29. Ger., 14.6.28).—Paper is impregnated, in some cases with addition of a carrier electrolyte (sodium chloride), with an aqueous or alcoholic, neutral or alkaline solution either of an organic nitroso-, nitro-, amino-, hydroxyl-amino-, or quinone compound, e.g., dimethylglyoxime, or of an organic colouring matter (sodium alizarin-monosulphonate), together with a mordant if required, so that on passage of a current with a soluble anode of silver, copper, lead, iron, nickel, aluminium, or alloys of these metals with each other or with zinc or tin, in the absence of a catalyst, either a sparingly soluble organic metal compound or a lake is produced, whereby the paper is marked. Alternatively, the paper, after treatment with a carrier electrolyte, a catalyst (vanadium pentoxide), and a mordant, is impregnated with an aqueous or alcoholic solution of an organic substance, e.g., aniline, which produces a sparingly soluble colouring matter or lake when oxidised with an insoluble anode or an anode rendered passive. F. R. ENNOS.

Coating paper. W. W. TRIGGS. From CHAMPION COATED PAPER CO. (B.P. 333,226, 3.5.29).—The binding agent consists of raw starch applied to the paper in aqueous suspension in admixture with the other constituents of the coating mixture, gelatinisation being subsequently effected by exposing the wet coated paper to substantially saturated steam. A dispersing agent (gum arabic) is preferably added to the coating mixture.

D. J. NORMAN.

Films or sheets for adhesive purposes. R. ARNOT (B.P. 333,194, 27.2. and 15.3.29).—Synthetic resins in a reactive state are applied to a porous carrier such as paper, fabric, or the like, or are mixed with film-forming solutions, *e.g.*, viscose solution or cellulose ester solutions, without the addition of any volatile solvents other than those which normally result from manufacturing processes, *e.g.*, those present in cellulose lacquers. Softening agents, metal powders, fireproofing agents, or other adhesives may be incorporated.

D. J. NORMAN.

[Spinning pot for] production or treatment of artificial threads or other textile yarns, *e.g.*, rayon, by the centrifugal spinning process. J. L. RUSHTON, H. HILL, and D. L. PELLATT (B.P. 335,049, 11.9.29).

Spinning centrifuges for artificial silk. A. E. O'DELL. From BERLIN-KARLSRUHER INDUSTRIEWERKE A.-G. (B.P. 335,307, 16.7.29).

Paper pulp and like beating engines. S. MILNE (B.P. 335,671, 26.7.29. Addn. to B.P. 207,716).

Carbohydrate compounds (B.P. 334,282).—See III. **Rubber goods etc.** (B.P. 335,271). **Rubber-paper products** (U.S.P. 1,756,035).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Kinetics of chlorine bleaching. H. KAUFFMANN (Z. angew. Chem., 1930, 43, 840—843).—Assuming that the active substance in bleaching liquors is free hypochlorous acid formed by the hydrolysis of hypochlorite, the velocity coefficient of the transformation of the ClO' ion (a reaction of the second order) is a measure of the bleaching power of the solution. It is shown that this coefficient must be inversely proportional to the hydroxyl-ion concentration, and this is in accord with the observation that bleaching power increases with increasing acidity. Since the destructive effect on cotton fibre increases with the alkalinity, it follows that neither hypochlorous acid nor chlorine monoxide can be responsible. It is assumed that the fibre is attacked by an unstable complex ion $[\text{ClOH}, \text{ClO}]'$, and a calculation involving the equilibrium constants of the dissociation of hypochlorous acid and of the hydrolysis of chlorine shows that the destructive effect should be at a maximum when the ratio $\text{Cl}_2 : \text{NaOH}$ is 1 : 1.5. This is in agreement with observation. F. L. USHER.

Liability of dyed wool to mildew with special reference to the resistance resulting from chroming. R. BURGESS (J. Text. Inst., 1930, 21, T441—452).—Samples of wool dyed under various conditions with and without addition of chromate were inoculated with mould fungi, maintained under conditions favourable

to mould growth, and their liability to mildew was examined. The solubility of chromed and unchromed wool in boiling water, and in an Alizarin Red dye-bath, the effects of various concentrations of potassium dichromate on mildew development on unscoured yarn, and enzyme activity on chromed and unchromed wool have also been investigated. From the results obtained it is concluded that the process of chroming imparts to wool a considerable resistance to the growth of mould fungi, which is due to impoverishment of the wool as a source of the nutriment required by the organisms. Thorough washing-off after dyeing or scouring reduces the liability to mildew. B. P. RIDGE.

Potentiometric control of the degree of mercerisation of cellulose. S. N. USHAKOV and A. D. SOKOLOV (J. Appl. Chem., Russia, 1930, 3, 31—46).—Electrometric titration of the liquors indicates that the rate of circulation is important in shortening the reaction time. CHEMICAL ABSTRACTS.

Colloid chemistry of laundering. L. ZAKARIAS (Rev. gén. Colloid., 1930, 8, 112—120).—A review of published work on the protective effect, surface tension, and foaming power of soaps and their relation to the detergent effect. The detergent action of soap is due to the peptisation and swelling of the colloidal impurities, which combine with the soap to form a mixed colloid, soluble in water and readily removed by rinsing. The addition of fats, sulphonated oils, or polysaccharides does not increase the detergent action of soap. The presence of polysaccharides protects soap from the action of calcium salts, so that soaps containing polysaccharides have simultaneously a softening and a detergent effect.

E. S. HEDGES.

Detergent power of soaps. BOSSHARD and STURM.—See XII.

See also A., Oct., 1260, **Effect of light on dyes** (KRAIS). 1309, **Fastness of dyes to human perspiration** (VASS and McSWINEY).

PATENTS.

Dyeing of animal fibres. DURAND & HUGUENIN Soc. ANON. (B.P. 334,508, 27.5.29. Addn. to B.P. 218,649; B., 1925, 708).—Dyeings fast to rubbing are obtained with esters of leuco-vat dyes on animal fibres by adding to the acid oxidising bath used for development an emulsifying agent which remains soluble in an acid medium ("Supamines," or a sulphonated fatty acid).

F. R. ENNOS.

Application of vat dyes to textile fibres. A. R. THOMPSON, JUN., ASSR. to ROHM & HAAS Co. (U.S.P. 1,759,071, 20.5.30. Appl., 27.2.26).—Streaky or uneven dyeing is prevented by addition of sodium formaldehyde-sulphoxylate to the alkaline dye bath reduced with sodium hyposulphite.

F. R. ENNOS.

Coloration of textile materials. BRIT. CELANESE, LTD. (B.P. 308,173, 18.3.29. U.S., 17.3.28).—Yarns, particularly those containing cellulose derivatives, are tinted with fugitive colours by contact with a wick or pad moistened with a solution of a dye in di- or polyhydric alcohols or compounds having a di- or polyhydric alcohol base of b.p. above 100°. Suitable solvents include ethylene glycol, diethylene glycol, and their

mono- or di-ethyl ethers. Since these compounds are hygroscopic it is preferred to add an amount of water equal to that which would be absorbed from the air during use; this addition prevents gradual subsequent changes in viscosity and volume. A. J. HALL.

Coloration of textile materials. BRIT. CELANESE, LTD. (B.P. 308,733, 27.3.29. U.S., 27.3.28).—Filaments, yarns, etc. are temporarily coloured, continuously with their production, by passing over a wick, pad, or roller supplied with a tinting fluid consisting of a fugitive colouring matter, a non-drying animal, vegetable, or mineral oil, and a sulphonated oil.

F. R. ENNOS.

Coloration of [artificial silk] textile filaments, yarns, threads, etc. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,565, 11.1.29. Cf. B.P. 332,263; B., 1930, 944).—Artificial silk yarns composed of cellulose or its esters or ethers are coloured intermittently with fast dyes at regular or irregular spaced intervals along their length by means of suitable devices which rotate or oscillate, or reciprocate in contact with the travelling yarn.

A. J. HALL.

Production of coloured resists under Aniline Black. I. G. FARBENIND. A.-G. (B.P. 316,316, 26.7.29. Ger., 28.7.28).—The fabric, padded as for Aniline Black, is printed with a paste containing zinc oxide, large excess of caustic alkali, and a mixture of a non-nitrated nitroso-amine alkali salt and a coupling component such as a 2:3-hydroxynaphthoic or acetoacetic arylamide. The fabric is steamed, whereby the Aniline Black is developed but not the azo colour, and then passed successively through an acetic acid bath and a chromate bath to develop the coloured resist. The production of yellow, orange, scarlet, and red resists free from aureole is described.

C. HOLLINS.

Production of thickening materials for use in printing [of textile fabrics]. H. BOLLMANN and B. REWALD (B.P. 333,959, 28.5.29).—A mixture of one or more of thickening agents, such as dextrin, gum tragacanth, starch, or gum arabic, with a solution of vegetable or animal phosphatides in a mineral oil is claimed. Suitable phosphatides are extracted from ground soya beans by means of benzene-alcohol (9:1), the solvent being afterwards removed by distillation and the phosphatides precipitated from the residual oil by blowing in steam; soya-bean oil adhering to the precipitated phosphatides is largely removed by centrifuging.

A. J. HALL.

Printing on wool [with leuco-vat dye esters]. I. G. FARBENIND. A.-G. (B.P. 334,419, 11.10.29. Ger., 11.10.28. Addn. to B.P. 298,648; B., 1929, 1012).—Wool is dyed with a dischargeable colour, and is then printed with leuco-vat dye ester and a reducing agent (formaldehydesulphoxylate), steamed, and developed with acid and persulphate. Coloured discharge effects are obtained.

C. HOLLINS.

Manufacture of artificial silk [of matte lustre] from viscose. I. G. FARBENIND. A.-G. (B.P. 312,687, 6.5.29. Ger., 31.5.28).—Subdued lustre is imparted to viscose silk by emulsifying with the viscose solution not less than 0.5% (calc. on the viscose) of one or more

organic bases (excluding aniline) which are more or less insoluble in water, *e.g.*, quinoline, dimethylaniline, toluidine.

D. J. NORMAN.

Production or treatment of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., W. I. TAYLOR, R. P. ROBERTS, and L. W. GREGORY (B.P. 334,563, 28.3.29).—Materials of reduced or subdued lustre are produced by dry-spinning solutions of cellulose esters or ethers containing a metallic (barium) salt, and subsequently treating the formed filaments with a fixing solution to precipitate therein an opaque, insoluble metallic compound (barium sulphate or phosphate).

F. R. ENNOS.

Improvement of fabrics made from or containing vegetable fibre. HEBERLEIN & Co. A.-G. (B.P. 334,756, 14.10.29. Ger., 12.10.28).—A linen-like appearance is imparted to the fabric, without loss of its natural softness, by pressing the fabric above 100° and then treating with a swelling agent (mineral acids, concentrated alkali lye with or without cooling, concentrated zinc chloride or ammoniacal copper oxide solutions).

F. R. ENNOS.

Waterproofing of fabrics. J. W. MACKENZIE (B.P. 334,020, 16.7.29).—A waterproofing composition, applicable to canvas fabric by painting is prepared by heating together a mixture of flour, water, sodium carbonate, and tallow or of a heavy mineral oil, and adding rosin; a sizing agent such as gum arabic may be added before use. The composition dries on the fabric within 1 day.

A. J. HALL.

Treating and ageing of fabrics. E. S. CHAPIN and A. H. JACOBY (B.P. 334,069, 29.8.29).—The steam supplied to the ageing chamber is admitted at successive stages in such manner that although heat is generated locally within the fabric (printed with vat dyes) due to chemical reactions, its temperature is maintained constant within satisfactory limits.

A. J. HALL.

Treatment of casein-varnish-finished coated fabrics or articles. M. N. NICKOWITZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,746,162, 4.2.30. Appl., 23.11.27).—Sheeting or fabric surfaced with rubber (or even with pyroxylin or oxidised oil) is treated with a casein varnish. After vulcanisation by dry heat the surface is treated with a 4% solution of bromine in carbon tetrachloride, the varnish thereby being softened and rendered smooth without affecting the under layer of rubber.

D. F. TWISS.

Dyeing [of regenerated cellulose]. J. BADDILEY, R. BRIGHTMAN, and P. CHORLEY, Assrs. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,776,802, 30.9.30. Appl., 4.6.28. U.K., 7.6.27).—See B.P. 296,485; B., 1928, 855.

[Dyeing of "mixed" cellulose acetate] threads, cords, and fabrics. C. W. PALMER and S. M. FULTON, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,777,445, 7.10.30. Appl., 16.6.27. U.K., 21.7.26).—See B.P. 278,116; B., 1927, 905.

Apparatus for cleaning liquid solvents used for dry-cleaning and other purposes. J. H. BEAUMONT. From R. R. STOLLEY CORP. (B.P. 334,208, 27.5.29).—See U.S.P. 1,711,829; B., 1929, 895.

Bleaching or otherwise treating fibrous material. B. P. VON EHRENTAL and K. SCHOLZ (B.P. 316,151, 23.7.29).

Washing machines. E. A. ALLIOTT, and MANLOVE, ALLIOTT & CO., LTD. (B.P. 335,028, 22.8.29). R. S. LANGE (B.P. 334,906, 13.4.29).

Centrifugal extractor (U.S.P. 1,760,775).—See I. Fatty acid esters [as wetting agents] (B.P. 315,832). Cleaning solvents (B.P. 333,217).—See III.

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

Chemistry of leaching chalcocite. J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper 473, 1930, 24 pp.).—Chalcocite (Cu_2S) can be leached with ferric sulphate with the production of copper sulphate and ferrous sulphate. Neither air with water alone nor ferrous sulphate dissolves the mineral, but sulphuric acid with access of air will dissolve it at a slower rate than ferric sulphate solution. The rate of dissolution of chalcocite is independent both of the concentration of ferric sulphate, provided enough reagent is present, and of the acid concentration, though a slight excess of acid is desirable as it prevents the formation of basic iron compounds. Increase in temperature greatly accelerates the reaction; *e.g.*, with a solution containing 0.5% Fe as ferric sulphate and 0.5% H_2SO_4 , 73% of the copper was dissolved in 1 day at 50°, in 5 days at 35°, and in 15 days at 23°. Variation in size of particles from 10- to < 200-mesh had little influence on the rate of dissolution if the particles were freely in contact with the solution. Products of the reaction do not interfere until large quantities of ferrous sulphate appear in the solution. When leached with ferric sulphate one half of the copper dissolves rapidly, leaving the residue with an approximate formula, CuS , but of entirely different properties from those of covellite. This compound is attacked at a much slower rate. (Cf. following abstract.) C. A. KING.

Innovations in copper leaching employing ferric sulphate-sulphuric acid. H. E. KEYES (U.S. Bur. Mines, Bull. 321, 1930, 67 pp.).—The leaching solution containing ferric sulphate and sulphuric acid is produced by autoxidation of ferrous sulphate and sulphur dioxide in the presence of air. The first reaction is to oxidise the iron and then to produce sulphuric acid, but intimate contact of air and solution is desirable. Most copper minerals, *e.g.*, chalcocite, bornite, and the oxidised forms of mineral, are amenable to leaching with acidified ferric sulphate, exceptions being chalcopyrite and certain silicates. Copper is precipitated from solution by means of sponge iron. Results of plant tests on different ores by open drainage and flood-leaching are described. (Cf. preceding abstract.) C. A. KING.

Heat transfer. OTHMER.—See I. Organic nitrogen from agricultural waste. DIEGO. Sulphur from coal gas. PETIT.—See II. Action of acids on non-rusting steels. SAUVAGEOT and LAUPRÊTE. Titration of potassium cyanide. SANIGAR.—See X. Measuring and recording p_{H} . WULFF and KOR-

DATZKI.—See XI. Ammonia from beet juice. ZAMARON.—See XVII. Base-exchange materials. MARTIN.—See XXIII.

See also A., Oct., 1258, Sputtered nickel films and the synthesis of ammonia (INGERSOLL). 1277, Determination of metal in organic compounds incapable of electrolysis (DOSIOS and PIERRI).

PATENTS.

Production of chemically pure phosphoric acid. I. G. FARBERIND. A.-G. (B.P. 334,790, 18.11.29. Ger., 20.2.29. Addn. to B.P. 319,656; B., 1930, 660).—The efficiency of the prior process is improved by electrolysis at 50° or above. When the crude acid contains insufficient heavy-metal salt small amounts of a copper salt, preferably copper phosphate, are added in order to facilitate the deposition of arsenic. The solution may contain 1 pt. of copper to at least 2 pts. (preferably 2.5–4 pts.) of arsenic. S. K. TWEEDY.

Digestion of phosphate rock. W. H. LOHMANN, ASSR. to GEN. CHEM. CO. (U.S.P. 1,747,588, 18.2.30. Appl., 17.5.27).—In the digestion of phosphate rock with liquor obtained by washing mud produced in a previous digestion and adding sulphuric acid and nitre cake to the resulting liquid, the precipitation of glau-berite (calcium sodium sulphate) is prevented by keeping the liquor below 66° throughout the digestion. S. K. TWEEDY.

[Continuous] production of dicalcium phosphate. A. HOLZ and T. V. D. BERDELL (B.P. 335,007, 30.7.29).—A solution of an alkali chloride is electrolysed, the liberated hydrogen is collected separately, and the chlorine passed into water into which an equivalent amount of sulphur dioxide is simultaneously introduced. The mixture of hydrochloric and sulphuric acids obtained is caused to react with crude phosphate rock, the solution, after filtration, being treated with the alkali hydroxide, derived from the electrolysis, and calcium hydroxide, and the precipitated dicalcium phosphate removed by filtration. The mother-liquor containing alkali chloride is utilised for the electrolysis. Alternatively, after treatment of the phosphate rock and filtration, the filtrate may be concentrated and caused to react with additional phosphate rock, and the solution of monocalcium phosphate precipitated with alkali hydroxide alone. W. J. WRIGHT.

Manufacture of dithiophosphates. C. T. ROMIEUX and H. P. WOHNSIEDLER, ASSRS. to AMER. CYANAMID CO. (U.S.P. 1,748,619, 25.2.30. Appl., 14.11.27).—Phosphorus pentasulphide is treated with an organic hydroxy-compound (*e.g.*, an aliphatic alcohol such as isopropyl alcohol). A solvent, such as solvent naphtha or other hydrocarbon, is then added to the crude product, after which an amine (*e.g.*, aniline or ammonia) is added and the resulting pure dithiophosphate of the amine is filtered off. Alternatively, the crude product is dissolved in an alkaline substance (*e.g.*, sodium carbonate solution), the aqueous layer formed being removed and treated with acid in order to regenerate the dithiophosphoric acid in the pure state. The acid is then dissolved in a solvent, such as solvent

naphtha or water, and a dithiophosphate precipitated therefrom by addition of an amine, a base, or a metal salt.

S. K. TWEEDY.

Manufacture of bleached white barytes. W. J. O'BRIEN (U.S.P. 1,758,009; 13.5.30. Appl., 28.5.27).—The ground barytes (1–10 pts.) is fused and roasted with nitre cake (1 pt.) in an oxidising atmosphere, up to 0.1 pt. of an oxidising agent (*e.g.*, sodium nitrate, potassium chlorate) being added if desired; the molten mass when passed into water preferably containing a reducing agent (sodium sulphite) yields a precipitate of bleached barytes. The nitre cake may be replaced by sodium chloride up to the extent of 50%.

S. K. TWEEDY.

Manufacture of barium carbonate. B. LAPORTE, LTD., I. E. WEBER, and V. W. SLATER (B.P. 334,709, 5.9.29).—A barium compound containing sulphur (*e.g.*, the sulphide or hydrogen sulphide) is added to a solution of carbon dioxide (under pressure, if desired) or barium hydrogen carbonate in such proportion that carbonic acid or barium hydrogen carbonate is always present, or that the p_H value of the solution is always less than 7. The process may be made continuous by simultaneously adding carbon dioxide and barium sulphide to water; the gas need not be free from oxygen.

S. K. TWEEDY.

Production of gels. Preparation of zeolites. W. W. TRIGGS. From GEN. ZEOLITE CO. (B.P. 334,676—7, 27.7.29).—(A) Small proportionate amounts of two concentrated reacting solutions are mixed in coalescing flow; the flow is continued in order to effect thorough admixture and the fluid is then collected in a receiving tank where the gel is allowed to develop. Preferably the two solutions come into contact as baffled jets. The method may be employed to produce gel zeolites, and is particularly adapted for use with solutions which react rapidly to form thick gelatinous precipitates. The operations may be carried out at normal temperature. (B) An alkali silicate solution containing about 0.25–2 mols. of silica per mol. of alkali oxide is combined with a solution of an amphoteric metal hydroxide containing 1–1.25 acid equivalents to each equivalent of amphoteric metal oxide. The gel formed is dehydrated, preferably below 100°, and thoroughly washed. The silicate solution may be obtained by mixing, during or before the zeolite-forming reaction, a soluble silicate and a non-zeolite-forming alkaline substance; or the amphoteric oxide solution by mixing a soluble amphoteric metal compound and a non-zeolite-forming acidic substance. In an example, sodium silicate, aluminium sulphate, sodium hydroxide, and sulphuric acid are caused to interact in admixture.

S. K. TWEEDY.

Manufacture of aluminium alcoholates containing aluminium chloride. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 334,820, 7.1.30. Ger., 13.6.29).—A mixture of aluminium and an alcohol is treated, if desired, at elevated temperature, and preferably in presence of an indifferent solvent such as ethyl acetate, with dry chlorine, or with a chlorine compound (*e.g.*, hydrochloric acid) capable of imparting chlorine to aluminium. Excess of aluminium is essential if the alkoxide produced is to be free from

alcohol of crystallisation. The product actively catalyses the conversion of acetaldehyde into ethyl acetate.

S. K. TWEEDY.

Manufacture of metal carbonyls. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,976, 24.6.29 and 10.3.30).—Carbon monoxide is passed in succession through a series of reaction vessels, the carbonyl formed in any vessel being condensed from the gas stream before it enters the next vessel. Fresh supplies of carbon monoxide are admitted first to the vessel in which the reacting material is almost exhausted and the gas stream almost denuded of carbon monoxide issues from the vessel containing the freshest reaction materials.

A. R. POWELL.

Manufacture of hydrogen. A. T. LARSON, ASSR. to LAZOTE, INC. (B.P. 311,737, 14.5.29. U.S., 15.5.28).—The reaction of steam with carbon monoxide at temperatures above 300° is accelerated by employing catalysts containing copper and an oxide of zinc, tungsten, molybdenum, cerium, vanadium, manganese, uranium, chromium, or magnesium.

W. J. WRIGHT.

[Electrolytic] manufacture of chlorine. F. S. LOW, ASSR. to WESTVACO CHLORINE PRODUCTS, INC. (U.S.P. 1,746,542, 11.2.30. Appl., 25.10.28).—Hydrochloric acid is electrolysed between submerged electrodes, the hydrogen chloride content of the solution being replenished, *e.g.*, by introducing gas below the surface of the electrolyte, preferably at a point which is so much below the surface that the gas introduced is almost completely absorbed. A cell for carrying out the process is described.

S. K. TWEEDY.

Vanadium catalysts for use in the oxidation of oxidisable gases [sulphur dioxide]. S. ROBSON and P. S. LEWIS (B.P. 334,862, 8.6.29).—A mixture of dried silica gel with 5–10% of vanadium pentoxide, ammonium vanadate, or an alkali vanadate and a small quantity of a promoter such as stannous chloride is bonded with sodium hydroxide or silicate and with a vegetable gum to form a thick paste which after partial drying is made into pellets. These are then treated cold with a stream of sulphur dioxide until they show an acid reaction, heated to 400° in a stream of hot air, and transferred to the catalyst chamber for the manufacture of sulphuric acid by the contact process.

A. R. POWELL.

Manufacture of carbon disulphide. H. OEHME, and CHEM. FABR. KALK G.M.B.H. (B.P. 314,060, 5.6.29. Ger., 23.6.28).—Hydrogen sulphide, absorbed in ammonia liquor from coal gases and liberated by heat or by a current of carbon dioxide, is passed over charcoal at 900–1000° at such a rate that 70% of the sulphur is converted into carbon disulphide; the unused hydrogen sulphide is absorbed in ammonia liquor and returned to the circuit.

C. HOLLINS.

Manufacture of carbon disulphide. H. OEHME, and CHEM. FABR. KALK G.M.B.H. (B.P. 334,856, 5.6.29. Ger., 2.10.28).—In the process described in B.P. 314,060 (preceding abstract), after removal of the reaction products when only partial decomposition of the hydrogen sulphide has taken place, the remaining hydrogen sulphide, separated from the hydrogen, if desired, is used for producing sulphur or its compounds.

other than carbon disulphide. Or the mixture of hydrogen and hydrogen sulphide may be returned to the crude gas, so that recovery of undecomposed hydrogen sulphide is effected at the same time as the recovery of the hydrogen sulphide from the crude gas itself.

W. J. WRIGHT.

Preparation of phosgene [carbonyl chloride].

R. WILLIAMS, ASSR. to DU PONT AMMONIA CORP. (U.S.P. 1,746,506, 11.2.30. Appl., 4.10.27).—Carbon monoxide and nitrosyl chloride are allowed to interact, preferably in presence of a catalyst comprising activated carbon and within the temperature range of 100–250°.

S. K. TWEEDY.

Manufacture of phosphoric acid. S. G. NORDEN-GREN, ASSR. to AKTIEB. KEMISKA PATENTER (U.S.P. 1,776,595, 23.9.30. Appl., 12.4.29. Ger., 7.7.28).—See B.P. 314,976; B., 1930, 861.

Manufacture of ammonium chloride. J. W. MOORE and W. G. POLACK, ASSRS. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,776,698, 23.9.30. Appl., 1.6.27. U.K., 25.6.26).—See B.P. 274,263; B., 1927, 701.

Manufacture of iron carbonyl compositions. A. MITTASCH and M. MÜLLER-CUNRADI, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,920, 30.9.30. Appl., 22.4.25. Ger., 5.12.24).—See Can.P. 262,601; B., 1927, 403.

Fixation of atmospheric nitrogen. J. BRIGHT-MORE (U.S.P. 1,776,308, 23.9.30. Appl., 23.6.27. U.K., 6.7.26).—See B.P. 283,232; B., 1928, 192.

Sulphur compounds from gases (B.P. 305,027).—See II. **Abrasive** (U.S.P. 1,747,579).—See VIII. **Extracting iron from ores** (U.S.P. 1,759,456). **Metal netting as catalyst** (B.P. 334,466).—See X. **Water-purifying material** (U.S.P. 1,757,372).—See XXIII.

VIII.—GLASS; CERAMICS.

Chemical and mineralogical character of stalactites from two glass works. D. S. BELIANKIN (Bull. Acad. Sci. U.S.S.R., 1930, 257–265).—Stalactites formed in the furnaces of two glass works have been examined and the chemical natures of dinas and glass compared. Chemically the two stalactites differ only in their lime and magnesia contents. Mineralogically, whilst one appears to consist of tridymite, cristobalite, and glass, the other consists of tridymite and pseudowollastonite.

A. FREEMAN.

Translucency and particle size in white enamels containing stannic and zirconium oxides. C. J. VAN NIEUWENBURG and J. SCHOTSMAN (Trans. Ceram. Soc., 1930, 29, 342–351).—To test the truth of the supposition that the opacity of enamels increases with decreasing particle size, provided that the latter remains about one half to one fourth of the wave-length of the kind of light used, the opacifying power was measured and curves for the complete cumulative distribution of three brands of tin oxide and two of zirconium oxide were obtained by the use of an ordinary visual photometer. Results show the above hypothesis to

be incorrect. It is concluded that a test of particle size is by no means sufficient as a means of predicting opacifying power.

R. J. CARTLIDGE.

Earthenware glaze preparation. A. TEICHFELD (Trans. Ceram. Soc., 1930, 29, 339–341).—In Poland, and, in general, on the Continent, the frit in the mill mixture of earthenware glazes varies between 65% and 80%. This necessitates a fritting operation once in every six weeks, the kiln being run for a whole week. When the content of frit in the mixture was reduced from 76% to 48%, fritting was then only necessary once in every three months, and the melted frit ran so easily that for trials the well-known perforated crucible was used.

R. J. CARTLIDGE.

Tenriuji yellow Celadon glaze. T. ISHII (Trans. Ceram. Soc., 1930, 29, 352–359).—Nine base glazes within the range $0.2K_2O$, $0.8CaO$, $0.3-0.5Al_2O_3$, $3.0-4.0SiO_2$ were prepared to find some which would be similar to the Tenriuji Celadon in lustre, transparency, and colour. The colorants used contained 3.0–3.5 pts. of ferrous silicate and 0.01–0.07 pt. of chromic oxide, added to 100 pts. of the batches. Experiments were conducted on glazes containing rutile and manganous carbonate as auxiliary colorants. The secret of producing this glaze is shown to lie in the composition of the base glaze and of the body, and in the nature of the kiln atmosphere during firing.

R. J. CARTLIDGE.

Kinuta blue Celadon glaze. T. ISHII (Trans. Ceram. Soc., 1930, 29, 360–387).—Firing is most important in conducting experiments on Celadon glazes, since the colours are strongly affected by the nature of kiln atmosphere. Test pieces were fired in a wood-fired laboratory kiln with reducing flame to about 1300° for 18–26 hrs. Results show the best base glazes for Kinuta Celadon ware are those which are more siliceous than porcelain glazes and tend to be devitrified. The felspar content should be high enough to prevent rapid melting, but not so high that crazing results. The best proportion for the bases is $0.5K_2O$ to $0.5CaO$, or $0.6K_2O$ to $0.4CaO$.

R. J. CARTLIDGE.

X-Ray examination of kaolins and clays. F. KÄSTNER and F. K. MAYER (Sprechsaal, 1930, 63, 118–120; Chem. Zentr., 1930, i, 2466).—Chemical and X-ray analysis showed that kaolinisation of granite buried for 1 year in garden soil had occurred. The clays and kaolins examined contained the same mineral (kaolin).

A. A. ELDRIDGE.

Firing of ceramics and faults due to firing. P. BREMOND (Trans. Ceram. Soc., 1930, 29, 330–338).—The bodies examined were fired slowly to given temperatures, and after each firing the vitrification was determined. Curves show that the hard porcelains are not readily vitrified until about 1200°, whereas felspar porcelains begin to vitrify at about 1050–1100°. Carburisation by carbon monoxide, methane, ethylene, and town's gas is discussed. Faults in all porcelain and stoneware bodies may be created or removed by carburising and decarburising these bodies at a stage in the firing below the 5% porosity point or above this limit. (Cf. B., 1927, 141.)

R. J. CARTLIDGE.

Influence of oxidising and reducing atmospheres on refractory materials. III. Experiments with a refractory mixture containing added amounts of colloidal ferric hydroxide. A. E. J. VICKERS (Trans. Ceram. Soc., 1930, 29, 290—298; cf. B., 1928, 570).—Refractory cones containing carbon and colloidal ferric hydroxide were heated in atmospheres of nitrogen, oxygen, carbon monoxide, carbon dioxide, sulphur dioxide, hydrogen, air, and water vapour and the effects were noted. An inspection of the graph shows that water vapour produces the greatest decrease in refractoriness, but such effect for any gas depends on the amount of ferric oxide added. R. J. CARTLIDGE.

Formation of mullite in refractories. P. P. BUDNIKOV and B. A. KHIZH (J. Appl. Chem., Russia, 1930, 3, 21—24).—A furnace lining originally containing SiO_2 57—58, Al_2O_3 36.4—37.3, Fe_2O_3 1.5—2, CaO 1.5—1.9%, after six months' service in a coal-fired furnace contained SiO_2 49.38, Al_2O_3 28.54, Fe_2O_3 20.82, CaO 0.91%. Crystals of mullite with embedded crystals of ferrous oxide were present. Under certain conditions the formation of mullite is favoured by the presence of iron compounds. CHEMICAL ABSTRACTS.

Advances in the decorative arts as applied to ceramics. F. H. RHEAD (Trans. Ceram. Soc., 1930, 29, 388—400).

Pan grinding. MELLOR.—See I.

See also A., Oct., 1241. **Silicate system based on crystal structure** (VON NÁRAY-SZABÓ).

PATENTS.

Colouring of clay. R. T. VANDERBILT, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,755,616, 22.4.30. Appl., 5.5.27).—The clay, which may be previously dried, is sprayed, in lump or granule form, and while hot from the drying operation, if desired, with a solution of a dye, and is then pulverised and thoroughly mixed.

S. K. TWEEDY.

Manufacture of [non-efflorescing] bricks. A. BONNINGTON, Assr. to ALTON BARIUM PRODUCTS CO. (U.S.P. 1,758,026, 13.5.30. Appl., 14.7.27).—As raw materials brick clay together with natural barium sulphate, or the residue from roasted or reduced barytes ore from which, if desired, the water-soluble ingredients have been extracted, are employed. The proportions of the admixture are preferably such that the water-soluble salts contained in the clay are converted into insoluble salts. S. K. TWEEDY.

Manufacture of [abrasive] material comprising anhydrous calcium sulphate and calcium pyrophosphate. C. B. DURGIN and P. LOGUE, Assrs. to FEDERAL PHOSPHORUS CO. (U.S.P. 1,747,579, 18.2.30. Appl., 16.11.25).—To hot sulphuric acid (d 1.06—1.07) is added crushed phosphate [of lime] (e.g., calcined, steamed bone), and, after stirring for several hours and adding milk of lime if desired, the precipitate of calcium sulphate and dicalcium phosphate is collected and calcined. The product (consisting of interlaced crystals) preferably contains about 15% P_2O_5 .

S. K. TWEEDY.

Bleaching or decolorising of clay or other

minerals. T. W. PARKER, Assr. to DARTMOOR CHINA CLAY CO., LTD. (U.S.P. 1,777,446, 7.10.30. Appl., 25.2.28. U.K., 28.2.27).—See B.P. 279,694; B., 1928, 15.

Manufacture of non-splintering laminated glass sheets, other compound inter-adherent laminated sheets, and the like. W. C. DOUGAN (B.P. 334,858, 6.6.29).

Manufacture of pottery ware. W. J. MILLER (B.P. 310,961, 22.4.29. U.S., 5.5.28).

IX.—BUILDING MATERIALS.

Fundamental synthesis of calcium aluminates and their hydration. VII. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 315—318 B).—Heating and compressive strength tests have been carried out in continuation of previous work (A., 1930, 436).

C. W. GIBBY.

PATENTS.

Manufacture of cementitious material of cellular structure. G. B. HINTON (U.S.P. 1,756,789, 29.4.30. Appl., 24.10.27).—A mixture of cement pulp and a frothing agent is aerated and agitated to form a froth of small uniform bubbles coated with cement (cf. U.S.P. 1,657,716; B., 1928, 216); bubbles of larger size are then incorporated in the froth so that the product contains bubbles of air coated with a film of cement having smaller bubbles of air embedded therein.

S. K. TWEEDY.

Seasoning of wood [prior to impregnation]. F. S. SHINN (U.S.P. 1,763,070, 10.6.30. Appl., 15.10.27).—The wood, contained in a retort, is successively treated with steam under raised pressure, e.g., at 20 lb./in.², treated with compressed air at, e.g., 80 lb./in.², and subjected to a high vacuum. The first and second treatments may be reversed and the various processes repeated if desired.

L. A. COLES.

Preservation of wood. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,202, 28.5.29).—Wood is impregnated with a hydroxyalkylamine salt of a nitrated phenol, e.g., mono- or di- β -hydroxyethylaniline, hydroxy-ethylamine or -propylamine, etc. salts of 2:4-dinitrophenol or 3:5-dinitro-*o*-cresol. The salts may be applied together with fluorides, hexamethylenetetramine, etc.

C. HOLLINS.

Production of [heat- and sound-insulating] organic material. E. C. BAYER (U.S.P. 1,777,247, 30.9.30. Appl., 31.1.27. Denm., 9.2.26).—See B.P. 265,968; B., 1928, 299.

Cold bituminous paving composition. O. H. BERGER, Assr. to W. P. McDONALD CONSTRUCTION CO. (U.S.P. 1,776,379, 23.9.30. Appl., 13.7.27).—See B.P. 293,830; B., 1928, 750.

[Concrete] mixing machine. P. P. YONE, Assr. to KOEHRING CO. (U.S.P. 1,760,101, 27.5.30. Appl., 9.4.27).

Fireproof or soundproof floors or ceilings. RHEOCRETE PUMICE STONE SLAB CO., LTD., and D. WHITAKER (B.P. 334,914—5, 12.6.29).

Drying of timber (U.S.P. 1,760,444).—See I.

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

High-frequency steel furnaces. D. F. CAMPBELL (Iron and Steel Inst., Sept., 1930. Advance copy. 10 pp.).—At present the largest furnaces of the Ajax-Northrup type are of 20–25 cwt. capacity per heat, giving an output of 20 tons of steel per day; there appears no technical difficulty in constructing larger units of 3–5 tons, or even 25 tons, though the latter might not be economically practicable. The rapidity of melting and absence of contamination are discussed with reference to the higher-melting alloys, and costs have been reduced by the greater efficiency of motor-generator sets. C. A. KING.

Open-hearth furnace steelworks. Comparison of British and Continental installations and practice. H. C. WOOD (Iron and Steel Inst., Sept., 1930. Advance copy. 32 pp.).—Production of steel in Great Britain is almost entirely due to the Siemens-Martin process, whilst on the Continent this process has a strong competitor in the basic Bessemer converter, which results in every incentive to improve practice to be applied. The outputs in Continental practice are generally in advance of those obtaining in Great Britain, and great importance has been placed on large tonnage outputs per unit at low fuel consumption, low repair and labour costs, and the general ease of administrative and scientific control. Modern mechanical equipment is almost universally employed, and every encouragement is offered to the introduction of improvements in design or method. C. A. KING.

What reasons compelled the Prague Ironworks Company to introduce thin-walled blast-furnaces? J. SARE (Iron and Steel Inst., Sept., 1930. Advance copy. 33 pp.).—The theory of blast-furnace working is discussed and in connexion with the irregular working and severe corrosion of the walls it was observed that places where the lining had been melted off to only a few cm. thick would last well for a comparatively long time. Five furnaces of the thin-walled type have been erected on practically the original lines, and have been eminently successful, as the lining has a long life, troubles in operation have been decreased, and the consumption of coke has been reduced contrary to general conceptions. The original thickness of the lining of the latest furnace was 250 mm., and after operating for 18 months the lining was 180–200 mm. thick in the middle of the shaft and 120–140 mm. in the belly. It was expected that after attaining this equilibrium this thickness would probably remain for a long period undergoing merely mechanical wear. The only disadvantage is the increased consumption of cooling water. Construction costs are not higher than those of the older type, as the decreased firebrick covers the extra cost of armouring the furnace. C. A. KING.

Stock distribution and gas-solid contact in the blast furnace. C. C. FURNAS and T. L. JOSEPH (U.S. Bur. Mines, Tech. Paper 476, 1930, 73 pp.).—The relative inefficiency of the blast furnace is due in part to poor contact between the gas stream and the ore, and this in turn depends largely on the difference in size of the

material in different parts of the furnace. Size segregation in a furnace is due to the greater ore-coke ratio at the centre caused by the greater angle of repose of the ore. The average particle size at the centre may be decreased by reducing the time taken to deposit the materials on the bed, *e.g.*, by keeping the stock line close to the bed and by alteration in the bell discharge. Conversely, the size at the centre is increased by reversed filling, alternating skips of ore and coke, and by decreasing the bell clearance. Material finer than 14-mesh is potential flue dust, and as about 25% of this is lost it would be more economical to separate the fine portions and sinter it before introduction into the blast furnace. It is also desirable to crush all material to pass 2-in. mesh. At one plant about 20% of fuel has been saved by crushing and grading the charge into three different sizes, and charging each size separately. C. A. KING.

Desulphurisation [of pig iron] in the basic open-hearth furnace. K. KÖHLER (Stahl u. Eisen, 1930, 50, 1257–1264).—The effect of lime and manganese in the slag on the desulphurisation of iron in the basic open-hearth furnace has been studied. The sulphur content of the finished steel is inversely proportional and that of the slag directly proportional to the lime content of the slag. The rate of removal of the sulphur is also a linear function of the lime content of the slag between 25 and 40% CaO. A further increase in lime increases the viscosity of the slag, and therefore reduces its reactivity. No definite relation between the manganese content of the slag and its efficiency as a scavenger for sulphur could be established. Slags with a low lime and a high manganese content have no desulphurising action, and a deficiency of manganese in no way impairs the desulphurising action of the lime. Addition of a large excess of manganese does, however, appear to have some accelerating action on the removal of sulphur from the metal bath. A. R. POWELL.

Influence of nickel and of chromium on the properties of cast iron. L. THIERY (Compt. rend., 1930, 191, 47–49).—As regards its influence on the transition temperature of cast iron, replacement of silicon by nickel yields a fine graphite more highly disseminated throughout the metal; nickel and chromium when simultaneously present give rise to an even finer structure. The cementite normally present in cast iron is replaced by pearlite as the nickel percentage increases, and by ferrite when the nickel content is increased to 1.40–2.46%. Nickel up to about 2% increases the tensile strength, extension, and elasticity, but diminishes the hardness (Brinell), whilst the subsequent addition of chromium increases the hardness and decreases considerably the extension and elasticity. H. F. GILLBE.

Nitrogen in technical iron. III. Precipitation of nitrogen and carbon from α -iron as an example of the decomposition of a doubly supersaturated solid solution. W. KÖSTER (Arch. Eisenhüttenw., 1930–1, 4, 145–150; Stahl u. Eisen, 1930, 50, 1401–1402; cf. B., 1930, 715).—The effect of ageing on the magnetic properties of mild steels containing nitrogen and quenched from just below the A1 point has been

studied in an attempt to explain the mechanism of the decomposition of a doubly supersaturated solid solution of the duralumin type. On ageing at 20°, only the effect of carbide precipitation on the magnetic properties can be detected; at 110–150°, however, the effect of nitride separation preponderates, but again above 150° the action of carbide precipitation is more marked. Measurements of the change of coercivity during ageing show that the presence of carbide in supersaturated solid solution retards the separation of nitride therefrom, especially at 110°. For any constant carbon content of the solid solution there is a linear relation between the total nitrogen content of the steel and the increase in coercivity on ageing. By plotting these curves for steels with varying nitrogen and carbon contents the effect of carbon on the amount of nitrogen which can be retained in solid solution has been determined; this varies from 0.002% N with 0.01% C to 0.02% N with 0.035% C. These results indicate that the isothermal precipitation of a constituent from a solid solution may be greatly influenced by the presence of a second constituent, and that in the case of steel the separation of nitrogen from solid solution at 100° comes to an end, independent of the total nitrogen content, when the amount still in solid solution reaches a definite value dependent on the carbon content of the solid solution.

A. R. POWELL.

Effect of contamination by nitrogen on the structure of electric welds. L. W. SCHUSTER (Iron and Steel Inst., Sept., 1930. Advance copy. 30 pp.).—It has been suggested from time to time that the needles found in welds are caused by oxygen, but in all the welds made with a Swedish iron electrode and also an electrode from which gases had been removed by heating in hydrogen, no needles were evident unless an appreciable amount of nitrogen was present. Welds made in an atmosphere of carbon dioxide were also free from needles, but in every weld made in nitrogen needles occurred either before or after heat-treatment in an atmosphere free from oxygen. In all cases in which needles have been formed as a result of a normalising treatment the original structure approached that known as a Widmanstätten structure, from which the needle structure develops. Freedom from needles by no means implies an absence of nitrogen, as the conditions may prevent nitride needles from coming out of solution, or a high nitrogen content may form braunite. C. A. KING.

Copper steel for dynamo and transformer sheets. A. KUSSMANN, B. SCHARNOV, and V. S. MESSKIN (Stahl u. Eisen, 1930, 50, 1194–1197).—Addition of up to 0.7% Cu to steel with 1.5 and 4% Si has practically no effect on the magnetic properties, but a larger proportion of copper has a deleterious effect. Less than 0.7% Cu improves the hardness and tensile strength and the resistance to corrosion of these steels, and is therefore a desirable addition.

A. R. POWELL.

Determination of chromium in special steels. P. KLINGER (Arch. Eisenhüttenw., 1930–1, 4, 7–15; Stahl u. Eisen, 1930, 50, 1166–1167).—The chromium content of 12 special steels with 1.5–12% Cr has been determined by nine different methods. Methods involv-

ing alkali fusions give the best results when the chromium is determined iodometrically, and of those based on dissolution of the steel in acids followed by oxidation to chromic acid the persulphate-silver nitrate procedure is the most satisfactory. The effect of other metals on the results given by the various procedures is briefly discussed.

A. R. POWELL.

Determination of manganese in cobalt steels. P. KRÜGER (Z. angew. Chem., 1930, 43, 687).—The method previously described (cf. Hallbauer and Krüger, B., 1927, 447) is modified to prevent the interference of organic substances derived from the carbon in the steel. The filtrate from the cobaltinitrite is boiled with a slight excess of nitric acid to destroy nitrites, neutralised with zinc oxide, filtered, and titrated directly with permanganate by Volhard's method. A. R. POWELL.

Diffusion of aluminium into iron. N. W. AGEEV and O. I. VHER (Inst. Metals, Sept., 1930. Advance copy. 13 pp.).—The reaction of iron with molten aluminium takes place in two stages: in the first of these the iron dissolves in the liquid until the solution is in equilibrium with the solid phase at a given temperature; in the second the alloy thus formed diffuses into the iron, forming the δ -solid solution of aluminium in iron and at temperatures above 1100° the ϵ -phase also. The rate of diffusion increases with the temperature according to Weiss' equation, but falls rapidly with the time of contact. These results are in agreement with the accepted equilibrium diagram of the system.

A. R. POWELL.

Gravimetric determination of vanadium in high-speed steel. S. CINBERG (J. Russ. Met. Soc., 1926, 173–176).—The steel is digested with hot 17% sulphuric acid, the residue (tungsten, vanadium, and carbides of iron and chromium) is collected and boiled with nitric acid (1:2) until all the tungsten is converted into trioxide. The liquid is evaporated to small volume and treated with alkali hydroxide, the residual chromium being reduced with alcohol and precipitated with ammonia. After removal of ammonia by boiling, the liquid is treated with a slightly acid solution of mercurous nitrate, the orange-yellow double salt which is precipitated being ignited in platinum to vanadium pentoxide. In steels containing no chromium, but 1.5–4.5% V, the vanadium can be determined by digesting with dilute sulphuric acid, igniting the insoluble residue in platinum, and weighing as V_2O_5 .

CHEMICAL ABSTRACTS.

Effect of titanium on the transformation points of steels. A. MICHEL and P. BÉNAZET (Rev. Mét., 1930, 27, 326–333).—Addition of titanium to mild steel raises the temperature of the A3 transformation and lowers that of the A4, so that it appears that the γ -field of the iron-titanium system is bounded by a closed curve extending outwards to about 1.5% Ti. Thus the system is somewhat similar to the iron-chromium system. In the iron-chromium alloy with 13% Cr addition of only 0.6% Ti is sufficient to suppress the $\alpha \rightarrow \gamma$ transformation.

A. R. POWELL.

Secondary tempering of over-tempered steels and stability of austenite. P. CHEVENARD and A. PORTEVIN (Compt. rend., 1930, 191, 523–525).—The

inferences that under conditions (*i.e.*, such that no point of transformation occurs) for over-tempering a hyper-eutectoid steel there should, as the percentage of carbon decreases, successively appear the points Ar₂ and Ar₁, and further, that between these points austenite should be specially stable, have been verified, and a case of secondary tempering was examined. A steel (1.5% C, 2% Cr) brought to the austenitic condition by over-tempering at 1180° in water was examined in the way previously described (*cf.* B., 1930, 950). It was heated to varying temperatures, θ_r , for 7 hrs., and the dilatation, hardness, and electrical and magnetic properties were determined and plotted against θ_r . The results for values of θ_r below 330° and above 500° are quite similar to those obtained (*loc. cit.*) for the corresponding manganese steel; but in the interval an entirely different phenomenon, a secondary tempering, is observed. This is discussed with the aid of the (amplitude of anomalies)/ θ_r curves (*cf.* B., 1921, 698 A), and the results are shown to be consistent with the following changes: with $\theta_r = 350^\circ$ austenite begins to change into martensite (Ar₂), the amount of cementite formed increasing as θ_r increases and the remaining austenite becoming more and more decarbonised. At 430° Ar₁ appears with formation of troostite. The double maximum of the hardness- θ_r curve indicates two causes of hardening: (a) production of the aggregate $\alpha +$ cementite, and (b) production of secondary martensite.

C. A. SILBERRAD.

Thermal hardening of grey castings. L. GUILLET, J. GALIBOURG, and M. BALLAY (*Compt. rend.*, 1930, 191, 538—541).—Austenitic grey castings acquire a martensitic structure as a result, *e.g.*, of prolonged heating at 400—600°, or of cooling in liquid air, and the change is accompanied by an increase in hardness. It is more usual in practice, however, to produce this effect by means of martensitic tempering followed sometimes by recovery (*cf.* B., 1928, 607). Data are given for the effects of varying temperature and conditions of recovery on the hardnesses of the castings after tempering in oil (700—950°), in a current of air (700—950°), and in still air (900°). The transformation points were determined dilatometrically. Simple tempering without recovery gave fragile castings with low resistance to shearing, bending, and compression. Recovery at 300—350°, though not greatly reducing the hardness, increased the above resistance by 20—25%.

J. GRANT.

Hardening of some metals [steel and monel metal] by cold-rolling. E. GREULICH (*Stahl u. Eisen*, 1930, 50, 1397—1401).—The hardness, yield point, and elongation of carbon and alloy steels begin to change after only a 1% reduction in thickness by cold-rolling, whereas the ultimate strength and reduction in area in the tensile test first show signs of a change after a 6% reduction. The hardness, yield point, and ultimate strength increase with the reduction in thickness by cold-rolling according to the logarithmic equation, $y = a + b \cdot \log \phi$, where a and b are constants and ϕ is the percentage reduction. The constant, a , depends on the original tensile strength and hardness, whereas b is characteristic of the capacity of the material for hardening by cold-work. For plain carbon steels,

5% nickel steel, and rustless chromium steels, $b = (9.5 \text{ kg./mm.}^2)/\phi$ and is independent of the carbon content, provided that the structure is pearlitic: austenitic steels and monel metal harden more rapidly, however.

A. R. POWELL.

Problem of ageing phenomena on the basis of recent research on iron alloys. W. KÖSTER (*Z. Metallk.*, 1930, 22, 289—296).—A summarised account is given of recent work of the author and others on the age-hardening of alloys of iron with carbon, nitrogen, and copper, especially of iron supersaturated with both carbon and nitrogen, and the bearing of the results on modern theories of age-hardening is discussed.

A. R. POWELL.

Heterogeneity of an ingot made by the Harmet process. A. KŘÍŽ (*Iron and Steel Inst.*, Sept., 1930. Advance copy. 16 pp.).—Results of a complete macro-graphic and chemical examination of an ingot pressed by the Harmet method are given. The typical V-segregations formed in the central zone of ingots cast in the usual manner were not formed, the funnel-like contraction of the residual molten steel being counteracted by the artificial compression. A thick horizontal segregation under the head, evidently due to the compression, is typical in ingots cast by this method. C. A. KING.

Permanence of dimensions [of steel] under stress at elevated temperatures. W. H. HATFIELD (*Iron and Steel Inst.*, Sept., 1930. Advance copy. 18 pp.).—With the view of bringing the comparative permanence of dimensions of steel within the scope of a test practicable in point of time, the method adopted consists in determining, by static loading, the stress within which, at the given temperature, stability of dimensions is attained within 24 hrs. for a further period of 48 hrs. with an extension not exceeding the elastic deformation of 0.5% on the gauge length, and with limitation of measurement of the order of 0.000001 in. per in. per hr. This value is termed the "time yield," and a single test for a given temperature is obtainable in 3 days.

C. A. KING.

Evaluation of the quality of railway rails. A. N. MITINSKY (*Rev. Mét.*, 1930, 27, 370—380).—Creep of the metal on the upper surface of a rail is due to the steel having too low an elastic limit, which is usually caused by rolling at too high a temperature. A sorbitic structure is the most resistant to wear and surface crushing, but a troostite structure is almost as satisfactory, especially as regards resistance to abrasion. Good rails may be made by rolling at a relatively low temperature, or by rolling at a high temperature, cooling, reheating at 700°, cooling in a current of cold air to below 600°, and then allowing the rails to cool normally in still air.

A. R. POWELL.

Chemical methods of testing zinc coatings on sheet and wire. E. H. SCHULZ (*Stahl u. Eisen*, 1930, 50, 1017—1022).—A critical review of the various methods which have been proposed for determining the regularity of zinc coatings on iron and steel, the amount of zinc per unit area, and the resistance of zinc coatings to corrosion. Methods of testing for porosity and for differentiating between hot-galvanised and electrolytic deposits are also discussed.

A. R. POWELL.

Determination of manganese in cobalt steels.

J. KASSLER (Chem.-Ztg., 1930, 54, 733—734).—The steel (2.5 g.) is dissolved in 30 c.c. of nitric acid (d 1.2), the solution diluted, and the iron precipitated with zinc oxide. An aliquot part of the solution is filtered, treated with 3 g. of ammonium chloride, and boiled for 2 min. with 1 g. of ammonium persulphate. The precipitated manganic hydroxide, which contains only traces of cobalt, is redissolved in dilute nitric or sulphuric acid with the aid of a little hydrogen peroxide and oxidised to permanganic acid by the persulphate-silver nitrate method, and the analysis is finished in the usual way by titration with arsenite. A. R. POWELL.

Principles governing the testing of steel for its resistance to corrosion. E. H. SCHULZ (Stahl u. Eisen, 1930, 50, 1266—1269).—Standardised procedures and apparatus for making the usual accelerated and time corrosion tests on steels are briefly described. A. R. POWELL.

Action of the common acids on various kinds of non-rusting steels.

M. SAUVAGEOT and (MLLE.) L. LAUPRÈTE (Rev. Mét., 1930, 27, 362—367).—The rates of dissolution in sulphuric, hydrochloric, and nitric acids of varying concentration of a 13% chromium steel, an austenitic nickel-chromium steel, an austenitic nickel-chromium-molybdenum steel, and a high-nickel steel have been determined at 15° and at 80°. The three steels containing chromium were all practically unattacked by nitric acid of any concentration at any temperature, but the nickel steel rapidly dissolved in hot 1—25% nitric acid and slowly in cold acid of the same concentration, although acid of a higher concentration than 30% was without action at any temperature. The nickel steel was by far the most resistant to the action of hydrochloric acid, but all the samples were badly corroded by hot 20% acid. Sulphuric acid had practically no action on the nickel steel whatever the temperature (up to 100°) or concentration; the other steels showed a maximum rate of dissolution in 40—45% acid. A. R. POWELL.

Attack of insects on metals. O. BAUER and O. VOLLENBRUCK (Z. Metallk., 1930, 22, 230—233).—Perforations in a lead water-pipe covered with felt were traced to the gnawing action of the beetles, *Dermestes peruvianus* and *D. larvatus*. Experiments with glass test tubes closed with thin sheet lead, tin, aluminium, zinc, and brass showed that the beetles could penetrate the two first-named metals, but were unable to make even the slightest impression on the others. A. R. POWELL.

Reactions in blowing of cupriferous nickel matt.

V. TAFEL and F. KLEWETA (Metall u. Erz, 1930, 27, 85—88; Chem. Zentr., 1930, i, 2618).—The reaction $\text{Cu}_2\text{S} + 2\text{NiO} = 2\text{Cu} + 2\text{Ni} + \text{SO}_2$ takes place above 1300°. When formed in the converter, nickel oxide may react more readily. A. A. ELDRIDGE.

New method of preparing copper wire-bars.

M. TAMA (Z. Metallk., 1930, 22, 207—209).—Direct melting of carefully washed copper cathodes in an Ajax-Wyatt induction furnace followed by casting into vertical moulds produces excellent copper ingots for drawing into wire. Owing to the neutral atmosphere

of the furnace, the oxygen content of the bars is extremely low and the other impurities should not exceed 0.002%. Wire 3.56 mm. in diam. drawn from these bars has a conductivity of 58 m./ohm-mm.², a tensile strength of 25 kg./mm.², and an elongation of over 35%.

A. R. POWELL.

Tensile properties of electrical conductor wires at low temperatures. F. PESTER (Z. Metallk., 1930, 22, 261—263).—The tensile strength of copper wire increases with fall of temperature from 20° to —60° by 9%, that of bronze wire by 6.3—8.9%, that of aluminium wire by 10.5%, and that of aldreys wire by 10.2%. The elongation in all cases increases considerably on cooling, but the bending test for copper, bronze, and aldreys shows minimum values at —20°.

A. R. POWELL.

Properties of extruded rods of electrolytic copper, smelter copper, arsenical copper, and 4% aluminium bronze, and their variation with the extruding conditions.

P. SIEBE and G. ELSNER (Z. Metallk., 1930, 22, 203—206, 238—245).—The tensile properties, resistance to impact shock, and grain size of extruded rods of electrolytic copper (99.86% Cu, 0.05% O), refined copper (99.66% Cu, 0.06% O), arsenical copper (99.49% Cu, 0.39% As, 0.09% P), and aluminium bronze (4.04% Al) have been determined immediately after extrusion at 750—850° and at 990—1020°, after subsequent drawing to reduce the diameter by 15%, and after annealing for various periods at 650—950°. All of the copper specimens had a higher tensile strength but lower ductility when extruded at 800° than when extruded at 1000°, but the differences were somewhat reduced by subsequent drawing; of the three varieties of copper, that containing arsenic had the highest strength and ductility in all cases. The tensile strength of the aluminium bronze remained the same after extrusion at 800° or at 1000°, but the higher temperature increased the elongation; in this case the drawn metal had better tensile properties at the lower extrusion temperature. Photomicrographs and a tabulated description of the structural changes which occur during extrusion, drawing, and annealing are included. A. R. POWELL.

Manufacture application of "Y" and "R.R." alloys. W. C. DEVEREUX (Nickel Bull., 1930, 3, 286—293).—Details are given of the methods of preparing copper and nickel hardener alloys for use in making "Y" and "R.R." aluminium alloys; then follows an account of the methods of melting, casting, heat-treating, and working these alloys and a brief outline of their properties. A. R. POWELL.

Electric melting of aluminium. E. F. RUSS (Z. Metallk., 1930, 22, 273—276).—Crucible, kettle, and hearth furnaces of the resistance type for the melting of aluminium are described and illustrated. They have the advantage over gas-, oil-, or coke-fired furnaces of a stationary atmosphere which reduces the loss by oxidation and prevents gas adsorption to a large extent. By means of automatic temperature regulators a steady temperature gradient may be maintained and all danger of overheating the metal is avoided. A tilting-hearth furnace with a capacity of 1500—2000 kg. will melt

8—10 tons of aluminium in 24 hrs. with a current consumption of 250 kw./hr. at 220 volts, equivalent to 600—700 kw.-hrs. per ton. The loss by oxidation should not exceed 1%. A. R. POWELL.

Remelted aluminium and aluminium alloys and their uses. R. STERNER-RAINER (Z. Metallk., 1930, 22, 269—272).—In remelting scrap aluminium alloys the charge should be made up to contain 5—7% Zn and 3—4% Cu for sand-castings, 2—3.5% Zn and 6—8% Cu for chill-castings, and not more than 2.5% Zn and 6—8% Cu for hollow castings. In no case should the impurities exceed 1.2% Fe + Si, 0.3% Pb, 0.3% Sn, 0.2% Ni, 0.1% Mn, and 0.1% Mg; the minimum aluminium content in all cases is 87%. The alloys are capable of improvement by heat treatment and are rather more easily worked on the lathe than are similar alloys made from virgin metal, as the turnings are shorter and there is less tendency for the tool to tear the metal surface. A. R. POWELL.

Improvement of an aluminium alloy as shown by X-rays. VON GÖLER and G. SACHS (Mitt. Materialprüf., 1930, 117; cf. A., 1929, 743).—X-Ray examination of a 5% Cu aluminium alloy after quenching from 525° and ageing at various temperatures shows that no change in the structure takes place below 100°; at 150° about 20% of the copper is precipitated out of solid solution in 24 hrs., at 200° 40% is precipitated in 30 min., and at 250° the whole of the copper separates within a short time. On the other hand the mechanical properties improve with ageing at below 100°; this improvement is characterised by an increase in the yield point and tensile strength without a corresponding reduction in the ductility. Precipitation of the copper is accompanied by a further increase in tensile strength but a reduction in the ductility. With single-crystal specimens precipitation of the copper causes considerable variations in the mechanical behaviour, so that the crystal behaves like fine-grained metal, although it has an X-ray structure similar to that of a single crystal, but single crystals aged at the ordinary temperature behave in the tensile test in a normal way. A. R. POWELL.

Gas removal and grain refinement of aluminium alloys. W. ROSENHAIN, J. D. GROGAN, and T. H. SCHOFIELD (Inst. Metals, Sept., 1930. Advance copy. 14 pp.).—By passing through molten aluminium or its alloys the vapour of carbon, silicon, titanium, or tin tetrachloride, aluminium chloride, ferric chloride, or tetrachloroethane, gas occlusions are almost completely removed from the metal. Ferric and tin chlorides cause the metal to be contaminated with the corresponding metals, as also does, to a very small extent, titanium tetrachloride, but in this case the titanium acts beneficially in causing the development of a very fine grain which persists after several re-meltings. This grain refinement can also be produced by adding small quantities of titanium-aluminium alloy to the metal, but this treatment does not remove dissolved gases. The chloride treatment of modified silicon-aluminium alloys results in their complete reversion to the normal state. Treatment of "Y" alloy with 2 c.c. of titanium tetrachloride per lb. provides a method of producing high-quality castings in sand or chill moulds and appre-

ciably improves the forging, rolling, and drawing properties of the metal. A. R. POWELL.

Eutectoid transformation of aluminium bronze. II. Effect of quenching velocity. I. OBINATA (Mem. Ryojun Coll. Eng., 1930, 3, 87—94; cf. B., 1930, 105).—Quenching in water from above 500° of the aluminium bronze with 12.5% Al does not completely suppress the β - β' transformation, which can still be detected by an evolution of heat as the alloy cools from 500° to 400°. The acicular structure obtained by quenching in water is therefore regarded as a heterogeneous mixture of β and β' . Quenching in toluene at -95° produces a homogeneous structure which is assumed to be that of pure β , and the apparently homogeneous structure produced by cooling at a somewhat slower rate than obtains in water-quenching is considered to be pure β' , which is much harder than β . A. R. POWELL.

Nickel-aluminium bronze. T. ISHIKAWA (J. Soc. Mech. Eng. Japan, 1928, 31, 215—233).—The hardness of forged bars of nickel (10%)—aluminium (2%) bronze rapidly increases by tempering at 600° after quenching in water or liquid bath below 400°.

CHEMICAL ABSTRACTS.

Hardening of beryllium-aluminium and magnesium-aluminium alloys. M. HAAS and D. UNO (Z. Metallk., 1930, 22, 277—279; cf. B., 1930, 716).—Dilatometric tests and micrographical examination of beryllium-aluminium alloys have confirmed the composition of the eutectic as 1.4% Be, 98.6% Al, m.p. 644°. The solid solubility of beryllium in aluminium increases from 0.2% at 20° to 0.8% at 640°. Schmid and Spitaler's equilibrium diagram of the aluminium-magnesium system has been confirmed by dilatometric analysis.

A. R. POWELL.

Notched-bar impact strength of some aluminium alloys, especially at low temperatures. W. A. GÜLDNER (Z. Metallk., 1930, 22, 257—260).—The notched-bar impact strength of aluminium alloys is of the order of 1—4 m.-kg./cm.², its actual value in any specific case being a linear function of the speed of the blow. The slope of the straight line showing this relation is a definite property of the metal and is not affected by heat treatment although it varies with the mechanical treatment. An increase in the weight of the hammer has no influence on the impact strength. A. R. POWELL.

Corrosion of light and ultra-light metals and alloys. R. CAZAUD. (A) Critical studies of corrosion tests. E. HERZOG and G. CHAUDRON. (B) Reduction of mechanical properties by corrosion. COURNOT and MOLNAR. (C) Development of the zone of action of galvanic couples. COURNOT and MOLNAR. (D) Method of [testing corrosion by] the E.M.F. of dissolution. AUBERT and PROT. (E) Relative tests on the rise of temperature of activated aluminium alloys. QUILLARD. (F) Effect of arsenic on the corrosion resistance of duralumin in sea-water. R. CAZAUD. (G) Use of fatty substances in the protection of aluminium against chemical action. A. TRILLAT. (H) Application of the "phenol method" to the protection of light aluminium alloys. AUBERT and A. PIGNOT. (I) Resistance to sea-water of aluminium sheet with various protective coatings. QUILLARD.

(J) Resistance to sea-water of special light aluminium alloys. LEGENDRE. (K) Practical corrosion tests on caissons. LECŒUVRE (Rev. Mét., 1930, 27, 285—289, 290, 291—296, 296—299, 299—300, 337—341, 341—343, 343—344, 344—347, 347—350, 350—351).—(A) Experiments with various light-metal alloys in numerous corrosive liquids show that the loss in weight is not a reliable method of measuring the resistance to corrosion; more reliable comparative figures are obtained by determining the diminution in the percentage elongation in the tensile test. (B) The salt-spray test is a more rapid method of determining the resistance of aluminium alloys to sea-water than is the total immersion test. In the spray test the reductions in tensile strength and elastic limit do not appear to be proportional to the progress of corrosion. (C) Cadmium and zinc protect aluminium from corrosion by sea-water, whereas iron, copper, and nickel accelerate the corrosion. For aeronautical purposes aluminium alloys may be rivetted with cadmium-plated duralumin rivets, which act as a protection against corrosion, causing the surrounding metal to become covered with a thin protective film of alumina. Aluminium and duralumin rivets act as protective agents in magnesium alloy structures. (D) The effect of inclusions in duralumin in promoting corrosion by the formation of local couples is discussed; incorrect thermal treatment may result in accelerated corrosion, owing to the segregation of more electropositive constituents. (E) The rate at which the temperature of the metal rises after dipping it in mercuric chloride solution and exposing it to the air is a measure of the resistance of an aluminium alloy to corrosion. This test shows that addition of 1—1.5% Mn to aluminium greatly reduces its rate of corrosion, but that 2% Mg increases the rate considerably. (F) Arsenic may be added to duralumin in the form of copper or manganese arsenides, but losses of arsenic by volatilisation occur during heat-treatment. In the microstructure arsenic appears as needles of arsenide which have little effect on the mechanical properties or resistance to corrosion. (G) Coating aluminium with various fatty substances affords efficient protection against corrosion; a water jet under 9—12 kg. pressure does not remove the greasy coating. (H) Aluminium alloys may be protected from corrosion by painting with a mixture of 30% of pitch, 5% of phenol, and 65% of light petroleum; two coats are required, the metal being allowed to dry for 48 hrs. after each application. A final application of aluminium paint is given to restore the original aluminium appearance of the surface. (I) Comparative tests on the protective action of films produced by treatment of aluminium with sodium silicate and of films of paraffin and varnish are recorded. (J) The effect of protective plates of zinc attached to structures in which duralumin is in contact with another metal or alloy under sea-water is illustrated, and the behaviour in sea-water of certain special aluminium alloys of unstated composition is briefly described. (K) Aluminium structures may be protected from corrosion by covering them with a resistant varnish or with cadmium by the Schoop spray method or by electrolysis. Good protection is also afforded by applying bands of zinc by the Schoop method to various parts of the structure. A. R. POWELL.

Transformation points in metals [zinc, bismuth, thallium, and cobalt]. A. SCHULZE (Z. Metallk., 1930, 22, 194—197, 308—311).—The electrical resistance, thermoelectric power, and thermal expansion of the above four metals have been determined at temperatures up to the m.p. The curves for "spectroscopically pure" zinc show no points of inflexion, whereas those for zinc containing cadmium, lead, and iron show one or more such points corresponding with the m.p. of the eutectic of zinc and the impurity. It would thus appear that zinc undergoes no allotropic transformation, previous observations to the contrary being due to the presence of impurities. Similarly, pure bismuth undergoes no allotropic transformation between -190° and 270° , but the presence of impurities may cause inflexions in the curves of some of its physical properties. Thallium shows abrupt changes in the thermal expansion and electrical resistance curves at 225.2° on heating, due to the change $\alpha \rightarrow \beta$ -thallium; on cooling, the reverse change takes place a few degrees lower. β -Thallium has a lower resistance and greater coefficient of expansion than α -thallium. Cobalt undergoes an allotropic transformation on heating at 444° , but the reverse change is subject to considerable hysteresis effects the magnitude of which is enhanced by the presence of impurities. A second change occurs at 1128° , when the metal loses its magnetic properties.

A. R. POWELL.

Diffusion of zinc and lead into liquid tin. Kinetics of soldering. A. MERZ and E. BRENNECKE (Z. Metallk., 1930, 22, 185—189, 234—237).—The diffusion constants of zinc and lead in molten tin have been determined at 260° , 310° , and 370° by the Graham-Stefan method. The constant for zinc at 260° is $2.66 \text{ cm.}^2/\text{day}$ and the temperature coefficient $0.0119 \text{ cm.}^2/\text{day}/^{\circ}\text{C.}$ between 260° and 370° . For lead the diffusion constant is $1.23 \text{ cm.}^2/\text{day}$ at 260° and the temperature coefficient $0.0066 \text{ cm.}^2/\text{day}/^{\circ}\text{C.}$ Within the above temperature range the rate of diffusion in both cases is independent of the concentration of the metals. When copper is dipped into molten tin a layer of Cu_3Sn is formed below 420° and this layer has a retarding effect on the rate of dissolution of copper in liquid tin. The above results are discussed in the light of Nernst's theory of reaction velocity in heterogeneous systems and their bearing on the theory of the mechanism of soldering is indicated.

A. R. POWELL.

Recovery of fine gold and silver from old gold and silver alloys. E. ROSENBAUM (Chem.-Ztg., 1930, 54, 743—744).—The method of refining gold-silver alloys by inquartation and parting with nitric acid is described and a detailed account given of the usual ammonium chloride-sulphur dioxide method for recovering platinum and gold from solutions obtained by dissolving alloys of these metals in aqua regia.

A. R. POWELL.

Electrodeposition of gold alloys. G. GRUBE (Heraeus Festschr., 1930, 34—44; Chem. Zentr., 1930, i, 2475).—The electrodeposition of alloys of gold with copper, silver, and with both metals, is described. The concentration of the electrolyte must be kept constant and air excluded.

A. A. ELDRIDGE.

Titration of potassium cyanide, and of free cyanide in silver-plating solutions, by means of silver nitrate. E. B. SANIGAR (Trans. Amer. Electrochem. Soc., 1930, 58, 49—68).—The effects of certain impurities (especially carbonates) on the sharpness and accuracy of the end-point in the titration of 0.5*N*-potassium cyanide and of a silver-plating solution (20.5 g. Ag per litre, 0.4*N* free cyanide) with silver nitrate, using potassium iodide as indicator, have been investigated. The presence of potassium carbonate in the simple cyanide solution did not interfere with the titration unless its concentration was as high as 0.18*N*, when precipitate formation began to mask the end-point. In the argenticyanide solution, however, this limiting concentration of carbonate was only 0.03*N*. Addition of 0.1*N*-sodium hydroxide is without effect on the sharpness of the end-point or on precipitate formation. Addition of ammonia raises the limiting carbonate concentration considerably, but tends to cause high results unless the concentration of potassium iodide is increased. It is found, however, that precipitate formation due to carbonates can be prevented without affecting the accuracy of the end-point, merely by diluting the argenticyanide solution sufficiently. With an average plating solution containing carbonates, a 35–50-fold dilution is recommended before titration with 0.1*N*-silver nitrate. This method is preferred to that in which carbonates are removed by precipitation with barium or calcium salts before titration, since any excess of these salts leads to the formation of precipitates which mask the end-point, or if the solution is sufficiently diluted to avoid this, the titration values obtained are somewhat low. H. J. T. ELLINGHAM.

Scientific industrial control of electrolytic deposits, especially of nickel and chromium. M. BALLAY (Rev. Mét., 1930, 27, 316—325).—A review of modern scientific methods of controlling the p_H of nickel-plating baths and the current yields in nickel- and chromium-plating baths, and of determining the adherence and resistance to corrosion of electrolytic deposits. A. R. POWELL.

Electrodeposition of cobalt-nickel alloys. I. S. GLASSTONE and J. C. SPEAKMAN (Trans. Faraday Soc., 1930, 26, 565—574).—The composition of the alloys deposited from solutions of definite p_H between 3.2 and 5.2, buffered by addition of acetic acid as required, and containing nickel and cobalt sulphates at concentrations varying from 2 to 0.41 g.-equiv. per litre has been determined for current densities ranging from 0.0028 to 5–6 amp./dm.² The deposit always contains a relatively greater proportion of cobalt than the solution from which it is deposited, on account of the retarding influence of the cobalt ion on nickel. The composition of the alloy at low current density is not influenced by changes of current density, by dilution, or by stirring the electrolyte. At current densities above about 1 amp./dm.², the cobalt content of the deposit decreases with the current density, as a result of the decrease of the cobalt-ion concentration caused by the preferential deposition of that metal; the effect becomes more marked on dilution, but is slightly diminished by stirring.

With increase of the p_H of the electrolyte the composition of the initial deposit remains unchanged, but the effect of the impoverishment in cobalt ions becomes operative at lower current densities, and the current efficiency increases. Added anions or cations do not appear to influence the composition of the alloy deposited. The results obtained are compared with those found for the simultaneous deposition of iron and nickel.

H. F. GILLBE.

"Pendulum" hardness tests of commercially pure metals. D. A. N. SANDIFER (Inst. Metals, Sept., 1930. Advance copy. 29 pp.).—The time and scale hardnesses and work-hardening capacities of 23 commercially pure metals have been determined with the Herbert pendulum tester. Time work-hardening capacity is measured by plotting induced hardness against the number of passes of the pendulum ball and subtracting the original time hardness from the maximum induced hardness read from the curve. There is no definite relation between the time and scale hardnesses of pure metals. The time work-hardening capacity of soft metals is usually low; it increases as the time hardness increases, then decreases again, finally becoming negative for very hard metals, e.g., manganese. The relation between the scale hardness (*S*) and the scale work-hardening capacity (*SC*) is expressed by the equation: $(S-29)^2 + 66(SC-51.1) = 0$ for a pendulum length of 0.21 mm. The time hardness (*T*) is a linear function of Young's modulus; for pure metals $E = 8.1 \times 10^{10} T$ dynes/cm.² for a pendulum length of 0.1 mm. For all metals for which $T < 30$ the relation $TV^{1.87} = 818.5$ (where *V* is the at. vol.) holds good, the sole exception being antimony. The gradual increase in hardness produced by cold-working a pure metal is accompanied by a slow decrease in the work-hardening capacity.

A. R. POWELL.

Effects of two years' atmospheric exposure on the breaking load of hard-drawn non-ferrous wires. J. C. HUDSON (Inst. Metals, Sept., 1930. Advance copy. 14 pp.).—Samples of wire (diam. $\frac{1}{16}$ in.) were exposed to the London atmosphere for 2 years and their tensile strength was then compared with their original strength and with the strength of similar wires kept indoors for the same period. The wires subjected to atmospheric corrosion showed an average loss in strength of 2–3%, except in the case of 70:30 brass, for which the loss in strength was much greater. The absolute values for the corrosion deduced from these results are of the same order of magnitude as those obtained in more prolonged field tests. The poor behaviour of brass wire is attributed to secondary action of the brass on the copper corrosion products first formed, resulting in the redeposition of copper and dezincification of the outer brass layers. The corrosion products which adhere to nickel-copper alloys contain a much higher proportion of copper than that in the original alloy; this is due to selective dissolution of the nickel in the rain water and removal of the soluble nickel salts, and is especially marked when the atmosphere contains a high proportion of sulphur compounds. A. R. POWELL.

Brinell, Rockwell, and scleroscope hardness of non-ferrous metals. O. SCHWARZ (Z. Metallk., 1930,

22, 198—202).—Hardness measurements of annealed and hard-worked copper, brass, aluminium, and light aluminium alloys have been made with the Brinell, Rockwell, and scleroscope hardness testers. The relation between the Rockwell *B* hardness ($\frac{1}{16}$ -in. ball, 100-kg. load) and the Brinell hardness (*H*) (load in kg. = $10 \times [\text{diam. of ball}]^2$) is $H = 8300/(140 - B)$ for values of *H* between 50 and 180, and between the Rockwell *C* hardness (2.5-mm. ball, 62.5-kg. load) and the Brinell hardness $H = 2900/(100 - C)$. The Brinell hardness number is roughly 4.5 times the scleroscope number for non-ferrous metals (mean deviation $\pm 5\%$), but the scleroscope does not measure exactly the same property of a material as the Brinell and Rockwell machines; the scleroscope number is a function of the product of the elastic limit and elongation. A. R. POWELL.

Treatment of accumulated concentrates from the Oriental Consolidated Mining Co., Hokuchin, Korea. W. G. CLARKE and B. H. MOORE (Bull. Sch. Min. W. Australia, 1930, 5, 31—38).—Fine-grinding and cyanidation by agitation of the raw concentrates (4.5 dwt. Au per ton) was preferred to roasting and cyanidation. CHEMICAL ABSTRACTS.

Leaching of chalcocite. SULLIVAN. **Copper leaching.** KEYES.—See VII.

See also A., Oct., 1245, **System nickel-chromium** (SEKITO and MATSUNAGA). **System iron-tungsten** (TAKEDA). **System iron-nickel-cobalt** (KÜHLEWEIN). 1253, **Electrolysis of amalgams, fused silver-lead alloys, and bronzes** (KREMAN and others). 1258, **"Explosive" antimony** (VON STEINWEHR and SCHULZE; COHEN and COFFIN).

PATENTS.

Production of metals [iron] from ores. E. A. A. GRÖNWALL and H. J. H. NATHORST (B.P. 334,500, 26.4.29).—The ore is smelted in the usual way in a shaft furnace, but the gases issuing from the shaft are cooled for the removal of carbon dioxide by condensation and/or washing and the residual gases are mixed with a proportion of oxygen insufficient for their complete combustion and returned to the lower part of the furnace, preferably after carburetting the mixture by passing it through a mass of glowing coal. A. R. POWELL.

Low-temperature reduction of iron ores. E. D. NEWKIRK and A. J. BRIGGS (U.S.P. 1,760,078, 27.5.30. Appl., 3.10.27).—A mixture of finely-ground iron ore and coke is passed downwards through a cylindrical, rotating tube furnace, inclined slightly to the horizontal, and a stream of highly reducing gases is passed counter-current to the ore stream. At intervals along the tube a limited supply of air is admitted, so that the gases are fractionally and progressively burned as they pass towards the feed hopper. The continuous production of a good grade of sponge iron in one operation is thus ensured without external heating of the furnace. A. R. POWELL.

Extraction of useful elements [iron] from their ores by the aid of gases. B. HOFSETH (U.S.P. 1,759,456, 20.5.30. Appl., 25.8.27. Norw., 10.9.26).—Iron ore is treated, *in situ*, with hot gases to preheat a section to 350—500°, and currents of hydrogen and

chlorine from separate pipes are allowed to impinge on the heated zone, whereby ferric chloride volatilises and may be collected in suitable chambers through which the issuing gases are drawn directly from the ore body. The heat evolved in the reaction is sufficient to maintain it continuously as long as hydrogen and chlorine are supplied in sufficient quantity. A. R. POWELL.

Melting of metal or heating molten metal in an electrical furnace, and a furnace therefor. K. G. WENNERSTRÖM (B.P. 334,201, 28.5.29).—The metal is melted above a layer of hot slag, through which an electric current is passed. The slag is contained in a series of narrow heating chambers in the lower part of the furnace, and the electrodes conducting the current to these chambers are internally water-cooled. A. R. POWELL.

Furnace for casting iron and other difficultly fusible metals under pressure. E. PETERSSON (U.S.P. 1,758,463, 13.5.30. Appl., 5.11.26. Swed., 9.1.25).—The furnace is provided with a casting pipe which terminates in a nozzle situated in a hole in an upper side-wall of the furnace so that it is exposed to the heat of the molten metal. The mould is pressed against this nozzle by means of a self-adjustment device which ensures a vacuum-tight connexion with the mould. A. R. POWELL.

Casting of readily oxidisable metals. W. W. SMITH (U.S.P. 1,761,498, 3.6.30. Appl., 18.4.28).—The casting mould is made from a mixture containing 50—90% of pulverised carbon, 2—15% of powdered soapstone, and the remainder magnesium oxide, the mixture being bound with a light mineral oil. The surface of the mould is dusted with magnesium oxide. C. A. KING.

Ingot mould. E. J. KAUFFMAN, Assr. to VALLEY MOULD & IRON CORP. (U.S.P. 1,762,098, 3.6.30. Appl., 19.7.29).—In casting an ingot mould, a mixture of cast iron and at least 5% of mild steel is used. A mould having a gradation from steel at the inner face to iron at the outer surface is made by casting first the iron centrifugally and then the mild steel. C. A. KING.

[Production of] metal [cast iron]. C. F. LAUENSTEIN, Assr. to LINK-BELT CO. (U.S.P. 1,760,241, 27.5.30. Appl., 15.10.28).—Cast iron having a structure consisting of grains of ferrite and graphite surrounded by an intercrystalline bond of sorbite or pearlite is claimed. This structure is obtained by annealing white cast iron at 800°, quenching, and annealing at 500—650°. A. R. POWELL.

Manufacture of cast-iron grinding balls. F. KRUPP GRUSONWERK A.-G. (B.P. 319,350, 18.7.29. Ger., 22.9.28).—The cast iron used contains 0.3—1.8% Si, 2.7—3.8% C, and 0.3—1.2% Cr, the actual proportion of these constituents being varied according to the diameter of the ball, so that the sand-cast metal has a ledeburitic structure without an exceptionally hard skin. This is obtained if $D = 250 + 250Cr/3 - 50(Si + C)$, where *D* is the diam. in mm. and Cr, Si, and C are the percentages of the alloying elements. A. R. POWELL.

Iron refining. G. S. EVANS, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,759,347, 20.5.30. Appl., 18.2.28).—After melting down the metal in an air

furnace, the slag is removed and a soda ash refining agent is introduced into the firing end of the furnace, allowed to flood the surface of the metal, and thereafter removed from the opposite end. The original slag may be further fluxed by an addition of the same reagent.

C. A. KING.

Metal [case]-hardening process. G. A. FLECKENSTEIN, E. M. HANF, and M. L. WATERMAN, ASSRS. to SINGER MANUF. Co. (U.S.P. 1,759,690, 20.5.30. Appl., 23.11.28).—The parts of an article which are to be kept soft during any of the usual case-hardening processes are first plated with a coating of chromium 0.00006 in. thick.

A. R. POWELL.

Treatment of ferrous metal articles. M. C. BAKER and W. A. DINGMAN, ASSRS. to PARKER RUST PROOF Co. (U.S.P. 1,761,186, 3.6.30. Appl., 30.6.28).—The articles are pickled, washed, heated at 260°, quenched in 33% phosphoric acid solution to which, if desired, metal phosphates may be added, and dried at 160–230° without washing.

A. R. POWELL.

[Inhibitor for use in the pickling] treatment of iron or steel. K. B. THEWS and J. S. CARLITZ (U.S.P. 1,759,634, 20.5.30. Appl., 7.12.29).—Substances of the general formula $CX(NR_2)_2$, where X is selenium or tellurium, and R is an alkyl, aryl, or acyl group, are claimed.

A. R. POWELL.

Metallurgical process [for treatment of copper sulphide ores]. R. D. PIKE (U.S.P. 1,761,641, 3.6.30. Appl., 11.2.26).—The ore is leached with a hot solution of ferric chloride and the filtered liquor is treated with sponge iron to remove copper. The ferrous chloride solution thus obtained is treated with calcium chloride to reduce its sulphate content to a value below that corresponding with the solubility of calcium sulphate, filtered, and electrolysed to obtain pure iron. The tailings from the leaching vats are dried, heated to remove sulphur by distillation, roasted, and leached with the spent electrolyte from the iron cells before it is returned to the main leaching vats.

A. R. POWELL.

Smelting of copper. A. D. WILKINSON, ASSR. to CANANEA CONSOL. COPPER Co., S.A. (U.S.P. 1,759,078, 20.5.30. Appl., 26.3.26).—A mixture of 25% of flotation concentrates, 66–70% of crushed copper ore, and 9–5% of converter chips, the whole containing 9–10% of moisture, is banked at a steep angle in a reverberatory and smelted under conditions which result in the formation of a rich copper matte. The presence of water in the charge eliminates a large proportion of the sulphur, and oxidises most of the iron into the slag.

A. R. POWELL.

Reducing the quantity of sulphurous acid gas set free from copper glance during roasting. M. MIYAKE (B.P. 334,761, 18.10.29).—Copper sulphide ore is mixed with a small proportion of bone ash and red-earth prior to roasting.

A. R. POWELL.

[Nickel-iron-copper] alloy and the process of forming the same. J. J. OLSEN (U.S.P. 1,759,169, 20.5.30. Appl., 24.8.27).—An alloy suitable for use in welding or brazing is made by melting 140–150 lb. of copper, adding 4–5 oz. of 85% ferrosilicon, 3.5–7 lb. of carbon-free iron, and 1.75–3.5 lb. of nickel, stirring

under a sodium nickel borate flux, and casting in a reducing atmosphere at just above the m.p.

A. R. POWELL.

Manufacture of [silicon-copper-tungsten or -molybdenum] alloys. J. GRAY. From A. PACZ (B.P. 335,081, 7.10.29).—An alloy for the manufacture of electrical contact terminals comprises 50–95% Mo and/or W, 5–50% Cu, and 0.5–5% Si. Part of the copper may be replaced by silver and 0.5–2% Fe, Co, or Ni may be added. The alloy is produced by precipitating a mixture of alkali silicate, tungstate, or molybdate with an acid, reducing the oxides in hydrogen, heating the resulting powder with magnesium in an inert atmosphere, extracting the magnesia with acid, again reducing the metals in hydrogen, and heating the sponge so formed with molten copper or a copper-silver alloy until the latter soaks into the mass.

A. R. POWELL.

[Electric] furnace for producing aluminium. P. E. FRØLAND (U.S.P. 1,761,442, 3.6.30. Appl., 1.3.28. Norw., 12.3.27).—The bottom of an electric furnace is of tamped carbon in which is embedded a more solid bar of carbon, *e.g.*, an electrode, which extends outside the furnace to form the cathode connexion. As an auxiliary contact, bars of aluminium preferably are let into recesses in the underside of the bed and connected also to the current supply.

C. A. KING.

[Zinc]-aluminium alloy. T. S. FULLER and D. BASCH, ASSRS. to GEN. ELECTRIC Co. (U.S.P. 1,760,549, 27.5.30. Appl., 13.12.23).—The alloy contains at least 80% Al, more than 6% Zn, 0.2–2.5% Mg, and 1–3% Fe. It is annealed at 450–575°, quenched, and aged at 150° for 1–3 hrs. After this treatment an alloy of 80.75% Al, 6% Zn, 2% Fe, and 1.25% Mg has a hardness of 65 Brinell and a tensile strength of 30,000 lb./in.²

A. R. POWELL.

Aluminium alloys. METALLGES. A.-G. (B.P. 335,105, 7.11.29. Ger., 7.12.28).—Alloys of aluminium with 1–6% Cu, 2–14% Zn, up to 1% Mg, and up to 0.5% Si, with or without up to 1% Cd and Mn, are claimed. The preferred range of composition is 3–4% Cu, 8–10% Zn, 0.1–0.5% Mg, 0.05–0.1% Li, 0.4–0.8% Mn, and 0–1% Cd.

A. R. POWELL.

Casting light metals, especially aluminium and aluminium alloys. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 334,435, 29.10.29. Ger., 29.10.28).—A rectangular melting furnace is provided with a number of small delivery chambers arranged in a row on one side of the furnace and connected therewith by means of small channels. These chambers are closed at the top by means of a cooling supporting plate on which the chill moulds are situated and which is provided with supply tubes which project into the molten metal in the delivery chambers. In this way the metal may be drawn up into the mould without disturbance of the metal surface by connecting the upper part of the mould to a vacuum pump.

A. R. POWELL.

[Silicon]-aluminium alloys [for pistons]. K. SCHMIDT GES.M.B.H. (B.P. 334,777, 6.11.29. Ger., 15.8.29).—An aluminium alloy consisting of more than 70% Al with 15–25% Si, 0.2–3% Cr, 0.5–3% Mn, 0.5–3% Co, 0.5–3% Ni, 0.5–3% Sb, and 0.5–1% Ti and/or Mo is claimed.

A. R. POWELL.

Removal of carbonyl-forming metals or carbides thereof from soot. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,190, 27.5.29).—The material is heated at 400–500° in hydrogen, an inert gas, or a gas mixture containing a small proportion of oxygen, and then at 50–210° in a current of carbon monoxide under elevated pressure and in the presence of a small amount of sulphur as a catalyst. A. R. POWELL.

Metal [lead] foil. W. J. HAWKINS, Assr. to AMER. MACHINE & FOUNDRY Co. (U.S.P. 1,760,645, 27.5.30. Appl., 16.5.28).—Lead containing 0.25–0.5% Mg may be rolled to foil having a thickness of only 0.0005 in. A. R. POWELL.

Lead alloy for electric cable sheaths etc. STANDARD TELEPHONES & CABLES, LTD., Assees. of R. S. DEAN (B.P. 314,522, 31.5.29. U.S., 29.6.28).—An alloy of lead with 0.02–0.07% Ca quenched from 310–325° and aged at below 200° is claimed. [Stat. ref.]

A. R. POWELL.

Electrolytic recovery of tin in the form of compact plates of any desired thickness, from alkaline lyes containing alkali stannate. SIEMENS & HALSKE A.-G. (B.P. 334,738, 1.10.29. Ger., 28.3.29. Addn. to B.P. 286,673; B., 1928, 452).—The addition of colloids to the bath as specified in the prior patent is unnecessary when lead and bismuth salts are absent.

A. R. POWELL.

[Nickel]-electroplating process. R. J. WIRSHING and H. R. FAAS, Assrs. to GEN. MOTORS RES. CORP. (U.S.P. 1,761,948, 3.6.30. Appl., 10.1.27).—Nickel may be plated from a bath containing 200 g. of nickel acetate and 20 g. of sodium chloride per litre, using a current density of 15–100 amp./ft.² at 35–70°.

A. R. POWELL.

Metal netting for use as catalyst in contact units [e.g., for ammonia oxidation]. BAMAG-MEQUIN A.-G. (B.P. 334,466, 12.12.29. Ger., 8.2.29).—Platinum and/or rhodium wires are inserted as warp and/or weft threads in a netting composed of platinum-rhodium alloy. J. S. G. THOMAS.

Soldering flux. F. E. HARRIS (U.S.P. 1,757,118, 6.5.30. Appl., 2.3.29).—A flux, suitable for use in soldering aluminium, copper, pewter, etc., comprises zinc stearate mixed with an oil (olive oil) and a light petroleum derivative such as benzol, or benzine admixed with carbon tetrachloride to destroy inflammability.

S. K. TWEEDY.

Reduction of iron ores. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,771,971, 29.7.30. Appl., 16.6.28).—See B.P. 313,597; B., 1930, 994.

Reduction of ores, oxides, and the like. H. E. COLEY (U.S.P. 1,777,993, 7.10.30. Appl., 2.11.23. U.K., 10.11.22).—See B.P. 215,400; B., 1924, 601.

Manufacture of steel. E. H. GRAF, Assr. to LUSIFER PRODUCTS Co. (U.S.P. 1,777,081, 30.9.30. Appl., 23.11.28).—See B.P. 332,890; B., 1930, 952.

Manufacture of [sand] moulds [for casting iron]. W. KLEPSCH (U.S.P. 1,777,975, 7.10.30. Appl., 15.9.27. Ger., 29.6.26).—See B.P. 302,254; B., 1929, 133.

[Refractory materials for] refining industrial metals in the molten state. J. A. JACK (U.S.P.

1,777,617, 7.10.30. Appl., 31.3.28. U.K., 6.3.28).—See B.P. 312,700; B., 1929, 645.

Refining of copper. H. H. ALEXANDER (U.S.P. 1,776,826, 30.9.30. Appl., 5.7.28).—See B.P. 310,356; B., 1929, 525.

Protection of metal bodies from corrosive agencies. P. F. SCHADE, Assr. to CHEMIEPROD. GES. M.B.H. (U.S.P. 1,777,107, 30.9.30. Appl., 11.11.27. Ger., 27.7.27).—See B.P. 293,835; B., 1929, 856.

Manufacture of bimetallic strips, plates, or the like. F. W. MILLER (U.S.P. 1,776,364, 23.9.30. Appl., 11.11.27. U.K., 26.11.26).—See B.P. 266,645; B., 1927, 338.

Etching of printing forms. J. BEKK, Assr. to BEKK & KAULEN CHEM. FABR. G.M.B.H. (U.S.P. 1,776,535, 23.9.30. Appl., 18.1.29. Ger., 24.1.28).—See B.P. 304,685; B., 1929, 687.

Apparatus for cleaning articles of metal etc. [by solvents]. CHEM. WERK ZÜRICH A.-G. (B.P. 313,599, 23.5.29. Switz., 15.6.28).

[Die]-casting of metals or alloys [with an aluminium base]. METAL CASTINGS, LTD., and A. H. NICHOLSON (B.P. 334,714, 12.9.29).

Welding [apparatus]. BUDD WHEEL Co., Assees. of J. P. TARBOX, A. F. HANSON, and G. B. REED (B.P. 335,491, 24.1.30. U.S., 26.1.29).

Fusing coatings to metal sheets. AJAX METAL Co., Assees. of H. NEUHAUSS (B.P. 335,769, 24.10.29. U.S., 24.10.28).

Carrying out reactions with gases etc. (B.P. 334,926).—See I.

XI.—ELECTROTECHNICS.

Instrument [“stato-ionometer”] for measuring and recording the concentration of hydrogen and other ions, employing the thermionic valve. P. WULFF and W. KORDATZKI (Chem. Fabr., 1930, 329–330, 342–345, 353–355).—The use of the emission valve in the measurement of, e.g., p_H offers the possibility of automatic recording of results as variations in the anode current. This is not the case with any form of bridge potentiometer. For a practical instrument it is essential that the zero point shall be reasonably constant and the current consumption should be as small as possible. Arrangements proposed by various investigators are discussed. The authors' apparatus employs three batteries (one for compensating current) and one valve, but the batteries may be very small in size. The voltmeter is placed in circuit with the compensating battery, which is so adjusted that no current passes. As, however, the *E.M.F.* of the heating current gradually falls and the emission is also reduced, but not linearly, the zero position changes slowly and should be adjusted daily. The apparatus is provided with alternative connexions by which the complete galvanometer scale may represent from 250 to 1000 millivolts, or it may be graduated in p_H values for a given temperature. The apparatus should not be exposed to sudden temperature changes, but a temperature fall of a few degrees alters the zero by only 1 or 2 millivolts. A suitable

hydrogen electrode for use with the instrument is illustrated and curves obtained in the hydrolysis of methyl acetate with caustic soda and in the ferrocyanide titration of zinc are given. C. IRWIN.

Steel furnaces. CAMPBELL. **Welding.** SCHUSTER. **Tensile properties of conductor wires.** PESTER. **Melting of aluminium.** RUSS. **Test for corrosion.** AUBERT and PROT. **Transformation points in metals.** SCHULZE. **Electrodeposition of gold alloys.** GRUBE. **Silver plating.** SANIGAR. **Control of electrodeposition.** BAILLAY. **Cobalt-nickel deposits.** GLASS-TONE and SPEAKMAN.—See X. **Ash determinations of cane and beet products.** FARNELL.—See XVII.

See also A., Oct., 1230, **Sensitising alkali metal photo-electric cells** (OLPIN). 1253, **Electrolysis of amalgams, fused silver-lead alloys, and bronzes** (KREMANN and others). 1258, "Explosive" **antimony** (STEINWEHR and SCHULZE; COHEN and COFFIN). 1265, **Coreless induction furnace** (ADCOCK). **Electric muffle with chromium-nickel wire heaters** (SIEBERT). 1268, **Decomposition of hydrocarbons in the electrodeless discharge** (AUSTIN).

PATENTS.

Regeneration of [sulphated] lead plates of electric accumulators. E. FEIGELSON (B.P. 334,393, 18.9.29).—The sulphated plates are charged and discharged for a number of short periods, the charging current density being about one fifth normal, until the density of the electrolyte ceases to increase and the plates appear normal. J. S. G. THOMAS.

Electric dry cell. SIEMENS BROS. & CO., LTD., and C. F. EDWIN (B.P. 334,420, 12.10.29).—A carbon electrode surrounded with manganese dioxide is arranged in an electrolyte consisting of a paste composed of lead chloride, flour, and gum, and contained in a lead tube forming the other electrode. J. S. G. THOMAS.

Photosensitive cells. ARCTURUS RADIO TUBE CO., ASSEES. OF S. RUBEN (B.P. 334,409, 30.9.29. U.S., 16.3.29).—A cuprous oxide electrode is arranged in contact with an electrolyte, *e.g.*, hydrogen peroxide, from which nascent oxygen is evolved independently of electrolytic action. J. S. G. THOMAS.

Thermionic cathodes. WESTINGHOUSE ELECTRIC & MANUF. CO., ASSEES. OF G. P. HALLIWELL (B.P. 334,460, 5.12.29. U.S., 14.12.28).—An intimate mixture of a metal of the nickel group, or its oxide, and a deoxidising agent, *e.g.*, titanium, tungsten, molybdenum, thorium, vanadium, tantalum, silicon, manganese, aluminium, chromium, or metals of the rare-earth group, is pressed into bars or slugs and heated in a non-oxidising atmosphere at a sufficiently high temperature to cause sintering and recrystallisation. J. S. G. THOMAS.

Electrically heated [tunnel] kiln. B. J. MOORE and A. J. CAMPBELL, ASSTS. TO GIBBONS BROS., LTD. (U.S.P. 1,776,321, 23.9.30. Appl., 26.11.27).—See B.P. 270,035; B., 1927, 492.

Electric cell. A. HELBRONNER and E. DUTT (U.S.P. 1,777,202, 30.9.30. Appl., 19.4.28. Fr., 11.8.27).—See B.P. 295,589; B., 1930, 153.

Impregnation of dielectric materials. GEN. ENG. CO. (RADCLIFFE), LTD., and S. TAYLOR (B.P. 335,576, 25.6.29).

Electrolytic writing and drawing paper (B.P. 313,561).—See V. **Pure phosphoric acid** (B.P. 334,790). **Dicalcium phosphate** (B.P. 335,007). **Chlorine** (U.S.P. 1,746,542).—See VII. **Furnace for melting metals** (B.P. 334,201). **Treatment of copper sulphide ores** (U.S.P. 1,761,641). **Alloys** (B.P. 335,081). **Furnace for aluminium** (U.S.P. 1,761,442). **Alloy for cable sheaths** (B.P. 314,522). **Recovery of tin from alkaline lyes** (B.P. 334,738). **Nickel plating** (U.S.P. 1,761,948).—See X. **Rubber-paper products** (U.S.P. 1,756,035).—See XIV. **Ozonising-tobacco** (U.S.P. 1,757,477).—See XX.

XII.—FATS; OILS; WAXES.

Component glycerides of stillingia (Chinese vegetable) tallow. T. P. HILDITCH and J. PRIESTMAN (J.S.C.I., 1930, 49, 397—400 τ).—Samples of stillingia tallow from Chinese sources and from plantations in Florida and Texas have been investigated with reference to fatty acid composition and glyceride structure. The crude American plantation sample had sap. equiv. 260.4, acid value 53.5, iodine value 20.0, and setting point 47.3°, whilst the neutralised fat had sap. equiv. 275.4, iodine value 22.1, and setting point 48.2°. The setting points are much higher than those recorded hitherto for stillingia tallow. Of two Chinese samples, I (neutralised) had sap. equiv. 260.9, iodine value 26.6, and setting point 25.6°, and II (a very crude sample) was purified by extraction with warm alcohol, the residual fat (77%) having sap. equiv. 270.6, acid value nil, iodine value 14.4, and setting point 42.4°, whilst the alcohol-soluble portion (23%) had sap. equiv. 254.0, acid value 87.6, and iodine value 29.8. The fatty acids present, determined by ester-fractionation, were mainly palmitic (57—69%) and oleic (21—34%), with small amounts of myristic (3—6%) and stearic (1—3%) acids; non-fatty matter of a semi-volatile ester nature appears to accompany the fat in small quantities. According to the relative amounts of saturated and oleic acids present in the whole fat, the fully-saturated glycerides are present to the extent of about 25—35%; in the mixed saturated-unsaturated glycerides, mono-oleodisaturated glycerides (mainly oleodipalmitins) predominate. The American sample contained 27.6% of fully-saturated glycerides (largely tripalmitin) and over 60% of mono-oleodisaturated glycerides (chiefly oleodipalmitins). Stillingia tallow should be an excellent candle- or soap-making material, but its value as a confectionery fat is uncertain in consequence of the presence of comparatively high proportions of tripalmitin.

Determination of the detergent power of soaps. E. BOSSHARD and H. STURM (Chem.-Ztg., 1930, 54, 762—763).—A standard soiled fabric is prepared by precipitating a known amount of iron oxide (from iron ammonium alum solution) on a flannel strip. The strip is washed by a standardised procedure, dried, and ashed, and the residual iron oxide in the ash is determined by the Zimmermann-Reinhardt method. The

amount of iron oxide removed by the washing operation, expressed as a percentage of the total soil, represents the detergent power of the soap used. If desired, the soiled test strip may be oiled by treating with a known amount of an ethereal solution of petroleum oil, and the fat left after washing determined by extraction. Reproducible results are obtained. The addition of perborate to a curd soap lowered the detergent value.

E. LEWKOWITSCH.

Is degreasing of raw bones with liquid solvents practicable? A comparison with oil-seed extraction. G. GÜNTHER (Chem.-Ztg., 1930, 54, 761—762).—Extraction of substances of such texture as raw bones with liquid media is impracticable; penetration and fat-extraction is incomplete, and displacement of the solvent from the relatively cold material by steam would lead to excessive condensation and spoil the material for further working up.

E. LEWKOWITSCH.

Greek tobacco-seed oil. J. D. KANDILIS and N. S. KARNIS (Praktika, 1929, 4, 475—481; Chem. Zentr., 1930, i, 2820).—The (drying) oil (36.85%) had: iodine value 117.8—137.9, d_{20}^{20} 0.9253—0.9440, n_D^{25} 1.4735—1.4828, acid value 2.25—16.93, saponif. value 186.8—201.6, Hehner value 95.21—96.34, Reichert-Meissl value 0.32—2.03, Polenske value 0.15—0.30.

A. A. ELDRIDGE.

Laundering. ZAKARIAS.—See VI. **Viscosity of linseed oil.** BARRY.—See XIII.

See also A., Oct., 1271, **Partial hydrogenation of linolenic acid** (BAUER and ERMANN). 1308, **Fat from Cypridina** (KOTAKE and KIMOTO). **Liver oil of thresher shark** (LOVERN). 1323, **African Chaulmoogra species** (PERROT and FRANÇOIS). **Wallflower-seed oil** (VAN LOON). 1326, **Determination of fats** (PINCUSSEN and KOLODNY).

PATENTS.

Removal of slime from oils and fats. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,089, 21.10.29).—The slime is precipitated by treating the fats, at temperatures from 70° to 200°, with small quantities of anhydrous sulphonic acids of aromatic hydrocarbons (e.g., β -naphthalenesulphonic acid) and, if desired, in an inert atmosphere and/or in the presence of adsorbents.

E. LEWKOWITSCH.

Treatment of [unsaturated] oils and other glycerides. W. C. ARSEM, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,760,535, 27.5.30. Appl., 22.5.29).—The polymerisation ("bodying") of drying or semi-drying oils can be greatly accelerated by the use of about 1% of an organic amine (preferably benzidine) as catalyst.

E. LEWKOWITSCH.

Fatty acid esters (B.P. 315,832).—See III. **Emulsions** (U.S.P. 1,758,651).—See XX. **Grease from garbage** (U.S.P. 1,764,390).—See XXIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Modern [lacquer] finishes. F. KOLKE (Farben-Ztg., 1930, 35, 2569—2570, 2618—2619).—An account is given of the general properties and methods of application of the following "novelty finishes":

colourless cracking lacquers, crystallising lacquers (oil and cellulose bases), crinkle, grain, and Lymnato finishes. The effects obtained are illustrated.

S. S. WOOLF.

Viscosity of lithographic varnishes and linseed oil. T. H. BARRY (J. Oil Col. Chem. Assoc., 1930, 13, 220—230).—The viscosity-temperature relations of representative grades of litho varnishes, alone and mixed with each other and with linseed oil, were observed over the temperature range 20—90°, using the falling-sphere method of determination. Even with mixtures of extreme grades no departure from regularity was noted, the curves being of the linear-logarithmic type throughout. From the absence of the relatively great increase in viscosity invariably produced when small additions of the colloid are made to colloidal systems, it is inferred that, within the limitations of the experiment described, the dispersions are of the "molecular solution" type.

S. S. WOOLF.

Rosin size manufacture. C. N. RIDLEY (Ind. Chem., 1930, 6, 369—371).—A brief summary of current methods of rosin size manufacture, including the Delthirna process. The effect of hard water on rosin size is illustrated.

T. T. POTTS.

Protection of light aluminium alloys. AUBERT and PIGNOT.—See X. **Hop resins.** GUTHRIE and PHILIP.—See XVIII.

PATENTS.

Production of lacquers and other coatings for absorptive material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,567, 6.5.29).—Solutions of practically water-insoluble cellulose derivatives in practically water-insoluble organic solvents or solvent mixtures, which may contain difficultly volatile constituents, are emulsified in aqueous solutions of organic water-soluble, film-forming, and protective colloids which are free from albumin, e.g., polymerised vinyl alcohols, starch, water-soluble urea-formaldehyde condensation products, ammonium salts of conversion products of phenol-formaldehyde condensation products with halogen fatty acids, methylcellulose. Softening agents, other emulsifying or wetting agents, fillers, colouring materials, resins, etc. may be incorporated.

S. S. WOOLF.

Casein-containing plastic composition. W. W. CHRISTMAS, Assr. to C. C. HINES (U.S.P. 1,758,500, 13.5.30. Appl., 9.6.24).—Powdered casein (1 lb.), powdered calcium hydroxide (1 lb.), calcium carbonate (1 lb.), acetic acid ($\frac{1}{2}$ oz.), an inorganic filler (3 lb.), and an organic fibrous filler (3 pints) are mixed with water to form a flowing mass.

D. WOODROFFE.

Treating waste and low-grade [zinc oxide] pigments. H. REINHARD (U.S.P. 1,776,689, 23.9.30. Appl., 8.9.28. Ger., 9.9.27).—See B.P. 321,675; B., 1930, 204.

Black azo pigments (B.P. 311,400).—See IV. **Adhesive films etc.** (B.P. 333,194).—See V. **Coated fabrics** (U.S.P. 1,746,162).—See VI. **Nitrocellulose-rubber composition** (U.S.P. 1,746,142).—See XIV. **Impregnation of leather etc.** (B.P. 333,759).—See XV. **Light-sensitive plates** (B.P. 315,355).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber mills and Banbury mixers. C. F. SCHNUCK (Ind. Eng. Chem., 1930, 22, 1007—1010).—A description is given of the essential features of construction and of the modern working and respective advantages of the ordinary mill and the Banbury machine for the breakdown and compounding of rubber. D. F. TWISS.

Effect of vulcanising temperature on the properties of a rubber-sulphur mixture. C. R. PARK (Ind. Eng. Chem., 1930, 22, 1004—1007).—Using a mixture of rubber (100 pts.) and sulphur (6½ pts.) and vulcanising over a temperature range of 135.5—162°, tests on tensile strength, extensibility, tear-resistance, and ageing, whether natural or accelerated, indicate that, within the limit of experimental error, the same result is obtained at equivalent degrees of vulcanisation whatever is the temperature of vulcanisation. The temperature coefficient of vulcanisation is 2.5 per 10°. Although accelerated ageing tests are of practical value, there is a marked difference between the changes induced by ageing naturally, in a Geer oven, and in a Bierer-Davis bomb, respectively. D. F. TWISS.

Surface application of age-resisters to vulcanised rubber versus mill incorporation prior to vulcanisation. W. L. SEMON, A. W. SLOAN, and D. CRAIG (Ind. Eng. Chem., 1930, 22, 1001—1004).—Comparative ageing tests at 70°, in an oven and in oxygen under a pressure of 21.1 kg./cm.², on a mixture of high zinc oxide and sulphur content and on a non-blooming motor-tread quality, respectively, using a large number of anti-oxidants, shows that the relative effectiveness of the various anti-ageing agents varies considerably with the test applied. Surface application of the anti-oxidant in solution, however, is generally less effective than incorporation by milling (cf. Moureu, Dufraisse, and Lotte, B., 1930, 625), the main advantages of the former being the possibility of treating finished articles and the avoidance of complications from interference by the anti-oxidant with the processing or vulcanisation of the stock. Incorporation by milling is easier, safer, and more effective generally. D. F. TWISS.

PATENTS.

(A) Production of aqueous rubber emulsion. (B) Dispersion of coagulated bodies. (C) Production of aqueous rubber dispersions. W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,755,890—2, 22.4.30. Appl., [A] 11.4.23, [B] 10.7.24, [C] 14.9.27).—(A) Raw or compounded rubber can be converted into an aqueous emulsion or artificial latex by dissolving, together with a saponifiable substance such as oleic acid, in a volatile solvent, *e.g.*, benzene, and dispersing in water containing an alkali or amine; the resulting soap facilitates the dispersion process. The volatile solvent can be eliminated by evaporation and the bulk of the soap and non-caoutchouc constituents may subsequently be removed by centrifuging the dispersion and separating the aqueous portion, after which the cream may be rediluted. (B) Rubber (or gutta-percha), compounded or otherwise, is dispersed in water by adding up to 15% of an inorganic colloid, such as colloidal clay, and subjecting in a mixing machine to a kneading and stretching operation with the gradual addition of water.

A paste, cream, and milk are thus successively obtainable. Although these dispersions are not easily coagulated by acid, they yield a coherent film on removal of water. (C) A hydrophilic colloid, such as Irish moss jelly, is worked into rubber together with water, so as to effect a preliminary separation of the rubber globules; the mass is then kneaded in a dough mixer with the gradual addition of a solution of the reaction product obtained by heating glue or albumin with caustic alkali. If the amount of Irish moss used in the first operation is sufficient, the second colloid is not necessary and water alone may be added during the kneading operation. The product is a smooth paste coagulable by acids.

D. F. TWISS.

Concentrating [rubber] latex. H. W. BANKS, ASSR. to UNITED STATES RUBBER Co. (U.S.P. 1,755,379, 22.4.30. Appl., 28.11.24).—Rubber latex mixed with colloidal materials, *e.g.*, gelatin or gum arabic, gradually separates into two layers, the lower containing little rubber, whilst the upper has a high concentration of rubber and a correspondingly smaller content of non-caoutchouc substances; the process may be repeated. Such concentrated latex yields highly transparent rubber films. D. F. TWISS.

Method of forming tacky rubber compound and products. M. C. TEAGUE, ASSR. to AMER. RUBBER Co. (U.S.P. 1,746,875, 11.2.30. Appl., 9.6.24).—An acidic ingredient capable of imparting tackiness to rubber, *e.g.*, pine tar, rosin, or coal tar, is converted into an easily decomposable, water-soluble form, *e.g.*, by treatment with a volatile base such as aqueous ammonia. The solution is then added to rubber latex. During the eventual drying operation the volatile base is lost, leaving the ingredient uniformly distributed in the rubber from the latex. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER Co., LTD., D. F. TWISS, G. W. TROBRIDGE, and W. E. GORHAM (B.P. 335,271, 25.6.29).—Cellulosic or fibrous containers are provided with protective coatings by applying an aqueous dispersion of rubber or analogous material mixed with an aqueous dispersion of wax (such as paraffin wax) or of a mixture of waxes, the wax preferably amounting to 20—95% of the dry rubber-wax mixture. Vulcanisation is not necessary. A similarly treated lid becomes sealed in position on such a container when warmed. D. F. TWISS.

Manufacture of surface-finished rubber goods. H. W. EMERY, ASSR. to ARCHER RUBBER Co. (U.S.P. 1,762,165, 10.6.30. Appl., 3.11.26).—A permanent, lustreless appearance is imparted to rubber-coated sheeting by applying to the surface of the compounded rubber a powder such as starch, asbestine, talc, or mica, with or without previous reduction of tackiness of the surface by treatment with soap solution. After removal of surplus powder by washing, *e.g.*, with a volatile solvent, such as carbon tetrachloride or benzene, the sheeting is dried and heat-vulcanised, the surface finally being hardened by halogenation, *e.g.*, by contact with an atmosphere or solution containing free halogen or a sulphur halide. D. F. TWISS.

Production of combinations of rubber and paper

and product obtained thereby. R. P. ROSE and H. E. CUDE, Assrs. to GEN. RUBBER CO. (U.S.P. 1,756,035, 29.4.30. Appl., 9.10.25).—A waterproof paper suitable for electric insulation is made by adding to paper pulp a protective colloid, such as alkaline casein, a dispersion of a paper waterproofing material, such as aluminium resinates or a synthetic resin, and latex. The addition of a coagulant then effects deposition of the dispersed material and rubber on the fibres, which are subsequently sheeted or shaped. D. F. TWISS.

[Nitrocellulose]liquefied rubber composition and its manufacture. H. P. BUTLER (U.S.P. 1,746,142, 4.2.30. Appl., 10.5.28).—A solution of rubber in a suitable solvent, *e.g.*, toluene, with or without the addition of carbon tetrachloride, is heated under reflux for several hours at 100–120°. The solution, of which the viscosity has thus been decreased, is then mixed with a cellulose preparation in order, *e.g.*, to produce a homogeneous solution containing $\frac{1}{2}$ –1 pint of soluble pyroxylin with 2–12 oz. of liquefied rubber. D. F. TWISS.

Curing of moulded rubber articles. H. R. MINOR, Assr. to INDUSTRIAL PROCESS CORP. (U.S.P. 1,746,357, 11.2.30. Appl., 28.8.26).—In the rapid vulcanisation of moulded rubber articles such as motor tubes, a preheated inert gas, *e.g.*, carbon dioxide, is applied as the internal pressure medium; the gas is preferably heated by use of a portion of the steam withdrawn from the jacket of the mould. D. F. TWISS.

Rubber-vulcanisation process. W. P. TER HORST, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,756,315, 29.4.30. Appl., 28.11.27).—Unsymmetrical trisubstituted guanidines of the type $\text{NRR}'\cdot\text{C}(\text{NH})\cdot\text{NHR}$, where R and R' are aryl and alkyl radicals, respectively, *e.g.*, diphenylethylguanidine obtained by the interaction of phenylecyanamide and ethylaniline hydrochloride, are effective vulcanisation accelerators. D. F. TWISS.

(A) Rubber-vulcanisation accelerator. (B) Vulcanisation of rubber. W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,747,187–8, 18.2.30. Appl., [A] 6.10.25, [B] 11.10.28).—(A) Incorporation of basic organic accelerators, such as di-*o*-tolylguanidine, is facilitated by employing these in the form of their compounds with fatty acids, *e.g.*, stearic acid. (B) Vulcanisation accelerators are prepared by treating the reaction products of an aliphatic aldehyde-ammonia, *e.g.*, hexamethylenetetramine or crotonaldehyde-ammonia, and a mercaptobenzthiazole with an aliphatic aldehyde such as formaldehyde or aldol. D. F. TWISS.

Treatment of rubber. P. I. MURRILL and W. W. EVANS, Assrs. to R. T. VANDERBILT CO., INC. (U.S.P. 1,755,703, 22.4.30. Appl., 23.9.27).—The condensation product of an aldehyde, such as formaldehyde or aldol, with a diarylalkylenediamine, *e.g.*, diphenylethylenediamine, is used as an antioxidant. D. F. TWISS.

Manufacture of vulcanised rubber and of materials for use therein. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,352, 7.10.30. Appl., 15.8.27. U.K., 3.9.26).—See B.P. 280,661; B., 1928, 101.

Coated fabrics (U.S.P. 1,746,162).—See VI. **Imitation leather (B.P. 334,361).**—See XV.

XV.—LEATHER; GLUE.

Analysis of leather. A. T. HOUGH (Cuir tech., 1930, 23, 47–48; Chem. Zentr., 1930, i, 2506).—The official method for the determination of ash is inexact. The insoluble constituents of the ash should be determined on fat-free leather which has been extracted with water. The extracted fat should also be ashed. The soluble ash should be heated to fuming with sulphuric acid in order to deduce the quantity of tans. A. A. ELDRIDGE.

Effect of temperature on single-bath chrome tanning. C. OTIN and G. ALEXA (Cuir tech., 1930, 23, 53–56; Chem. Zentr., 1930, i, 2504).—Quantities of hide powder (10 g.) were shaken for 48 hrs. with 200 c.c. of chromium sulphate solution of various concentrations at 25°, 32°, and 40°, the mixture being filtered with suction and the residue washed with distilled water (200 c.c.). The quantity of dissolved chromic oxide and sulphur trioxide increased regularly with the concentration of the tanning solution. Rise of temperature increased the quantities adsorbed, particularly from dilute solutions owing to hydrolysis. A. A. ELDRIDGE.

Diffusion of oak and chestnut extract into gelatin jellies. M. DE LA BRUÈRE (Cuir tech., 1930, 23, 73–75; Chem. Zentr., 1930, i, 2505).—Differences in appearance, due to differences in p_{H} value, are described. A. A. ELDRIDGE.

Photographic gelatin. LAWRIE.—See XXI.

See also A., Oct., 1295, **Tea tannin** (TSUJIMURA), 1303, **Combination between dyes and gelatin granules.** (RAWLINS and SCHMIDT).

PATENTS.

Tanning preparation. R. W. FREY, Assr. to U.S.A. (U.S.P. 1,757,040, 6.5.30. Appl., 31.7.28).—Ferrochrome or other iron-chromium alloy is dissolved in hydrochloric acid, the solution rendered basic by the addition of an alkali carbonate, and the product used in the tanning of leather. On completion of the tannage an oxidising agent is added to the tan liquor to convert Fe^{++} into Fe^{+++} . D. WOODROFFE.

Impregnation of leather or waste leather and manufacture of articles therefrom. J. TAYLOR and A. V. KELLER (B.P. 333,759, 29.8.29).—A condensation product of urea, thiourea, substituted ureas or their derivatives, or phenolic compounds or their derivatives with formaldehyde, paraformaldehyde, or other aldehydic compounds and derivatives is prepared, *e.g.*, by mixing the paraformaldehyde with water to a paste, adding the finely-powdered urea etc., and concentrating the product at not above 90° until a transparent syrup is produced. This is mixed with the leather or waste leather, allowed to dry, ground to powder, with or without the addition of pigment or colouring matter, and moulded. D. WOODROFFE.

Glueing of leather, and adhesive materials therefor. W. STELKENS (B.P. 314,867, 27.6.29. Ger., 3.7.28).—The surfaces to be glued together are first moistened with a solvent for the adhesive material or are softened by heat alone or in conjunction with the moistening process. The adhesive materials, which consist of mixtures of finely-powdered dry cellulose esters and ethers, *e.g.*, acetylcellulose, with solid softening

agents, *e.g.*, triphenyl phosphate, or which may be a film, *e.g.*, nitrocellulose, phenyl phosphate, and butyl phthalate, are then placed between them. Finally the surfaces are subjected to pressure. D. WOODROFFE.

Manufacture of imitation leather having a natural grain. O. G. BOHLIN (B.P. 334,361, 10.8.29).—The surface of imitation leather consisting of compounded caoutchouc and leather fibres is subjected to cold vulcanisation, the whole being subsequently subjected to hot vulcanisation. This procedure ensures a superficial layer which is more solid and more highly vulcanised than the underlying mass. D. F. TWISS.

Manufacture of glue. I. F. LAUCKS and C. N. CONE, Assrs. to I. F. LAUCKS, INC. (U.S.P. 1,757,805, 6.5.30. Appl., 13.9.24).—Vegetable protein-containing material, *e.g.*, ground soya-bean cake, is mixed with hydrated lime, powdered rosin or resins, caustic soda, water, sodium silicate, and potassium permanganate, the amount of the last-named compound being based on that of the protein matter present.

D. WOODROFFE.

Manufacture of adhesives. RÖHM & HAAS A.-G. (B.P. 311,339, 8.5.29. Ger., 9.5.28).—The use of one or more polymerised esters of acrylic acid, dissolved in acetone or formed into thin sheets (*e.g.*, a 10% solution of polymerised ethyl acrylate in acetone), is claimed. [Stat. ref.] D. WOODROFFE.

Treatment of hides, skins, etc. D. L. LEVY (B.P. 335,252, 21.6.29).—See U.S.P. 1,742,514; B., 1930, 434.

Tanning and tanning composition. F. S. LOW, Assr. to F. S. LOW and A. W. BERRESFORD (U.S.P. 1,764,516—7, 17.6.30. Appl., 9.8.28).—See B.P. 256,979; B., 1927, 497.

Polyazo dyes [for leather] (B.P. 313,562).—See IV. **Casein plastic** (U.S.P. 1,758,500).—See XIII.

XVI.—AGRICULTURE.

Microbiological analysis of soils. T. BAUMGÄRTEL and H. BUTENSCHÖN (Landw. Jahrb., 1930, 72, 256—268).—Previous work (Baumgärtel and Simon, B., 1930, 341) is extended, and results are confirmed by cylinder experiments. Cultures in Beijerinck's medium from soils treated with increasing amounts of potash fertilisers show increasing development of *Azotobacter chroococcum* and, where heavy dressings of potash have been used, larger numbers of protozoa. A. G. POLLARD.

Biochemical researches on fertile soil. II. **Adsorption capacity.** A. J. J. VANDE VELDE and A. VERBELEN (Natuurwetensch. Tijds., 1930, 12, 147—154; cf. B., 1930, 524).—The methods proposed for determination of adsorption capacity of soils by other authors are outlined, and results given of determinations carried out with four different soils with (1) aniline dyes, viz., methylene-blue, methyl-violet, and methyl-green; (2) peptone solution; and (3) centrifuged milk. Whilst the results were not concordant, the behaviour towards each of the reagents used was of the same order. S. I. LEVY.

Chemical phases of submerged soil conditions. W. O. ROBINSON (Soil Sci., 1930, 30, 197—217).—

Examination of soil solutions of bog and similar soils is recorded. These differ from aerated soil solutions in containing relatively high proportions of ferrous and manganous carbonates, calcium, magnesium (brought into solution by microbiologically produced carbon dioxide), together with some hydrogen sulphide. Submergence of soils for short periods does not increase their acidity, excepting that due to carbon dioxide. Gas production is characteristic of all submerged soils and is largely influenced by algæ of the *Chlamydomonas* group. Hydrogen and methane are produced in the absence of algæ, whilst in their presence carbon dioxide, nitrogen, and variable amounts of methane are formed. A. G. POLLARD.

Soils in the plastic state. G. B. BODMAN and M. TAMACHI (Soil Sci., 1930, 30, 175—195).—The Atterberg method of determining soil plasticity is modified and a method devised for determining the moisture content at which soils exhibit similar "stiffness." Relationships exist between these values and the air-dry moisture contents, the moisture equivalents, and the colloid contents for all degrees of stiffness. Organic and inorganic colloids produce similar effects on the moisture-stiffness relationships, but the p_H value of soil is without influence in this respect. A. G. POLLARD.

Analytical methods in base-exchange investigations on soils. C. J. SCHOLLENBERGER and F. R. DREIBELBIS (Soil Sci., 1930, 30, 161—173).—Analytical details and technique are recorded for the leaching of exchangeable bases from soil with ammonium acetate solution and the determination in the extract of sodium, potassium, ammonium, magnesium, calcium, aluminium, and manganese. A. G. POLLARD.

[Relation between] soil, fertilisers, and chemical composition of plants. D. J. HISSINK (Chem. Weekblad, 1930, 27, 529—533).—Examples are given to show that whilst the proportions of mineral constituents in the plant depend primarily on the plant itself, they are also dependent on the minerals present in the soil or added as fertilisers. Analysis of the plant and the soil cannot, however, suffice to determine the nature and amount of fertiliser which will produce the best crop. S. I. LEVY.

Loss of residual chlorates from soil. C. R. MEGEE and R. W. LIPSCOMB (Mich. Quart. Bull., 1930, 12, 84—87).—The period necessary between application of sodium chlorate and sowing is considered. CHEMICAL ABSTRACTS.

Ammonium sulphate and sodium nitrate in a sod orchard. R. D. ANTHONY (Penn. Agric. Exp. Sta. Bull., 1930, No. 249, 3—24).—Little difference between the effects of the two fertilisers was observed. When a heavy sod is present the tree suffers from lack of nitrates. CHEMICAL ABSTRACTS.

Fertilisation of shade trees. II. **Conifers.** H. L. JACOBS (Davy Tree Expert Co. Res. Bull., 1930, 5, 15 pp.).—Advantageous chemical fertilisation is recorded. CHEMICAL ABSTRACTS.

Comparison of some nodule-forming and non-nodule-forming legumes for green manuring. L. T. LEONARD and H. R. REED (Soil Sci., 1930, 30, 231—236).—No decided difference was observed in the

effects of nodule and non-nodule legumes used as green manures. A. G. POLLARD.

Legumes as a source of nitrogen in crop rotations. T. L. LYON (Cornell Univ. Agric. Exp. Sta. Bull., 1929, No. 500, 22 pp.).—Legume residues promote the availability of soil nitrogen more markedly than those of other mature crops. The nitrogen content of lucerne was unaffected by fertilisation with superphosphate and potassium chloride, but the crop was much heavier.

CHEMICAL ABSTRACTS.

Varying effect of lime on grassland with different schemes of manuring. W. E. BRENCHELY (J. Min. Agric., 1930, 37, 663—673).—On grass land dressed repeatedly with dung only, liming decreased the hay yields. Where dung and artificial fertilisers had been used the ill-effects of lime were delayed for one or two seasons according to the amount of lime applied. On plots receiving only artificial fertilisers, liming tended to decrease the yield where sodium nitrate was the source of nitrogen, and to increase yields where ammonium sulphate was used. In the latter case there was a notable increase in the ratio of grass to weeds in the herbage. On unlimed soils treated with minerals and heavy dressings of ammonium sulphate, sodium silicate proved beneficial. In a season of severe frost and drought ammonium sulphate tended to kill much of the grass. Liming to a large extent prevented this injury. A. G. POLLARD.

Influence of manuring and spacing on the soundness of potatoes. T. REMY, F. VON MEER, and H. VÖLKER (Landw. Jahrb., 1930, 72, 209—246).—Heavy dressings of potash do not affect the quality of potatoes adversely, but extreme deficiency of potash tends to produce tubers of greater soundness. Increasing applications of nitrogenous fertilisers increase the contents of both total and amino-acid-nitrogen and of the nitrogen precipitable by copper hydroxide in the tubers. The ratio of the three quantities is not altered in a definite manner, but the proportions of amino-acid- and precipitable nitrogen tend to increase with larger dressings of nitrogen. No relationship exists between soundness and the quantity and nature of the nitrogenous substances in the tubers. A. G. POLLARD.

Effect of harvesting in different stages of ripeness on the valuable constituents of cereals. F. BERKNER and W. SCHLIMM (Landw. Jahrb., 1930, 72, 269—291).—Late-harvested grain has the greater dry weight per 1000 grains. There is a migration of starch to the grain in the later stages of ripening and a rapid increase in the protein content between the "milky" stage and full ripeness. A. G. POLLARD.

New sulphur-resin spray. W. NEWTON and R. J. HASTINGS (Sci. Agric., 1930, 11, 26—28).—Sulphur (4 pts.), resin (4 pts.), potassium hydroxide (7 pts.), and water (1 pt.) by wt. are mixed together. Sufficient heat is developed to fuse the mass, which must be vigorously stirred to prevent charring. The melt is poured into thin layers to cool, and is subsequently dissolved in water (16 lb. per 100 gals.). The spray has good spreading and adhesive properties and is an effective fungicide and contact insecticide. Satisfactory trials are recorded against *Botrytis tulipæ* and also aphids, mites, and red spider. A. G. POLLARD.

Properties and composition of the water-soluble fruit-tree carbolic sprays. J. WEICHERTZ (Chem.-Ztg., 1930, 54, 702—704).—Various commercial preparations (phenol content 5—13%) contained 5—9% of water; a further 4—5.5% could be added before the clear solutions showed turbidity. The addition of water produces an unstable water-in-oil emulsion; this gradually disappears to give a homogeneous solution (at, e.g., 54.2% water content) which on further addition of water leads to a very stable oil-in-water emulsion (cf. A., 1929, 1379). The view that the injurious action of the sprays is attributable to the presence of high-boiling tar-products, e.g., anthracene oil, must be abandoned (cf. Neodendrin preparation).

E. LEWKOWITSCH.

Organic nitrogen from agricultural waste. DIEGO.—See II. Nicotine insecticides and vine culture. FERRÉ.—See XVIII.

See also A., Oct., 1322, **Influence of temperature on composition and quality of peas** (BOSWELL). 1323, **Improvement of metabolism of plants by physiological culture** (BARBIERI). **Development of seedlings from seeds** (AXENTSEV). 1326, **Precipitation of silicic acid in plants** (FREY-WYSSLING).

PATENTS.

Treatment of alfalfa [lucerne], clover, and other hard-shelled seeds to promote prompt germination and remove weed seeds therefrom. H. R. WARREN, ASSR. to WARREN-TEED SEED CO. (U.S.P. 1,756,497, 29.4.30. Appl., 15.11.28).—Seeds of the same kind but of varying hardness are soaked in water or glucose solution (*d* 1.18) at 21—46°, and when the soft seeds have swollen, these are removed by flotation or screening and dried. The remaining seeds are scarified, soaked, and separated as before, and the balance of hard seeds is again scarified.

A. G. POLLARD.

Treatment of seeds to secure uniform germination. J. L. KELLOGG, ASSR. to WARREN-TEED SEED CO. (U.S.P. 1,756,488, 29.4.30. Appl., 15.11.28).—The process described in U.S.P. 1,756,497 (cf. preceding abstract) is improved by soaking the seed in a colour-fixing solution, e.g., alum, and subsequently scarifying in the presence of the solution. A. G. POLLARD.

Ripening of sugar cane. D. P. J. BURGIERES (U.S.P. 1,746,190, 4.2.30. Appl., 13.2.28).—To enhance the sucrose content of sugar canes in subtropical countries, their vegetative growth is checked shortly before harvesting by application of powdered lime or other suitable finely-divided substance to the tops of the plants, so that the "buds" are attacked without being destroyed. J. H. LANE.

Treatment of alfalfa [lucerne], clover, and other hard-shelled seeds to promote prompt germination and remove weed seeds therefrom. WARREN-TEED SEED CO. (B.P. 335,102, 1.11.29. U.S., 15.11.28).—See U.S.P. 1,756,488 and 1,756,497; preceding.

Fertiliser with a base of phosphate and sulphur. B. BODRERO (U.S.P. 1,777,908, 7.10.30. Appl., 15.7.26. Ger., 4.11.25).—See B.P. 262,017; B., 1927, 171.

Dicalcium phosphate (B.P. 335,007).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Drying of [sugar] beetroots by the Oxford process. P. ORTH (Bull. Assoc. Chim. Sucr., 1930, 47, 241—247).—The construction and operation of the dryer in use at Eynsham beet-sugar factory are described. The drying process is considered satisfactory. A control over the period Oct. 9—Dec. 25, 1929, showed an average increase of only 0.089% of invert sugar (calc. on fresh beets) as the result of the process, the weekly averages ranging from 0.037 to 0.101%. J. H. LANE.

Dextrorotatory matters other than sucrose in [French] beets harvested in 1929—1930. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 247—251).—Analysis of 32 samples of beets at various dates throughout this campaign showed an average direct polarisation (by hot aqueous digestion) of 15.03, compared with average sucrose contents of 14.19, 14.36, and 14.47%, as determined by the Herzfeld-Creydt, Clerget, and Sailard methods, respectively (cf. B., 1930, 343). Part of this abnormal difference is attributed to the presence of raffinose and part to the influence of nitrogenous substances. Sidersky, however, considers that it is due to pectic substances, which are dissolved to a greater extent in analysis by hot aqueous digestion than in the technical diffusion process. In the preceding three campaigns the author found that the total amount of sugar entering a factory, as calculated from the direct polarisation of the beets, agreed closely with that calculated from the polarisation of the diffusion juice, with due allowance for losses in diffusion, but last campaign the latter control indicated 1.24% less sugar (calc. on beets) than the former, and the actual yield of sugar confirmed the lower value rather than the higher. J. H. LANE.

Evolution of ammonia during evaporation [of beet-sugar juices]. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 252—254).—The amount of ammonia evolved in the working of beet juices is estimated at about 382 kg. per 1000 tons of beets, and some suggestions are made for its recovery. J. H. LANE.

Physico-chemical purification of sugar products by activated carbons in presence of sulphurous and phosphoric acids. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 259—263).—Beet syrup from the centrifugals, having purity 83% and alkalinity 0.03% CaO, was treated at 75—80° with 0.33 g. of Norit per 100 g. of sugar, sulphited to an alkalinity of 0.005% CaO, heated to 80—90°, and filtered. The colour was reduced in the ratio 180:100, the purity was raised by 2.02%, and there was no trace of inversion. When 0.05% of lime was used instead of Norit the decolorisation was only 180:130, and there was no increase of purity. Good results were obtained with 1.5 g. of carboraffin and 0.25—0.5 g. of commercial phosphoric acid per 100 g. of sugar. The rise in purity was over 1%, the decolorisation was 180:95 or 90, and filtration was greatly improved. The final alkalinities were 0.01—0.008% CaO, and no inversion occurred, although the syrup was heated at 90° for 25 min. J. H. LANE.

Ash determinations [of cane and beet products] by the conductivity method using the "Salometer." R. G. W. FARNELL (Internat. Sugar J.,

1930, 32, 347—349).—For such determinations the author recommends the "Salometer," which is accurate, easy to manipulate, portable, and independent of a lighting circuit. In a *résumé* of applications of the method in sugar work, reference is made to the control of clarification by comparison of the ash per 100° Brix of raw and clarified juices, to the control of continuous carbonatation processes, of remelts, and of fresh water for diffusion and boiler feed, to general refinery control, to the calculation of sugar yields from juices and mass-cuites based on the relative ash contents of the juices or syrups and corresponding centrifugal run-offs, to the influence of organic non-sugar on conductivity in relation to the various methods of cane juice clarification, and to use of ash determinations in controlling the sulphur dioxide content of raw cane sugars. J. H. LANE.

Semi-commercial production of xylose. W. T. SCHREIBER, N. V. GEIB, B. WINGFIELD, and S. F. ACREE (Ind. Eng. Chem., 1930, 22, 497—501).—Under the auspices of the U.S. Bureau of Standards, large-scale production of xylose was investigated in 1928 in an experimental factory plant. The raw material was cottonseed-hull bran, which consists of cottonseed hulls freed from "fuzz" and contains 30—35% of pentose. The material was heated with water under 1 atm. pressure for 2 hrs. to dissolve gums, then washed with cold water, agitated with cold 0.25*N*-sulphuric acid for 1 hr. to remove mineral matters, washed again, and digested with 0.2*N*-sulphuric acid under 10 atm. pressure for 2 hrs. to hydrolyse the xylan. After draining from the undissolved matters, the extract, containing xylose equal to about 26% of the weight of the original bran, was neutralised with milk of lime, filter-pressed, concentrated to *d* 1.28 in a vacuum evaporator, again filtered, concentrated to *d* 1.35 at 45—50°, and cooled gradually over a period of 30—48 hrs. in a rotating crystalliser. About half of the xylose in the extract was thus obtained as a brown crystalline product containing 90—94% of xylose and 1—2% of ash. One recrystallisation of this yielded about 65% of a white product of high purity. The process of manufacture is straightforward and does not require very elaborate plant. (Cf. Emley, B., 1930, 681.) J. H. LANE.

Consumption of nitric acid in the oxidation of xylose. G. M. KLINE and S. F. ACREE (Ind. Eng. Chem., 1930, 22, 975—980).—The action of nitric acid on xylose with the production of dibasic sugar acids was investigated. The temperature used was 90—95° and oxides of nitrogen were recovered by scrubbing with caustic soda. The ratio of xylose to nitric acid was found to be about 1:2. Nitric acid may be used at 25—30% concentration. Too prolonged heating increases the loss of nitric acid, which under the best conditions amounts to about 15%. The sugar acids were approximately differentiated into mono- and di-basic acids by the use of bromophenol-blue and phenolphthalein as indicators. Some decomposition of dibasic acid into oxalic acid etc. seemed to occur. C. IRWIN.

Conversion of sucrose into dextrose under the influence of moisture and heat, and the necessity of drying sugar after discharge from the centrifuges.

L. RAIMBERT (Bull. Assoc. Chim. Sucr., 1930, 47, 215—220).—A lecture. J. H. LANE.

Manufacture of rice starch. A. E. WILLIAMS (Ind. Chem., 1930, 6, 387—389).—Descriptive.

Use of "Lystonol" in beet-sugar manufacture. J. ZAMARON (Bull. Assoc. Chim. Sucr., 1930, 47, 254—258).

Fermentation characteristics of *Rhizobium* species. WALKER and BROWN. **Cane molasses fermentation.** HILDEBRANDT and BOYCE.—See XVIII. **Boiler feed-water for sugar factories.** MOUSCADET.—See XXIII.

See also A., Oct., 1248, **Formation of starch paste** (GORBATSCHEV). 1256, **Velocity of inversion of sucrose** (TAKETOMI). 1324, **Rye starch** (TAO).

PATENTS.

Treatment of raw sugar-beet shavings. ARCOS, LTD. From I. TISCHTSCHENKO and V. TCHEFRANOV (B.P. 329,112, 11.4.29).—Before desiccation, beet slices are treated with pulverised or dissolved alkali to prevent inversion of sucrose by acids in the juice or in the drying gases. J. H. LANE.

Treatment of beet juice for manufacture of sugar. J. FRIEDRICH (B.P. 331,308, 24.4.29).—Diffusion juice is heated under a pressure of 1—2.5 atm. and at temperatures up to 138° for 15 min. with about 0.15% of lime (calc. on beets). Impurities are thus precipitated, and the small quantity of lime remaining in solution may be removed by carbonating to the usual alkalinity or by treatment with oxalic acid at the boiling temperature. (Cf. B., 1930, 75.) J. H. LANE.

Manufacture of sugar. A. W. BULL, Assr. to DORR Co. (U.S.P. 1,755,165, 22.4.30. Appl., 4.12.28).—Beet juice is limed and carbonated practically simultaneously, so that it never becomes saturated with lime. Prior to liming and carbonating, raw juice is mixed with already carbonated juice, the precipitate in which adsorbs some of the impurities from the raw juice. The juice may, in part at least, be repeatedly circulated through a zone of liming and carbonation. These features are combined in a process of continuous carbonation in which beet juice, descending through a tall carbonation tank, is treated with a regulated supply of milk of lime in the upper part and carbonated by gas entering near the bottom. The juice leaves the tank at the bottom and a portion of it, e.g., one eighth, is conducted to filters or settling vessels while the remainder passes upwards through a second tank, where it is mixed with raw juice, and then flows again into the top of the carbonation tank. The volume of raw juice thus introduced into the circuit is equal to the volume of carbonated juice withdrawn as mentioned for filtration or sedimentation. J. H. LANE.

Manufacture or refining of sugar. J. B. TALBOT-CROSBIE and H. WISE (B.P. 331,314, 30.4.29).—Sugar solutions obtained by the extraction of desiccated beet or other sugar-containing plant, with or without previous purification, are used in place of water as solvent for raw sugar which is to be refined. J. H. LANE.

Syrup and its manufacture. J. K. DALE, Assr. to SUN-MAID RAISIN GROWERS OF CALIFORNIA (U.S.P.

1,746,993, 11.2.30. Appl., 11.3.27).—Purified syrups of neutral flavour are produced from raisins or other dried fruits by extracting with water, concentrating to about 55° Brix, cooling to allow crystallisation of tartaric acid compounds, filtering, diluting to 35—50° Brix, rendering slightly alkaline with sodium hydroxide, heating to 65—75°, filtering, acidifying to about p_H 5.5, and concentrating to 55° Brix. Decolorising agents may be applied at a suitable stage, and sucrose or a material containing it incorporated to prevent crystallisation of dextrose. J. H. LANE.

Raisin syrup and its manufacture. H. W. DENNY, Assr. to SUN-MAID RAISIN GROWERS OF CALIFORNIA (U.S.P. 1,746,994, 11.2.30. Appl., 8.10.25).—Raisin syrup substantially free from raisin flavour and from tartaric acid and tannin is produced preferably by concentrating raisin juice to about 60° Brix to allow part of the tartaric acid compounds to crystallise, then filtering, diluting, liming to an alkalinity of 0.5% CaO, carbonating in one or two stages to a final alkalinity of 0.02—0.05% CaO, and eliminating remaining soluble calcium salts from the filtered juice by treatment with phosphoric acid followed by sodium or potassium hydroxide. Decolorising agents may be applied at any suitable stage. J. H. LANE.

Manufacture of dextrose. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,750,938, 18.3.30. Appl., 6.8.24. Renewed 20.6.29).—To produce pure dextrose hydrate from the impure hydrate, such as the third crop referred to in U.S.P. 1,471,347 (B., 1924, 27), the latter is partly remelted by heating with or without addition of water or light dextrose liquor, so that some of the crystals remain undissolved, and the magma is then crystallised in motion, without seeding, and at a temperature favourable to the crystallisation of the hydrate, preferably about 40° or below. J. H. LANE.

Production of dry laevulose or carbohydrate mixtures. W. C. ARSEM, Assr. to INDUSTRIAL TECHNIQS CORP. (U.S.P. 1,763,080, 10.6.30. Appl., 31.1.27).—Dry laevulose is produced by heating inulin in a closed vessel with the calculated amount of water for hydrolysis and with a suitable hydrolytic agent; e.g., 1000 lb. of inulin mixed with 11.55 U.S. galls. of water are heated at about 100° with sulphur dioxide or carbon dioxide, under a pressure of 10—100 lb./in.² for 1 hr. or until hydrolysis is complete. J. H. LANE.

Continuous conversion of starch. INTERNAT. PATENTS DEVELOPMENT CO. (B.P. 310,924, 11.5.29. U.S., 3.5.28).—A suitable mixture of starch, water, and acid is gelatinised by heat and forced through long tubes ($\frac{3}{4}$ in. bore, 15 ft. long) which are surrounded by steam under pressure. Four bundles of tubes in all are used, giving a total travel of the material of 60 ft. The pressure employed is usually higher than in batch-working, e.g., 50—80 lb./in.² in the production of liquid glucose or 50—100 lb./in.² for solid grape sugar. In the tubes the material attains approximately the same pressure and temperature as the surrounding steam, and when discharged it liberates steam, part of which may be used for gelatinising fresh starch material. Suitable apparatus is described and figured. The densities of

the starch suspensions and the amounts of acid used may be substantially the same as in batch-working. The material is forced through the apparatus in 4–8 min. according to the temperature of the steam and the degree of conversion required. The tubes may be of heat-resisting glass or metal with enamel linings to prevent any catalytic influence of bare metal on the conversion. J. H. LANE.

Increasing the output in extracting crystallised sugar from molasses and syrups. E. RICARD, Assr. to SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,776,819. 30.9.30. Appl., 23.8.26. Belg., 5.9.25).—See F.P. 621,075; B., 1928, 137.

Ripening of sugar cane (U.S.P. 1,746,190).—See XVI. 2:3 [β]-Butylene glycol by fermentation (B.P. 315,263).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Colorimetric method of determining the soft resins of hops. J. M. GUTHRIE and G. G. PHILIP (J. Inst. Brew., 1930, 36, 455–459).—Minced hops are extracted with industrial methylated spirit and the α - and β -resins are extracted from the spirit solution by light petroleum. The last-named is evaporated and a solution in industrial methylated spirit of the residual resins is coloured by a methyl-alcoholic solution of uranyl acetate and compared in a Hellige universal colorimeter with a wedge of a standard colour prepared from a known solution of α -resin. The colorimetric reading permits the calculation of the sum of the α -resin and of the α -resin colorimetrically equivalent to the β -resin expressed as a percentage on the hops. C. RANKEN.

Fermentation cabinet. C. H. BAILEY (Cereal Chem., 1930, 7, 341–345).—A cabinet is described suitable for carrying out dough fermentation under carefully controlled conditions of temperature and humidity.

W. J. BOYD.

Some fermentation characteristics of various strains of *Rhizobium meliloti* and *R. japonicum*. R. H. WALKER and P. E. BROWN (Soil Sci., 1930, 30, 219–229).—The fermentation of dextrose and galactose by numerous strains of *R. meliloti* and *R. japonicum* is examined by means of changes in the p_H value of the media. Whilst individual strains of the organisms produced consistent results, different strains of the same species caused considerable variations in the degree of acidity or alkalinity produced. In general, *R. japonicum* produced a more alkaline reaction than did *R. meliloti*. Fermentation tests with the two sugars are insufficient to distinguish between the two species of organisms.

A. G. POLLARD.

Influence of the p_H value of the medium on alcoholic fermentation. I. Influence exerted by the products of fermentation on the velocity of multiplication of the yeast cell. II. Alcoholic fermentation in liquids of high hydrogen-ion concentration. III. L. CASALE (Annali Chim. Appl., 1930, 20, 336–353, 353–357, 357–361).—I. Sugar solutions containing inorganic yeast nutrients and of initial p_H values ranging from 2.8 to 8 have been fermented at 22°, the amount of fresh yeast added per litre corresponding with 0.0296 g. of yeast dried at 100°. The

maximum velocity of fermentation occurred at p_H 3.3–4, and the maximum spirit yield at p_H 2.8 and 5, whilst the greatest fermenting power corresponded with the initial p_H 6.93 and the final value 4. The sugar was completely utilised in all the solutions except that with initial and final values 2.8 and 2.6, respectively; in this solution the production of yeast was equal to that obtained in the other solutions, so that at p_H 2.6 inhibition of the action of the zymase, but not of cell multiplication, occurs. The amount of lactic acid formed increased with the initial p_H of the fermenting liquid, but about the same amount of volatile acid was produced in all the solutions, and the formation of glycerol followed the same course as the fermenting power. The activity of multiplication of the yeast was measured by two methods: (1) making use of the fermentative activity, and (2) using the increase in weight of the yeast. Since the first method measures the multiplication during a short period of the fermentation, whilst the second gives the total activity, different results were obtained by the two methods. By method (1) the activity of multiplication was greatest at p_H 6.9, whereas the weight of dry yeast formed was highest when the p_H fell during fermentation from 8 to 4, or from 5 to 2.8, or from 4 to 3. The effect of the p_H value of a fermenting solution consists in modifying the relation between the weight of dry yeast and that of the weight of mineral substances absorbed by the yeast, and also the relation between the cations and anions of the mineral substances.

II. Determination of the constants of yeast cell multiplication during alcoholic fermentation in solutions containing inorganic yeast nutrients and of various p_H values shows that cell multiplication gradually falls off as the fermentation proceeds, except in alkaline solutions (initial p_H 8), with which the rate of multiplication increases during the early stages of the fermentation.

III. A p_H value below 2.5 arrests both yeast cell multiplication and the action of the enzymes determining the formation of alcohol, but does not prevent the action of the other enzymes present, such as those giving rise to lactic and formic acids and acetaldehyde. The acetaldehyde present in highly acid fermented liquids appears to be, not an intermediate product of fermentation derived from pyruvic acid by the action of carboxylase, but a product of the action of another enzyme, probably on lactic acid (cf. Schade, B., 1906, 1112).

T. H. POPE.

Malting and brewing trials with a 6-rowed winter barley. H. L. HIND (J. Inst. Brew., 1930, 36, 435–439).—A British 6-rowed winter barley (F. 112) has been grown which gives a notable yield, and can be malted to produce a brewing material as satisfactory as that obtained from foreign barleys of the same type. Except for its high moisture content the variety closely resembles Californian Bay brewing barley. It requires a longer resting period before malting than that necessary for a 2-rowed English barley, and on that account is not fit to malt so early in the season as is imported foreign barley. It has also the defect that it is liable in wet weather after ripening and before cutting to develop *Cladosporium* on the pales at the germinal ends of the grains. C. RANKEN.

Prediction of extract [yielded by a barley as malt]. L. R. BISHOP (J. Inst. Brew., 1930, 36, 421—434).—For any one variety there is an inverse relation between the nitrogen contents of barley and the extract yield of the resulting malt, whilst the extract increases with increase of grain size. Soil and season have no direct influence. An "extract equation," which permits the prediction of the amount of extract a barley should yield as malt, can be constructed for each separate variety of barley, and for a pure line of Plumage—Archer barley $E = 110.1 - 11.2N + 0.18G$, where E is the extract in lb. of dry malt made from the screened barley, N the nitrogen content on dry unscreened barley, and G the weight in g. of 1000 dry corns of unscreened barley. For Spratt—Archer barley 0.5 lb. must be added to the extract value. Variations in malting conditions are the chief source of differences between prediction and analysis. When adjustments are made for any given maltster, 68.5% of the predictions are within 0.8 lb. of the actual results obtained, whilst 95.5% of the predictions are within 1.6 lb. C. RANKEN.

Acid formation during the fermentation of beer wort. W. WINDISCH, P. KOLBACH, and R. ILLIES (Woch. Brau., 1930, 47, 417—421, 427—431, 437—441).—Worts prepared from several malts, and worts prepared from the same malt but varied as regards protein degradation, acidity, and buffering, were fermented under comparable conditions. Active, titratable, and volatile acidity, esters, and total, formol, and ammoniacal nitrogen were determined on worts and beers. For the esters an improved form of the method of Windisch, Reimers, and Hirschbruch (B., 1915, 812) was employed. The results obtained from different malts and from worts prepared under varying conditions were too complicated to lead to definite conclusions, but strong assimilation of ammonia was frequently associated with higher acid production. Addition of hydrochloric, sulphuric, phosphoric, or lactic acid to wort within practical limits of p_H , is without effect on nitrogen assimilation, but reduces the production of acid, particularly of volatile acid. The p_H of the beers varies with, but to a smaller degree than, that of the worts. Added acetic acid in part disappears during fermentation and the volatile acidity may be less in the beer than in the wort; nitrogen assimilation is also decreased. Addition of citrate buffer without alteration of the reaction of the wort leads to increased acid production, but acetate is partly destroyed. The latter case and that of one malt were the only two in which notable ester formation occurred. F. E. DAY.

Stimulation of cane molasses fermentation by certain metallic salts. F. M. HILDEBRANDT and F. F. BOYCE (Ind. Eng. Chem., 1930, 22, 1011—1014).—Yeast treated with suitable concentrations of copper sulphate (1 in 2000), manganous sulphate (1 in 5000), or sodium cyanide (1 in 12,000) in a solution of cane molasses, and then used as seed in a second solution to which no salts have been added shows a small but definite increase in the yield of alcohol, as compared with untreated yeast. The effect is not continued on seeding a third solution of molasses from the second.

W. J. BOYD.

[Contamination of wines by] nicotine [insecticides used] in vine culture. L. FERRÉ (Ann. Falsif., 1930, 23, 337—344).—The findings of Viala and Marsais (cf. Rev. Viticulture, 1928, 233; 1929, 70) that wines are highly contaminated by nicotine from insecticidal washes have been critically examined. From a consideration of the amount of nicotine deposited on the grapes it is shown that the amounts of nicotine reported to be present in the wines (231 mg. and 330 mg./litre) are impossible with the concentration normally used for the wash. The nicotine is deposited on the grapes in a soluble form and is readily removed by rain even when an adhesive is used. In hot, dry seasons the nicotine is rapidly lost by volatilisation, for after 8 days only traces could be detected. In wines made from grapes which had received one or two sprayings with nicotine washes not the slightest trace of nicotine could be found. The method of determination consisted in (i) steam-distillation after concentrating the wine, (ii) precipitation as silicotungstate, (iii) steam-distillation of the precipitate in the presence of magnesia, (iv) titration with 0.02N-sulphuric acid. It is concluded that the results obtained by Viala and Marsais are untenable as the presence of nicotine in wine would be discovered by taste alone. H. J. DOWDEN.

Buffering power of wines; keeping and plastering. V. MORANI and L. MARIMPIETRI (Annali Chim. Appl., 1930, 20, 313—335).—The potentiometric method for detecting addition of mineral acids to wine (cf. B., 1930, 390), with measurement of the specific buffering powers during neutralisation, may be applied to wines which, after addition of acid, have deposited solid substances as the result of keeping or plastering. Where extraneous causes do not intervene, the two processes, keeping and plastering, always determine a lowering of the p_H of the wine, this being caused by diminution of the extractive substances, especially of the proteins and of salts of organic acids. The method suggested should be applicable also to vinegar. T. H. POPE.

Oxidimetric determination of ethyl alcohol. E. KOSTUK (Bull. Assoc. Chim. Sucr., 1930, 47, 231—237).—For rapid routine determinations of alcohol in fermented liquids or vinasses, a quantity of the sample containing not more than 0.06—0.07 c.c. of absolute alcohol is distilled so that all the alcohol is obtained in 3—10 c.c. of distillate. Thus 100 c.c. of vinasses are distilled (apparatus described), and 10 c.c. of distillate collected, or 10 c.c. of 10- or 20-fold diluted wines or fermented worts are distilled and 3—4 c.c. collected in a test tube. Ten c.c. of a solution containing 33.832 g. of potassium dichromate, 250 c.c. of concentrated sulphuric acid, and 130 c.c. of 85% syrupy phosphoric acid (d 1.7) per litre are measured into another test tube and then heated nearly to boiling and poured into the tube containing the distillate previously warmed to 40—50°, the latter tube being rotated during the pouring to rinse down the walls. After 5 min. the mixture is transferred to a large vessel, into which both test tubes are thoroughly rinsed with water, and the liquid is finally diluted to about 300 c.c., treated with 6—8 drops of a solution of diphenylamine in concentrated sulphuric acid (1 g. per 100 c.c.), and titrated with a solution

containing 70 g. of ferrous ammonium sulphate and 20 c.c. of sulphuric acid per litre until the original brown colour, after passing through bluish-violet, changes sharply to green. The titre of the ferrous solution is ascertained by a blank titration of 10 c.c. of the dichromate solution under similar conditions, whence the volume of dichromate solution reduced by the distillate can be calculated (1 c.c. \equiv 0.01 c.c. of absolute alcohol).

J. H. LANE.

Boiler feed-water for distilleries. MOUSCADET.—See XXIII.

See also A., Oct., 1256, **Velocity of inversion of sucrose** (TAKETOMI). 1303, **Identification and determination of methyl alcohol in presence of ethyl alcohol** (IONESCO-MATIU and POPESCO). 1316, **Enzyme purification; pancreatic amylase** (SHERMAN and others). 1317, **Crystalline pepsin** (NORTHROP). 1318, **Fourth and fifth fermentation processes of sugar** (NEUBERG and KOBEL). **Co-zymase** (VON EULER and MYRBÄCK). 1322, **Enzymic change in ripening barley** (LÜERS and others).

PATENTS.

Manufacture of yeast. A. W. HIXSON and N. E. A. HUGOSON, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,745,693, 4.2.30. Appl., 14.1.21).—Bakers' yeast is propagated with aeration in a solution containing molasses with an added oxidising agent such as calcium hypochlorite. An approximately constant p_H is ensured by the addition of phosphates or salts of a polybasic acid, whilst ammonium sulphate and urea are added as nutrients.

C. RANKEN.

Preparation of yeast. G. O. W. HEIJKENSJÖLD, Assr. to AKTIEB. BÄSTA (U.S.P. 1,757,568, 6.5.30. Appl., 7.3.29. Swed., 14.3.28).—Fermentation is started by seeding yeast into a sugar solution in a fermentation tank in greater amount than that required for the quantity of sulphite waste liquor which will fill the fermentation vessel. Sulphite waste liquor is added to the tank, and, periodically, solution containing yeast is withdrawn, from which the yeast is separated and returned to the tank, while the separated solution is run to waste. The withdrawn wort is replaced by fresh sulphite waste liquor, and the concentration of the sugar and nutrients regulated as desired.

C. RANKEN.

Manufacture of vitamin products. J. TAKAMINE, J. TAKAMINE, JUN., and N. FUJITA, Assrs. to TAKAMINE FERMENT CO. (U.S.P. 1,756,574, 29.4.30. Appl., 18.4.24).—Moistened protein-containing material, such as cereals, clover, starch residues, etc., is sterilised by steam and fermented by a fungus such as *Aspergillus oryzae*. The developed vitamin products and those already in the culture are adsorbed from an aqueous extract of the fermented mash by inert earths, from which they are subsequently dissolved by barium hydroxide. The barium is precipitated by sulphuric acid and protein matters are removed by lead acetate, the excess of which is removed by hydrogen sulphide. After purification by alcohol and acetone, the supernatant liquid is precipitated by ammoniacal silver nitrate, the metallic element of the resulting vitamin compound removed by hydrogen sulphide, and the vitamin solution concentrated under vacuum.

C. RANKEN.

[Microbiological] preparation of 2:3[β]-butylene glycol from carbohydrates. T. H. VERHAVE, SEN. (B.P. 315,263, 25.6.29. Holl., 10.7.28).—In the fermentation of carbohydrates (potato mash, cane molasses, etc.) with *Clostridium polymyxa*, *Aerobacter aerogenes*, or other bacteria producing β -butylene glycol, a sterilised mash is used containing the carbohydrate, a nitrogen compound (malt, ammonium sulphate), a phosphate (superphosphate, phosphorite), and a carbonate (limestone).

C. HOLLINS.

Centrifugal separator (U.S.P. 1,760,071).—See I.

XIX.—FOODS.

Baking characteristics of various types of wheat as reflected by different baking procedures. G. MOEN (Cereal Chem., 1930, 7, 351—357).—Using six flours of varying characteristics, the Standard Baking Procedure of the American Association of Cereal Chemists was compared with two other methods, one of which differed in having longer time and lower temperature of fermentation, with a modified formula, and the other in having shorter time and lower temperature of fermentation, with the standard formula. The standard procedure was the most efficient, giving the required information in the shortest time. In order to differentiate between flours of different strength, time and temperature of fermentation should be so adjusted that diastatic capacity is severely taxed. Addition of bromate to the standard formula emphasises desirable or undesirable characteristics in wheat which the ordinary standard procedure fails to reveal.

W. J. BOYD.

Effect of temperature on the dough and its influence on the standard baking test. V. E. FISHER (Cereal Chem., 1930, 7, 367).—In using the Standard Baking Test, the temperatures most suitable for creaming and doughing are respectively 23° and 21°.

W. J. BOYD.

Determination of carotene in flour with the Pulfrich photometer. ANON. (Mühle, 1930, 67, 209—210; Chem. Zentr., 1930, i, 2816).—The flour is compared photometrically with barium sulphate.

A. A. ELDRIDGE.

Analysis of egg yolk. M. MONHAUPT (Chem.-Ztg., 1930, 54, 697—698).—Moisture is determined by mixing the liquid yolk with a known weight of dry sand and heating at 100—105° to constant weight. Direct carbonisation of the yolk leads to loss of chlorine owing to the action of the metaphosphoric acid formed during combustion of the yolk on the sodium chloride added as a preservative; this is prevented by the addition of 6—10% of calcium hydroxide prior to heating. The ash is extracted with water, the filtered solution neutralised with sulphuric acid, and the chloride determined by titration with silver nitrate.

A. R. POWELL.

Keeping qualities of butter. VI. **Production of metallic flavour in butter and milk.** VII. **Microbic flora of off-flavoured butter.** G. L. A. RUEHLE (Mich. Agric. Exp. Sta. Tech. Bull., 1930, No. 102, 2—12, 13—46).—Metallic flavour may be imparted by iron or copper lactate and by decomposi-

tion of protein, *e.g.*, by bacterial or enzyme action. Numerous organisms were isolated.

CHEMICAL ABSTRACTS.

Decrease in interior quality of hens' eggs during storage as indicated by the yolk. P. F. SHARP and C. K. POWELL (Ind. Eng. Chem., 1930, 22, 908—910).—During storage the yolk membrane of an egg is progressively weakened owing to the stretching produced by the transference of water from the white to the yolk, and furthermore the viscosity is reduced, with the result that when the yolk is placed on a flat plate it assumes a flatter shape and, finally, a condition is reached when the egg cannot be opened without breaking the yolk. The "flatness" of the yolk as measured by the ratio of the height to diameter has been used as an index of the interior quality of eggs. The influence of temperature of storage was most pronounced. The average value for freshly laid eggs was 0.41, but after 3 days at 37° the value fell to 0.30, whereas it had only reached 0.34 after 100 days at 2°. The limit at which the egg could not be opened without rupturing the yolk was about 0.25. The total solids content of the yolk is influenced by the ingress of water and the effect of the storage temperature on this value resembles closely that on the "yolk index."

H. J. DOWDEN.

Copper content of certain foods. F. GRENDL (Pharm. Weekblad, 1930, 67, 913—921).—The average copper content of cows' milk, determined by the sodium diethyldithiocarbamate method, is 0.11 mg. per litre, and of goats' milk 0.15 mg. per litre. Pasteurised milk contains on an average 0.22 mg. per litre, which is practically the same as that found in human milk. The copper content of a variety of other common food-stuffs varies from 0.07 to 0.22 mg./100 g., calves' liver being exceptional in that it contains about 0.5 mg./100 g.

H. F. GILLBE.

Determination of husk in analysis of cocoa. M. WAGENAAR (Chem. Weekblad, 1930, 27, 533—539).—The various methods prepared for identifying and determining husk in cocoa are reviewed; the optical method based on examination in polarised light, worked out by the author (*cf.* B. 1929, 955), is declared to be the only satisfactory quantitative test.

S. I. LEVY.

Flavour of orange honey. E. K. NELSON (Ind. Eng. Chem., 1930, 22, 448).—The pleasant odour of orange honey suggested the presence of methylanthranilate and tests for this substance gave positive results.

J. H. LANE.

Vitamins in canned foods. IX. Tomato products. E. F. KOHMAN, W. H. EDDY, and C. ZALL (Ind. Eng. Chem., 1930, 22, 1015—1017; *cf.* B., 1930, 34).—Feeding experiments on rats and guinea pigs were used for testing the vitamin content of tomatoes. The vitamin-A content of green tomatoes almost doubles on ripening. Canning of either green or ripe tomatoes has little effect on their vitamin-A content, and there is little loss in concentrating the juice *in vacuo* or in an open pan. The vitamin-A is largely removed with the pulp on filtering. There is little difference in the vitamin-B content of green and ripe tomatoes, raw or canned. Filtration of the juice through celite results in much loss of vitamin-B, but vacuum concentration causes

very little loss. 7 G. of green tomatoes, raw or canned, are about the equivalent in vitamin-C of 5 g. of ripened, green-picked tomatoes, raw or canned. Considerable loss of vitamin-C results from the various commercial processes of canning, except in the case of tomatoes canned whole. When the process involves the mixing of tiny air bubbles with the product, as in cycloning, this loss is greater than in processes in which intimate contact with air is avoided. The vitamin-B content is affected to a smaller degree in the same way. When the cycloning is carried out on the hot juice the atmosphere of steam appears to exert a protective action.

W. J. BOYD.

Drapes of *Schinus molle* as adulterant of *Piper nigrum*. A. CREMONINI (Annali Chim. Appl., 1930, 20, 309—312).—This adulteration of ordinary pepper by false (red or Peruvian) pepper may be detected by immersing a small portion, grain by grain, in Fehling solution kept boiling for a time. Reduction of the solution is effected by the sugar present in *Schinus molle* (*cf.* B., 1928, 873). If the grains on which the cuprous oxide has deposited are removed from the Fehling solution, washed in water, and cut through with a sharp knife, the internal, woody involucre of irregular stellate form, surrounded by vacuoles, which in the just ripe fruit are full of essential oil, is visible to the naked eye. If the drupes are freshly and not too highly torrefied, the small amount of essential oil remaining in the vacuole may be detected by heating with nitric acid (*d* 1.40), copious evolution of red vapours occurring; if the drupes are then removed, crushed, and heated with water, vapour with a strong odour of terpenes is emitted. Ground material may be tested by the above reactions and may be examined microscopically for the very hard, woody fragments furnished by the interior of the drupe. The extent of the adulteration may be judged approximately from the reduction of Fehling solution, 1 g. of the lightly torrefied, air-dried drupes (26—28) containing about 0.1 g. of reducing sugar (dextrose).

T. H. POPE.

Fermentation cabinet. BAILEY.—See XVIII.

See also A., Oct., 1295, Tea tannin from green tea (TSUJIMURA). 1321, Vitamin-A of butter (MORTON and HEILBRON).

PATENTS.

Preservation of eggs and the yolks and whites thereof. J. FOUSEK (U.S.P. 1,756,188—9, 29.4.30. Appl., 11.5.27).—Equal weights of sugar and (A) egg yolks or (B) egg whites are mixed at about 60° until the mass is smooth and of a honey-like consistency.

E. B. HUGHES.

Improvement of tea. J. P. CROFT (B.P. 334,224, 1.3.29).—The caffeine content of tea is increased by mixing with it a hot, concentrated, aqueous solution of caffeine, kneading till dry, and blending the product with more tea in suitable proportion. The preparation of a malted tea is described.

E. B. HUGHES.

Preservation of grain and the like. R. A. LEGENDRE (U.S.P. 1,777,044, 30.9.30. Appl., 21.1.29. Fr., 2.4.27).—See B.P. 310,980; B., 1929, 535.

Manufacture of aerated waters, sparkling drinks, and the like. C. HOLLINS and E. CHAPMAN, Assrs. to

BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,777,365—7, 7.10.30. Appl., [A] 5.8.27, [B, C] 9.7.28. U.K., [A—C] 19.8.26).—See B.P. 280,302; B., 1928, 67.

Purification of phosphatides [from soya beans etc.]. H. BOLLMANN (U.S.P. 1,776,720, 23.9.30. Appl., 4.9.26. Ger., 6.10.25).—See B.P. 259,166; B., 1926, 993.

Reconditioning of musty grain and the like [by ultra-violet rays]. E. VON HORVÁTH (B.P. 335,445, 30.11.29).

[Apparatus for] preservation of fruits, vegetables, and other foods. J. MAYOR (B.P. 335,013, 7.8.29).

Vitamin products (U.S.P. 1,756,574).—See XVIII. **Emulsions (U.S.P. 1,758,651).**—See XX. **Liquid treatment (U.S.P. 1,756,275).**—See XXIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Evaluation of tincture of iodine. F. HALLERMANN (Apoth.-Ztg., 1930, 45, 252—253; Chem. Zentr., 1930, i, 2459).—For the determination of total iodine 2 g. of the tincture are treated with 10 c.c. of chloroform and with a solution of ferric chloride, the solution being gently mixed at intervals for 1.5 hrs. Phosphoric acid (25%, 25 c.c.) is then added, and the solution titrated with 0.01N-thiosulphate. A. A. ELDRIDGE.

Solution of magnesium citrate. E. W. REES (Amer. J. Pharm., 1930, 102, 496—505).—Of 69 samples of solution of magnesium citrate, U.S.P. X., having ages varying from 1 to 3 years, 64% contained a deposit which consisted chiefly of the tribasic citrate, $Mg_3(C_6H_5O_7)_2 \cdot 14H_2O$. This compound is formed in the solution made according to U.S.P. X., and, if these directions are adhered to, the product remains clear for 3 months. Attempts to prepare a permanently clear solution were not successful. Samples containing the crystalline deposit can be restored by warming and agitating. Freedom from mould growth and the stability of the solution are dependent on the presence of carbon dioxide. The U.S.P. X. samples contain free citric acid and not magnesium acid citrate, as is evidenced by the fact that the sugar present is inverted under the same conditions as a plain sugar solution containing the same amount of free acid.

E. H. SHARPLES.

Citric acid assay of solution of magnesium citrate, U.S.P. H. V. ARNY and H. H. SCHAEFER (J. Amer. Pharm. Assoc., 1930, 19, 941—945).—Results of the U.S.P. X. tests for free and total acid in commercial and experimental samples of this solution are given. The tests are satisfactory, but the limits imposed for the former value are unnecessarily severe. Statements that citrate is rapidly lost from the solution by decomposition in presence of air are not confirmed. The U.S.P. IX. formula is preferred to that at present official for this preparation. H. E. F. NOTTON.

Methods of assay of chloral hydrate. H. A. WATSON (Amer. J. Pharm., 1930, 102, 506—525).—Seven classes of methods of assay of the compound have been compared. Determination of the formic acid produced on treatment with magnesium oxide is unreliable owing to the instability of magnesium formate, which substance is a probable cause of error in Kippenberger's method

in which the carbon monoxide produced on decomposition of the chloroform formed by treatment with magnesium oxide is measured. Conversion of the chloral hydrate into chloride either with zinc dust (Self's method) or with aqueous or alcoholic alkali gives good results, using the author's modifications. Ruff's iodometric method is satisfactory when the interfering secondary reactions are reduced to a minimum by diluting the alkali to ten times the volume and then adding it drop by drop with constant shaking. A modification of the method of Archangelsky, in which the formic acid produced by the action of alkali is determined by the reduction of mercuric to mercurous chloride, gives uniform results about 0.5—1% low. E. H. SHARPLES.

Quinizarinsulphonic acid (rufianic acid) as a precipitant for alkaloids. L. ROSENTHALER (Pharm. Zentr., 1930, 71, 561—562).—Whilst precipitates are obtained with most alkaloids, often at very great dilutions, they are very similar in appearance, and the reagent is not likely to be important for microchemical differentiation. S. I. LEVY.

Sterilisation of hexamethylenetetramine solutions. G. H. NIJHOFF and H. D. VAN OORT (Pharm. Weekblad, 1930, 67, 884—888).—Sterilisation by boiling causes decomposition with formation of ammonia and formaldehyde. It is recommended that 2% of sodium bicarbonate should be added to the solution and sterilisation effected by Tyndallisation. The extent of the decomposition under various conditions has been determined. S. I. LEVY.

Removal of the bitter principle of lupin seed by Thom's process. M. GERLACH (Landw. Jahrb., 1930, 72, 247—253).—The bitter principle of yellow lupin seeds cannot be removed from whole seed by 50% methyl or ethyl alcohol, nor by denatured spirit. Spirit (50% denatured) containing a small amount of hydrochloric acid removed 75% of the alkaloid from crushed seeds, but with considerable losses of total dry matter, fat, and protein. Satisfactory extraction without attendant losses was obtained by heating the whole seeds in water containing a little hydrochloric acid and subsequently treating with 50% denatured spirit. A. G. POLLARD.

Preparation of extractum hydrastis fluidum and methods of analysis. Z. CSIPKE (Ber. Ungar., pharm. Ges., 1929, 5, 73—85; Chem. Zentr., 1929, i, 1583).—A report on the known methods of preparation and on the official determination of the alkaloid content in the drug and the extract. L. S. THEOBALD.

Determination of theophylline and ethylenediamine-theophylline. P. W. SCHMITT (J. Amer. Pharm. Assoc., 1930, 19, 821—825).—A method for the analysis of ethylenediamine-theophylline ("Euphyllin") consists in the determination of theophylline by precipitation with a standard ammonio-silver chloride reagent followed by determination of the excess of silver in the filtrate and a determination of the ethylenediamine by calculation from the total nitrogen content.

E. H. SHARPLES.

Comparative methods of assay of Chinese ephedras. Y. D. Hsu (J. Amer. Pharm. Assoc., 1930, 19, 817—820).—From an examination of various methods, the following is recommended as being con-

venient and satisfactory for the determination of total alkaloids in ephedras. A mixture of the dried, powdered drug (20 g.), distilled water (40 c.c.), and freshly slaked lime (5 g.) is macerated for 10 min., 100 c.c. of 2% ammonium chloride solution in 70% alcohol are added, and the mixture is shaken. Five further quantities of 100 c.c. are periodically added with shaking and the mixture is allowed to macerate for 12 hrs. After filtration, the alcohol is evaporated and the filtered concentrate after the addition of 10 c.c. of ammonia solution is extracted three times for 1 hr. each with chloroform, 50, 40, and 40 c.c., respectively. The combined extracts are evaporated *in vacuo*, the residue being dissolved in neutral alcohol and titrated with 0.1*N*-hydrochloric acid. A sample consisting of a mixture of *E. equisetina* and *E. sinica* yielded 1.999% of total alkaloid. Further extraction with 200 c.c. of alcoholic ammonium chloride gave 0.056% of alkaloid.

E. H. SHARPLES.

Jasmine. ANON. (Parf. mod., 1929, 22, 691—705; Chem. Zentr., 1930, i, 2177).—A description of manufacture and use.

L. S. THEOBALD.

Zdravet in modern perfumes. ANON. (Parfums de France, 1929, 7, 315—316; Chem. Zentr., 1930, i, 2177).

Balsamic mass from acetic acid tar. PALLAS.—See II. Tobacco-seed oil. KANDILIS and KARNIS.—See XII. Adulterant of *Piper nigrum*. CREMONINI.—See XIX.

See also A., Oct., 1286, Homologues and analogues of phenylpropanolamine (HARTUNG and others). 1294, Preparation of thymol from piperitone (KIMURA). Sagittol (YANOVSKY). 1295, α -Tetrahydrofurfuryl chloride and ethers (KIRNER). 1300, Catalytic reduction of nicotine and metan nicotine (HARLAN). 1303, Identification and determination of methyl alcohol in presence of ethyl alcohol (IONESCO-MATTU and POPESCO). 1304, Micro-detection of alkaloids (LANDER). 1315, Pharmacology of gold compounds (VON ISSEKUTZ and others). Local anæsthetics derived from dialkylaminopropanediols (RIDER). 1320, Vagotonin (PÉNAU and SANTENOISE). Crystalline insulin (GEILING and DE LAWDER; JENSEN and DE LAWDER). Oestrin (MARRIAN). Crystalline menoformone (DINGEMANSE and others). Relaxative hormone of the corpus luteum (FEVOLD and others). 1323, Leaves of *Solanum pseudocapsicum* (BREYER-BRANDWIJK).

PATENTS.

Manufacture of [pyro]catechol [4-(3:4-dihydroxyphenyl)thiazoles. T. B. JOHNSON, Assr. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,743,083, 7.1.30. Appl., 3.2.28).—Chloroacetylpyrocatechol [3:4-dihydroxyphenyl chloromethyl ketone] is condensed with thioamides, thiocarbamides, or thiocarbamates to give thiazoles which have therapeutic value. 4-*mp*-Dihydroxyphenylthiazoles having various substituents in the 2-position already described (cf. Johnson and Gatewood, A., 1929, 943) are claimed. C. HOLLINS.

Manufacture of basic bismuth salts or organic arsinic acids. I. G. FARBERMUND. A.-G. (B.P. 334,449, 14.11.29. Ger., 22.11.28).—Non-mercurated alkali arylarsinates react with solutions of bismuth salts (excluding

bismuthyl potassium sodium tartrate) to give alkali-insoluble basic bismuth arylarsinates. Examples are basic bismuth salts of 3-acetamido-4-hydroxybenzene-arsinic acid, methylbenziminazolonearsinic acid, and 3-amino-4-hydroxybenzenearsinic acid. C. HOLLINS.

Manufacture of pharmaceutical compounds. W. T. WINCKLER, Assr. to VON WINKLER LABS., INC. (U.S.P. 1,751,964, 25.3.30. Appl., 7.11.28).—Sulphoricinoleic acid, *i.e.*, sulphonated castor oil, is converted into a silver salt by the addition of silver ammonium formate. The product is specific for gonorrhœa.

C. HOLLINS.

Ozonising tobacco. S. ROSENHOCH (U.S.P. 1,757,477, 6.5.30. Appl., 9.7.28. Ger., 11.7.27).—The tobacco is brushed clear of foreign matter, slightly moistened with sterilised water, and, after 5—10 hrs., the leaves are separated, foreign plant matter is removed, and the leaves are again moistened. After 10—16 hrs. they are spread thinly on an electrically charged conveying device and treated with a countercurrent of ozone in an amount varying with the nicotine content of the tobacco. The electrical treatment breaks up the cells without destruction of aromatic resins or oils, so that the ozone can enter the leaf and effect its refinement. Suitable apparatus is described.

E. H. SHARPLES.

Chemical emulsion. J. R. CONOVER, Assr. to LAB. OF RES. CHEMOTHERAPY (U.S.P. 1,758,651, 13.5.30. Appl., 29.5.24).—Emulsions are made from substances which are identical with those found in animal tissue or tissue-producing substances, such as lanolin, other animal fats and waxes, casein, egg and serum albumins, etc. The emulsions closely resemble living tissue and provide efficient carriers for germicidal and chemotherapeutic agents. *E.g.*, the colloidal potassium silver salt of polypeptides derived from casein, which has a germicidal activity equal to that of a 1.33% phenol solution and which retains its activity indefinitely, is prepared by mixing an aqueous silver nitrate solution with a solution of casein in dilute sodium or potassium hydroxide under suitable conditions, and adding the mixture to an emulsion made by stirring lanolin with a solution of casein in alkali or alkaline-earth hydroxide or carbonate solution.

E. H. SHARPLES.

After-treatment of sterilised catgut for surgical use. R. GRAF & Co., A.-G. (B.P. 333,847, 10.12.29. Ger., 14.12.28).—Catgut, sterilised in the usual manner, particularly with iodine, is treated with solutions which contain protein, such as bacteriological meat-broth solution, for 12—24 hrs., and then immediately with 96% alcohol for a similar period. The catgut is thereby freed from injurious chemicals, particularly free iodine, and is also rendered non-irritant. E. H. SHARPLES.

Production of medicaments containing bismuth. A. LIEBRECHT, Assr. to CHEM.-PHARM. A.-G. BAD HOMBURG (U.S.P. 1,777,173, 30.9.30. Appl., 24.7.25. Ger., 29.7.24).—See B.P. 237,912; B., 1927, 60.

Manufacture of inactive menthol. K. SCHÖLLKOPF and A. SERINI, Assrs. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,776,667—1,776,671, 23.9.30. Appl., 20.9.28. Ger., [A—D] 15.2.27, [E] 15.12.26).—See B.P. 285,403; B., 1928, 873.

Phenanthridone etc. (B.P. 333,173).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Jelly strength of photographic gelatin. J. P. LAWRIE (J.S.C.I., 1930, 49, 403—404 r).—In testing jelly strengths of gelatins by Tracey's apparatus (B., 1928, 310), considerably divergent results were obtained. Strictly controlled experiments were carried out in an attempt to ascertain the cause of the irregularities, but it was found to be impossible to repeat or duplicate results. The irregularities are attributed to the important influence of time and temperature during the making of the gel, and it is suggested that it is essential to use a proper room, thermostatically controlled. Apparently no consideration has been given to the possibility of the irregularities being produced by mechanical defect or faulty design of the apparatus used.

Antifogging and antisensitising compounds. S. E. SHEPPARD (Phot. J., 1930, 70, 439).—The effect of the presence of such substances on the *E.M.F.* of silver-ion concentration cells, using concentrations of the order of those afforded by saturated solutions of the silver halides, has been investigated at various p_H values. A removal of silver ions, presumably by complex formation, was shown by the antifogging agents thioacetanilide and nitrobenzimidazole, the desensitising dyes phenosafranine and chrysoidine, and, in an extension of the work to sensitisers, by orthochrome-T, kryptocyanine, erythrosin, and the general sensitiser allylthiourea. This supports the suggested common initial behaviour of such compounds in forming complexes with the silver halide. A tentative explanation of the inability of chrysoidine bases to desensitise is given.

J. W. GLASSETT.

Desensitising properties of Basic Scarlet N. (Miss) F. M. HAMER (Phot. J., 1930, 70, 436—437).—Basic Scarlet N may be separated by extraction with ether into two constituents; the soluble and insoluble components react similarly to chrysoidine and safranine, respectively. The fact that the desensitising properties of a 1:5000 bath are unaffected by sodium carbonate and sulphite present in normal developer concentrations is not in accordance with the presence of chrysoidine, but the striking increase in loss of image, or inhibition of development, obtained by increasing the bath from 1:5000 to 1:500 is strong confirmation of its presence. Comparative tests indicate that Basic Scarlet N is not superior to safranine as a practical desensitiser.

J. W. GLASSETT.

See also A., Oct., 1260, **Effect of light on dyes** (KRAIS). 1266, **Emulsions on glass for recording distances** (COOKSEY and COOKSEY).

PATENTS.

Manufacture of light-sensitive layers. KALLE & Co. A.-G. (B.P. 333,820, 2.11.29. Ger., 3.11.28).—Aromatic azides (e.g., azidosalicylic acid) which are decomposed by light action to form a dye are dissolved in water or an organic solvent, or incorporated in gelatin or collodion solutions, and applied to a paper or film support and dried. After exposure the unchanged azide is removed by application of a solvent. The colour of the image may be modified by the inclusion of acids, bases, or metallic salts in the sensitive layer.

J. W. GLASSETT.

Light-sensitive plates or the like. INTERNAT. GEN. ELECTRIC CO., INC., ASSECS. to ALLGEM. ELEKTRICITÄTS-GES. (B.P. 315,355, 27.6.29. Ger., 12.7.28).—The support for the light-sensitive layer is prepared from artificial resins produced from the condensation products of polyhydric alcohols and polybasic acids or their anhydrides, e.g., glycerol-phthalic anhydride resins. The transparency, flexibility, and colour of the support can be controlled during manufacture by the addition of fillers, softening agents, and dyes.

J. W. GLASSETT.

Photographic reducers. I. G. FARBEIND. A.-G. (B.P. 334,144, 29.11.29. Ger., 29.11.28).—The retardation of the reducing action in the higher densities, caused by either the localised concentration of the reaction products or the more rapid change in p_H at those parts, is minimised, in the first case, by the incorporation of a certain amount of the reaction products in the reducer, and, in the second, by the addition of a buffer salt. The following reducers are claimed: (A) 10% sodium thiosulphate solution 100 c.c., ferric oxalate 4 g., ferrous oxalate 4 g.; (B) potassium dichromate 2 g., potassium bisulphate 0.6 g., potassium sulphate 9 g., water 100 c.c.

J. W. GLASSETT.

Chemically transforming photographic silver images. F. LIERG (B.P. 333,361, 8.7.29).—The process depends on the use of such compounds as thiocarbamide, thiosinamine, etc. which readily form water-soluble complexes with the silver halides, but which form insoluble or only slightly soluble complexes with other silver salts. The silver image is converted first into a silver halide, and is then precipitated as the new salt by treatment in a bath containing thiosinamine and the new salt-forming agent such as potassium ferrocyanide, tartaric acid, sodium nitrite, sodium phosphotungstate. The process may be adapted to the formation of azo-dye images and to the preparation of dye-repellant images suitable for dye-printing processes.

J. W. GLASSETT.

Imbibition processes for producing pictures in colour. UVACHROM A.-G. F. FARBEIND. (B.P. 383,392, 8.8.29. Ger., 16.2.29).—Improved reproduction is obtained by (a) using only the more highly-dispersed fractions of the dyes obtained by known methods by ultrafiltration, fractional precipitation, etc.; (b) treatment of the relief with aqueous glycollic acid; (c) exclusion of the yellow rays from the printing light used for the preparation of the relief; (d) addition of casein to the gelatin layer for the reception of the dye; and (e) regeneration of the relief during printing by treatment with an oxidising agent which destroys the strongly absorbed dye.

J. W. GLASSETT.

Manufacture of monochromatic photographic pictures. C. ROEHRICH (B.P. 308,361, 21.3.29. Ger., 22.3.28).—A dichromated gelatin plate is exposed behind a negative of the subject and treated with a suitable red dye which is absorbed most strongly in the unhardened parts. This dyed image is used in the subsequent copying process and exerts a non-uniform filtering action which varies in accordance with the light and shade of the original picture. Control of contrast may be obtained by suitably dyeing the unhardened and hardened parts. [Stat. ref.]

J. W. GLASSETT.

Colour photography. W. V. D. KELLEY (B.P.

333,931 and 333,933, 23.5.29).—(A) The blue- and green-sensitive layer is separated from the red-sensitive emulsion by a thin layer of gelatin dyed with congo-red, which may be readily bleached subsequent to exposure by treatment with a dilute solution of sodium hyposulphite. (B) If the different colour-sensitive emulsions are coated on separate supports and placed together face to face for the exposure, the red-sensitive layer is bathed in a solution of congo-red, which does not penetrate, but forms an effective filter in the surface layers. J. W. GLASSETT.

Colour photography. SPICERS, LTD., J. W. GOLD-SMITH, T. T. BAKER, and C. BONAMICO (B.P. 333,865, 1.6.29).—In order to apply a multicolour screen to a film of cellulose acetate, nitrocellulose, or other cellulose esters, the surface of the film is treated in an alkaline bath composed of a 0.5–5% solution of caustic potash in methylated spirit, which renders the film capable of absorbing dye solutions very readily and facilitates their removal or bleaching. J. W. GLASSETT.

Production of films in natural colours by a single exposure. F. LIERG (B.P. 333,697, 2.7.29).—A film support is coated on one side with a blue-sensitive emulsion and on the other with superimposed green- and red-sensitive emulsions with the latter uppermost. By a suitable optical device both sides of the film are exposed simultaneously, and, after development and fixation, the blue screen image is mordanted, dyed yellow, and protected by a coat of lacquer. The green and red screen images are then mordanted, dyed with a bluish-red dye (rhodamine), treated in a hardening bath, and dried. The film is finally immersed in a bath of a bluish-green dye, which combines with the bluish-red dye to give a pure blue; owing to the hardness of the gelatin layer it does not penetrate to the red image underneath. J. W. GLASSETT.

Multicolour photographs. H. and C. HERZOG (J. HERZOG & Co.) (B.P. 333,668, 4.6.29).—The positive material consists of silver halide emulsions dyed with non-diffusing, highly transparent, colloidal dyes coated upon a transparent support. Printing is made from the separate colour-record negatives through the support side of the appropriately coloured positive material, and, after development in a tanning developer (sulphite-pyrocatechol), the unhardened parts are removed in hot water. A series of these monochrome images are then transferred in register to a common support. The colour may be controlled by treating the finished monochrome images with solvents for or reducing agents of the dye. J. W. GLASSETT.

[Lens unit for] production of photographic images. H. D. BEACH (B.P. 335,696, 27.8.29. U.S., 5.3.29).

Method of enabling pictures or the like to be exhibited in substantially natural colours. A. BERNARDI, and RAYCOL, LTD. (B.P. 335,310, 18.7.29).

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Charge for blasting caps. W. DE C. CRATER, ASSR. to HERCULES POWDER Co. (U.S.P. 1,759,565, 20.5.30. Appl., 13.8.29).—Nitrolactose is employed as the main charge for blasting caps, in conjunction with an initiat-

ing charge of fulminate and chlorate, lead azide, diazo-dinitrophenol, etc. If desired, the nitrolactose may be mixed with the initiating charge. W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Vapour pressures of fumigants. IV. $\alpha\alpha\beta\beta$ -Tetra-, penta-, and hexa-chloroethanes. O. A. NELSON (Ind. Eng. Chem., 1930, 22, 971–972; cf. Young and Nelson, B., 1929, 456).—The vapour pressures of tetra- and penta-chloroethanes were determined by the static method at temperatures ranging from 25° to 146° and 160°, i.e., the b.p. of these compounds. Hexachloroethane being a solid which sublimes rapidly at its m.p. (185°), its vapour pressure over the range 15–60° was determined by saturating air with the vapour and cooling to –78°. C. IRWIN.

Water softening: certain properties of some base-exchange materials. A. R. MARTIN (J.S.C.I., 1930, 49, 389–394 r).—There are two main types of base-exchange material, the synthetic products and certain minerals. The minerals may be subdivided into non-porous glauconites and porous minerals. The bulk densities, volumes of interstitial spaces, and exchange values at varying rates of flow of water have been determined. The regeneration of the base-exchange materials and their salt consumption have been studied. The facts can be interpreted by supposing that with a glauconite the exchange of bases is wholly confined to the outer surface of the material, where it is very rapid. In the case of the synthetic products there is, in addition to the action at the outer surface, an action in the interior of the particle, diffusion to and from the interior being a relatively slow process. The interior action may be either in the mass of the material or at capillary surfaces. The mechanical disintegration and solubility of the base-exchange materials have been investigated, and in particular the effect of the original silica content of the water on their solubility.

Purification of [boiler feed]-waters in sugar factories and distilleries. G. MOUSCADET (Bull. Assoc. Chim. Sucr., 1930, 47, 207–215).—One or two coats of an aqueous suspension of finely-divided graphite, preferably colloidal, applied to the interior of sugar-factory boilers, after they have been cleaned and dried at the end of a campaign, will reduce incrustation during the following campaign, the salts precipitated from the water tending to form a sludge instead of a hard deposit. "Magnetine," a mixture of a special graphite with finely-powdered metals such as aluminium and copper is recommended for preventing corrosion as well as incrustation of boilers. In the discussion on the paper the use of phosphates for the treatment of boiler feed-water was advocated by Zamaron. J. H. LANE.

Water treatment for raw water ice-making. L. M. CULTER (J. Amer. Water Works' Assoc., 1930, 22, 1215–1219).—During the freezing of raw water the carbonates of magnesium and calcium and oxides of iron and aluminium as well as silica and suspended matter gradually accumulate in the core of the block and can be readily removed. The more soluble carbonate of sodium which replaces those of magnesium and calcium when the raw water is previously softened by base exchange or by soda ash treatment remains trapped in the body of the

ice block. Removal of colour is of great importance, and with some waters may be effected by the use of lime. Simultaneous removal of bacteria takes place. When the water has a low p_H and free carbon dioxide the colour is removed by treatment with sodium aluminate and aluminium sulphate to bring the final alkalinity to 10 p.p.m. H. INGLESON.

Protozoological examination of water. (MME.) N. L. WIBAUT (Chem. Weekblad, 1930, 27, 526—529).—Tabulations of the numbers of bacteria (*B. coli*) and protozoa present in previously sterilised waters at successive intervals after seeding show that in most cases both increase at first, the latter much faster than the former; after the period of increase both decrease. The conclusion is drawn that the protozoa can entirely eliminate the bacteria in most cases. Comparative tests of the number of protozoa and bacteria present in a water may be used to determine the date of infection of the water with bacteria. S. I. LEVY.

Effect of iron in the accuracy of the determination of dissolved oxygen [in water] by the Winkler method O. M. URBAIN and J. N. MILLER (J. Amer. Water Works' Assoc., 1930, 22, 1261—1265).—In such determinations the error due to the presence of iron increases with the time which elapses between the addition of sulphuric acid and the titration of the iodine liberated and with the amount of iron present in the water. A graph of corrections for iron contents up to 100 p.p.m. and for times up to 24 hrs. is given. Waters containing more than 50 p.p.m. of iron give wholly unreliable results, whilst those containing less than 50 p.p.m. give inaccurate results unless the titrations are made immediately. H. INGLESON.

Hydrogen sulphide in industry: occurrence, effects, and treatment. W. P. YANT (Amer. J. Pub. Health, 1930, 20, 598—608).

Acute response of guinea-pigs to vapours of some new commercial organic compounds. II. Ethylbenzene. W. P. YANT, H. H. SCHRENK, C. P. WAITE, and F. A. PATTY (U.S. Pub. Health Rep., 1930, 45, 1241—1250).

PATENTS.

Sewage disposal plant. T. B. SIMS, Assr. to L. J. WARDLAW (U.S.P. 1,757,262, 6.5.30. Appl., 23.4.28).—A central scum chamber or gas vent in a double-storey settlement tank is fitted with a gastight cover and is maintained under reduced pressure by means of a suitable pump. The increased rate of removal of dissolved and fermentation gases thus induced causes a separation of the lighter particles of sludge and forms a scum which after mechanical agitation is withdrawn intermittently for further treatment. The degassing of the sewage also facilitates the dissolution of oxygen at a later stage with consequently increased stability. C. JEPSON.

Treatment of [sewage] sludge. Sewage disposal system. T. B. SIMS (U.S.P. 1,757,263—4, 6.5.30. Appl., [A] 10.12.28, [B] 29.4.29).—(A) The digestion of sludge is facilitated by mixing, preferably with vigorous agitation, the heavier and more decomposed sludge from the bottom of the tank with the less decomposed scum. The use of an intermittent vacuum is suggested to carry out this purpose and also to skim floating matter from the surface of the sewage and introduce the same

into a centrally situated sludge-digestion chamber normally maintained under partial vacuum to remove the gases generated. (B) In a system which includes the treatment of settled sewage on a percolating filter intermittently operated by means of a dosing chamber, the latter is so arranged that the rise and fall of the water therein may be utilised to supply compressed air for the aeration of the sewage prior to its entrance into the closing chamber, and also to create a partial vacuum over a portion of the sedimentation tank (cf. U.S.P. 1,757,262; preceding), by means of which hydrogen sulphide is removed from solution and the liquor rendered more amenable to stabilisation by aeration at a later stage. C. JEPSON.

Recovery of grease from garbage. W. D. CLEARY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,764,390, 17.6.30. Appl., 19.11.26).—The mixture of water, grease, and insoluble matter expressed from digested garbage is heated to 82° and centrifuged to remove the suspended solids. It is then allowed to separate and the floating grease is passed to a centrifugal separator, whence it is obtained in a fairly pure condition but may be washed and re-separated if desired. C. JEPSON.

Water-purifying material and its manufacture. H. KRIEGSHEIM and W. VAUGHAN, Assrs. to PERMUTIT Co. (U.S.P. 1,757,372, 6.5.30. Appl., 19.9.22).—The base-exchange, water-softening properties of glauconite are improved by successive treatment with a weak solution of sodium silicate and an acid-reacting solution, e.g., aluminium sulphate. An improved product, stated to contain a higher proportion of combined silica and to be harder and less affected by heat than the natural product, is made by treating the mineral with a strong solution of sodium silicate, drying for about 1 hr. at 200°, and subsequently washing the crushed mass with calcium chloride solution (5%) before treatment with aluminium sulphate. C. JEPSON.

Purifying water. L. L. HEDGEPEETH, Assr. to W. C. OLSEN (U.S.P. 1,757,526, 6.5.30. Appl., 17.4.28).—In the treatment of coloured waters etc., considerable improvement over the usual procedure is claimed if, after the water has been coagulated, e.g., with alum, and the p_H adjusted to the isoelectric point of the precipitated lakes to prevent peptisation, a further addition of a caustic coagulant, e.g., an alkaline aluminate, is made. This reacts with any unaffected alum and produces a secondary precipitation which is also brought to a maximum by adjustment of the p_H value. The dispersal of the matter carried down by the first precipitation is prevented by the coating of aluminium hydroxide produced by the secondary reaction. C. JEPSON.

Liquid treatment [with zeolites]. A. S. BEHRMAN, Assr. to GEN. ZEOLITE Co. (U.S.P. 1,756,275, 29.4.30. Appl., 23.5.29).—Soluble impurities, e.g., alkali carbonates and bicarbonates, may be removed from water by conversion into calcium or magnesium bicarbonates by treatment with the zeolites of these metals and subsequent precipitation as carbonates by any known method, e.g., addition of calcium hydroxide. Deposition of the carbonates on the grains of zeolite is prevented by pre-carbonation if required. The removal of sodium bicarbonate is of importance in ice manufacture, as it renders the product brittle and opaque. C. JEPSON.