

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JUNE 5 and 12, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Applications of the phthaleinoscope.** P. BRUÈRE (Ann. Falsif., 1930, 23, 532—534).—Further applications to those already described (A., 1930, 731) are to the determination of the alkalinity of liquid manures and the acid value of oils. E. B. HUGHES.

**Continuous control of salt content of boiler feed-water by means of visual conductivity tests.** E. ROTHER and G. JANDER (Z. angew. Chem., 1930, 43, 952—954).—A form of conductivity apparatus, which permits the continuous reading of electrolytic concentration of solutions over a wide range, is described. It is particularly useful in determining the salt content of boiler feed-water, in which case the concentration range which can be examined corresponds to 0.50—50.0 g. Cl/litre. E. S. HEDGES.

**Nomogram for use in gas analysis.**—See II. **Concentration by submerged combustion.**—See VII. **Testing of metallic materials.**—See X.

### PATENTS.

**Open-hearth furnace.** S. NAISMITH (U.S.P. 1,778,505, 14.10.30. Appl., 31.7.26).—A reverberatory furnace is constructed with the whole of one side wall in the form of doors without even supporting columns to obstruct access. B. M. VENABLES.

**Recuperative soaking pit.** G. R. McDERMOTT, Assr. to CHAPMAN-STEIN FURNACE Co. (U.S.P. 1,777,125, 30.9.30. Appl., 4.4.28).—The main exhaust ports from the pit are in the same wall as the inlets, so that the bulk of the gases take a horse-shoe course; to enable the furthest portion of the pit to be equally heated, auxiliary ports are placed in the opposite wall and the gases from these are led through passages to a point in the recuperator which is subjected to greater suction than the main inlet to the recuperator, *i.e.*, to a point nearer the chimney. B. M. VENABLES.

**Rotary kiln.** R. W. P. HORN and J. H. NISSLEY (U.S.P. 1,779,626, 28.10.30. Appl., 5.12.28).—The claims refer to means for the outlet of gases and inlet of material at the upper end of the kiln. The end of the kiln is closed except for a minor aperture through which the material is blown by compressed air. The gases are exhausted through two apertures in the cylindrical wall which, though at opposite ends of a diameter, are not opposite longitudinally and are provided with surrounding walls within the kiln to prevent exit of material. The compressed air admitted with the feed completes the combustion of partly burnt fuel. B. M. VENABLES.

**Sintering machine and the like.** E. CUDDIHY (U.S.P. 1,777,398, 7.10.30. Appl., 5.6.28).—A method

of lubricating the sliding pallets of a sintering machine is described, the oil being delivered by compressed-air ejectors. B. M. VENABLES.

**Device for the distribution of charges in a shaft furnace.** W. W. TRIGGS. From Soc. ANON. AWANS-FRANÇOIS (B.P. 345,121, 11.4.30).—Adjustable inclined blades are placed beneath the bell of the charging hopper. B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. P. COULSON (B.P. 345,583, 16.6.30. U.S., 14.6.29).—In a boiler of the type described in B.P. 319,220 (B., 1931, 139), a device is inserted in the top end of each Field tube which causes a substantial separation of liquid and vapour while they are still within the tube; in this way the bulk of the liquid is made to circulate rapidly within the tube without leaving it. B. M. VENABLES.

**Heat exchange.** C. B. GRADY, Assr. to METROPOLITAN ENG. CORP. (U.S.P. 1,779,538, 28.10.30. Appl., 24.1.27).—An exchanger suitable for transferring heat from a hot fluid, *e.g.*, gases of combustion, simultaneously to a liquid, *e.g.*, feed water, and to a gas, *e.g.*, combustion air, comprises a number of vertical, double-concentric tubes, the hot fluid flowing downwards through the annular spaces, the liquid upwards through the cores, and the gas transversely and zig-zag upwards outside the tubes. B. M. VENABLES.

**Heat-exchange apparatus.** F. HEATHER (B.P. 345,279, 17.9.29 and 11.3.30).—A form of baffle suitable for insertion into the passages of a heat exchanger, to improve the contact of the fluid with the walls, is described. B. M. VENABLES.

**Apparatus for heating and cooling fluids.** R. MORTON & Co., LTD., and P. ROBINSON (B.P. 344,687, 29.3.30).—In a heat exchanger of the filter-press type, the dividing walls between the fluids are formed of deeply corrugated thin plates. Sealing means are described. B. M. VENABLES.

**Tunnel drying ovens.** J. G. OLSSON and F. I. E. STENFORS (B.P. 345,531, 17.4.30).—A tunnel kiln is provided with at least two tracks with heaters are situated between them. The main circulations are transverse and are produced by individual fans driven by a common shaft, the flows being reversible by flaps. A fresh-air inlet is provided at one end and moist air is withdrawn by independent fans. B. M. VENABLES.

**Drying apparatus.** H. HAAS (U.S.P. 1,778,318, 14.10.30. Appl., 21.11.28).—In an apparatus of the type in which the material is supported on a pervious conveyor and subjected to currents of air circulated by

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

low-pressure fans, the material is first loosened up by smaller quantities of high-pressure air applied underneath the conveyor. B. M. VENABLES.

**Drying apparatus.** J. D. ULLGREN, ASST. to AKTIEBOLAGET KARLSTADS MEKANISKA VERKSTAD (U.S.P. 1,779,571, 28.10.30. Appl., 17.12.28. Swed., 10.11.27).—A bowl is covered by a hood within which is a conical baffle upon which the material is fed and under which hot gases are admitted. The material is moved from the centre to the circumference of the bowl by scrapers running on the bottom; the angle of the scraper blades can be varied by altering the height of their supporting arms. From the hood depend one or more circular baffles so that vapours may be exhausted from two or more zones at different temperatures. B. M. VENABLES.

**Drying apparatus.** G. W. RILEY, and G. SCOTT & SON (LONDON), LTD. (B.P. 345,302, 20.12.29).—Material such as powdered milk is produced by evaporation and grinding without removing it from a vacuum. The evaporation may be effected on a belt conveyor interleaved with heating plates and enclosed in a casing which is connected to a vacuum pump through a condenser. The flaked milk drops off the conveyor into other casings containing the grinding mills and connected direct to the same vacuum pump. B. M. VENABLES.

**Dryer.** F. A. and G. P. JADIN (U.S.P. 1,777,493, 7.10.30. Appl., 10.2.30).—The goods to be dried are contained in a stationary, cylindrical casing with perforated bottom; heat is applied to a solid bottom below it, and provision is made for catching condensate dripping from the cold roof. B. M. VENABLES.

**Desiccating apparatus.** J. O. B. COULLING and P. N. RYLANDER, ASSS. to OTATO CORP. (U.S.P. 1,778,310, 14.10.30. Appl., 5.12.29).—A spray-drying chamber having a roof of concrete or other rigid material is provided with an inner false roof which is constructed of flexible sheets to permit expansion and contraction. The centre part is rigidly held to the outer roof and supports the spraying device and apertures for admission of drying air. B. M. VENABLES.

**Drying of grass, peat, and other divided materials.** N. TESTRUP, O. SODERLUND, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 344,762, 1.11.29, 17.1.30, and 16.7.30).—The material is conveyed through a sinuous conduit in a current of heated air or gases of combustion. The speed is so high (10–25 m./sec.) that an initial temperature of 800–1100° may be used without harm to the material. The conduit is designed to prevent the formation of pockets of stationary material. B. M. VENABLES.

**Temperature-regulating means.** A. A. STOW and E. H. P. JONES (B.P. 344,481, 28.11.29).—A thermostat comprising concentric tubes alternately of different metals operates, through doubly magnifying levers, electric switches which control heating resistors directly or through relays. The outer tube of the thermostat is perforated to permit direct access of the hot gases to the inner tubes. B. M. VENABLES.

**Heat-insulating casings for pipe conduits.** H. SCHNELL (B.P. 344,882, \*5.12.29).—Electrical heating

elements are placed in grooves in the insulating material and adjacent to the surface of the pipe so that the steam or other vapour may be maintained in superheated condition. The jacket for the heating element may be made of mica, hard porcelain, solidified water-glass, and/or oiled paper. B. M. VENABLES.

**Refrigerating medium.** H. MANNHARDT, ASST. to W. MANNHARDT (U.S.P. 1,778,033, 14.10.30. Appl., 8.8.29).—The use of sulphur hexafluoride is claimed. L. A. COLES.

**Machine for treating materials. [Disintegrator.]** F. KUTASZEWICZ (U.S.P. 1,777,205, 30.9.30. Appl., 15.3.27. Ger., 1.2.26).—The rotor of a disintegrator acts mainly as a fan, its blades being shorter axially than the casing so that air currents can return towards the axis at the ends. Disintegration is effected by the material swirling through pockets formed in the circumferential wall, these being pear-shaped and inclined towards the material flung by the fan; the openings are at the small end. B. M. VENABLES.

**Crushing mill.** H. E. WHIPPLE (U.S.P. 1,777,061, 30.9.30. Appl., 29.10.28).—A rotating cylinder contains a number of loose, hollow, open-ended crushing rollers of decreasing diameter and placed one within the other. The material enters and leaves through opposite ends of the mill, and screens may be provided at the latter end. B. M. VENABLES.

**[Crushing or pulverising] mill.** E. H. DIMLER, ASST. to BETHLEHEM FOUNDRY & MACHINE Co. (U.S.P. 1,777,605, 7.10.30. Appl., 10.8.27).—The crushing surfaces are formed of material of the stellite type, viz., an alloy of cobalt with another member of the chromium group, e.g., tungsten, preferably applied as a welded-on lining. B. M. VENABLES.

**Grinding or crushing mills.** F. E. MARCY (B.P. 345,192, 7.1.30).—A lining for a ball mill is constructed of rolled material; the plates are wavy or corrugated, the part of the plate on the slope of the corrugations that wears most being made thicker than the rest, but any axial section is of uniform thickness. B. M. VENABLES.

**Grinding, crushing, and pulverising machinery.** G. CLARK (B.P. 344,890, 12.12.29).—A toothed rotor effects disintegration of the material in three stages, viz., against a plain or ribbed surface well clear of the rotor; against the ends and sides of curved channels intercalating with the teeth; and against grid bars. B. M. VENABLES.

**Grinding mills.** E. BARTHELMESS (B.P. 345,388, 14.1.30).—A rotor produces high-speed circular currents of air between two concentric rings and in addition drives mullers, rolls, or other positive crushing elements on the inside of the inner ring. The rotating air current effects the final grinding, and conveys away the finely-crushed material without the use of another fan; partly ground material drops out of the air current into the path of the positive crushers. B. M. VENABLES.

**Pulveriser.** C. E. BRAINARD, ASST. to WESTERN FOUNDRY Co. (U.S.P. 1,776,411, 23.9.30. Appl., 2.8.28).—The machine comprises a rotating shell containing loose, segmentally divided grinding rollers alternating with

distributing rings of bladed form. Charging and discharging is effected through hollow trunnions.

B. M. VENABLES.

**Pulveriser.** C. A. DREISBACH, ASSR. to STANDARD EQUIPMENT CO., INC. (U.S.P. 1,776,654, 23.9.30. Appl., 27.2.28).—A pair of crushing rolls, preferably fluted, are mounted so that the upper one can rise and fall and both are surrounded by a rotating drum which is provided with screening and lifting devices, and dips into a bath of water; the driving shaft and arms for the rolls are inserted through large openings in the ends of the drum.

B. M. VENABLES.

**Pulveriser.** W. W. PETTIBONE (U.S.P. 1,778,564, 14.10.30. Appl., 19.7.29).—In a disintegrator comprising a beater chamber, an inlet chamber for additional air, and one or more fan chambers in series around the same shaft, the air-inlet ports are provided with deflectors which divert coarse particles from the outer part of the whirling currents to positions near the axis, whence they re-enter the grinding chamber.

B. M. VENABLES.

**Pulverisers.** SOC. FRANÇ. DE CONSTRUCTIONS MÉCANIQUES ANC. ETABL. CAIL (B.P. 345,534, 22.4.30. Fr., 24.4.29).—A ball or pebble mill has the ground material removed by a current of air which is admitted to the lower part of the grinding mass through ports which are actually covered by it. The fixed air-inlet pipe inserted through the trunnion communicates only with ports that happen to lead to that part of the mass that has the most kinetic energy, and the air stream carries the feed material with it.

B. M. VENABLES.

**Pulverising apparatus.** CHAUFFE ET COMBUSTION, SOC. ANON. (B.P. 345,465, 5.3.30. Belg., 6.3.29).—A conical grinding element is gyrated in a conical casing in such a manner that its axis generates a cone; a spherical portion attached round its apex works in a corresponding spherical recess in the casing, and the point of oscillation lies on or near the spherical surface.

B. M. VENABLES.

**Screening plant.** V. REYNOLDS and J. MASEFIELD (B.P. 344,673, 13.3.30).—The use of rubber bands on the tyres and/or rollers of a rotary screen is claimed.

B. M. VENABLES.

**Separation of dry materials.** C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 343,946 and 344,802—4, [A] 19.8.29, [B—D] 28.8.29).—Devices in which a bed of the mixed materials is kept fluid by air currents flowing upwards through a pervious support are described. In (A) a number of fingers are provided upstanding from the deck of a reciprocating table or dependent from a grid above it, the fingers in either case partaking of the movement of the table and extending right through the bed of material. In (B) the device comprises an inclined conveyor, or rakes running over an inclined surface. The feed is near the top of the slope, the heavy material being dragged upwards and the light running downwards by gravity and being discharged over a weir at the foot of the slope. In (C) a substantially level trough has two runs of a single conveyor or of two separate conveyors running through it at different levels and moving the light and heavy material to opposite ends. In (D) a cylindrical tank is provided with a

pervious diaphragm above which are rotated arms, curved in opposite directions in different strata and delivering the lighter material to the circumference and the heavier to a central outlet; the feed is also central at a height which is adjustable above the edge of the tank.

B. M. VENABLES.

**Separation of dry materials.** I. L. BRAMWELL, and BIRTLEY CO., LTD. (B.P. 345,275, 10.9.29).—A pneumatic shaking table is supplied with a steady current of air which is rendered pulsating as it passes through the deck by forming the bottom of the air chamber as a bellows and reciprocating it in a vertical direction simultaneously with the horizontal reciprocation of the whole table.

B. M. VENABLES.

**Centrifugal machines for separating solids and liquids.** F. DUPUIS, and ATELIERS J. HANREZ SOC. ANON. (B.P. 344,658, 3.3.30).—A method of continuous discharge of solids is described. It involves raking by worms in two stages, in the second stage of which the rakes moving at centrifugal speed work adjacent to a stationary surface; the gearing also operates at high speed.

B. M. VENABLES.

**Separation of solids from liquids.** M. P. REYNOLDS, ASSR. to W. S. TYLER CO. (U.S.P. 1,775,830, 16.9.30. Appl., 7.1.27).—Sugar cane or similar material containing liquid amenable to such treatment is crushed to liberate the juice, the solids are removed by vibratory screens, and the residue is re-crushed to remove further liquor.

B. M. VENABLES.

**Apparatus for conveying powdered materials in pipes.** I. E. and E. LANHOFFER, ASSRS. to FULLER CO. (U.S.P. 1,777,043, 30.9.30. Appl., 29.10.25. Fr., 24.9.25).—When powdered material is conveyed in a stream of compressed air, expansion of the air may so disperse the powder that it settles out; to avoid this the conveying conduit is gradually contracted in the direction of flow. Means for charging the powder and air into the conduit are described.

B. M. VENABLES.

**Agitation of materials.** I. T. THORNTON and J. H. CROWELL, ASSRS. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,778,049, 14.10.30. Appl., 4.6.28).—A mixer is described which shears the material away from the wall, changes the peripheral movement to a radial one, and gives a downward motion to the centre of the mass.

B. M. VENABLES.

**Inclined mixers comprising a bowl-like rotating drum.** F. MOSER (B.P. 345,169, 12.6.30. Ger., 15.6.29). The fixed axle-pin is extended within the bowl and on it is placed a deflecting plate which catches material dropping from near the closed end of the bowl and deflects it towards the open end.

B. M. VENABLES.

**Centrifugal separators and methods of operating same.** H. W. FAWCETT (B.P. 344,464, 9.9.29, 16.10.29, and 4.7.30).—In a centrifuge adapted to the continuous separation of solids from liquids, the waste of lighter liquid is prevented and the discharge of solids aided by the use of a carrier liquid of intermediate density. This is supplied to an outer zone partitioned off from the separating zone except for passages opposite the apertures for discharge of solids; the outer zone is vented over a weir which is adjustable, and the carrier liquid

is so balanced against the lighter liquid that the dividing surface comes within the transfer ports between the inner and outer zones. B. M. VENABLES.

**Centrifugal machines for separating milk or liquids of different density.** ECREMEUSES MELOTTE SOC. ANON. (B.P. 344,710, 29.4.30. Belg., 28.5.29).—An elastic restraint for the shaft of a centrifuge is described. B. M. VENABLES.

**Production of conical discs for centrifugal separators.** BERGEDORFER EISENWERK A.-G. ASTRA-WERKE (B.P. 344,473, 4.11.29. Ger., 3.11.28).—Methods of manufacture of the discs are described, involving spinning so that the conical part is thinner than are the flanges. B. M. VENABLES.

**Device for separating light liquids.** W. LINN-MANN, JUN. (B.P. 344,604, 21.1.30).—The tangential inlet of a settling vessel is continued right round the circumference in the form of a gutter open to the interior of the vessel. B. M. VENABLES.

**Filters.** H. S. HELE-SHAW, W. J. RIDGWAY, and W. N. WILLIAMS (B.P. 345,251, 14.11.29).—The elements of an edge-filter are formed of wire-gauze sheets having porous granular material attached to their edges by the natural roughness, assisted, if desired, by interleaving them with discs of solid sheet of rather larger diameter. B. M. VENABLES.

**Filters.** METALLGES. A.-G., G. MÜLLER, and E. SIEBERT (B.P. 344,502, 5.12.29).—In a filter of the type comprising a mass of active or other material restrained between perforated diaphragms, provision is made to permit any gas that may accumulate on the inlet side to pass to the outlet side through a small controllable by-pass, the incoming liquid being guided so as to sweep towards the entrance of the by-pass. B. M. VENABLES.

**Filter device.** R. M. THOMPSON (U.S.P. 1,778,342, 14.10.30. Appl., 3.1.21).—The material is filtered through two pervious conveyors running together which may approach, recede, and approach each other. During one or more of the recessions a washing or extracting liquid may be applied to the partly pressed material, and gutters are provided to catch separate filtrates so that, *e.g.*, in the pressing of fish or fish offal, the oils may be collected separately from the glue stock. B. M. VENABLES.

**Thickening filters.** J. B. VERNAY (B.P. 345,383 and Addn. B.P. 346,078, [A] 10.1.30, [B] 3.2.30. Fr., [A] 10.1.29, [B] 18.5.29).—In (A) the filter comprises a number of hollow, rotating, cylindrical elements entirely submerged in a trough of the prefilter, the whole of each element being subjected either to a vacuum for filtering or to a fluid under pressure (preferably air) to dislodge the thickened mud. In (B), in order to reduce the amount of air that has to be exhausted, to re-establish the vacuum, and to enable wet vacuum pumps alone to be used, a hollow hermetically sealed spacer is placed within each element, leaving only a small annular space for filtrate or air. B. M. VENABLES.

**Mechanical filters.** S. J. RICKS and C. E. DOUGLAS (B.P. 345,514, 4.4.30. Addn. to B.P. 322,148; B., 1930, 126).—A less expensive form of the filter is

described, in which the stirring and cleaning apparatus is applied only to the upper part of the sand.

B. M. VENABLES.

**Filtration process and apparatus.** C. T. CABRERA, ASST. to ELECTRO DIALYZER CORP. (U.S.P. 1,776,883, 30.9.30. Appl., 1.6.28).—The liquid, *e.g.*, sewage or waste liquors from pulp mills, canning plant, etc., is passed upwardly through a chamber, or preferably through a number of chambers in parallel, having open top and bottom and containing a mass of filtering material such as stranded metal which is compacted to any desired extent by a screw and pressure plate at the top and may be flushed through closable openings in the sides of the chamber; provision is made for rapidly changing the lower section independently of the upper part. B. M. VENABLES.

**Apparatus for dialysing liquids.** O. M. URBAIN, ASST. to OHIO SANITARY ENG. CORP. (U.S.P. 1,777,057, 30.9.30. Appl., 13.7.28).—The apparatus is suitable for tests on putrescible matter which may take several days. The membrane is in the form of a shallow tray that floats on the dialysing water contained in a closed vessel. The water is constantly renewed from a closed reservoir; the air, which must be admitted to permit outflow of water, is freed from oxygen by an absorbent such as alkaline pyrogallate and the dissolved oxygen is removed from the water before or during use. B. M. VENABLES.

**Distillation apparatus.** D. T. WILLIAMS, ASST. to PETROLEUM DERIVATIVES, INC., OF MAINE (U.S.P. 1,777,950, 7.10.30. Appl., 12.7.24).—The apparatus comprises a number of straight inclined tubes arranged zig-zag in a setting for heating by gases of combustion. Vapour outlets are provided at the return bends down one side of the setting and, as the apparatus is specially intended for the continuous distillation of mineral oils under high vacuum, provision is made for breaking up foam, comprising inner tubes, spaced from the walls of the outer tubes, having closed upper ends and perforations on the top. The spacer in the annular space at the lower end of any tube is arranged to permit flow of liquid through the annular space, but to confine vapour to the core. The feed is at the uppermost end, and the first tube serves only as a preheater. There may be a number of vertical rows of tubes in parallel in the same setting. B. M. VENABLES.

**Distillation apparatus.** D. T. WILLIAMS (U.S.P. 1,778,177, 14.10.30. Appl., 20.11.25).—In a distillation system, especially for mineral oils, the still and condenser are maintained at a higher vacuum than is the receiver, the difference being obtained by placing the last-named at a lower level. B. M. VENABLES.

**Fractionating tower.** J. C. BLACK (U.S.P. 1,776,061, 16.9.30. Appl., 10.11.26).—A system for the fractionation of hydrocarbons or other liquids is described. Above a main fractionating tower is an analyser producing reflux liquid and the final vapour. Below the main part of the tower are two other compartments heated by steam coils and provided with bubble trays producing final liquid fractions. B. M. VENABLES.

**Fractionating column.** R. E. WILSON and W. H. BAHLKE, ASSNS. to STANDARD OIL CO. (U.S.P. 1,778,475,

14.10.30. Appl., 6.8.27).—A form of bubbling tray is described in which the pool of liquid is held at a definite depth. B. M. VENABLES.

**Dephlegmator or bubble towers.** H. MOORE, and J. B. JOHNSON & Co. (REFINERY ENGINEERS), LTD. (B.P. 345,565, 19.5.30).—In a bubble tower, the liquid is caused to flow in a circular direction round the trays, flow across the centre being prevented by a large bubble cap. The downflow port is in the form of a radial opening protected on one side by a wall, the plate next below being rotated slightly with respect to the one above. B. M. VENABLES.

**Bubble cap for gas and liquid contact apparatus.** C. M. ALEXANDER (U.S.P. 1,777,869, 7.10.30. Appl., 18.1.27).—The cap is shaped like a hollow gear wheel in plan view, and has slot-shaped gas outlets in both the flanks and ends of the teeth. B. M. VENABLES.

**Bubble caps.** J. E. KOBERNIK (U.S.P. 1,776,032—3, 16.9.30. Appl., 5.1.28).—In (A) the inner part of a duplex cap has a closed top and the outlet for the gases is through slots in the lower part of the vertical cylindrical wall; the bubbles are caught under an attached annular cap with serrated lower edges and perforations in the top. In (B) the cap comprises a perforated dished top supported by a pair of uprights, and is formed out of a single sheet of metal. B. M. VENABLES.

**Heating and cooling of fluids and viscous liquids.** C. W. STANCLIFFE (B.P. 345,312, 20.9.29).—In apparatus of the type described in B.P. 286,757 (B., 1928, 352), the scrapers, rollers, or spiral springs are subdivided into comparatively short lengths so that they will effectively operate irrespective of slight untruth of the tubes. B. M. VENABLES.

**Apparatus for recovery of the energy transmitted to liquids in the conveyance of the same against high pressures.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 344,842, 30.8.29).—The pressure on an earlier batch of liquid is reduced, and that on new liquid raised, on opposite sides of a piston or diaphragm, the pump side being smaller than the motor side by the area of the piston rod; in cases where a diaphragm is employed, booster pumps may be used to make up friction losses. Hydraulic and electrical means are described for controlling the valves. [Stat. ref.] B. M. VENABLES.

**Apparatus for vaporisation of liquids.** F. C. CALVERT & Co., LTD., and B. LE N. FOSTER (B.P. 344,821, 7.12.29).—A liquid, such as disinfectant, is slowly dripped on to an electrical hot-plate, the resistance wire of which is insulated by means of magnesium metal (or alloy) which has been converted into hydroxide by heating in steam; claim is also made for the form of the apparatus. B. M. VENABLES.

**Concentrating and homogenising apparatus [for prefill].** E. HEIBIG, Assr. to FILTRES PHILIPPE (U.S.P. 1,778,596, 14.10.30. Appl., 14.3.29. Fr., 18.6.28).—A thickening filter for providing concentrated and thoroughly mixed feed for a rotary filter is described. The pulp dropping from vertical cylindrical vacuum elements is collected by a pair of rotating cylinders provided with vanes so that they act as a gear-wheel

pump and force the thick pulp to a further stirrer and conveyor. The cylinders not only mix and deliver the pulp, but also prevent the disturbance in the bottom of the vessel from reaching the filtering zone.

B. M. VENABLES.

**Catalytic gas reactions.** DU PONT AMMONIA CORP., Assees. of J. A. ALMQUIST (B.P. 344,576, 31.12.29. U.S., 31.12.28).—In a process involving the use of a main catalyst and a purifier catalyst, the flow of gases is in the following order: in heat exchange with, and within the same pressure-resisting wall as, but not in direct contact with, the main catalyst; through the purifier catalyst in a separate pressure-resisting vessel; through a device to remove the converted impurity and to regulate the temperature; through the main catalyst to the place of recovery of the desired constituents.

B. M. VENABLES.

**Apparatus for treatment of gases or mixtures of gases and vapours, in the presence of catalytic or purifying agents and for the reactivation of such agents.** COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 345,496 and Addns. B.P. 345,511 and 346,967, [A] 25.3.30, [B] 1.4.30, [C] 28.3.30. Fr., [A] 2.8.29, [B] 13.1.30, [C] 5.12.29).—In (A) the catalytic or purifying material is supported between perforated disc partitions in a cylindrical casing and the gases to be treated pass through it from end to end, but the gas for regeneration is admitted and exhausted through perforated tubes embedded in the mass; thus the gas passes transversely through it in a well-distributed manner. In (B) coils for heating or cooling are also embedded in the mass. In (C), the casing of the apparatus is formed as a parallelepiped having small width compared with length and height; the distributing roses for the regenerating gases are placed horizontally along the long sides with their apertures facing the walls, which may there be indented to act as reflectors.

B. M. VENABLES.

**Apparatus for separating and recovering vapours and gases with adsorbent carbon.** O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,777,460, 7.10.30. Appl., 2.7.24. Renewed 21.12.27).—A vertical cylindrical casing is divided into three compartments by vertical foraminous walls: the outer compartments serve as inlet and outlet headers for the various reacting and regenerating fluids, and the inner contains the active carbon in which are embedded heating-cooling coils served from headers formed in the lower end of the shell. B. M. VENABLES.

**Separation of [acidic] gases.** GIRDLER CORP. (B.P. 344,786, 4.12.29. U.S., 4.12.28).—Gases such as carbon dioxide, sulphur dioxide, and/or hydrogen sulphide are absorbed by definite chemical combination in a liquid which is afterwards regenerated by heat. Suitable absorbents are mono- and di-ethylamine and "triethanolamine"; the last-named has the advantages that it is a viscous non-volatile liquid soluble in water and its salts are decomposed at about 100°.

B. M. VENABLES.

**Gas washer.** G. W. VREELAND (U.S.P. 1,776,644, 23.9.30. Appl., 24.2.28).—The apparatus comprises a tower containing a number of trays and lifting-spraying

cones. Moisture eliminators may be placed in an exit passage wrapped round the tower. B. M. VENABLES.

**Gas scrubber.** A. G. MCKEE (U.S.P. 1,778,426, 14.10.30. Appl., 15.3.26).—The apparatus comprises a tower containing centrifugal devices which spray the liquid to the walls, from which it is collected and re-sprayed. B. M. VENABLES.

**Apparatus for treatment of industrial gases.** COMP. GÉN. DE CONSTRUCTION DE FOURS (B.P. 345,457, 1.3.30. Fr., 2.3.29).—The gas is caused to pass through either a ring of jets or a narrow annular orifice and then to impinge on a film of liquid spread centrifugally over the surface of a rotating dish; the flow of the gas is inwards to a central exhaust passage, and therefore countercurrent to the film of liquid, this film being also sufficiently strong to prevent puncturing by the jets of gas. The apparatus is suitable for the chemical as well as physical removal of a constituent or impurity of the gas. B. M. VENABLES.

**Centrifugal means for treating [separating dust from] gases.** W. W. TRIGGS. FROM INTERNAT. PRECIPITATION Co. (B.P. 344,421, 28.11.29).—The apparatus comprises a number of vortical separating chambers, of which the length is many times the diameter, operated in parallel and having common inlet, outlet, and dust-collecting chambers. The clean air outlets are  $\frac{1}{2}$ — $\frac{3}{4}$ , and the dust outlets are tapered down to  $\frac{1}{4}$ — $\frac{3}{8}$ , of the diameter of the largest part of the cyclone chambers. It is stated that a double vortex is formed, the outer one producing a downward force on the dust many times that of gravity, and the inner producing an upward flow of clean air which originates in the dust-settling chamber at the bottom. B. M. VENABLES.

**Gas pumps particularly for use in gas-analysis apparatus.** AKTIEBOLAGET CARBA, ASSEES. OF PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 344,952, 14.12.29. Swed., 17.12.28).—A water-operated suction pump acting on the broken-column system is described. B. M. VENABLES.

**Apparatus for solidifying gases.** H. B. RUDD (B.P. 345,229, 14.11.29. U.S., 17.11.28).—Liquid carbon dioxide or other gas to be solidified is passed through a cooling coil and expansion jet into a chamber lined with a cartridge of hard rubber or other smooth non-conducting material. The gas produced by the expansion passes over the cooling coil, and when the chamber becomes full of solid the increase of pressure operates an automatic valve to cut off the supply of liquid. B. M. VENABLES.

**Lubrication of steam turbines.** G. W. BURKHARDT, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,778,447, 14.10.30. Appl., 16.3.27. Ger., 13.10.26).—The lubricant comprises a mixture of a refined mineral oil distillate with, e.g., 3% of high-molecular alcohols of wool grease. L. A. COLES.

**Hydraulic-brake fluid.** R. M. BAGLEY, ASSR. to R. M. HOLLINGSHEAD Co. (U.S.P. 1,779,460, 28.10.30. Appl., 8.3.29).—A mixture of equal volumes of ethylene glycol monoethyl ether and castor oil is claimed; part or all of the latter may be replaced by glycerin. A. R. POWELL.

**Charge for fire extinguishers.** C. A. THOMAS and C. A. HOCHWALT (U.S.P. 1,777,339, 7.10.30. Appl., 12.11.26).—A charge for anti-freeze fire extinguishers for use at temperatures down to  $-40^{\circ}$  comprises a solution containing 25% of sodium potassium carbonate, 36% of ethylene glycol, and 39% of water in one portion of the receptacle and a solution of chlorosulphonic or similar acid in the other. A. R. POWELL.

**Chemical thermoscope composition.** T. L. DAVIS, ASSR. to W. O. SNELLING (U.S.P. 1,777,353, 7.10.30. Appl., 19.12.28).—A solution of 1 g. of hexapyridine cobaltous cyanate in 100 c.c. of chloroform to which are added 16 c.c. of pyridine is pink below  $20^{\circ}$ , lilac at  $20$ — $30^{\circ}$ , violet at  $30$ — $40^{\circ}$ , and blue above  $40^{\circ}$ . With increasing amounts of pyridine the colour changes occur at higher temperatures, and with less pyridine at lower temperatures. A. R. POWELL.

**Reducing the atmospheric moisture content in confined spaces.** R. A. COCK (B.P. 345,358, 23.12.29).—A pad impregnated with a 1:3 solution of calcium chloride is used to prevent steaming of shop windows etc. B. M. VENABLES.

**Container for corrosive materials.** R. M. MEIKLEJOHN, ASSR. to GEN. CHEM. Co. (U.S.P. 1,778,532, 14.10.30. Appl., 24.8.27).—The invention relates to the closure of a lined tank. The non-corrodible lining, or a collar-like extension of it, is brought out through the bung hole and bent back over the edge of the mouth, which is, if necessary, reduced in thickness so that the lining lies below the roots of the screw thread if such be used. The closure is completed by a female cap lined with a disc of the same material, no additional gasket being provided. B. M. VENABLES.

**Polarimeter indicator.** L. MEYER (U.S.P. 1,777,216, 30.9.30. Appl., 11.2.29. Ger., 20.1.28).—The graduated drum is geared to the polarimeter proper by worm and wheel, and is provided with two scales each of which reads in both directions: one is graduated in plus and minus degrees of angle; the other, for use in urine analysis, is marked in percentage of sugar on the positive side and percentage of albumin on the negative. To prevent confusion as to positive and negative and to count complete revolutions of the worm or drum, a pointer is attached to the wheel or polarimeter proper. B. M. VENABLES.

**Continuously indicating and recording viscosimeters.** J. L. HODGSON and H. E. DALL (B.P. 345,210, 14.12.29).—A continuous sample of the liquid is brought to a standard temperature and caused to flow under a constant head through an orifice with turbulent (or mixed turbulent and laminar) flow into a vessel having a capillary outlet giving purely laminar flow. The amount of liquid accumulating in the second vessel under these conditions will be a measure of the head producing the discharge, and hence of the viscosity; the weight is determined by mounting the vessel on a scale beam. In each case the flow from the outlet is in a horizontal direction to avoid effects of momentum. Alternatively to the first vessel and orifice a pump having constant discharge may be used. B. M. VENABLES.

**Apparatus for aerating and/or mixing liquids.** W. N. JONES (B.P. 346,455, 13.1.30).—See U.S.P. 1,765,338; B., 1931, 140.

**Furnaces for cremating or incinerating.** K. LUDWIG (B.P. 344,946, 16.12.29).

**Filtering device for liquids under high pressure, particularly fuel oil for internal-combustion engines.** F. V. HAUGSTED (B.P. 344,932, 14.12.29).

**Tunnel kiln for charcoal.**—See II. **Filtration of suspended sediments.** Dry kiln.—See IX. **Furnace parts.**—See X. **Purification of gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Classification of Rumanian coals.** I. BLUM (Bul. Chim. Soc. Române, 1931, 31, 3—11).—Classification on the basis of geological and "chemical age" (*i.e.*, the extent of chemical transformation of the primal material) is discussed. Comparison of the elementary composition of Rumanian coals and lignites, the temperatures at which they evolve gases, and the amounts and composition of these gases, indicates that Lupeni coal is of a bituminous nature. Comparison of the effects of reagents (*e.g.*, potassium hydroxide, dilute nitric acid, mixtures of nitric and sulphuric acids) leads to similar conclusions. The contents of bitumen, lignin, humic acids, and cellulose in the coals and lignites were determined by extraction with benzene-alcohol mixture, sulphurous acid, ammonia, and Schweitzer's reagent, respectively; lignin and humic acids characteristic of lignite were absent in Lupeni coal. It is concluded that Lupeni coal should be classed as bituminous.

H. E. BLAYDEN.

**Influence of the constituents of Rumanian coals on the composition of the gases obtained by distillation to 500°.** I. BLUM (Bul. Chim. Soc. Române, 1931, 31, 13—19).—Four Rumanian coals were exhaustively extracted in succession with benzene-alcohol mixture and with benzene under pressure, to extract bitumens; with normal sulphurous acid to extract lignin; and with ammonia to extract humic acids. The untreated coals and the residues from each extraction were distilled up to 500° and the volume and composition of the gas evolved determined in each case. The coals richest in humic acids and lignin evolved the most gas, and the amounts of unsaturated hydrocarbons decreased on extraction of the bitumen. Carbon dioxide was evolved in least quantities from the final extraction residue, which also evolved large amounts of methane; but carbon dioxide and monoxide were evolved in large amounts from the residues rich in lignin and humic acids. Bitumen could be extracted from the ligneous coals by use of benzene-alcohol mixtures alone.

H. E. BLAYDEN.

**Low-temperature carbonisation of Donetz coals.** A. P. SCHACHNO and I. B. RAPOPORT (Brennstoff-Chem., 1931, 12, 121—122; cf. B., 1930, 42).—Six coals of varying rank from the Donetz region have been carbonised at 200°, 350°, 425°, and 500° in the aluminium assay apparatus. A flaming gas coal (moisture 3.5%, ash 11.4%, volatile matter 43.1%) proved most suitable for low-temperature carbonisation; its semi-coke was little hygroscopic, easily combustible, relatively hard,

and stable on storage. Distillation of this coal at 500° yielded 14.1% of tar and about 8% of gas.

A. B. MANNING.

**Lignite III. Analysis of lignite from Kankyohokudo, Korea.** R. ODA (J. Soc. Chem. Ind., Japan, 1930, 33, 398—399 B).

**Lignite. IV. Low-temperature distillation of lignite, humic acid, lignin, and cellulose.** R. ODA (J. Soc. Chem. Ind., Japan, 1930, 33, 441—444 B; cf. B., 1930, 973).—Tables and curves are given showing the evolution of gas, analysis of gas, and the proportion of coke, tar, water, and gas obtained in the distillation between 0° and 500° of lignite (mediæval lignin, cf. B., 1930, 973), humic acid I prepared from the foregoing, humic acid II prepared from dextrose by Conrad's method, lignin isolated from *Picea ajanensis* according to Willstätter, and cotton-paper. L. J. HOOLEY.

**Destructive hydrogenation in bomb-polymerisation in cracking.** S. A. KISS (Ind. Eng. Chem., 1931, 23, 315—318; cf. B., 1930, 175).—A theoretical discussion of the results obtained by Waterman and Percuin (B., 1925, 746).

C. B. MARSON.

**[Carbonisation of coal and dehydration of gas.]** H. C. SMITH (Gas J., 1931, 193, 787—794).—Results obtained by carbonisation of cleaned coal, and by gasification of the resulting low-ash coke, are briefly described. Working data and costs are also given for a gas-dehydration plant dealing with 3 million cub. ft. per day.

R. H. GRIFFITH.

**Partial dehydration of town gas.** R. L. GREAVES (Gas J., 1931, 193, 797—799).—A review of the arguments for and against drying of town gas. Labour and capital costs are given for a plant working with calcium chloride and dealing with 100,000 cub. ft. per hr.

R. H. GRIFFITH.

**Catalytic reduction of carbon monoxide to methane in mixtures of carbon monoxide and hydrogen and in illuminating gas, in presence of nickel-asbestos, whereby carbon dioxide, heavy hydrocarbons, and oxygen are removed.** H. A. J. PIETERS (Chem. Weekblad, 1931, 28, 209—210).—An experimental confirmation of the method of Larson and Whittaker (B., 1925, 400). The carbon monoxide content of carbon monoxide-hydrogen and carbon monoxide-hydrogen-methane-nitrogen mixtures may be accurately determined by hydrogenation. The results agree satisfactorily with those obtained by the ammoniacal cuprous chloride method. H. F. GILLBE.

**Nomogram for use in gas analysis.** J. SCHMIDT (Chem. Fabr., 1931, 137—138).—The nomogram is constructed to enable methane and hydrogen content to be read separately from the volume contraction and carbon dioxide absorbed after the combustion of an aliquot part of the gas sample with air. Small values such as may be obtained for carbon dioxide absorption after the combustion are multiplied by 10 or 100 in order to yield accurate results.

C. IRWIN.

**Aqueous liquors from low-temperature carbonisation of coal. II.** G. T. MORGAN and A. E. J. PETTET (J.S.C.I., 1931, 50, 72—74 T).—*o*-Cresol, *m*-cresol, homocatechol, isohomocatechol, resorcinol, and quinol

were isolated from the liquor produced at 625° from a Warwickshire coal, and in the same liquor determinations were made of the forms in which sulphur occurs. In a liquor obtained from Dalton Main coal at 450°, phenol, *o*-cresol, pyrocatechol, resorcinol, quinol, and acetic acid were identified. Comparisons are drawn between liquors produced from several coals at 625°, and the total phenolic content is shown to depend on the oxygen content of the coal used. In comparing liquor from high-temperature carbonisation with that from low-temperature carbonisation, it is found that the former has a lower total phenolic content, but a higher content of phenol itself.

#### Determination of the paraffin content of asphalts.

W. VON PIOTROWSKI and H. BURSTIN (*Petroleum*, 1931, 27, Asphalt, [3], 3—7).—The paraffin contents of three samples of asphalt (obtained by concentration of a paraffin-free mineral oil, by air-oxidation of the residues from the cracking of mineral oils, and by concentration of a paraffin-containing mineral oil, respectively) were determined in three independent laboratories by (a) the Engler-Holde, (b) the Marcusson, and (c) the Schwarz methods, details of which are given. A comparison of the results indicates that method (c) is unreliable on account of the disturbing influences of the high temperatures used, that method (b) is too complicated for technical purposes and gives results difficult to reproduce, and that method (a) is simple and rapid, and gives relatively good reproducibility of results. On the basis of the results, the Mineral Oil Section of the Polish Standardisation Committee have accepted the Engler-Holde method as standard. H. E. BLAYDEN.

#### Cracking of Rangoon paraffin in vapour phase.

J. W. HEYMANS (*J. Inst. Petroleum Tech.*, 1930, 16, 661—677).—Rangoon paraffin (av. mol. wt. 407) was cracked under vacuum at 450°, 600°, and 700°. The products were examined for quantity, mol. wt., bromine absorption, m.p., and refractive index. At 450°, 2.1 mols. per mol. of paraffin were obtained, 6 mols. at 600°, and 13 mols. at 700°. Splitting takes place at the end of the molecules and the yield of unsaturateds increases with rise of temperature. A certain amount of polymerisation takes place at the lower temperatures. At 700° the non-gaseous products are almost entirely unsaturated. T. A. SMITH.

#### Cracking and condensation of hydrocarbons.

II. Acetylene. E. BERL and K. W. HOFMANN (*Z. angew. Chem.*, 1931, 44, 259—261; cf. *A.*, 1931, 596).—In presence of carbon as catalyst, acetylene polymerises at 600—700° to aromatic hydrocarbons, with high yield. Metallic catalysts, and those containing silica, stannic oxide, or calcium oxide, are unsatisfactory, whilst carbon catalysts of large active surface promote decomposition of the acetylene and formation of methane. The yield of liquid products of low b.p., suitable for use as fuel, is increased in presence of water vapour, a 98.8% conversion being obtained at 740° without appreciable loss of activity of the catalyst even after prolonged use. H. F. GILLBE.

Determination of the gum content of cracked spirits. W. LITTLEJOHN, W. H. THOMAS, and H. B. THOMPSON (*J. Inst. Petroleum Tech.*, 1930, 16, 684—695).

—As the present methods were found to give discordant results for samples of the same spirit, the following standard method of carrying out the test has been developed. A hemispherical glass dish (wt. 20 g., diam. 7 cm., depth 3.5 cm.) is placed on a steam-bath with an arrangement for giving a standard current of air over the surface of the spirit. The sample (25 c.c.) is evaporated for 1—1¼ hrs. If the sample yields more than 11 mg. of gum, the test is repeated with a fresh diluted sample which will give not more than this weight of gum. Gum-free spirit must be used for blending. Precise details are given in the original. T. A. SMITH.

Lubricating fractions of Cabin Creek (W. Va.) petroleum. Action of solvents on the heavy constituents of petroleum. H. M. SMITH (*U.S. Bur. Mines, Tech. Paper*, 1930, No. 477, 48 pp.).—Lubricating oil stock was prepared from the crude oil by distillation in steam after topping by natural gas, and removal of wax by cold-settling following addition of acetone-*sec*-butyl alcohol mixture. The lubricating stock was then repeatedly extracted with acetone at 37.8° until 60 fractions had been taken, and then a further 35 fractions were obtained at 50° as the solubility had fallen too low at the lower temperature for satisfactory extraction. A further six fractions were then extracted by ethyl acetate at 21.1°. The physical constants and empirical formula of each fraction were determined, and the fractions were further examined by fractionation with acetone and vacuum distillation. The tables of data show that the lubricants are roughly divided into two types by selective extraction with acetone. The more soluble portions have relatively high sp. gr. and C:H ratio, low mol. wt., and low or medium viscosity, the less soluble portions having the converse of these properties. For paraffin-base crude it is shown that sp. gr., refractive index, sulphur content, and carbon residues are dependent on  $x$  where  $C_nH_{2n+x}$  is the empirical formula of the oil. The other properties are functions of the molecular constitution.

[With F. W. LANE, I. H. NELSON, and J. M. DEVINE.] Extractions on 3 crude oils were carried out with acetone and with ethyl alcohol. The extraction tends to divide the oils into two types, that extracted first being the more naphthenic in its qualities. Distillation alone gives only a series of oils of increasing density and viscosity. Distillation is necessary, however, for fractionation after the initial separation into types has been made by means of solvents. T. A. SMITH.

Desulphurisation of Tschusovskie Gorodki petroleum distillates, using silica gel. G. I. TSCHUFAROV and M. R. LITOVTSCHENKO (*J. Chem. Ind., Moscow*, 1931, 8, 65—68).—Silica gel removes only mercaptans from petroleum distillates; by a combination of treatment with sulphuric acid and with this adsorbent 70% of the sulphur can be removed from this material. R. TRUSZKOWSKI.

Determining the viscosity of oils by the Tausz falling-ball viscosimeter. F. GEIGER (*Petroleum*, 1931, 27, 209—214).—After multiplying the time taken by the ball to fall, as measured by this instrument, by a factor, the result differs by 0—5% from that obtained by Ostwald's method. The figures obtained vary with



(a) the weight of the ball, (b) the oils used, and (c) temperature and the length of tube. Neither the Stokes nor the Ladenburg formula is sufficiently accurate, but these formulæ may be made more accurate by using them in conjunction with a factor. Experiments are still in progress to determine the viscosity of turbulent oils and liquids.

E. DOCTOR.

**Viscosity-temperature relations of lubricating oils.** W. L. BAILLIE (J. Inst. Petroleum Tech., 1930, 16, 643—660).—The equations  $\log A = m \log \theta + K$  and  $\log V = n \log \theta + C$ , due to Eckart (Day, "Handbook of the Petroleum Industry," 1, 390), in which  $A$  and  $V$  are the absolute and kinematic viscosities and  $m$ ,  $n$ , and  $C$  are constants, agree to within  $\pm 2.5\%$  with results obtained experimentally. The expression is a special case of a general expression due to Slotte. The values of the constants for different oils are given. The values of  $m$ ,  $n$ , and  $C$  increase with the viscosities of the oils,  $C$  being always positive and  $m$  and  $n$  negative. The expression  $\log (\bar{V}_1/\bar{V}_2) = \rho \log \theta_1/\theta_2$  is more convenient for determining the viscosity-temperature relations, where  $V$  may be either the absolute or kinematic viscosity and  $\rho$  denotes  $m$  or  $n$ , depending on the basis of calculation adopted;  $\theta$  is measured in °F. The relation of the time of efflux of an oil from a short-tube viscosimeter to its kinematic viscosity is given by the expression  $V = AT - B/T$ . Since  $V = \theta^n$ , where  $\log w = C$ , then  $AT - B/T = \theta^n$ . This equation gives the relation between efflux time and temperature and agrees with experiment. For the Redwood viscosimeter the values of  $A$  and  $B$  are 0.0026 and 1.715, respectively. The effects of inaccurate temperature and time measurements with the Redwood instrument are shown to be considerable with oils of low viscosity and with more viscous oils at the higher temperatures. A table is given showing the kinematic viscosities of oils and the Redwood efflux times.

T. A. SMITH.

**Detonation as affected by mineral lubricating oils.** R. O. KING and H. MOSS (Engineering, 1931, 131, 1—4).—The action of various typical constituents of mineral lubricating oil blends, including distillates of naphthenic, asphaltic, and paraffin-base crudes and a cylinder stock and spindle oil, on fuels improved in anti-knock value by the addition of "ethyl fluid" or benzol has been determined at normal and high induction temperatures. For a fuel the highest useful compression ratio (H.U.C.R.) of which has been increased by the addition of ethyl fluid, consisting of a 3:2 mixture (by vol.) of lead tetraethyl and ethylene dibromide, the detonation-inducing effect is greatest with the asphaltic-base oil, being represented by a fall of 0.75 compression ratio for 12% of oil in the fuel-oil mixture. The behaviour of the cylinder stock is exceptional and resembles that of rape and castor oils in having no deleterious effect on the detonation-delaying action of ethyl fluid. Raising the induction temperature results in the different oil effects, including that of the cylinder stock, becoming practically equal. Using a 50/50 petrol-benzol mixture, all the oils except the spindle oil were of nearly equal deleterious action, which was less at normal induction temperatures than when the fuel was given an equally high anti-knock value by

ethyl fluid, but on raising the induction temperature it became greater. At induction temperatures of 90° the oils destroy one third of the beneficial effect of benzol as against one quarter of that of ethyl fluid in similar circumstances. For a fuel consisting of cyclohexane alone the deleterious oil effect varied from one half to two thirds of that obtained for a 50/50 petrol-benzol mixture, and taking the combined oil and temperature effects at 50° induction temperature, the H.U.C.R. for the cyclohexane had fallen from 6.0 to 5.5 and for the petrol-benzol mixture to 5.10. With ordinary commercial petrols having a H.U.C.R. of 4.3—4.5 and containing up to 8% of oil in solution, the deleterious oil effect was barely measurable, even in one variety containing 20% of benzol. The deleterious effect becomes evident at higher compression ratios only.

H. S. GARLICK.

**Hydrocyanic acid.**—See VII. **Corrosion of iron by hydrogen cyanide.**—See X. **Carbolic powder.**—See XXIII.

PATENTS.

**Vertical retort with crossovers.** J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,779,958, 28.10.30. Appl., 26.8.24).—The alternate vertical chambers of a vertical retort battery are provided with a bridge block across the charge opening so that combustion gases pass up the side heating flues, through the bridge block, and down the opposite side flue. The coal is preheated by passage over the sides of the bridge block, and the flues work in conjunction with regenerators.

H. E. BLAYDEN.

**Coking oven retort.** J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,780,803, 4.11.30. Appl., 31.7.23. Renewed 27.3.30).—The heating walls of each oven of a battery contain vertical heating flues divided into groups (e.g., two), each group consisting of two sub-groups which are joined by horizontal flues and work in conjunction with separate inflow and outflow regenerators.

H. E. BLAYDEN.

**Heating of stills or coke ovens with hot gases.** CHEM.-TECHN. GES.M.B.H. (B.P. 344,920, 9.12.29. Ger., 7.12.28).—Stills or ovens which are heated by circulating hot gases in helical or circular paths are provided with a rotating propeller placed at a turning point in the path of the gas. Fresh heating gases are introduced before or after the propeller and are mixed with the circulating gas, and an amount of waste gas equal to the fuel gas introduced is by-passed to waste and preheats the fuel gas.

H. E. BLAYDEN.

**Tunnel kiln [for activating charcoal].** O. L. BARNEBEY and M. B. CHENEY (U.S.P. 1,778,747, 21.10.30. Appl., 21.2.25. Cf. U.S.P. 1,541,099; B., 1925, 581).—The kiln, through which the charcoal is conveyed on trays placed on movable carriages, is separated into a lower compartment (provided with coolers) in which the carriages run, and an upper compartment (containing the trays) which receives supplies of heating, cooling, and reactive fluid along its length so that the temperature gradient rises gradually from each end to the middle section of the kiln. Airtight vestibules are situated at each end for charging and discharging the kiln, and means are provided for atomising water into the exit end

of the kiln, and for regulating the return to the kiln of the exit gases. L. A. COLES.

**Carbonisation of carbonaceous material.** E. B. EDWARDS (B.P. 344,455, 6.12.29).—Carbonaceous material which is to be subjected to heat treatment is carried through an oven or furnace in a number of metallic containers. These containers are forced from one end of the furnace to the other, in contact with each other so that the material in them cannot move freely. If desired, the containers may be made to return through the hot zone by means of a cyclic path. Means are provided for filling each vessel and for removal of the semi-coke produced; interlocking devices may be employed to ensure perfect movement through the hot zone. R. H. GRIFFITH.

**Production of carbon for absorption and decolorising.** E. URBAIN, ASS. TO URBAIN CORP. (U.S.P. 1,778,343, 14.10.30. Appl., 17.7.28. Fr., 20.7.27).—Carbon is activated by passing over it phosphorus vapour, with or without admixed steam, above 350° and washing out the absorbed reaction products. H. E. BLAYDEN.

**Manufacture of practically dry carbon.** M. GOBERT (B.P. 345,237, 10.12.29).—Carbon black, obtained by the action of an electric arc on a hydrocarbon oil, is purified by heating to 350–400°, so that it is freed from water and unchanged oil. The necessary heat can be provided by gases formed in the cracking process; if necessary, unsaturated hydrocarbons in this gas may be previously removed and used as additional raw material for production of carbon. Similarly, liquid hydrocarbons driven off during purification may be condensed and returned to the arc. R. H. GRIFFITH.

**Hydrogenation of carbonaceous materials.** IMPERIAL CHEM. INDUSTRIES, LTD., R. HOLROYD, and C. COCKRAM (B.P. 344,975, 28.12.29 and 23.10.30).—Coal is hydrogenated with the aid of massive catalysts (*e.g.*, a member of the copper sub-group or of group II, in the presence of metals of groups III–VII) in the form of strips or grids of the metals used, one of which may form a coating upon a base metal, *e.g.*, tin plate. H. E. BLAYDEN.

**Conversion of liquid carbonaceous materials into more valuable products by destructive hydrogenation.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 345,282, 15.10.29).—High-boiling hydrocarbons or tars which contain considerable amounts of asphaltic materials are subjected to a preliminary separation before hydrogenation in the presence of a catalyst, in order to avoid troubles due to deposition of pitch. The treatment consists in heating, preferably in the presence of hydrogen under pressure, so that a distillate alone comes into contact with the catalyst; the residue withdrawn from the apparatus is mixed with fresh material for further use. R. H. GRIFFITH.

**[Catalysts for] destructive hydrogenation.** H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 345,305, 20.12.29).—Catalysts for production of low-boiling hydrocarbons consist of nickel or cobalt, alloyed or coated with tin; alloys of nickel or cobalt with each

other or with iron can similarly be coated with tin. Such materials can be employed for working with oils in either the liquid or vapour phase. R. H. GRIFFITH.

**Manufacture of water-gas.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,858, 29.4.30. Addn. to B.P. 214,544; B., 1924, 549).—In the production of water-gas by treating a moving bed of granular fuel with steam, secondary air is admitted to the space immediately above the fuel bed, during the air blast, so that carbon monoxide and solid particles of fuel are completely burned before leaving the generator. For a vessel 3 m. in diam., and with a grate 7 m.<sup>2</sup> in area, 1400 m.<sup>3</sup> of primary air and 1600 m.<sup>3</sup> of secondary air are required per hr. The latter is supplied from 12 channels in the side walls, the openings in which are placed so that combustion cannot occur on the walls, but attains a maximum at a central point above the fuel bed. R. H. GRIFFITH.

**Manufacture of oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., ASSEES. OF J. A. PERRY (B.P. 344,742, 19.6.30. U.S., 27.7.29).—For gasification of large quantities of heavy oils, in association with water-gas manufacture, modifications in plant are made to avoid blockage by carbon or pitch. Twin generators are provided, having a relatively large free space above the fuel bed, which serves as a primary cracking zone. Heat is stored in the fuel bed and in an adjacent superheater, during an air-blow through both generators in parallel; during the steam-run, oil is added to the top of one generator while steam passes in at the base. The resulting gas stream is divided, part of it going through the superheater and part through the second generator, where excessive cracking occurs; after an interval this process is reversed, steam and oil being fed to the second generator. Owing to the flexibility of the method, gas of almost any calorific value can be obtained, and a further modification may include back-run steaming through the superheater. R. H. GRIFFITH.

**Production of hydrogen by decomposition [separation] under pressure of coke-oven gas.** PATENTVERWERTUNGS A.-G. "ALPINA" (B.P. 345,755, 8.2.30. Ger., 8.2.29).—The yield of hydrogen obtained by separation of coke-oven gas, by cooling under pressure after a preliminary washing with water under pressure, is materially increased by returning to the process the first gaseous fraction which escapes on releasing the pressure on the water used. This gas contains 30–35% of hydrogen. R. H. GRIFFITH.

**Production of acetylene.** J. FERGUSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 342,771, 8.11.29).—Hydrocarbons other than methane (with or without methane) are passed with large excess of steam through a tube heated above 1000° at such a rate that the time of heating of the gases does not exceed 2 sec. *E.g.*, oil gas passed with 10 vols. of steam at 220 litres per hr. through a 5-mm. tube heated for 22 cm. of its length to 1250° gives 0.37 vol. of acetylene per vol. of oil gas. [Stat. ref.] C. HOLLINS.

**Conversion of methane into liquid hydrocarbons by thermal treatment.** N.V. SILICA EN OVENBOUW

MIL., and C. OTTO (B.P. 344,443, 27.11.29).—An apparatus for subjecting methane to a high temperature consists of a modified coke-oven setting, in which the gas is passed through channels in the brickwork, or in which the ovens themselves are used as contact chambers. Heating is provided by the combustion of gas in suitable adjoining chambers, and the process may be operated so that carbonisation of coal is carried out in the usual way, conversion of the methane obtained being achieved in another part of the same plant. Heat exchange between ingoing and outgoing gases increases the economy of the system, and means for condensation of any liquid products are provided. Hydrogen which is formed as a by-product may be added to that separated in the primary treatment of the carbonisation gases.

R. H. GRIFFITH.

**Gas calorimeters.** S. W. PARR, Assr. to BURGESS-PARR Co. (U.S.P. 1,779,371—2 and 1,778,958, 21.10.30. Appl., [A] 9.3.26, [B] 26.8.27, [C] 19.4.28).—(A) The liquid (e.g., water) used in the calorimeter is preheated to a constant temperature in a storage vessel, and the gas to be tested is heated to the same temperature by passage through heat-interchange coils immersed in the vessel. Equal volumes of gas and liquid, respectively, are made to flow alternately into each of two vessels, from which equal volumes of gas (at constant pressure) and liquid are alternately withdrawn through suitable valves, the gas being burned in a heat exchanger through which the liquid flows. The volumes of liquid and gas used may be measured. The water leaving this heat exchanger passes into a mixing chamber where a stream of air bubbles thoroughly mixes it. Means are provided for observing the temperature of the liquid entering the heat-exchange vessel and of the liquid leaving the mixing chamber, and the corresponding heat of combustion of the gas may be continuously recorded. (B) The liquid in use is continuously circulated from the heat exchanger and mixing chamber over cooling coils through which cold water flows and into the storage tank, where the gas to be tested is brought, by heat exchange, to the same temperature as the liquid. The air for combustion is heated to a constant temperature by passage through a vessel containing the preheated liquid, which, if water is used, ensures constant humidity of the air. (C) The cooling and heating sections of the calorimeter and valve mechanism are mounted between heat-insulating partitions to insulate them from each other and from the atmosphere. Improvements are claimed in the electro-mechanical control of the valves and measuring vessels.

H. E. BLAYDEN.

**Gas calorimeter.** J. P. FISHER, Assr. to BURGESS-PARR Co. (U.S.P. 1,779,373, 21.10.30. Appl., 9.3.28).—The gas and water are metered in fixed proportions to the calorimeter by means of a screw pump in connexion with inlet and outlet chambers containing the gas and water. Pressure regulators maintain the pressures in each chamber constant and equal, and the density of the outlet gas is maintained constant with varying temperature of the water.

H. E. BLAYDEN.

**[Automatically controlled] gas production.** H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,772,641, 12.8.30. Appl., 16.5.21).—Combustible gas is obtained

by passing an air blast through a bed of carbonaceous material at a high temperature; the blast is operated electrically and is controlled by the amount of gas present in a storage holder. Means are also provided, involving a thermostatic device heated by burning a portion of the gas, for passing to waste any low-grade gas that is made on restarting the plant.

R. H. GRIFFITH.

**Oxidation of natural gas.** E. H. BOOMER, Assr. to GOVERNORS OF THE UNIVERSITY OF ALBERTA (U.S.P. 1,776,771, 30.9.30. Appl., 23.7.28).—In order to obtain alcohols, aldehydes, etc. by direct oxidation of natural gas, the latter is mixed with oxygen equivalent to half the volume of hydrocarbons present in the gas. The mixture is compressed to 60—300 atm. and passed over a catalyst at 250—500°; suitable contact materials are zinc and lead chromates, or a salt of a metal of group II or III, together with a salt of a metal of group IV, VI, or VIII.

R. H. GRIFFITH.

**Gas purifier for gas works, coke-producing plants, and the like working with dry [purifier] mass.** J. CHATWIN. From THYSSEN'SCHE GAS- u. WASSERWERKE G.M.B.H. (B.P. 345,880, 22.5.30).—The purifying material is supported in a number of double trays, mounted one above another in a vessel which is preferably of tower form, and through the centre of which passes a gas-distributing main having perforations at regular intervals. The crude gas enters the numerous small chambers by this path, penetrates upwards and downwards through the purifying material held by each double tray, and is carried into an annular space formed by the outer shell of the whole vessel. Renewal of the purifying material is carried out by withdrawing the trays, but sulphur can also be extracted, without dismantling, by treatment with carbon disulphide or a similar solvent.

R. H. GRIFFITH.

**Purification of gas from sulphur.** HUMPHREYS & GLASGOW, LTD., Asses. of W. H. FULWEILER and T. K. CLEVELAND (B.P. 345,067, 27.2.30. U.S., 17.5.29).—Organic sulphur compounds are removed from gases by eliminating the hydrogen sulphide in the usual manner and passing them over a zinc oxide catalyst (which may contain promoters such as chromium, vanadium, cerium, etc.) at 300—500° so as to convert the sulphur present into hydrogen sulphide, which is removed. Undecomposed organic sulphur compounds are eliminated by passing the gas through sulphuric acid.

H. E. BLAYDEN.

**Purification of exhaust gases.** L. VECCHIO (U.S.P. 1,775,876, 16.9.30. Appl., 12.12.25).—Waste gases from an internal-combustion engine are purified by contact with ammonia, aqueous alcohol, and a vegetable oil in successive compartments of a bubble-washer. Oily, acidic, or tarry particles are removed in the first stages, and any material that is mechanically suspended is finally held back.

R. H. GRIFFITH.

**Testing the inflammable condition of a mixture of air or oxygen with an inflammable gas or vapour.** N. J. THOMPSON (U.S.P. 1,779,569, 28.10.30. Appl., 14.9.26).—The inflammable properties of mixtures of air and gases or vapours are tested continuously by passing the mixture over a heated surface or suitable

catalyst and making the rise in temperature resulting from combustion to indicate (*e.g.*, by means of a thermocouple and suitable calibration) the heat developed in terms of a fraction of that produced by the combustion of mixtures of hydrocarbons at the lower explosive limit. The last-named amount of heat is stated to be the same for all hydrocarbons. The indicator may be adapted to control automatically the amount of air in the mixture tested.

H. E. BLAYDEN.

**Distillation of coal tar.** BARRETT Co., Assees. of G. T. GAMBRILL, JUN. (B.P. 345,654, 21.11.29. U.S., 30.11.28).—The lower-boiling constituents of a coal tar are continuously removed, to produce a pitch of any desired characteristics, by means of a hot stream of inert gas which is passed through the heated liquid. The necessary apparatus comprises a tube still, where the tar is heated, a vessel for ensuring contact with hot gases, condensers and heat exchangers for cooling the escaping vapours and for preheating the gases, and also means for circulating the permanent gases back to the contact vessel. It is also advantageous to preheat the crude tar, and to supply fresh material as pitch and distillate are withdrawn.

R. H. GRIFFITH.

**Bitumen and tar emulsions.** J. LEBEN, and ORMUL PRODUCTS, LTD. (B.P. 344,562, 18.12.29).—Bituminous materials are mixed with emulsifying agents in such a way that the product will give a good suspension when shaken with water. The agents employed for this purpose are fatty acids and/or resin acids, together with an alkali silicate and < 9% of water. The amount of alkali present, beyond that required to neutralise the organic acids, must be 3–5 mols. per mol. of silicic acid.

R. H. GRIFFITH.

**Removal of tar acids and the like from oils.** SOUTH METROPOLITAN GAS Co., and H. STANIER (B.P. 344,614, 31.1.30).—Tar acids are extracted from mixtures which also contain hydrocarbons by treatment with an organic base which is practically insoluble in liquid hydrocarbons. This type of substance is obtained by the interaction of an alkylene oxide and ammonia, or in the form of its hydrochloride by treating an alkylene monochlorohydrin with ammonia; "triethanolamine" is particularly useful. The extract is treated with water, preferably in the presence of a weak acid such as a solution of carbon dioxide, for the separation of the tar acids, and further purification from small quantities of hydrocarbon is accomplished by distillation or by extraction with a petroleum oil. The triethanolamine is regenerated by hydrolysis of its salts.

R. H. GRIFFITH.

**Purification of water containing tar, such as water used in coke manufacture.** P. GRUHL (B.P. 345,579, 6.6.30).—Waste water produced during carbonisation of coal etc. is freed from suspended or dissolved tarry impurities by distillation and fractional condensation. For this purpose a boiler, holding the water to be treated, is connected to a tower filled with a packing of ceramic material or coke; the heavy carbonaceous substances carried by the steam are condensed more easily than is the water vapour, so that they return to the boiler and can be drawn off from time to time. Clean water is condensed at the top of the column, and, by

controlling the rate of heating, a satisfactory separation can always be obtained.

R. H. GRIFFITH.

**Purification of mineral oils, tars, their distillation products, and the like.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,738, 23.1.30).—Previous to the purification of oils by means of high-pressure hydrogenation, asphalts or resins, or substances which would polymerise under the conditions employed, are removed from the raw material by a pretreatment which consists in mixing the crude oil, at temperatures below 100°, with dilute sulphuric, acetic, oxalic, or malonic acid, or in employing polymerising catalysts such as sulphur, potassium bisulphite, zinc chloride, or aluminium chloride. At higher temperatures, preferably 350°, the same results may be achieved by using substances with a large superficial area such as silica gel, pumice, etc. Liquid or gaseous diluents can then be added to the material and the liquid or vapour obtained can be hydrogenated without any loss in activity of the catalyst due to the clogging of its surface.

R. H. GRIFFITH.

**Distillation of hydrocarbons.** BARRETT Co., Assees. of C. G. STUPP (B.P. 345,272, 18.12.29. U.S., 18.12.28).—A still for the treatment of tars, oils, etc. contains a large volume of the material to be distilled, in which a roller is immersed close to one wall of the vessel. The rapid rotation of this roller throws a film of oil against the side of the still and causes a constant fall of spray. Into the centre of this spray is passed a stream of hot gas, which supplies the heat for distillation and carries off the volatile products. At the same time, any chance of overheating of part of the still is precluded, so that no risk of coking can arise. The top of the still is preferably curved to assist in the thorough distribution of the spray; the apparatus can be worked continuously by adding fresh tar and withdrawing pitch.

R. H. GRIFFITH.

**Treating hydrocarbon oils.** C. W. WATSON, Assr. to TEXAS Co. (U.S.P. 1,780,286, 4.11.30. Appl., 1.7.27).—The oil is passed through a battery of series-connected stills provided with fractionating towers, where the oil is exposed to progressively rising temperatures. The vapours evolved from the first few of the stills of the series are condensed and collected, and divided into a smaller and a larger portion. The smaller portion is used as a reflux medium to those fractionating towers corresponding to the stills from which the vapours forming such fractions are obtained. The larger portion is introduced into heat-exchange with other hot products formed in the system and then passed through the first still in the series.

H. S. GARLICK.

**Treatment of [liquid] hydrocarbons.** C. J. FRANKFORTER, Assr. to FRANKFORTER OIL PROCESS, INC. (U.S.P. 1,780,873, 4.11.30. Appl., 27.8.29).—A gas containing hydrogen is activated by a preliminary passage over the exposed heated surface of a catalytic contact material situated in an external conduit, and then passed in a finely-divided condition in contact with an electrically heated contact surface into a hydrocarbon oil undergoing distillation in a cracking or other still. Most of the sulphur and sulphur compounds in the oil

are volatilised and can be separated from the gas by known means prior to its return to the system.

H. S. GARLICK.

**Treatment of [heavy] hydrocarbons.** E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,780,536, 4.11.30. Appl., 5.6.26. Fr., 9.4.26).—Heavy hydrocarbons, under slight pressure, are preheated by waste heat to approx. 200° and then passed through alternative atomisers into one of a pair of cracking chambers, maintained at 500—600°, containing a catalyst which aids decomposition and purification. Atomisation is effected by use of the residual gases of the process supplied under pressure. The resultant products are fractionally condensed and separated from the residual gases, which are collected; the heavy oil fractions are returned to the preheating reservoir. The coke deposited during cracking is burned from the inner surfaces of one of the cracking chambers by passing through a stream of air while cracking is proceeding in the alternative chamber, the heat generated by such combustion being employed to heat the residual gases.

H. S. GARLICK.

**Manufacture of products from hydrocarbons.** H. R. ROWLAND, Assr. to C. & C. DEVELOPING Co. (U.S.P. 1,779,402, 21.10.30. Appl., 21.11.23).—Oil vapours mixed with hydrogen are subjected at about 370° to a high-tension discharge (frequency 25—150 cycles per sec.). The vapours are then fractionated.

H. S. GARLICK.

**Cracking of hydrocarbons.** E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,777,307, 7.10.30. Appl., 4.6.29).—Raw lubricating oil stock, mixed with caustic alkali, is brought in direct contact with the vapours from a vapour-phase cracking unit in a dephlegmator. The lubricating fraction is vaporised and recovered in a second dephlegmating tower as a refined distillate. Fractions too light to be included in the petrol are returned to the cracking plant from a third dephlegmating tower.

T. A. SMITH.

**Cracking of hydrocarbons.** IMPERIAL CHEM. INDUSTRIES, LTD., and W. R. MADEL (B.P. 345,664, 24.12.29).—Tars, mineral oils, etc. are sprayed into a cracking chamber maintained at 650—750°, at a pressure not much above that in the chamber, by means of a jet of cracked gases from the process itself. The cracking chamber may be packed with a catalytic material, whilst the material to be treated may be subjected to a separate initial cracking in the liquid phase and may preferably be a middle oil from a destructive hydrogenation process.

H. S. GARLICK.

**Cracking of [petroleum] oils.** A. E. WHITE. From BETHLEHEM STEEL Co. (B.P. 345,005, 18.1.30).—The vapours from the petroleum still are fractionally condensed under pressure and the condensate is removed from the system, instead of returning it to the still. Remaining condensable vapours are condensed at atmospheric pressure. The first-named distillate may be further distilled to give high- and low-boiling fractions, the high-boiling fractions being returned to the petroleum still.

H. E. BLAYDEN.

**Hydrocarbon oil conversion.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,779,465, 28.10.30. Appl., 30.7.27).—Oil is passed through a

series of chambers in which it is brought in contact with the vapours from the preceding chamber and thus preheated. It is then passed through a cracking coil to a reaction chamber, from which vapours can be removed to the preheating chambers. The vapours from the chambers may be passed as desired through a vapour-phase cracking coil and returned to another chamber of the series.

T. A. SMITH.

**Recovery and purification of lubricating oils from mineral oils.** INDIAN REFINING Co. (B.P. 345,632, 16.9.29. U.S., 18.10.28).—A wax-bearing fraction obtained by distilling a wax-containing petroleum oil under reduced pressure is treated with acetone and the mixture allowed to separate. The upper acetone layer, containing most of the wax in solution, is drawn off. The lower layer is treated with a selective solvent having practically complete solvent action on the fraction at approx. 32° and substantially no solvent action on the solid components of the fraction at temperatures below -20°, e.g., approx. equal parts of acetone and benzol, in proportion greater than the volume of the wax-bearing fraction, and the resulting solution is cooled to below -17° in order to precipitate the wax. The dewaxed fraction is subsequently distilled (pressure < 1 atm.).

H. S. GARLICK.

**Breaking of petroleum emulsions.** M. DE GROOTE, L. T. MONSON, and (B, C) A. F. WIRTEL, Assrs. to W. S. BARNICKEL & Co. (U.S.P. 1,780,343—5, 4.11.30. Appl., 21.1.29).—Emulsions of water in petroleum are broken by agitation with (A) neutralised sulphite-cellulose waste liquor and dipropyltetrahydronaphthalene-sulphonic acid, (B) the ammonium salt of amylnaphthalenesulphonic acid or analogous compound which forms insoluble calcium and magnesium salts, or (C) a compound of the type XFRSO<sub>3</sub>H, where X is a polycyclic aromatic nucleus, e.g., naphthalene, F is a detergent-forming group, e.g., hydroxystearic acid, and R is an alkyl (e.g., propyl) group attached to the nucleus.

A. R. POWELL.

**Manufacture of liquid fuels.** COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 345,864, 2.5.30. Fr., 15.5.29. Cf. B.P. 267,512; B., 1928, 470).—In the process described in the prior patent for obtaining low-boiling hydrocarbons by low-temperature carbonisation of coal, followed by catalytic hydrogenation of the product at ordinary pressures, a second hydrogenation of all the light oil obtained either by distillation, cracking, or primary hydrogenation is effected. The resulting spirit is suitable, without further refining, for use as a motor fuel.

R. H. GRIFFITH.

**Disposal of sulphuric acid materials [from petroleum refining].** C. HINDE (U.S.P. 1,776,070, 16.9.30. Appl., 3.12.28).—Waste sulphuric acid from petroleum refining is sprayed through a heated chamber in such a way that the water is evaporated in the cooler parts and the carbonaceous material burned to carbon dioxide and sulphur dioxide in the hotter parts; finally, in the hottest zones, the remaining sulphur trioxide is caused to dissociate into sulphur dioxide and oxygen. The gases may be passed to a sulphuric acid plant or discharged into the air from a high stack.

A. R. POWELL.

**Filtering material [for purifying and bleaching mineral oils].** H. BLUMENBERG, JUN. (U.S.P. 1,777,546, 7.10.30. Appl., 10.8.27).—A finely-ground (20–100-mesh) mixture of 65 pts. of zinc sulphate crystals, 35 pts. of calcium hypochlorite, and 10–30 pts. of kieselguhr is claimed.  
A. R. POWELL.

**Regulation of the gas supply to coke ovens.** N.V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., G.M.B.H. (B.P. 346,254, 29.9.30. Ger., 28.9.29).

**Heat exchanger. Drying of peat etc. Separation of dry materials. Distillation apparatus. Fractionation tower. Bubble caps. Treating gases. Gas scrubber. Turbine lubrication.**—See I. **Fibre board.**—See V. **Oiling of fibres.**—See VI. **Metal aminocarbonyls. Catalyst.**—See VII. **Water-proofing concrete.**—See IX. **Protection of pipes etc. Inhibitor for pickling baths.**—See X. **Spray oils.** See XVI. **Removing carbon monoxide from gases.**—See XXIII.

### III.—ORGANIC INTERMEDIATES.

**Synthesis of methyl alcohol, using copper, zinc oxide, and chromic oxide as catalysts.** V. A. PLOTNIKOV, K. N. IVANOV, and D. A. POSPECHOV (J. Chem. Ind., Moscow, 1931, 8, 119–126).—The most active catalyst for the reduction of carbon monoxide by hydrogen contains 70 at.-% Cu, 27.5 mol.-% ZnO, and 2.5 mol.-% Cr<sub>2</sub>O<sub>3</sub>, whilst the greatest yields (98%) of liquid products (chiefly methyl alcohol) are obtained when the proportions 60 at.-% Cu, 35 mol.-% ZnO, and 5 mol.-% Cr<sub>2</sub>O<sub>3</sub> are employed. Increase in the hydrogen content of the reaction mixture above the theoretical amount necessary for the reaction  $\text{CO} + 2\text{H}_2 \rightleftharpoons \text{CH}_3\cdot\text{OH}$  leads to increase in the velocity of reaction and of the proportion of carbon monoxide converted into alcohol and methane, and to decrease in the amount converted into carbon dioxide. The optimal temperature is 265°; above 280° the yield falls owing to increased formation of water, carbon dioxide, and methane, whilst below 250° the velocity is very small. The addition of sodium silicate increases the stability of the catalyst, but decreases its activity.

R. TRUSZKOWSKI.

**Detection and determination of methyl alcohol.** L. SEMICHON and M. FLANZY (Ann. Falsif., 1931, 24, 80–87).—The present methods of determination based on oxidation to formaldehyde are unsatisfactory, owing to further oxidation to formic acid or even to carbon dioxide. A suggested improvement lies in the use of chromic acid to determine the oxygen equivalent of the solution. This amount of chromic acid is added to another part of the solution for the oxidation of the methyl alcohol to formaldehyde, which takes place to the extent of 32% under the conditions specified in the paper. The formaldehyde is characterised and may be determined colorimetrically by means of the red colour formed with phenylhydrazine and potassium ferricyanide in presence of hydrochloric acid.

E. B. HUGHES.

**Tests for impurities in ether. II. Acetaldehyde. III. Acetone.** G. MIDDLETON and F. C. HYMAS (Analyst, 1931, 56, 238–245).—II. Various

tests for aldehyde show the following sensitivity (parts per million): ammoniacal silver nitrate 1000, potassium hydroxide 40, diazobenzenesulphonic acid 3, Schiff's reagent with the addition of 0.1% of pyrogallol 2, Tollens' reagent 1, Nessler's reagent 1, Nessler's reagent diluted (1:4) 1. (Cf. B., 1928, 425.)

III. Reagents for acetone show: mercuric chloride and baryta or hydroxylamine and hypochlorite 100, sodium nitroprusside 20–25, acid mercuric sulphate 5, vanillin (Dutch pharmacopœia) 1, Nessler's reagent 0.5 (upper limit 20). Schiff's reagent, modified as above, is recommended as an official test for aldehyde, and the vanillin test is suggested for acetone, in order to combine specificity with sensitivity.  
T. McLACHLAN.

**Volumetric determination of small quantities of formic acid in acetic acid.** R. G. C. OLDEMAN (Pharm. Weekblad, 1931, 68, 379–380).—The use of 0.5N-alkali for titration of the hydrochloric acid liberated in the Fuchs method (A., 1929, 1323) is troublesome if less than 2.5% of formic acid is present, as the mercuric oxide formed at the end-point is difficult to observe and, further, it obscures the colour change of an indicator. If an excess of sodium chloride be added to the solution mercuric oxide does not separate, owing to complex formation, and by titration (phenolphthalein) 0.2% of formic acid may be determined.

H. F. GILLBE.

**[Manufacture of] 1:3:5-trinitrobenzene ("benzite").** L. DESVERGNES (Chim. et Ind., 1931, 25, 3–17, 291–306).—1:3:5-Trinitrobenzene is most cheaply and conveniently manufactured by oxidation of 2:4:6-trinitrotoluene with chromic acid. Other methods studied are reduction of picryl chloride, nitration of *m*-dinitrobenzene, nitration of benzyl chloride. For the manufacture of picryl chloride 1-chloro-2:4-dinitrobenzene is dissolved in 2 pts. of 40% oleum and heated for 3 hrs. at 150° with 5.6 pts. of 100% sulphuric acid and 1.5 pts. of 48% nitric acid, the process being an improvement on the Griesheim method (G.P. 78,309); a similar yield and quality are obtained by the Frankland and Garner method (J.S.C.I., 1920, 39, 257  $\tau$ ). The picryl chloride is reduced to 1:3:5-trinitrobenzene (64% yield) by heating for 1 hr. with 0.32 pt. of copper powder in 3 pts. of 96% alcohol and for a further 0.5 hr. after adding 1 pt. of water. The crude product is crystallised from hot benzene; the mother-liquors contain 2:4-dinitro- and 2:4:6-trinitro-phenol. In Köhler's method (private communication) for the oxidation of trinitrotoluene to trinitrobenzoic acid, the starting material is dissolved in 16.6 pts. of 92% sulphuric acid and 1.58 pts. of powdered potassium dichromate are added in small portions during 3 hrs. at 40° with constant stirring. The washed trinitrobenzoic acid is dissolved in 2–2.2 pts. of water at 90°, and the solution, filtered at 70–75°, is boiled for 5–6 hrs., the trinitrobenzene being skimmed off the surface at intervals; the yield is 73% (calc. on the trinitrotoluene). Oxidation with nitric acid (1 pt. of trinitrotoluene in 6 pts. of 94% sulphuric acid, heated at 190° for 40 hrs. with 5 pts. of 90–92% nitric acid and 4 pts. of 94% sulphuric acid) is dangerous, since trinitrotoluene in presence of air decomposes violently at 209–210°

(Köhler, private communication; Verola, *ibid.*) *m*-Dinitrobenzene, nitrated with 4.5 pts. of 93% nitric acid in 9 pts. of 67% oleum at 80–120° for 8 hrs. gives 50% yield of 1:3:5-trinitrobenzene. A table of setting points for mixtures of di- and tri-nitrobenzenes is given. 1:3:5-Trinitrobenzene has at present no commercial application. Added in small amount (3%) to trinitrotoluene it has a plasticising effect, and the mixture, after stoving at 40–55°, may be compressed to  $d$  1.60–1.63; detonation is complete. The solubilities of trinitrobenzene in water at 15° (0.028%), 50° (0.10%), and 100° (0.498%), and in various organic solvents at 17° and 50° [*e.g.*, alcohol (2.09% and 4.57%), pyridine (112.6% and 194.2%)] are determined. With pyridine it yields a compound, not melted at 240°.

C. HOLLINS.

**Mercury catalyst in nitration processes.** A. I. ZACHAROV (J. Chem. Ind., Moscow, 1931, 8, 30–37).—Yields of 60% of *p*-nitrophenol are obtained, using 3–6% nitric acid in the presence of mercuric nitrate (10% of the weight of phenol taken) and maintaining the temperature at –5°. No dinitrophenol is obtained, either in more concentrated acid or using more catalyst. 93% yields of nitrosalicylic acid, containing 80–82% of the *o*-isomeride, are obtained, using the same catalyst, 10–12% nitric acid, and maintaining the temperature below 45°, when the reaction is completed in 12–14 hrs. 85% yields of 1-nitro- $\beta$ -naphthol are similarly obtained, using 2% acid at 35°; the reaction time in this case is 8–9 hrs. 65–70% yields of 1:8-dinitronaphthalene are obtained by passing oxides of nitrogen at 20–25° through a solution in carbon tetrachloride of naphthalene, in the presence of mercuric nitrate. R. TRUSZKOWSKI.

**Bromo-potentiometric titration of  $\beta$ -naphthol-sulphonic acids in presence of each other. III. Mixtures containing oxy-Tobias acid, F acid, and trisulpho-acid.** J. S. HARLAND, S. D. FORRESTER, and D. BAIN (J.S.C.I., 1931, 50, 100–102 T).—Oxy-Tobias acid and F acid have been determined by bromine potentiometrically in 10*N*-sulphuric acid. Trisulpho-acid does not brominate under the same conditions. Those products to be found in sulphonation mixtures of  $\beta$ -naphthol which do not contain a sulphonic group in the 8-position can be determined in the presence of those which do contain a sulphonic group in the 8-position. F acid exists as the tetra-, tri-, and monohydrates and as the anhydrous form, the m.p. of which are given.

**Diphenyl: a starting point in organic synthesis.** G. T. MORGAN and L. P. WALLS (J.S.C.I., 1931, 50, 94 T).—Attention is directed to the possible utilisation in organic synthesis of the hydrocarbon, diphenyl, now available commercially at a moderate price. The following intermediates are obtainable from this source: *o*- and *p*-xenylamines, benzidine, carbazole, 2-amino-carbazole, phenanthridine and its homologues and derivatives.

**Lactic-acetic fermentation.**—See XVIII.

#### PATENTS.

**Catalytic processes. [Preparation of methyl alcohol].** BRIT. CELANESE, LTD., W. BADER, and E. B. THOMAS (B.P. 345,649, 18.9.29).—Metal oxides or

hydroxides, employed as catalysts, are prepared by electrolysis of the corresponding metal salt solutions, using an *E.M.F.* in excess of that required to deposit the metal. In the preparation of methyl alcohol from carbon monoxide and hydrogen, the catalyst may consist of zinc oxide or hydroxide, with or without the oxide or hydroxide of chromium. W. J. WRIGHT.

**Acetylene. Oxidation of natural gas. Sulphonated hydrocarbons.**—See II. **Metal aminocarbons.**—See VII. **Products from fish-liver oils.**—See XII. **Anethole.**—See XIII. **Organic acids by fermentation.**—See XVIII.

#### IV.—DYESTUFFS.

**Possible adulteration of saffron with annatto.** P. BRETIN, P. MANCEAU, and M. NAMDAR (J. Pharm. Chim., 1931, [viii], 13, 369–371).—The colouring matter of annatto may be separated from that of saffron by extraction with benzene, toluene, trichloroethylene, tetrachloroethane, or carbon disulphide, and can be detected in the extract by evaporation and addition of concentrated sulphuric acid to the residue. A greenish-blue coloration is formed. E. H. SHARPLES.

#### PATENTS.

**Manufacture of azo dyes for wool.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 340,640, 10.10.29).—*N*-Acylcyclohexylaminoarylamines (cf. B.P. 340,495; B., 1931, 334) are diazotised and coupled with a sulphonated or carboxylated coupling component. The acyl group may be aminobenzoyl (etc.), in which case disazo dyes are obtained; or the acyl group may be removed after coupling. Examples are dyes from the following *N'*-acyl-*N'*-cyclohexyl-*p*-phenylenediamines; acetyl derivative  $\rightarrow$  Schäffer acid (orange), R-acid (orange), benzoyl-K-acid (red), tetrahydronaphthalene- $\beta$ -sulphonyl-H-acid (bluish-red), salicylic acid (red-yellow chromed), or H-acid (red); hydrolysed, greenish-blue; benzoyl derivative  $\rightarrow$  *o*-anisyl- $\gamma$ -acid (brown); 2:5-dichlorobenzoyl derivative  $\rightarrow$   $\beta$ -naphthol-8-sulphonic acid (yellow-orange) or 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow); *m*-aminobenzoyl derivative  $\rightarrow$  2 mols. of  $\beta$ -naphthol-8-sulphonic acid (orange).

C. HOLLINS.

**Manufacture of acid disazo dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,807, 19.11.29).—A 4:4'-diaminodiarylmethane in which the methylene residue may carry an aryl or two methyl substituents is tetrazotised and coupled with 2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid or its monoarylsulphonate, or with 1 mol. of this acid or ester and 1 mol. of another component; the arylsulphonyl group may be introduced after coupling. Examples are: 4:4'-diamino- $\beta\beta$ -diphenylpropane  $\rightarrow$  2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid (red on wool; *p*-toluenesulphonated, bright yellowish-red); 4:4'-diamino-3:3'-dimethoxy- $\beta\beta$ -diphenylpropane  $\rightarrow$  1 mol. of *N*-*p*-toluenesulphonyl-H-acid and 1 mol. of 2:8-dihydroxynaphthalene-6-sulphonic acid (bluish-claret); 4:4'-diamino-di- or -tri-phenylmethane  $\rightarrow$  2 mols. of 2:8-dihydroxynaphthalene-6-sulphonic acid (red; brighter and yellower on *p*-toluenesulphonylation). [Stat. ref.] C. HOLLINS.

**Colouring foodstuffs.**—See XIX.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Difference in physico-chemical properties of aqueous solutions of [silk] cocoons which show yellow and violet fluorescent colours under ultraviolet light.** H. KANEKO and M. MIYASAKA (Bull. Sericult. Silk Ind., Japan, 1931, 3, 1—3; cf. B., 1930, 857).—Measurements of the physical properties of aqueous solutions of sericin prepared from each type of cocoon shows that cocoons showing a yellow fluorescence have by far the better reeling properties. The effect of storage of the cocoons on the properties of the aqueous extract has also been examined.

R. CUTHILL.

**Cotton for nitration from native [Italian] conifers.**

F. C. PALAZZO (Giorn. Chim. Ind. Appl., 1931, 13, 61—68).—Subjection of the crude cellulose of conifers, such as is used for paper-making, to chemical and physico-mechanical treatments readily applied industrially yields a product which contains a very high proportion of  $\alpha$ -cellulose and very little hemicellulosic material and is virtually equivalent to bleached cotton cellulose. The resemblance extends to the external physical characters and to the state of aggregation of the fibres, which are quite similar to those of linters and suggest the name "conifer cotton-waste." The behaviour of this material on nitration also corresponds closely with that of linters, the cellulose nitrate obtained being of the same titre and capable of stabilisation by the same procedure.

T. H. POPE.

**Highly nitrated cellulose.** G. DE BRUIN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 84).—Priority is claimed for the preparation of nitrocellulose containing 14% N. The product, which can be used in the manufacture of smokeless powder, is unaffected by boiling water, dilute caustic soda, and very dilute acids. Nitrocellulose containing 13.87% N loses only traces of nitrogen on boiling for 72 hrs. and is about 2% soluble in ether-alcohol. (Cf. Berl and Rueff, also Lenze and Rubens; A., 1931, 203; B., 1931, 368.)

A. RENFREW.

**Measurement of swelling criteria of wood pulp.**

A. NOLL (Papier-Fabr., 1931, 29, 114—116).—The capillary rise of sodium hydroxide solutions of mercerising concentration is found to be complete after 10 min. The mean of the values for strips of pulp  $15 \times 180$  mm., cut in the "length" and "cross" directions, is determined at 20°. The swelling of discs of pulp is measured by means of an apparatus described. The discs are held under slight pressure between nickel plates, one plate being free to move as the pulp swells. The linear expansion is measured directly, being of the order of 250—500%. It is found that swelling is complete in 5 min. The amount of mercerising solution absorbed by the pulp is measured by draining the discs and weighing. Results are corrected for "density" or "bulk" of the pulp examined. The results of several determinations are given.

T. T. POTTS.

**Mechanism of the [paper pulp] beating process.**

I, II. K. KANAMARU (J. Soc. Chem. Ind., Japan, 1931, 34, 39—50 B).—The mechanism of the beating process has been investigated by studying changes in the  $\zeta$ -potential and surface conductance of sulphite pulp

during beating. The relation between the former ( $\zeta$ ) and the time of beating,  $t$ , is expressed by the equation  $-d\zeta/dt = a(\zeta - \zeta_\infty)$ , where  $a$  is a constant depending on the conditions of beating, and  $\zeta_0$  and  $\zeta_\infty$  are the values of  $\zeta$  when  $t = 0$  and  $\infty$ , respectively. A similar relation between  $\zeta$  and  $t$  is found on immersing previously dried fibres in water; the velocity coefficient  $a$  is here determined by the conditions of immersion, and a theoretical significance is found for it on assuming that the reaction causing hydration of the cellulose micelles is monomolecular. The relation between the surface conductance,  $\kappa_F$ , at the cellulose-water interface and the time of beating,  $t$ , is given by  $d\kappa_F/dt = k(\kappa_F - \kappa_{F0})/t$ , where  $\kappa_{F0}$  is the value of  $\kappa_F$  when  $t = 0$ , and  $k$  is a constant characteristic of the sample used. When fibres are immersed in water without further treatment the conductance first increases to a maximum with time and then decreases. Surface conductance is valuable as a measure of the degree of beating and as a means of studying the beating mechanism. B. P. RIDGE.

**Blood-albumin.**—See XIX.

## PATENTS.

**Production of cellulose.** A. FRANZ, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,777,751, 7.10.30. Appl., 16.2.28. Ger., 5.8.26).—Vegetable material is treated below 80° with a concentrated solution of a chlorate (preferably sodium chlorate), together with a moderate quantity of a mineral acid and, where this acid is not hydrochloric acid, in the presence of a catalyst capable of producing hydrochloric acid from chloric acid (copper sulphate); the resulting pulp is washed with water.

F. R. ENNOS.

**Manufacture of  $\alpha$ -cellulose.** L. I. L. DURCHMAN (U.S.P. 1,780,347, 4.11.30. Appl., 8.5.29).—Wood chips are cooked with black and white sulphate liquor containing 0.8—1.0%  $\text{Na}_2\text{SO}_3$  (based on the dry wt. of wood), and the resulting pulp is boiled under pressure (1 atm.) with a solution of caustic soda and common salt, bleached, and treated at ordinary temperature for 3—6 hrs. with a 5—12% solution of sodium hydroxide. The latter is recovered for re-use and the pulp washed and treated with oxalic acid.

F. R. ENNOS.

**Manufacture of viscose.** S. WEIN, Assr. to N. RAFF (U.S.P. 1,779,521, 28.10.30. Appl., 1.3.24).—Cellulose is treated with zinc chloride solution to hydrate it without dissolution. After draining off the excess of solution the hydrated cellulose together with zinc chloride is treated with caustic soda and carbon disulphide to form cellulose xanthate, which is dissolved in alkali to yield viscose, the contaminating sulphur salts being precipitated as zinc salts which are removed by filtration.

F. R. ENNOS.

**Sealing material.** L. RADO (B.P. 344,469, 28.10.29).—Cellulose hydrate film, optionally in association with metal foil, paper, fabric, etc., is used in the manufacture of adhesive tape.

D. J. NORMAN.

**Production of foils from solutions of cellulose compounds, gelatin, etc.** WOLFF & Co. KOMM.-GES. AUF AKT., and M. KÖNIG (B.P. 345,179, 1.7.30. Ger., 22.7.29).—The film-forming solution, e.g., viscose, is fed on to a rotating cylinder, the lower part of which is



immersed in a coagulating bath, and the film is stripped off before the coagulating liquor has penetrated to the pouring base, so that during the whole operation the cylinder retains a coating of uncoagulated viscose.

D. J. NORMAN.

**Manufacture of foils from viscous solutions [containing cellulose, gelatin, etc.].** ZELLSTOFF-FABR. WALDHOF, and A. BERNSTEIN (B.P. 344,982, 2.1.30. Ger., 28.2.29).—The solution is fed into the gap between oppositely rotating rollers, and is then coagulated in the usual way.

D. J. NORMAN.

**Production of artificial filaments or threads.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 344,351, 26.11.29).—The output of the spinning machine is increased without additional apparatus, particularly pumps, by using with each pump a single disc-type jet with its spinning orifices arranged in groups so that two or more threads may be spun at the same time.

D. J. NORMAN.

**Manufacture of artificial fibres and films.** HEEBERLEIN & Co. A.-G. (B.P. 345,811—2, 21.3.30. Ger., 21.3.29).—To prevent degradation of regenerated cellulose when precipitated by means of concentrated mineral acids, addition is made to the precipitating bath of (A) formaldehyde, or a condensation product, polymerisation product, or a derivative which yields formaldehyde in the bath; or (B) a pyridine base or a salt thereof which is soluble in the concentrated acid, no stretch being applied to the product. F. R. ENNOS.

**Manufacture of artificial silk.** W. HARRISON (B.P. 344,873—4, 8.11.29).—(A) To improve their tensile strength, threads of regenerated cellulose or cellulose derivatives are treated with reagents (*e.g.*, caustic soda with Glauber's salt and aqueous acetone, respectively) to remove  $\beta$ - and  $\gamma$ -cellulose or their derivatives, and are stretched after treatment with a swelling agent (zinc chloride, acids, or aqueous acetone) which is subsequently removed. (B) To render them flexible and pliable, threads having a high tensile strength, *i.e.*, above 2 g. per denier, produced from cellulose esters other than nitrates are wholly or partly hydrolysed, whereby spaces are created between the cellulose crystallites in the formed threads. F. R. ENNOS.

**Washing and subsequent treatment of threads of artificial silk spun in the form of cakes.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 345,076, 6.3.30. Ger., 12.3.29).—The spun cakes are wrapped or tied up in wide-meshed or reticulated fabrics such as net, flattened, and, after squeezing under water to remove air or gas bubbles occluded between the threads, are suspended on rods which are continuously or intermittently rotated or moved, and subjected to the usual processes for washing and subsequent treatment. F. R. ENNOS.

**Manufacture of flexible gramophone records.** G. HILHOUSE (B.P. 345,875, 16.5.30).—Two discs of celluloid with a sheet of paper between are heated at 120—140° for 30—40 sec. under pressure (1700 lb./in.<sup>2</sup>) without the use of an adhesive, and the blank thus produced is allowed to cool. The paper should be adapted to receive print, and its diameter should not be less than that of the discs. F. R. ENNOS.

**Acetylation of cellulose.** E. ROTH (B.P. 345,069, 28.2.30. Fr., 28.2.29).—The time required for acetylation is much reduced if, as soon as the reaction mixture becomes capable of being pumped, it is forced repeatedly through one or more nozzles to effect a more complete mixing of the reactants than is possible by kneading. The nozzles are preferably of the injector type, a current of air or other gas being used to prevent clogging. It is advantageous to use more glacial acetic as diluent than is usual. Suitable apparatus is described.

D. J. NORMAN.

**Manufacture of cellulose acetate.** KODAK, LTD. From EASTMAN KODAK Co. (B.P. 345,800, 11.3.30).—Cellulosic material mixed with a catalyst (sulphuric or *p*-toluenesulphonic acid) is heated with saturated acetic anhydride vapour in a closed vessel from which the air has been substantially removed, the acetic acid formed during the reaction being retained in contact with the material until acetylation is complete. F. R. ENNOS.

**Pulp digester.** E. MORTERUD (U.S.P. 1,777,217, 30.9.30. Appl., 10.10.29).—The digester is provided internally at the middle with two annular sieve tubes, one of which is fixed to the digester wall, the other, of considerably smaller diameter, being fastened thereto by stays. These two sieve tubes are both connected to the inlet side of a circulating pump (preferably mounted inside the digester), the outlet from which is connected to other sieves at the top and bottom of the digester.

D. J. NORMAN.

**Treatment of fibre of non-coniferous origin.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,780,539, 4.11.30. Appl., 11.3.27).—To increase its softness and tear resistance, the fibre liberated, *e.g.*, by the sulphite process, after pretreatment with a lignin-reactive agent (chlorine) is digested at ordinary temperature with a solution of caustic soda, washed, and bleached in alkaline solution. F. R. ENNOS.

**Preparation of cellulose pulp.** H. L. HORN, Assr. to BAGASSE DEVELOPMENT, INC. (U.S.P. 1,780,750, 4.11.30. Appl., 2.12.29).—Cellulose-bearing materials such as bagasse, straw, grass, wood, cornstalks, etc. are simultaneously cooked by introduction of steam into the digester containing the material with water beaten by loose rods within the digester, and electrically and chemically treated by passing a current through the water to which sodium chloride has been added; lime and soda ash are added at a later period of the treatment and the pulp is drained from the liquor for use.

F. R. ENNOS.

**Production of sulphite pulp.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,780,842, 4.11.30. Appl., 5.1.27).—Raw cellulosic material is digested at raised temperature and pressure with sulphurous acid and ammonium sulphate solution. After separation of the pulp, the spent liquor is neutralised, preferably with basic calcium phosphate, and evaporated to *d* 1.16—1.26 for use as fertiliser. F. R. ENNOS.

**Producing low-density pulp [for explosives].** L. W. BABCOCK, Assr. to HERCULES POWDER Co. (U.S.P. 1,777,710, 7.10.30. Appl., 4.9.26).—The pulp, particularly bagasse, is ground and boiled with water under

atmospheric pressure for  $\frac{1}{2}$ —1 hr. to induce swelling without materially changing the porosity of the fibre. The dried pulp may be used in the preparation of low-density explosives. D. J. NORMAN.

**Making articles of wood and other pulp and drying same.** G. E. COBLENS and A. W. MORRIS (B.P. 344,547, 16.12.29).—The wet pulp is moulded into the required shape between mesh dies and subjected to high pressure to remove as much water as possible and establish intimate contact between the pulp and the dies. The pressure is then released and a current of electricity (preferably 110 volts A.C.) is passed through the moist pulp (the two-mesh dies serving as electrodes) to complete the drying by electro-endosmosis.

D. J. NORMAN.

**Absorbent paper and like felted fabrics.** J. STRACHAN (B.P. 344,846, 5.12.29).—Finely-divided colloidal cellulose, *e.g.*, waste vegetable parchment or cellulose silk that has been ground to pass 60-mesh, is mixed with rag, wood, or esparto pulp and the resulting stock run on a paper machine. The ratio of colloidal cellulose to wood or rag cellulose may conveniently be 2:1 for blotting paper and 1:1 for stereo-moulding paper.

D. J. NORMAN.

**Manufacture of fibre board.** R. W. CUTLER and J. R. COOLIDGE, III, Assrs. to MONTAN, INC. (U.S.P. 1,779,532, 28.10.30. Appl., 27.5.27).—The strength and water-resistance of fibre boards made from unvulcanised fibre are considerably increased by impregnating the board with a wax which remains hard below 60°, *e.g.*, montan wax. Various methods of effecting the impregnation are described.

D. J. NORMAN.

**Coating for writing tablets, boards, or like articles.** J. B. E. LAMBRECHT, C. DECORTE, and SOC. ANON. CHEMIFAX (B.P. 344,489, 2.12.29).—The coating consists of a cellulose derivative, *e.g.*, nitrocellulose, and/or any substance which is substantially insoluble in water and gives a continuous compact film, *e.g.*, rubber, in admixture with resin, *e.g.*, dammar, and/or acid-resisting agents, *e.g.*, paraffins, barium sulphate, or quartz. The coating solution is prepared by mixing solutions of the cellulose derivative and resin in different organic solvents (preferably volatile) and suspending or emulsifying in this mixture the acid-resisting agents. For very resistant coatings the quantity of resin etc. should exceed that of the cellulose derivative.

D. J. NORMAN.

**Filtration process.**—See I. Paper.—See VI.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Use of liquid chlorine in the bleaching of cotton fabrics.** BLANCHISSERIE ET TEINTURERIE DE THAONLES-VOSGES (Sealed Note No. 2301, 31.1.14. Bull. Soc. Ind. Mulhouse, 1931, 97, 60—62). Report by C. SUNDER and J. LICHTENBERGER (*Ibid.*, 62—64).—During the bleaching of cotton fabrics by treatment with a calcium or sodium hypochlorite solution the concentration of this liquor is maintained constant by continuous circulation downwards in the form of a shower through a vertical chamber where it meets an upward flow of chlorine gas issuing from a cylinder of liquid chlorine.

SUNDER and LICHTENBERGER cite numerous similar processes. A. J. HALL.

**Blood-albumin.**—See XIX.

### PATENTS.

**Mordanting hair or the like for production of felt.** V. BÖHM (B.P. 345,404, 24.1.30. Austr., 30.1.29).—The skins are treated with a solution containing nitric acid, an oxidising agent capable of acting independently of and simultaneously with nitric acid, *e.g.*, chlorates, perchlorates, permanganates, or persulphates, and a metal salt, *e.g.*, of aluminium, tin, or zirconium in quantity greater than would be necessary for catalytic acceleration of the action of the oxidising agents. A suitable solution contains (g./litre) zinc nitrate 100, nitric acid 100, and potassium chlorate 15—50.

D. J. NORMAN.

**Colouring of paper.** I. G. FARBENIND. A.-G. (B.P. 314,068, 12.6.29. Ger., 23.6.28).—Paper is coloured, either in the mass or by immersion, with the oxazines (?) obtainable by heating diarylamino-benzoquinones in a high-boiling solvent or according to B.P. 313,094 (B., 1931, 337), and sulphonating the product. Examples are: sulphonated oxazines from 3:6-dichloro-2:5-di-(*N*-ethyl-3-carbazolylamino)-*p*-benzoquinone (blue), 3:6-dichloro-2:5-di- $\beta$ -naphthylamino-*p*-benzoquinone (violet), and 3:6-dichloro-2:5-di-(*o*-phenoxyanilino)-*p*-benzoquinone (red).

C. HOLLINS.

**Coloration of cellulose ester materials.** BRIT. CELANESE, LTD. (B.P. 339,997, 10.6.29. U.S., 8.6.28. Cf. B.P. 313,404; B., 1930, 1107).—Cellulose acetate silk (etc.), after hydrolysis to the extent of 56% of the ester, is dyed by means of those dyes commonly used for cellulose ester materials.

A. J. HALL.

**Manufacture of artificial silk of dull lustre from viscose.** I. G. FARBENIND. A.-G. (B.P. 345,405, 24.1.30. Ger., 24.1.29. Addn. to B.P. 312,687; B., 1930, 1063).—An acid amide or a substituted acid amide, *e.g.*, di-*o*-tolyl-diethylcarbamide, which is insoluble in water and in viscose, but soluble in the acid spinning bath, is dispersed in the viscose solution prior to spinning.

D. J. NORMAN.

**Production of white or coloured matt surfaces on ethers or esters of cellulose or its transformation products.** I. G. FARBENIND. A.-G. (B.P. 345,673, 23.12.29. Addn. to B.P. 309,194; B., 1930, 859).—The lustre of cellulose ester or ether yarns and fabrics is reduced by impregnating (instead of printing as described previously) with a solution of urea, then drying, steaming for 20—30 min., washing, and drying. The delustring effect of the urea may be increased by adding water-soluble aliphatic hydroxycarboxylic acids (glycollic and lactic acids), or their salts, together with suitable dyes if desired.

A. J. HALL.

**Oiling and dressing of fibres for spinning.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 341,710, 16.1.30).—Condensation products, or salts thereof, *i.e.*, esters, amides, or ester-amides of alkylolamines (*e.g.*, mono-, di-, or tri-ethanolamine, hydroxyethylaniline, cyclohexyldiethanolamine, or acid amides thereof, hydroxyethylmorpholine, and the condensation products of these amines with aldehydes) with organic

carboxylic acids, sulphonic acids, or sulphonated carboxylic acids of high mol. wt. are used for oiling wool etc.; they do not form insoluble soaps with hard water, and can be easily removed from the fibre subsequently by washing with water. Suitable acids are those derived from fats or coal-tar oils. A. J. HALL.

**Sizing of textile fibres.** I. G. FARBENIND. A.-G. (B.P. 345,207, 10.12.29. Ger., 24.12.28).—Polyvinyl alcohols are used optionally in conjunction with other sizing agents and oils, fats, waxes, etc. An aqueous solution containing 2–3·33% of the alcohol and 0·2–0·33% of sulphonated oil is suitable. The size is equally suitable for cotton, artificial silk, and wool, and is readily removed by washing with warm water or 0·1–0·2% soap solution. D. J. NORMAN.

**Treatment [sizing] of textile materials.** BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 344,775, 27.8.29).—Yarns etc. intended for weaving are sized with aqueous solutions of carboxylic esters of cellulose containing ester radicals having as substituents carboxyl and/or hydroxyl groups; such esters may be degraded, if desired, to render them water-soluble. A. J. HALL.

**Manufacture of crêpe yarn.** D. PÄCHT (B.P. 345,396, 18.1.30).—The raw fibres are sized and, if desired, coloured on the way to the twisting machine, and then twisted and wound while still moist. The resulting yarn shows increased strength and elasticity and is free from internal stresses. Suitable apparatus is described. D. J. NORMAN.

**Treatment of silk.** C. H. KIDWELL, ASSR. to KIDWELL & Co., INC. (U.S.P. 1,777,203, 30.9.30. Appl., 28.2.30).—Hosiery knitted from real silk (degummed or not) are made smoother and less subject to "slippage" by treatment for about 1 min. in a solution containing 99 vol.-% of 85% formic acid, 1% of 95% sulphuric acid, and 2·5% of glycerin, followed by washing with dilute ammonia solution. Materials knitted with cotton, when so treated, acquire increased tensile strength and elasticity of fibre. A. J. HALL.

**Gasproof balloon fabric.** C. M. CARSON, ASSR. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,779,389, 21.10.30. Appl., 9.3.29).—Light, strong, closely woven fabric is given 8–10 successive coats of a mixture of vulcanised latex, gelatin, and a softener. A suitable mixture contains gelatin 25 g., glycerin 50 g., vulcanised latex (30% rubber) 100 c.c., water 500 c.c. The fabric is finally treated with a flexible varnish and dusted with aluminium powder. D. J. NORMAN.

**Manufacture of [rubbered] threads of textile material.** J. E. C. BONGRAND and L. S. M. LEJEUNE (B.P. 344,414–5, 25.11.29).—Spun, twisted, or stranded threads or fabrics are impregnated *in vacuo* or under pressure, or by a combination of these two methods, with (A) an aqueous dispersion of rubber, guttapercha, or the like, or (B) a cellulose ether either in aqueous dispersion or in solution in an organic solvent. A further surface-coating of rubber or cellulose ether may be applied if desired. The treated threads show high strength even when made from relatively short fibres,

and in the case of (A) may be used, after vulcanisation, in the manufacture of fabrics for pneumatic tyres.

D. J. NORMAN.

**Dyeing, washing, degreasing, and like machines.** C. CALLEBAUT and J. DE BLICQUY (B.P. 346,542, 8.3.30).

**Artificial silk for treatment in cake form.**—See V. Bleaching composition.—See VII. Compound from fish-liver oil.—See XII.

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

**Production of nitric acid by the oxidation of ammonia.** J. FAUSER (Chim. et Ind., 1931, 25, 556–569).—The use of increased pressure to accelerate the further oxidation of nitric oxide is twice as economical in power as the use of artificial cooling, and no difficulty now exists in the construction of turbo-compressors of chromium steel for this purpose. In modern practice the oxidation of the ammonia is performed at atmospheric pressure and the gases are then compressed. The efficiency of oxidation of ammonia is, other things being equal, reduced by increase of pressure, and at 4 atm. not more than 91% conversion is attainable and the life of the catalyst is much shorter. The author uses a gas mixture enriched with oxygen, containing 11% NH<sub>3</sub> and 22% O<sub>2</sub>, which enables the optimum temperature of 850–950° to be reached without heat exchangers. The removal of heat, which is necessary in an absorption tower, can be effected only by the use of an excessive liquid feed, which adds greatly to pumping costs. It is therefore proposed to replace the towers by horizontal cylinders of resistant steel. The lower part of the cylinder is filled with acid, through which the gas passes in a number of jets; the empty upper part serves as oxidation space. The total volume required is 30% less than with towers packed with Raschig rings. An empirical equation is given for the coefficient of absorption of a cylinder at a given pressure and concentration of acid. At 3 atm. and 20°, 12 cylinders will give 95% absorption and 19 cylinders 99% without the use of alkali. The acid runs in countercurrent to the gas by U-pipes from cylinder to cylinder, and the cylinders are cooled externally with water; the exit gases pass through a turbine to recover their residual energy. Some difficulty has occurred owing to oscillations set up in the acid in the cylinders, which, if in resonance with the general oscillations of the system, not only interfere with absorption, but are mechanically dangerous. Costs are discussed and the conclusion is reached that a pressure of 3·0–3·5 atm. is the most economical. C. IRWIN.

**Concentration of phosphoric acid by submerged combustion.** C. F. HAMMOND (Inst. Chem. Eng., Mar., 1931. Advance proof. 6 pp.).—The author's apparatus has a central vertical burner in a refractory-lined combustion chamber. An intimate mixture of gas and air in theoretical proportions renders combustion complete and the products pass directly into the liquid to be heated. The burner is calculated to have a theoretical flame temperature of 2145° and the inner wall of the combustion chamber (suitably insulated) to be at 1470°. The burner casing is surrounded by

an open-ended cylindrical column within which the exit gases rise and cause a liquid current by air-lift effect, so cooling the casing. The containing vessels are made of chemical stoneware. This leaves only the burner casings as sources of contamination, which must be excluded especially for acid of B.P. quality. After various materials had been tried certain corrosion-resisting steels were found to be satisfactory, the best results being obtained with casings machined from the solid metal rolled, annealed, and pickled. The behaviour of a given steel is not necessarily the same with crude phosphoric acid as with pure acid, but steels have been obtained giving negligible corrosion with either. The increase in sulphate content due to sulphur in the (town's) gas used was only slight. Pure acid can be concentrated with a loss of  $P_2O_5$  in fume of less than 1%. With crude acid yielding an exhaust containing silicon tetrafluoride, flushing of the exhaust main was necessary, but the phosphorus loss was still very small. Crude acid concentration requires good circulation as deposits of calcium sulphate having an eroding effect may otherwise be formed. The yield is thus much better than with standard-type evaporators and the small size of the burner casing, the only part exposed to attack, is an important advantage. The capital cost is competitive as would be the fuel cost if producer gas were used.

C. IRWIN.

**Formation of hydrocyanic acid in the electric discharge.** K. PETERS and H. KÜSTER (Brennstoff-Chem., 1931, 12, 122—127; cf. B., 1931, 429).—By subjecting a 1 : 1 mixture of methane and ammonia to an electric discharge under reduced pressure (20—40 mm. Hg), and using high gas velocities, yields of hydrocyanic acid up to 70% of theory have been obtained ( $CH_4 + NH_3 \rightarrow HCN + 3H_2$ ). The yield increased with increasing input of electrical energy, the rate of increase corresponding with the increase with rising temperature as calculated from the thermal equilibrium constant. The yield, calculated on the input of methane, increased with diminishing concentration of methane and was practically quantitative for concentrations of less than 15%; with mixtures containing more than 85% of methane and, therefore, less than 15% of ammonia the latter was similarly converted quantitatively into hydrocyanic acid. With high electrical energy inputs the greater part of the excess methane was converted into acetylene, whilst excess ammonia was decomposed into its elements. By passing the discharge through a 3 : 7 methane-ammonia mixture a good yield of crystalline ammonium cyanide was obtained. In the production of acetylene by this process the addition of some ammonia to the initial gas diminished the amount of carbon deposition without affecting the yield of acetylene, the tar and deposited carbon reacting with the ammonia to give hydrocyanic acid. The mechanism of the reaction is discussed.

A. B. MANNING.

**Volumetric determination of hydrofluoric acid with especial reference to the determination of hydrofluoric and sulphuric acids in etching baths.** F. H. ZSCHACKE (Chem.-Ztg., 1931, 55, 246—247).—The acid solution (10 g. weighed in a platinum dish) is diluted to 250 c.c. and 25 c.c. are titrated with *N*-sodium hydro-

oxide, first cold, then hot, using phenolphthalein as indicator. The solution is cooled, treated with 20 g. of sodium chloride and 5 c.c. of 20% potassium thiocyanate, and titrated with *N*-ferric chloride until a pale yellow colour persists. A mixture of 10 c.c. of alcohol and 10 c.c. of ether is then added and titration continued until the ethereal layer, after thorough shaking, acquires a pale pink colour. The first titration gives the total acidity and the second the total fluorine content. Soluble salts are determined by evaporating the solution to dryness and igniting the residue, which is then extracted with hot water to remove alkali sulphates, again ignited and weighed, and analysed as usual for lead, iron, aluminium, and lime.

A. R. POWELL.

**Determination of calcium oxide.** J. ZAWADZKI and W. LUKASIEWICZ (Rocz. Chem., 1931, 11, 154—157).—Calcium oxide present in Portland cement is extracted with a 1 : 1 mixture of absolute alcohol and phenol, the alcohol is distilled off from the filtrate, and dissolved calcium oxide is titrated with standard acid.

R. TRUSZKOWSKI.

**Determination of calcium carbonate in lime marl.** A. VÜRTHEIM and G. H. JONGEN (Chem. Weekblad, 1931, 28, 206—209).—The method described depends on the reaction  $2NH_4Cl + CaCO_3 \rightleftharpoons CaCl_2 + (NH_4)_2CO_3$ , which, as a result of the decomposition of the ammonium carbonate, is displaced to the right when the mixture is heated. The sample (1 g.) is finely powdered and treated with 300 c.c. of a 5% ammonium chloride solution. The mixture is distilled with a small flame until about 50 c.c. remain, care being taken to avoid the deposition of solid on the walls of the flask. To the distillate are added 200 c.c. of 0.1*N*-hydrochloric acid, and the excess of acid is titrated, using lacmoid as indicator. Since part of the ammonia is derived from decomposition of the excess of ammonium chloride, the residue from the distillation should be filtered and titrated, and the result, after deduction of 0.1 c.c. for the alkali liberated by the glass, deducted from the ammonia titre of the distillate. The results obtained are more concordant than those of Scheibler's method, and the sample used is larger.

H. F. GILLBE.

**Preparation and properties of colloidal aluminium hydroxide for medicinal use.** J. J. L. ZWIKKER (Pharm. Weekblad, 1931, 68, 328—343).—Colloidal aluminium hydroxide, possessing the necessary ease of peptisation in dilute hydrochloric acid, slow power of neutralisation, and the property of gelatinising in more strongly acid solution, and thus resembling "Alucol," may be prepared by Kohlschütter's method (B., 1919, 174 A; 1923, 655 A). A preparation suitable for medicinal use may be obtained by using a 10-fold excess of a 5% ammonia solution containing 5% of ammonium sulphate, stirring the mixture for 30 min., and setting it aside for 4—5 days at the ordinary temperature or for 18 hrs. at 40°. Care must be taken to avoid adsorption of organic material (including alcohol) by the colloid during drying. After drying for 18 hrs. at 40° the material contains about 35% of water.

H. F. GILLBE.

**Content of iron soluble in hydrochloric acid in asbestos for Gooch crucibles.** E. CATTELAÏN (Ann.

Falsif., 1930, 23, 530—531).—The necessity for treating asbestos with hot concentrated hydrochloric acid before use in Gooch crucibles etc. is emphasised. Samples tested contained 1.19—8.55% Fe (soluble in hydrochloric acid) and traces of manganese.

E. B. HUGHES.

**Determination of sulphur in pyrites.** I. A. LEVIN and G. V. RABOVSKI (J. Chem. Ind., Moscow, 1931, 8, 159—161).—0.7—0.8 G. of pyrites is dissolved in 20 c.c. of nitric acid, and the solution is repeatedly evaporated to dryness, with the addition of hydrochloric acid, in order to expel oxides of nitrogen. The final residue is moistened with 3—5 c.c. of dilute hydrochloric acid, diluted to 100—150 c.c., and excess of ammonia is added. The precipitate of ferric hydroxide is filtered off and washed, and the filtrate is diluted to 200 c.c. To 25 c.c. of the solution 7 c.c. of formalin, previously neutralised to methyl-red, are added, and the acidity is determined, using 0.25N-sodium hydroxide in the presence of phenolphthalein. The results so obtained for sulphuric acid are practically identical with those obtained gravimetrically.

R. TRUSZKOWSKI.

**Volumetric determination of selenium.** B. ORMONT (Z. anal. Chem., 1931, 83, 338—339).—Polemical against Benesch (A., 1929, 42). Complete extraction of selenium from lead-chamber slimes by digestion with sodium sulphide is impossible as part is retained as lead selenide in the lead sulphide formed by the action of the sodium sulphide on the lead sulphate in the slime.

A. R. POWELL.

**Distillation of phosphorus from tricalcium phosphate in the presence of alkali-metal salts.** N. E. PESTOV and A. E. SDOBNOV (J. Chem. Ind., Moscow, 1931, 8, 129—132).—The amount of phosphorus distilling off at a given temperature when phosphorite is heated with charcoal is greatest if a mixture of the composition: 1 mol.  $\text{Ca}_3\text{P}_2\text{O}_8$ , 3 mols.  $\text{SiO}_2$ , 1 mol.  $\text{Na}_2\text{SO}_4$ , and 0.5 mol.  $\text{Al}_2\text{O}_3$  be used. Inferior yields are obtained if sodium carbonate or chloride is used in place of sulphate.

R. TRUSZKOWSKI.

**Manufacture of bromine in France.** M. KALTENBACH (Chim. et Ind., 1931, 25, 543—555).—The mother-liquors of Stassfurt, of Alsace, and of Zarzis in North Africa each contain 2.0—4.5 g. of bromine per litre. That of the last-named lake can be concentrated up to 8 g./litre by solar evaporation. The water of the Dead Sea contains 4.8 g./litre. During the war period a number of bromine-recovery plants were built in Tunis. These ceased work in 1918, and French bromine production in Alsace only commenced in 1926. The six plants built or building in Alsace are described. Mother-liquor preheated to 75° is passed down a column against a countercurrent of steam and chlorine. The heavy bromine vapour is forced upwards by a reversal of the general direction of the gases in each tray of the column, the inlets being at the top and the outlets at the bottom. The column is packed with perforated tiles, the steam is introduced at the bottom, the chlorine at the third tray up. The stripped mother-liquor is passed through iron turnings to recover any entrained bromine, neutralised with limestone, and treated with a little sodium thio-sulphate. The condensed bromine drawn off from the

top is separated from water in a divider and the water returned to the column. The bromine passes into a lead receiver filled with Lunge plates, maintained at 55°, and fitted with a reflux condenser. Here it is freed from chlorine and bromine chloride. The colour of the bromine water gives an indication of the correct adjustment of the chlorine feed. The earlier plants erected gave a yield of about 70%. A more recent design provides a better contact between the gas and the liquid by an elaboration of the distribution channels and the staggering of the perforated tiles. This considerably increases the capacity. Heat exchangers have been abandoned in favour of steam preheating. A chlorine regulator is described in which the chlorine flow is maintained constant by the attendant by observation of a water-level. Absorption of excess chlorine is now performed in a small separate column fed with cold mother-liquor. Bromine is condensed with as little cooling as possible to restrict dissolution of chlorine. Alsatian mother-liquors contain organic matter which necessitates a final purification of the bromine with concentrated sulphuric acid.

C. IRWIN.

**Manufacture of potassium chloride and bromine at the mines of Kali Ste.-Thérèse.** BOURDIEU (Mem. Soc. Ing. Civ. France, 1930, 83, 1010—1028).

**Antimony yellow.**—See XIII.

PATENTS.

**Manufacture of chemically pure sulphuric acid.** W. S. ALLEN, Assr. to GEN. CHEM. CO. (U.S.P. 1,777,025, 30.9.30. Appl., 14.11.27).—The sulphuric anhydride concentration of gases from the contact process is increased by bringing them into contact with oleum, the temperature of which is regulated so as to cause liberation of sulphuric anhydride. The enriched gases are treated with steam, the temperature of the reaction preventing the formation of mist, and the gaseous sulphuric acid produced is condensed. The diluted oleum is utilised for absorbing sulphuric anhydride from further amounts of the gases. W. J. WRIGHT.

**Manufacture of concentrated nitric acid.** "MONTECATINI" SOC. GEN. PER L'IND. MINERARIA ED AGRICOLA, Assees. of G. FAUSER (B.P. 345,662, 23.12.29. It., 27.2.29).—Ammonia is oxidised at atmospheric pressure, the nitrogen oxides, after condensation of the aqueous vapour, being passed through a chromium-steel compressor into an absorption tower in countercurrent to water. The dilute nitric acid containing nitrogen oxides passes into another tower, where it is brought into contact with compressed oxygen and converted into concentrated nitric acid.

W. J. WRIGHT.

**Smelting of phosphoric material in an electric furnace.** W. R. SEYFRIED, Assr. to SWANN RESEARCH, INC. (U.S.P. 1,777,582, 7.10.30. Appl., 14.5.25).—A mixture of phosphate rock and carbon is smelted by means of an arc at the lower part of the furnace, from which air is excluded, and the resulting gases are oxidised in the upper zones of the furnace by the admission of preheated air sufficient to convert the phosphorus into pentoxide and thus preheat the charge. The carbon monoxide-phosphorus pentoxide mixture from the furnace is burned in a combustion chamber through

which pass the pipes leading the air to the electric furnace, and the pentoxide is absorbed in water in a scrubbing tower.

A. R. POWELL.

**Manufacture of phosphoric acid and cyanamide.** T. F. BAILY (U.S.P. 1,775,802, 16.9.30. Appl., 5.2.29).—A 5:3 mixture of phosphate rock and coke breeze is smelted in an arc furnace to produce calcium carbide, carbon monoxide, and phosphorus. The gases are passed through a condenser to remove the phosphorus and the carbon monoxide is used for heating purposes. The phosphorus is burned with just sufficient air to form phosphorus pentoxide and the latter is absorbed in water to form phosphoric acid, leaving almost pure nitrogen; this gas is passed into the cyanamide furnace together with the carbide formed in the first stage, which has meanwhile been finely ground.

A. R. POWELL.

[Fumigation by] evolution of (A) hydrocyanic acid, (B) hydrocyanic acid and an irritant gas. H. LEHRECKE, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,196—7, 4.11.30. Appl., 20.10.25. Ger., [A] 27.10.24, [B] 31.10.24).—(A) Hydrogen cyanide is absorbed in a porous substance such as kieselguhr or coke impregnated with a metal salt, *e.g.*, ferric or aluminium chloride, which forms a compound with the cyanide, and with a highly deliquescent salt, *e.g.*, aluminium, zinc, or calcium chloride. On exposing the material to moist air, hydrogen cyanide is evolved owing to the heat generated by the absorption of water by the deliquescent salt. (B) A hypochlorite is added to the mixture so as to produce an irritant gas (cyanogen chloride) which serves as a danger signal when using the material for fumigation.

A. R. POWELL.

**Manufacture of concentrated caustic alkali lyes.** I. G. FARBENIND. A.-G. (B.P. 344,545, 14.12.29. Ger., 15.12.28).—An aqueous suspension of strontium or barium hydroxide and a solid alkali carbonate is heated at the boil for, *e.g.*, 2 hrs.; the alkaline-earth carbonate is collected, washed with water (the washings being used for a fresh charge), and calcined to oxide for re-use in the process.

L. A. COLES.

**Manufacture of ammonium carbamate.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,494, 24.3.30).—Ammonia and carbon dioxide at 5–50° are caused to react in an aqueous solution of urea, which may already contain ammonium carbamate.

W. J. WRIGHT.

**Manufacture of diammonium phosphate.** SOC. D'ETUDES SCIENTIF. ET D'ENTREPRISES INDUSTR., Asses. of E. VOITURON (B.P. 345,451, 25.2.30. Ger., 26.2.29).—Phosphoric acid is treated in a saturator at the b.p. with more than 1 mol. of ammonia per mol. of phosphoric acid, the proportion of ammonia not exceeding that which can be completely fixed. The generated steam and heated air pass to a heat exchanger, and the liquor passes to another saturator into which sufficient ammonia to promote crystallisation of diammonium phosphate is introduced. The crystals are dried in a hydro-extractor, and the mother-liquor is led to the heat exchanger in which it is heated to 100°. It is then mixed in a scrubber with fresh phosphoric acid, which also combines with any ammonia escaping from the

second saturator and condensed steam from the heat exchanger.

W. J. WRIGHT.

**Hypochlorite bleaching composition.** L. T. HOWELLS, Assr. to ELECTRIC SMELTING & ALUMINUM CO. (U.S.P. 1,779,274, 21.10.30. Appl., 24.7.28).—Mixtures of sodium hypochlorite and fluosilicate and of calcium hypochlorite, sodium fluosilicate, and sodium fluoride, either in the powder state or in solution, are claimed.

A. R. POWELL.

**Flotation concentration of phosphatic material.** J. W. LITTLEFORD, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,780,022, 28.10.30. Appl., 3.7.28).—Ground phosphate rock is washed to remove slimes, agitated with a dilute solution of sodium hydroxide, and subjected to flotation in a cascade series of cells using oleic acid and/or soap, with or without pine oil or fuel oil, as a frothing agent.

A. R. POWELL.

**Slaking of lime.** W. H. HENDERSON (U.S.P. 1,780,821, 4.11.30. Appl., 6.4.28).—Ground quicklime is automatically fed into the top of a conical vessel up which is passed a continual stream of steam-heated water from a pipe introduced into the apex of the vessel. A continuous stream of milk of lime overflows from the upper wide end of the vessel, and the impurities settle to the bottom and are withdrawn from time to time.

A. R. POWELL.

**Manufacture of barium sulphide solutions.** H. HOWARD, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,776,029, 16.9.30. Appl., 2.12.26).—The solution obtained by lixiviating with water the product obtained by heating barytes with carbon is treated with a slight excess of barium hydrogen sulphide solution over that required to convert any barium hydroxide in the leach liquor into barium sulphide. The lithopone made from the solution of barium sulphide thus obtained is free from oxides soluble in acetic acid.

A. R. POWELL.

**Separation of metallic compounds [zinc sulphate from copper and iron sulphates].** O. A. FISCHER (U.S.P. 1,779,841, 28.10.30. Appl., 13.7.26).—Roasted sulphide ores containing zinc and copper sulphates with more or less ferric sulphate are heated at 450–600° with carbonaceous material, whereby copper and iron sulphates are decomposed and the zinc sulphate remains unchanged and may subsequently be leached out.

A. R. POWELL.

**Manufacture of metal [iron diethyl-]amino-carbonyls.** P. S. DANNER and J. E. MUTH, Assrs. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,780,643, 4.11.30. Appl., 26.9.27).—Equimolecular quantities of iron pentacarbonyl and diethylamine are heated at 100° under 3 atm., until the pressure falls almost to atmospheric, whereby *iron diethylaminopentacarbonyl*, (?)  $\text{Fe}(\text{CO})_3\text{NHEt}_2$ , useful as an anti-knock material for petrol, is formed.

A. R. POWELL.

**Working up beryllium minerals.** I. G. FARBENIND. A.-G. (B.P. 345,902, 28.6.30. Ger., 28.6.29).—The minerals are treated with hydrogen fluoride at above 100°, preferably at 600°, until silicon is expelled as its tetrafluoride; beryllium fluoride is extracted from the residue with hot water, and the hydroxide is precipitated by the addition of ammonia.

L. A. COLES.

**Working up beryllium sublimates containing sulphur and aluminium to obtain beryllium compounds.** H. LÖWENSTEIN (U.S.P. 1,777,122, 30.9.30. Appl., 8.10.29. Ger., 25.9.28).—The sublimates obtained by heating beryllium silicates with carbon and pyrites in an electric furnace are extracted with alkali, and the filtered solution is treated at room temperature with sufficient acid to precipitate beryllium compounds, aluminium compounds remaining in solution.

W. J. WRIGHT.

**Recovery of soluble beryllium compounds [from minerals].** H. LÖWENSTEIN (U.S.P. 1,777,267, 30.9.30. Appl., 18.9.29. Ger., 25.9.28).—Beryl or other beryllium mineral is heated in an arc furnace with iron pyrites and carbon sufficient to convert all the iron and silicon into ferrosilicon and to volatilise the beryllium and aluminium as sulphides partly separable by fractional condensation.

A. R. POWELL.

**Treatment of dolomite and other carbonates of magnesium.** J. BLUMENFELD, ASSR. to SOC. DE PROD. CHIM. DES TERRES RARES (U.S.P. 1,778,659, 14.10.30. Appl., 13.3.30. Fr., 11.3.29).—Calcined dolomite is digested with an aqueous solution of ammonium chloride sufficient to dissolve only the lime, the ammonia evolved is mixed with part of the carbon dioxide formed in the calcination, and the mixed gases are passed into a solution of calcium chloride formed in the first stage of an earlier operation to regenerate ammonium chloride and calcium carbonate. The undissolved magnesia is separated from the calcium chloride solution and treated with a slight deficiency of hydrochloric acid to obtain pure magnesium chloride solution, which is evaporated to recover the solid salt. The latter is electrolysed to obtain magnesium and chlorine which is converted into hydrochloric acid for use again in the process.

A. R. POWELL.

**Separation of calcium and magnesium chlorides.** A. K. SMITH and C. F. PRUTTON, ASSRS. to DOW CHEM. CO. (U.S.P. 1,780,098, 28.10.30. Appl., 16.6.25).—The calcium and magnesium chloride mother-liquor obtained in the treatment of brine is evaporated and any tachydrite which separates is removed and treated for the separation of calcium as described in U.S.P. 1,627,068 (B., 1927, 628). The solution, containing the chlorides in the ratio  $\text{CaCl}_2 : \text{MgCl}_2 < 2 : 1$ , is further evaporated at  $120^\circ$  under reduced pressure, whereby the salt  $\text{MgCl}_2 \cdot 2\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$  crystallises. Evaporation is continued until the crystals begin to be contaminated with  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ; the temperature is then raised to  $140^\circ$ , causing the latter salt to dissolve, and the double salt crystals are collected and purified according to U.S.P. 1,768,797 (B., 1931, 246).

A. R. POWELL.

**Dehydration of magnesium chloride.** E. LACELL (B.P. 345,518, 5.4.30. Ger., 5.4.29).—The hydrated chloride is added in small portions to a relatively large mass of the fused chloride, or to a mixture of at least 1 mol. of potassium chloride with 2 mols. of magnesium chloride, maintained at or above  $500^\circ$  by the passage of an alternating current through the material.

L. A. COLES.

**Catalyst of high mechanical strength.** R. WIETZEL and E. WILLFROTH, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,875, 30.9.30. Appl., 29.2.28. Ger.,

3.3.27).—A component of a catalyst for the destructive hydrogenation of distillable carbonaceous materials is mixed into a paste with a soluble magnesium salt, preferably in aqueous solution, the product being heated to expel acid, and further components being then incorporated.

W. J. WRIGHT.

**Manufacture of [adsorptive] metal oxide [catalysts].** R. GRIESSBACH and J. EISELE, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,775,640, 16.9.30. Appl., 29.11.26. Ger., 11.12.25).—Thorium, thorium and aluminium, or aluminium and ferric hydroxides freshly precipitated by means of ammonia from a nitrate solution are washed free from soluble salts and beaten up to a thin paste with boiling water; nitric acid is added cautiously until a clear colloidal solution is obtained, which is evaporated to a jelly and on drying yields a glassy mass. Ignition of this mass at  $500$ – $600^\circ$  affords a highly adsorption oxide mixture of great catalytic activity.

A. R. POWELL.

**Improving the properties of activated bauxite and product thereof.** R. C. BENNER and A. P. THOMPSON, ASSRS. to GEN. CHEM. CO. (U.S.P. 1,778,517, 14.10.30. Appl., 21.1.27).—Bauxite is heated at  $400$ – $450^\circ$  for 4 hrs. to expel water of hydration and, while still warm, soaked in a 20–25% solution of ferric nitrate *in vacuo*, so that the solution enters the pores. After draining off excess solution the mass is again heated at  $400$ – $450^\circ$  for 4 hrs. The product is less friable than untreated bauxite and is highly active in promoting the reaction between sulphur dioxide and hydrogen sulphide.

A. R. POWELL.

**Production of titania [from bauxite residues].** R. M. PALMER (U.S.P. 1,760,992, 3.6.30. Appl., 25.5.26).—The residues from the Bayer process or from the sulphuric acid digestion of bauxite are concentrated on shaking tables and the concentrates, with or without a preliminary heat-treatment, are separated magnetically to obtain an ilmenite concentrate and a rutile tailing, which may be utilised in known manner for the recovery of ferrotitanium and pure titania, respectively.

A. R. POWELL.

**Purification of [fused] aluminium oxide.** J. S. MASIN, ASSR. to SWANN RESEARCH, INC. (U.S.P. 1,777,570, 7.10.30. Appl., 1.6.25).—Artificial corundum obtained by fusing bauxite with carbon in the electric furnace is purified by crushing it to the size desired for abrasive purposes and treating the powder with a solution containing 6% of hydrofluoric acid and 16% of sulphuric acid. After washing and drying, the finest particles which are very impure are removed by screening.

A. R. POWELL.

**Preparation of activated adsorptive clay.** W. S. BAYLIS, ASSR. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,776,990, 30.9.30. Appl., 1.9.27).—Bentonite is treated with sufficient acid to remove one half of the alumina in the principal hydrosilicate, and the product is separated from the water-soluble salts formed.

W. J. WRIGHT.

**Recovery of arsenic, antimony, and tin from mixtures of their salts [obtained in the Harris lead-refining process].** C. A. HEBERLEIN, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,779,272,

21.10.30. Appl., 21.5.26).—The alkaline slag from lead refineries is crushed and digested with cold water to remove the excess alkali and the greater part of the sodium stannate relatively free from arsenate and antimonate. The residue is then leached with hot water, which dissolves all the arsenate and the remaining stannate, leaving the antimonate insoluble. The solution is evaporated and cooled to recover sodium arsenate and the mother-liquors containing stannate are returned to the cold-leaching vats. A. R. POWELL.

**Manufacture of tin chlorides.** W. S. SMITH (U.S.P. 1,777,132, 30.9.30. Appl., 18.6.27).—Chlorine is passed into molten tin and the molten stannous chloride is removed. The vapours of volatile chlorides are fractionated to condense impurities, and the stannic chloride vapour is condensed to a liquid. W. J. WRIGHT.

**Production and employment of oxygen [for use, e.g., in gas-protection appliances].** W. W. TRIGGS. From INHABAD-GES.M.B.H. (B.P. 304,292, 22.10.29).—A mixture comprising approx. 58% of potassium chlorate, 23% of potassium perchlorate, 8% of ferrosilicon, 7.2% of asbestos fibre, 0.3% of cobalt carbonate, and 3.5% of calcium hydroxide, which has been compressed moist and then dried, is ignited by a primer in a container, and the oxygen liberated is passed through a filter before use. L. A. COLES.

**Conversion of carbon dioxide into monoxide particularly in the reduction of iron ores.** B. M. S. KALLING and C. VON DELWIG (B.P. 345,234, 7.12.29. Swed., 10.12.28).—The carbon dioxide or gas containing it passes through a rotating, inclined shaft furnace partly filled with carbon, preferably charcoal mixed with a little coke to increase the electrical conductivity, and the mass is heated locally by two sets of radially disposed electrodes which, by their shape, raise the carbon into the gas space above. L. A. COLES.

**Production of carbon dioxide at low temperatures.** C. A. THOMAS and C. A. HOCHWALT (U.S.P. 1,777,338, 7.10.30. Appl., 16.11.25).—Chlorosulphonic acid is caused to react with sodium hydrogen carbonate in the presence of a solution of calcium chloride. The mixture will generate carbon dioxide at temperatures down to  $-40^{\circ}$ . A. R. POWELL.

**Manufacture of cyanogen chloride.** J. P. SCHMITT-NÄGEL, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,779,984, 28.10.30. Appl., 25.1.27).—A suspension of finely-ground sodium cyanide in six times its weight of carbon tetrachloride containing 0.2% of 95% ethyl alcohol is treated with dry chlorine at  $0^{\circ}$  to  $-10^{\circ}$  until the cyanide is almost, but not quite, completely decomposed. Fractional distillation of the product affords pure cyanogen chloride, or the filtered liquid may be used directly in the manufacture of organic derivatives in which the presence of water is deleterious. A. R. POWELL.

**Obtaining hydrogen sulphide from aqueous solutions of alkali carbonate, bicarbonate, and hydrosulphide.** Soc. ANON. HOLLANDO-BELGE POUR LA FABR. DU COKE, Asses. of C. OTTO & Co. G.M.B.H. (B.P. 345,873, 13.5.30. Ger., 13.5.29).—After the aqueous solution has been treated with carbon dioxide to give a gas mixture rich in hydrogen sulphide, it is treated with this gas mixture to convert the carbonate

into bicarbonate and hydrosulphide. The solution is then treated with gas rich in carbon dioxide, the hydrosulphide being thus converted into bicarbonate and hydrogen sulphide. W. J. WRIGHT.

**Electrical fine-cleaning of sulphur dioxide-containing gases.** LODGE-COTTRELL, LTD. From METALLGES.A.-G. (B.P. 344,748, 26.6.30).—The reactions, e.g., deposition of sulphuric acid mist and arsenic compounds, initiated in an electrical precipitator are completed in a reaction space following the precipitator, the end of the space containing an electrical or mechanical separator, preferably a filter packed with quartz, coke, etc., to remove precipitated impurities. L. A. COLES.

**[Catalyst for] manufacture of sulphur trioxide.** CALCO CHEM. Co., INC., Asses. of A. P. BEARDSLEY and N. A. LAURY (B.P. 345,556—7, 13.5.30. U.S., 13.5.29).—(A) A catalyst for the contact process is prepared by repeatedly immersing calcined cellite in ammonium metavanadate solution, drying the product at  $110^{\circ}$ , treating it with rubidium chloride solution, and again drying. (B) Cæsium chloride is substituted for rubidium chloride in the above process. W. J. WRIGHT.

**Production of phosphorus [in ferrophosphorus manufacture].** J. W. WALTON, Assr. to TENNESSEE PRODUCTS CORP. (U.S.P. 1,772,849, 12.8.30. Appl., 22.9.27).—In the production of ferrophosphorus by smelting iron ore and phosphate rock in the blast furnace the former is reduced to metal much higher in the furnace than the zone in which the rock is reduced to phosphorus; hence the gases from the lower zones just above the tuyères have a high content of phosphorus vapour. These gases are withdrawn from the furnace and passed through a cooling system to condense the phosphorus, or they may be mixed with air to convert the phosphorus into pentoxide, which is absorbed in water in a suitable absorption system. A. R. POWELL.

**Liberation of iodine in iodine-containing mineral waters.** F. ELLINGER, Assr. to MIJNBOW EN HANDELSMAATS. "WONOH GEDEH" (U.S.P. 1,775,897, 16.9.30. Appl., 7.3.29. Holl., 10.3.28).—The water is treated with lime to remove bicarbonates as insoluble calcium carbonate and is then neutralised with sulphuric acid. The iodine is liberated from this solution by treatment with the theoretical quantity of permanganate and a little more than the theoretical quantity of sulphuric acid. Absorption of the liberated iodine is effected with activated charcoal. A. R. POWELL.

**Refrigerating medium. Separation of acidic gases. Solidification of gases. Container for corrosive materials.**—See I. Acid from petroleum refining. Filtering material.—See II. Catalytic processes.—See III. Cements and fertiliser materials.—See IX. Metal-hardening baths. Vanadium from its compounds.—See X. Granulation of fertilisers.—See XVI. Potassium salt from distiller's waste.—See XVIII.

## VIII.—GLASS; CERAMICS.

### PATENTS.

**Production of mirror glass.** PILKINGTON BROS., LTD., F. B. WALDRON, and A. HARRISON (B.P. 345,676, 27.12.29).—Glass sheets are conveyed automatically



through apparatus in which they are successively cleaned, coated with silver, coppered electrolytically, washed, and sprayed with paint. L. A. COLES.

**Solution for and method of polishing glass.** I. METH (U.S.P. 1,777,321, 7.10.30. Appl., 24.9.28).—The glass is immersed in a mixture of 7–9 vols. of sulphuric acid, 2–4 vols. of water, and 2–4 vols. of hydrofluoric acid, to which is added 3–5% of oleum and 0.1% of phosphorus pentoxide. A. R. POWELL.

**Manufacture of reinforced glass sheets.** N. B. MATTINGLEY, and LANCEGAYE SAFETY GLASS, LTD. (B.P. 344,454, 6.12.29).—The glass sheets are coated with a composition comprising a cellulose derivative (the nitrate), camphor, a gum (gum benzoin), and a high-boiling solvent of cellulose acetate and nitrate (triacetin); the sheets are then united with an interposed cellulose acetate sheet by compression and heat.

L. A. COLES.

**Reinforcement of fragile [glass and ceramic] articles and materials.** BRIT. CELANESE, LTD. (B.P. 345,758, 11.2.30. U.S., 12.2.29).—The articles are provided with an external coating 0.005–0.03 in. thick, which may be built up in layers of different composition, comprising synthetic resins (phenol- or diphenylolpropane-aldehyde; urea- or toluenesulphonamide-formaldehyde) and cellulose derivatives.

L. A. COLES.

**Strengthened glass and the like.** J. CHAMBERLAIN and H. PERIAM (B.P. 345,688, 2.1.30. Addn. to B.P. 316,755).—Two or more layers of solutions containing cellulose derivatives and/or synthetic resins are applied to glass sheets, the solvents are removed by evaporation after each application, and the whole is united by heat and compression.

L. A. COLES.

**Manufacture of objects from plastic particularly ceramic substances by rotation and pressure.** PORZELLANFABR. KAHLA ZWEIGNIEDERLASSUNG FREIBERG (B.P. 346,148, 31.3.30. Ger., 22.11.29).

**Fused alumina.**—See VII. Enamelling iron etc. —See X. Refractory supports for electric tubes.—See XI.

## IX.—BUILDING MATERIALS.

**Furnace dust in the manufacture of Portland cement.** O. REBUFFAT and V. MAZZETTI (Giorn. Chim. Ind. Appl., 1931, 13, 74–76).—Furnace dust is composed mainly of two constituents: (1) A more readily fusible silico-aluminate less rich in silica and lime, but richer in alumina, iron, and magnesium than the clinker; this predominates in the coarser portion of the dust. (2) A very fine, distinctly crystalline silico-aluminate richer in silica than the clinker and practically free from magnesia. The concentration of the magnesia in the more fusible part and the marked increase of silica content of the more refractory part are characteristic of furnace dust and are related to the mechanism of its formation. The finer portions of the dust are of uniform type in the different samples of furnace dust examined, and agree in composition with a mixture of 4 mols. of dicalcium silicate and 1 mol. of tricalcium aluminate.

T. H. POPE.

**Cement chemistry in theory and practice.** IV. Problem of hardening and its signification in

cement research. V. Chemistry of high-strength cements. VI. Technical aspect of high-strength cements. H. KÜHL (Cement, 1931, 4, 3–15, 161–168, 272–287; cf. B., 1931, 159).—IV. Conclusions in favour of the crystalloid theory of hydraulic hardening of cement, reached by reason of the observation of crystals of monocalcium hydrosilicate and calcium aluminium sulphate, together with an amorphous gel when cement reacts with a considerable quantity of water, are fallacious as no trace of these needles and plates are evident in thin sections of old cement test pieces. Michaelis' view that hardening is due to shrinkage of a gelatinous mass is accepted. It is suggested that a gel is formed on the surface of cement particles which cements the particles together, and the very slow hydration of the grain abstracts water and hardens the gel. Such internal adsorption is not completed for a number of years, as evidenced by the complete change of specimens 20 years old towards polarised light, the almost optically isotropic gel gradually becoming microcrystalline in structure.

V. The transition between sound and unsound cement is gradual with no clearly-defined boundary. Increase in lime up to a certain limit increases both the tensile and crushing values and then tends to cause unsoundness. The mixture should be adjusted so as to form alite and celite in the greatest possible quantities, and with this in view the composition of the clay should be carefully controlled. The highest strengths are obtained from cements either very rich in silica or very low in silica, e.g., % SiO<sub>2</sub>/%(Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>) = 3–4, or 1.2–1.4 for the low-silica product. The burning and grinding processes should be efficient, and in addition to gypsum other catalysts, e.g., chlorides of the alkali and alkaline-earth metals and of cadmium, aluminium, and iron resulted in increased strength, whilst manganese, tin, and especially zinc, copper, and lead were injurious to strength.

VI. The "ideal" raw mix, theoretically, is given by the expression  $x = [2 \cdot 8\text{SiO}_2 + 1 \cdot 1\text{Al}_2\text{O}_3 + 0 \cdot 7\text{Fe}_2\text{O}_3 - \text{CaO (for clay)}] / [2 \cdot 8\text{SiO}_2 + 1 \cdot 1\text{Al}_2\text{O}_3 + 0 \cdot 7\text{Fe}_2\text{O}_3 - \text{CaO (for limestone)}]$ , in which  $x$  represents the limestone to be used with 1 pt. of clay. A decided danger exists in a too finely-ground clinker, not only on account of the reduced density, but also of the reduced strength. More accurate methods of determining the grade than the usual screening are discussed. C. A. KING.

**Effect of storage conditions on the properties of cement.** D. A. G. REID (Cement, 1931, 4, 261–263).—The loss on ignition of cement stored in a closed timber shed, in a warm oven representing extreme conditions of heat, and in a cold-storage chamber was in all cases found to increase and may be taken as indicative of the degree of deterioration. No serious damage resulted provided the loss on ignition was less than 2%, or possibly 4% for rapid-hardening cement. The quantity of water required increased and the time of setting fluctuated without any definite trend. The ultimate strength was not so much affected as the rate of attaining that strength. A metal drum was definitely the best container and a wooden cask the next best, but there was little to choose between a four-ply paper and a jute sack.

C. A. KING.

**Wood staining.** A. C. HEINEMANN (*Farben-Ztg.*, 1931, 36, 1259—1260).—The technology of the process is discussed and various types of water-stain in use are described. The drawback of raising the grain of the wood, to which all of these are prone, is avoided by the use of "wax-stains," *i.e.*, partly saponified emulsions of beeswax, carnauba or other hard wax, coloured with potassium chromate, copper sulphate, or acid dyes, together with pigments and fillers if desired.

S. S. WOOLF.

**Calcium oxide.**—See VII. **Blood-albumin.**—See XIX.

## PATENTS.

**Production of hydraulic cement.** G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 345,103, 26.9.29).—A mixture of calcium carbonate (limestone) with clinker prepared by burning calcareous and argillaceous material is ground for use as cement, the quantities being such that the proportion of limestone to free calcium oxide in the clinker gives a mixture containing, approx.,  $\text{CaCO}_3 : \text{CaO} = 8 : 1$ , and  $\text{CaCO}_3 : \text{aluminium silicate and free silica} = \text{about } 3 : 1$ . L. A. COLES.

**Production of calcium aluminate cements and fertiliser materials.** H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,778,224, 14.10.30. Appl., 18.6.27).—A mixture of calcium phosphate and alunite (basic potassium aluminium sulphate), preferably with the addition of carbon, is heated at 1200—1600° in an electric furnace to yield a slag which, on grinding, serves as a calcium aluminate cement; potassium compounds and phosphorus volatilise and are worked up to a potash-phosphate fertiliser, or the phosphorus may be recovered separately as phosphoric acid or as the element.

L. A. COLES.

**Filtering of suspended sediments [e.g., cement slurry].** C. PONTOPPIDAN (B.P. 344,412, 21.11.29).—Filtration is facilitated by the addition of an electrolyte comprising a soluble compound of calcium, magnesium, barium, aluminium, iron, potassium, sodium, or ammonium, *e.g.*, calcium sulphate. [Stat. ref.]

B. M. VENABLES.

**Waterproofing of concrete constructions.** G. C. MARTIN (U.S.P. 1,779,481, 28.10.30. Appl., 14.5.28).—Freshly laid wet concrete is coated with a layer of hot asphaltum, whereby the steam produced causes rapid setting of the cement and effects an intimate bond between the concrete and the asphaltum.

A. R. POWELL.

**Coating material [for roof shingles].** J. V. FREEMAN (U.S.P. 1,775,193, 9.9.30. Appl., 8.6.27).—Calcined fireclay from coal-mine dumps is ground coarsely, saturated with a solution of ferrous sulphate or spent pickling liquor, dried, and again calcined to obtain a homogeneous, granular, red material suitable for the manufacture of red tiles. A. R. POWELL.

**Dry kiln [for timber etc.].** F. M. CREIGHTON, Assr. to MOORE DRY KILN Co. (U.S.P. 1,777,255, 30.9.30. Appl., 16.9.25).—A kiln for the continuous drying of timber etc. is operated with positive circulation by means of a fan and suitable ducts, the air currents being upwards at the dry end and downwards at the green end; humidifying sprays, directed in the same direction as the air,

are provided at the dry and green ends. Heating means such as steam pipes are provided in the lower part of the kiln, graduated from nothing at the green end to a maximum at the dry end. Air is vented out at positions that may be varied according to the class of timber being treated. B. M. VENABLES.

**Floor-covering materials.**—See XIII.

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

**Constitution and formation of the phosphide eutectic in cast iron.** M. KÜNKELE (*Mitt. Kaiser-Wilh.-Inst. Eisenforsch.*, 1930, 12, 23—31; *Chem. Zentr.*, 1931, i, 516).—In grey cast iron a ternary (mixed crystals— $\text{Fe}_3\text{C}-\text{Fe}_3\text{P}$ ) and a binary (mixed crystals— $\text{Fe}_3\text{P}$ ) eutectic occur; the latter is, however, actually a degenerated eutectic of which the third constituent, graphite, is crystallised on graphite flakes already present. A. A. ELDRIDGE.

**Influence of silicon content and speed of cooling on the constitution of the phosphide eutectic in cast iron.** P. BARDENHEUER and M. KÜNKELE (*Mitt. Kaiser-Wilh.-Inst. Eisenforsch.*, 1930, 12, 33—38; *Chem. Zentr.*, 1931, i, 516).—In alloys free from manganese, up to 2% Si the ternary phosphide eutectic is formed under the conditions employed; when more than 2% Si is present the "pseudobinary" eutectic is formed. A manganese content of 0.7% is without influence. Slow cooling or increase in phosphorus content favours the formation of the pseudobinary eutectic.

A. A. ELDRIDGE.

**Effect of speed of cooling on the transformation temperature, structure, and fine structure of iron-carbon alloys.** F. WEVER and N. ENGEL (*Mitt. Kaiser-Wilh.-Inst. Eisenforsch.*, 1930, 12, 93—114; *Chem. Zentr.*, 1931, i, 515).—An apparatus for the quenching of wire in a current of hydrogen is described; cooling speeds up to 5000° per sec. can be attained. A three-dimensional model expresses the relation between carbon concentration, temperature, and speed of cooling. The appearance of a martensitic structure is connected with that of a tetragonal space-centred phase, the axial ratio increasing from unity for pure iron to 1.064 for 1.8% C. A. A. ELDRIDGE.

**Rôle of hydrogen cyanide in the corrosion of iron.** J. F. G. HICKS (*J. Physical Chem.*, 1931, 35, 893—904; *cf. B.*, 1929, 521).—The relative susceptibility to the action of hydrogen cyanide of various iron compounds which are possible intermediates formed during internal corrosion in gas mains has been determined. The accelerating effect of oxygen and carbon dioxide on corrosion of iron by hydrogen cyanide has also been studied. Hydrogen cyanide is the chief factor in the internal corrosion of gas mains, with carbon dioxide and oxygen next in order of attack; it is not, however, the cause of corrosion, which it cannot initiate. The cause is the dissolution of metallic iron in water. Corrosion by hydrogen cyanide does not differ from other similar processes with respect to mechanism. The final product is Prussian-blue. L. S. THEOBALD.

**Influence of metallic coatings on the mechanical properties of steel undergoing nitridation.** T. YOSIKI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 15, 143—154).—Nitridation of steel was effected by heating the steel at 650° for various time intervals in a stream of ammonia. The tensile strength of the specimens increased with the duration of the treatment, whilst the ductility decreased at first, afterwards becoming nearly constant. The values of these properties were reduced by previously plating the steel with tin. Bending tests showed that coatings of tin, tin-lead alloys, cadmium, and cadmium-lead alloys applied by hot-dipping offer considerable resistance to the penetration of nitrogen into the steel. Some results of impact tests on a chromium-nickel steel are recorded.

E. S. HEDGES.

**Apparatus for the works' determination of the total zinc coating on steel.** GOEKE (Chem. Fabr., 1931, 147—148).—The apparatus comprises a 100-c.c. glass bottle provided with a large hood-shaped glass stopper in which the specimen is placed prior to a test. The stopper carries a short tube with stopcock and a long tube terminating at the bottom of the bottle in a short upward bend and extending horizontally outside the bottle to a measuring burette, above which it terminates in a stopcock. The sample of galvanised steel is placed in the hood, the bottle and long tube are filled with *N*-hydrochloric acid containing 3% of antimony trichloride, and after fixing in the stopper, the specimen is dropped into the acid and the stopcock above the burette opened. The volume of acid displaced when multiplied by 0.00272 gives the weight of zinc present in g.; this weight divided by 7100 times the area of the specimen gives the thickness of the coating in mm.

A. R. POWELL.

**Influence of carbon on the critical points and hardness of 12% chromium steels.** T. MURAKAMI and Y. FUJII (Chikashige Anniv. Vol., 1930, 25—47).—By measurement of the thermal dilatation and magnetic properties of steels containing about 12% Cr the influence of carbon on the critical points has been studied. Increase of the carbon content lowers slightly the temperature of the A1 transformation and raises that of the A2 transformation; the A<sub>1</sub> change is not indicated in the magnetic analysis. The lowering of the critical point or the extent of the Ar<sub>2</sub> transformation depends on the maximum heating temperature and on the rate of cooling; at low rates the lowering is more pronounced in the low-carbon steels, but with rapid cooling it becomes marked with high-carbon steels. The hardness (Brinell) of fully annealed specimens increases as the carbon content is increased from 0.09% to 2.04%. The hardness-quenching temperature curves exhibit maxima at 1000°, independently of the carbon content; the curves for different specimens intersect only at higher temperatures, and there is a maximum hardness at 0.3% C, and a minimum at 1% C when the quenching temperature is 1200°. The cooling rate, provided that it is greater than that of air-cooling, has but little influence. The hardness-tempering temperature curves of the low-carbon steels, quenched from 900°, exhibit maxima at 500°; all the specimens exhibit these maxima when quenched from 1100°, and

a 2.04% carbon steel attains a considerably greater hardness than when in the quenched condition. Photomicrographs of the steels are reproduced and discussed.

H. F. GILLBE.

**Microstructure and hardness of quenched manganese steels.** T. MURAKAMI and K. HATSUTA (Chikashige Anniv. Vol., 1930, 229—236).—Manganese steels containing 2.07—12.44% Mn and 0.38—1.19% C, quenched from 900° and 1200°, have been investigated. The quenched low-manganese steels consist of martensite, and the austenite increases, at the expense of the martensite, as the manganese content increases; free carbide exists in the high-carbon steels quenched from 900°. The transformation austenite→martensite takes place readily during quenching in low-carbon, high-manganese steels, and the austenite content increases with increase of the carbon content owing to suppression of the transformation. Martensite is readily formed by quenching at 1200° specimens which consist of austenite if quenched from 900°. Quenching in oil or water produces practically the same structure. The effects described are attributable, not to the rate of cooling, but to quenching stress, which favours the production of martensite. The Shore hardness number of the quenched steels decreases with increase of the manganese content, especially with high-carbon steels, and is independent of the quenching liquid; the quenching temperature has but little influence on the hardness of the high-carbon steels, but its effect is appreciable with steels containing 0.4—0.7% C.

H. F. GILLBE.

**Determination of silver in steel.** W. BERG (Chem.-Ztg., 1931, 55, 259).—The steel (10—15 g.) is dissolved in 250 c.c. of 1:1 hydrochloric acid, the solution evaporated to dryness, the residue dissolved in dilute hydrochloric acid, and the insoluble silver chloride and silica are collected. The filtrate is treated with hydrogen sulphide, the precipitate dissolved in aqua regia, and the solution made ammoniacal and filtered. The filtrate is joined with the solution obtained by treating the siliceous residue with ammonia and the silver precipitated by making the solution feebly acid with hydrochloric acid. After keeping overnight in a dark place, the silver chloride is collected in a glass filter crucible, washed, dried at 130°, and weighed.

A. R. POWELL.

**Treatment of Hollinger precipitate to produce fine gold.** M. SCOTT (Bull. Inst. Min. Met., 1931, No. 318, 10 pp.).—The mill solution of gold etc. in calcium cyanide is treated with 0.045 lb. of zinc per ton, whereby a precipitate is obtained containing 35% Au, 7% Ag, 1.5% Cu, 9.3% Pb, 14.6% Zn, 11.7% CaO, 4.4% S, 0.8% (Fe,Al)<sub>2</sub>O<sub>3</sub>, and 0.7% SiO<sub>2</sub>. The precipitate is digested with hydrochloric acid and steam, such treatment removing 53% of its weight and leaving a residue containing 75.8% Au, 15.4% Ag, 2.3% Cu, 1.54% Zn, 0.76% S, and small amounts of lead, lime, and silica. This residue is digested with hot sulphuric acid as in the usual parting method, and the resulting sponge gold is washed first with hot water, then with sodium acetate solution to remove lead sulphate, and is finally melted with silica, borax, and manganese dioxide to obtain gold bars of 99.4—99.7% purity. The silver sulphate liquor is diluted with water and the

silver fractionally precipitated with aluminium powder, dried, briquetted, and melted with soda and borax. The filtrate from the silver is treated with more aluminium to precipitate the copper and last traces of silver. All the waste solutions from the process are passed to a settling tank in which the lead collects as sulphate together with silver chloride and a little fine gold. This precipitate is periodically smelted in a small water-jacketed cupola to obtain lead bullion, which is cupelled to obtain doré bars for further refining.

A. R. POWELL.

**Extraction of copper from oxidised ores by cyanide solution.** E. T. DUNSTAN (J. Chem. Met. Soc. S. Afr., 1931, 31, 190—193).—Leaching tests with a crude commercial cyanide solution on Rhodesian oxidised copper ores showed that a good extraction of copper could be obtained, but that cyanide consumption was exceedingly high and no cyanide could be regenerated by electrolysis of the liquors to recover the copper.

A. R. POWELL.

**Cyanidation of a copper-gold ore.** E. H. SMITH (Canad. Chem. Met., 1931, 15, 45—46).—The importance of an adequate addition of lime to an ore pulp containing copper sulphide minerals is illustrated with reference to the cyanidation of an Ontario gold ore; using 6 lb. of lime per ton, the cyanide consumption was 1.5 lb./ton and the zinc consumption for the complete precipitation of gold about 0.1 lb./ton, whilst the melted bullion was 700-fine. With only 1.5 lb. of lime per ton the corresponding figures were 6 lb./ton, 0.22 lb./ton, and 400-fine, respectively, and the cyanide liquors contained such a large proportion of copper that part had to be run to waste in every cycle.

A. R. POWELL.

**Effect of combinations of strain and heat treatment on properties of age-hardened copper alloys.** W. C. ELLIS and E. E. SCHUMACHER (Amer. Inst. Min. Met. Eng., Tech. Pub., 1931, No. 395, 13 pp.).—Hard drawing after heat treatment increased the tensile strength of a copper (96%)–nickel–silicon alloy. The properties were further improved by low-temperature ageing. Copper–cobalt–silicon alloys showed less pronounced ageing effects.

CHEMICAL ABSTRACTS.

**Changing over from acid to alkaline circuit in flotation practice at the mill of the Zinc Corporation, Ltd., Broken Hill, N.S.W., Australia.** R. D. NEVETT (Proc. Austral. Inst. Min. Met., 1930, 343—353).—The ore consists of galena, marmatite, calcite, and quartz with small quantities of pyrite, fluorspar, and rhodonite. Excellent separation of the galena and marmatite from one another and from the pyrites have been obtained by selective flotation in an alkaline circuit of  $p_{H}$  8.1—8.8. The ground ore pulp after thickening is agitated with 0.8 lb. of "Aero-cyanide" per ton for 6 min., then passed to a series of 14 flotation cells in which the lead is floated with 0.08 lb. of "Vallo C" oil and 0.01 lb. of potassium xanthate per ton. The tailings from these cells are treated with 2 lb. of copper sulphate, 0.8 lb. of sodium aerofloat, and 0.25 lb. of eucalyptus per ton, whereby the marmatite is floated. The average grade of lead concentrate contains 75.7% Pb and 3.6% Zn, and that of the zinc concentrate 50.4% Zn, 8.9% Fe, and 2.2% Pb. Flow-sheets

for the slime and sand sections of the plant are included.

A. R. POWELL.

**All-flotation [of ores] at North Broken Hill, Ltd.** A. LOWRY (Proc. Austral. Inst. Min. Met., 1930, 355—377).—The ore is crushed so that 16% remains on a 40-mesh screen, and the slimes are removed by a series of classifiers. The coarse sand is made into a 2 : 1 pulp with a 0.005% solution of sodium carbonate in a vortex mixer in which is also incorporated 0.04 lb. of eucalyptus-coal tar (1 : 1), 0.02 lb. of potassium xanthate, and 0.06 lb. of sodium silicate per ton of ore. The pulp passes to the flotation cells of the impeller type with additional aeration by means of compressed air passed down the impeller shaft; flotation of galena as a clean product containing 75% Pb is almost instantaneous and the cost of reagents is only 1.6d. per ton of ore (14% Pb) treated. The tailings from the lead cells are treated in an acid circuit for the recovery of their zinc content. The slime overflow from the classifiers is thickened and floated in a separate unit in an alkaline circuit for the recovery of the galena as a concentrate containing 69.3% Pb, 8% Zn, and 45 oz. of silver per ton. The total recovery of lead from both sections exceeds 95%.

A. R. POWELL.

**Determination of calcium and magnesium in roast blende.** L. SCHREIBER (Ann. Chim. analyt., 1931, [ii], 13, 98—102).—The mineral is extracted with hydrochloric acid for  $\frac{1}{2}$  hr. to remove hydrogen sulphide, nitric acid is added, and the whole taken to dryness to render silica and barium insoluble. Successive treatment is given with hydrochloric acid, a large volume of water, hydrogen peroxide, and ammonia. After filtration, if all the lead has been precipitated, calcium and magnesium are precipitated directly as oxalate and phosphate, respectively; otherwise ammonium acetate is added.

T. McLACHLAN.

**Oxygen in aluminium and its determination.** T. HARADA (Chikashige Anniv. Vol., 1930, 237—243).—Grard's method for the determination of oxygen in aluminium has been modified by passing the chlorine, before use, over heated carbon, in order to convert any free oxygen into carbon monoxide, and by purification of the residue, prior to its analysis, with hydrochloric acid. The alumina present in aluminium containing less than 0.38% O is in the form of a solid solution, whilst above this limit it exists merely as an admixture. Melting the metal under ordinary conditions always raises the oxygen content to above this critical value, and microscopical examination thus serves to indicate whether or not a sample has been remelted. Various commercial samples of aluminium contained 0.29—0.48% O.

H. F. GILLBE.

**Selective dissolution of magnesium corrosion products.** L. WHITBY (J.S.C.I., 1931, 50, 83—85 T).—A boiling 20% aqueous solution of pure chromic anhydride may be used to remove rapidly corrosion products from magnesium and many magnesium-base alloys. Solubility of the metal in this reagent may be neglected for most purposes. The activating effect of chloride present in some corrosion products may be inhibited by adding a small amount of silver chromate to the chromic acid used; small quantities of sulphate exert no activating action.

**Anisotropy of zinc sheets.** E. SCHMID and G. WASSERMANN (Z. Metallk., 1931, 23, 87—90).—Rolled zinc has a non-homogeneous structure, so that the physical properties vary according to the orientation of the specimen to the direction of rolling. Thus with sheet 2.27 mm. thick a specimen taken parallel to the direction of rolling had a yield point of 148 kg./mm.<sup>2</sup>, a tensile strength of 28.5 kg./mm.<sup>2</sup>, an elongation of 12%, and a coefficient of thermal expansion, between 30° and 50°, of  $30.5 \times 10^{-6}$ , whereas the corresponding values for a specimen taken transverse to this direction were 18.8, 35.9, 3, and  $18.7 \times 10^{-6}$ , respectively. The surface structure of rolled sheets of zinc comprised a simple fibrous texture with the hexagonal axis in the normal of the sheet with a scattering of 80°. The existence of this surface texture tends to diminish the anisotropic behaviour of commercial rolled sheets of zinc.

A. R. POWELL.

**"Flash-point" test for lead.** R. S. RUSSELL (Proc. Austral. Inst. Min. Met., 1930, 307—319).—The test consists in noting the temperature at which rapid dissolution takes place when the lead is heated with concentrated sulphuric acid at a uniform rate. Tests on numerous specimens of lead of varying degrees of purity and of different ages have shown that the test is of no value in determining the resistance of the metal to corrosion. In general, antimony tends to lower the "flash point" and copper tends to raise it. Samples cut from a piece of lead which had been 22 years in a sulphuric acid chamber gave "flash points" varying from 183° to 313.°

A. R. POWELL.

**Changes in the mixed crystals in heavy-metal alloys.** D. UNO (Chikashige Anniv. Vol., 1930, 215—221).—A discussion, showing the characteristic differences in the hardening processes of steel and light- and heavy-metal alloys.

H. F. GILLBE.

**Testing of [metallic] materials for apparatus to be used at high temperatures and pressures.**

E. FRANKE (Chem. Fabr., 1931, 133—135).—Materials suitable for high temperature and pressure, such as cast steels containing molybdenum or vanadium or 3.5% nickel steel, require much more elaborate mechanical tests than were formerly considered necessary. In particular the effect of ageing at high temperatures must be determined. For this purpose the notched-bar test is employed. Another piece of the metal is then stretched 10% in the cold, immediately raised to 250°, maintained at that temperature for 30 min., and then similarly tested. Determinations of the creeping and stretching limits under the temperature to be used in practice may be necessary to ensure that no deformation can occur. The requirements of suitable testing machines are discussed.

C. IRWIN.

**Effect of rate of bending in notched-bar bending tests [of metals].** J. G. DOCHERTY (Engineering, 1931, 131, 347—350, 414—415).—For all the ductile metals tested, the energy absorbed in bending or fracture increased with the speed of the test and the "impact" effect is merely the increase due to the normal velocity effect. With the less ductile metals the decrease in load began much earlier in the test and the rate was much more rapid, indicating that the cracking effect pre-

dominates. Certain relations between the notched-bar and ordinary tensile tests were noted. C. A. KING.

**Hardness testing of electrodeposits and other thin metallic coatings.** H. O'NEILL (Trans. Faraday Soc., 1931, 27, 41—51).—A machine similar to that described previously (B., 1928, 787) has been found suitable for the indentation and scratch testing of coatings down to less than 0.0005 in. in thickness provided they are not much harder than 400 Brinell. Harder deposits might be tested by the use of a 0.5-mm. hemispherical diamond indenter. Scratch tests are described on nickel deposits from baths of different composition. F. G. TRYHORN.

**Arrangement of the micro-crystals of white tin deposited by electrolysis.** H. HIRATA, H. KOMATSUBARA, and Y. TANAKA (Chikashige Anniv. Vol., 1930, 261—273).—X-Ray and photomicrographic examination of electrolytically deposited white tin shows that at low-current densities and concentration of the electrolyte the crystals grow to a considerable size, but that when both these factors are high the deposit assumes a fibrous structure. The normals to the octahedral faces (111) of the crystals are oriented parallel to a common axis.

H. F. GILLBE.

**Stainless metals.** (SIR) H. C. H. CARPENTER (J. Roy. Soc. Arts, 1931, 79, 557—592).—A lecture.

**Nickel-plating baths.**—See XI.

## PATENTS.

**Hardening baths for iron and steel.** A. STÄHLER (B.P. 345,677, 27.12.29. Ger., 29.12.28).—A cementation bath consists of equal weights of calcium and potassium chlorides, together with 8% of potassium carbonate or hydroxide, 1.5% of potassium cyanide or ferrocyanide, and 4% of finely-divided carbon. The hardening mixture is preferably made into lumps, which are immersed in the bath inside a perforated cartridge. To prevent oxidation and as a heat-insulating medium a layer of calcium cyanamide is kept on the surface of the bath. A. R. POWELL.

**Enamel coatings for iron and steel articles.** O. HOMMEL (B.P. 344,979, 31.12.29).—The metal surface is wetted, by spraying or dipping, with a solution of a cobalt, nickel, or manganese salt, with or without a binder (e.g., tannin or aluminium silicate), and the article is then heated at a temperature at which the solution reacts with the metal surface to form thereon a thin film of iron oxide and the oxide of the coating metal. The enamel coating is then applied in the usual way to this treated surface. A. R. POWELL.

**Manufacture of stainless cast-iron articles.** B. VERVOORT (B.P. 344,399, 27.8.29).—The casting is made from an iron alloy containing 10—20% Cr, with or without nickel, and the parts which are to remain stainless are given a high polish. A. R. POWELL.

**Protection of iron pipes, tubes, bars, etc.** J. & O. KREBBER, GES.M.B.H. (B.P. 345,379, 9.1.30. Holl., 10.1.29).—The pipes are coated with a mixture of asphalt with 10—15% of mineral oil and 40% of benzene, then, after drying, with a layer of millboard strip wound spirally, and finally with a layer of bitumen and talc, stone dust, sawdust, or lime. A. R. POWELL.

**Removing a lead coating from [iron] articles.**

G. DE DUDZEELE (B.P. 345,107, 29.3.30).—The coated article is treated with a saturated solution of sodium chloride containing 5% of sodium hydrogen sulphate, 2–5% of sodium nitrate, 2–5% of sodium acetate, and 1–10% of sulphuric acid. A. R. POWELL.

**Acid inhibitors [for steel-pickling baths].**

E. I. DU PONT DE NEMOURS & Co. (B.P. 344,419, 28.11.29. U.S., 21.6.29).—A solution in concentrated sulphuric or hydrochloric acid of the condensation product of an amine, *e.g.*, aniline or toluidine, an aldehyde, *e.g.*, butaldehyde, and carbon disulphide is claimed. A. R. POWELL.

**Protective compound [inhibitor for steel-pickling baths].**

L. J. CHRISTMANN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,779,961, 28.10.30. Appl., 18.9.29).—The condensation product of aldehyde-ammonia and phenylthiocarbamide is claimed. A. R. POWELL.

**Cleaning and preservation of metallic [iron or steel] surfaces. [Inhibitor for pickling baths.]**

J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,779,787, 28.10.30. Appl., 2.8.28).—The inhibitor claimed is made by heating creosote oil with sulphuric acid at 120–125°. A. R. POWELL.

**Preventing the dissolution of metal [iron] in acids [pickling baths].**

J. H. GRAVELL (B.P. 344,404, 26.10.29).—A solution of a condensation product of an aldehyde with ammonia, an amine, or a derivative of thiocarbonic acid is added to the pickling bath in such a way that it forms therein a finely-divided suspension which is stabilised by the addition of a protective colloid, *e.g.*, sulphite-cellulose waste liquors, starch flour, gums, etc. [Stat. ref.] A. R. POWELL.

**Alloys [low-carbon special steels].**

(SIR) R. A. HADFIELD (B.P. 344,500, 5.12.29).—Steel resistant to oxidation at high temperatures and having a high creep limit contains less than 1–1.5% each of carbon, chromium, and copper and less than 3% Mo, the copper content being less than the chromium content; *e.g.*, the steel may contain 0.09–0.12% C, 0.43–0.58% Cr, 0.34–0.38% Cu, 0.9% Mo, 0.05% Si, and 0.15–0.35% Mn. A. R. POWELL.

**Steel [alloy for rails etc.].**

P. P. MARTHOREY (B.P. 344,822 and Addn. B.P. 344,893, [A] 11.12.29, [B] 13.12.29. Fr., [A] 11.12.28).—(A) The steel contains 0.2–0.6 (0.325)% C, 0.2–4 (3.1)% Ni, 0.4–2.5 (1.4)% Cr, 0.3–2.0 (0.4)% Mn, 0.1–0.5 (0.225)% Si, 0.1–1.5 (0.2)% Mo or W, and the usual small amounts of phosphorus and sulphur. Up to 0.5% V, 0.1% Ti, and/or 0.3% Cu may also be added. (B) The nickel content is raised to 6% and the manganese content reduced to 0.3%; the surface layers are hardened by heating rapidly with a non-oxidising oxy-acetylene flame followed by air-cooling. A. R. POWELL.

**Alloy steel.**

J. A. NELSON, Assr. to BARBER-COLMAN Co. (U.S.P. 1,778,226, 14.10.30. Appl., 14.9.25).—Steel for cutting tools, files, etc. contains 1.05–1.15% C, 7.5–8.5% Cr, 8–9% W, 1–1.5% V, 7–9% Co, 0.2–0.3% Si, 0.2–0.3% Mn, and less than 0.03% S and 0.03% P. The tools are soaked at 1040°, quenched, and tempered at 535°. A. R. POWELL.

**Compositions of matter for making tools and the like.**

FIRTH-STERLING STEEL Co., Asses. of E. B. WELCH (B.P. 345,167, 11.6.30. U.S., 27.9.29).—Tungsten carbide, fused zirconia, or carborundum powder is mixed with cobalt powder which has been coated with a film of copper or chromium or with films of nickel or copper and of chromium, the mixture is ground together until homogeneous, pressed into the desired shape, and heated in a non-oxidising atmosphere at 1500–1600° until thoroughly sintered. A. R. POWELL.

**Manufacture of [cutting] tools.**

F. KRUPP A.-G. (B.P. 345,171, 14.6.30. Ger., 6.11.29).—Between the steel shank and the cutting edge of hard alloy is inserted a sheet of molybdenum, nickel-iron, or chromium-iron alloy having a coefficient of expansion equal to that of the hard alloy and the whole is welded together. A. R. POWELL.

**Manufacture of iron-nickel alloys [for loading coils].**

GEN. ELECTRIC Co., LTD., G. C. MARRIS, G. R. POLGREEN, and S. V. WILLIAMS (B.P. 345,736, 22.1.30).—A mixture of nickel and iron oxides in the desired proportions (*e.g.*, 80:20) is ground finely and reduced at 850° in hydrogen. The resulting slightly sintered sponge is pulverised, washed with 5% hydrochloric acid at 50°, then with cold water, dried, and reheated at 1000° for 1 hr. in a reducing atmosphere. The product is ground in a ball-mill to obtain smooth even particles which are mixed with 2.5% of fine talc powder, annealed at 900°, and formed into magnetic cores in known manner. A. R. POWELL.

**[Iron-aluminium] alloy and articles [furnace parts] manufactured therefrom.**

H. E. COLEY, LTD., and C. J. NEWTON (B.P. 345,693, 4.1.30).—An alloy of 25% Al and 75% Fe with less than 0.1% C is claimed for use in the construction of metal retorts and furnace parts which come into contact with gases containing sulphur compounds. A. R. POWELL.

**Nitrogenising of alloys of iron or steel.**

F. KRUPP A.-G. (B.P. 345,238, 16.12.29. Ger., 20.12.28).—The ammonia atmosphere in the usual nitrogenising containers is kept in violent motion by means of a revolving screw or the like. A. R. POWELL.

**[Nitrogenisation] treatment of iron and steel.**

M. HIRSCH (U.S.P. 1,772,866, 12.8.30. Appl., 2.7.27).—Steel, especially molybdenum steel, may be case-hardened by heating it with carbamide, cyanamide, hexamethylenetetramine, or other nitrogenous compound at a temperature sufficient to cause the liberation of nascent nitrogen. A. R. POWELL.

**[Nitrogen case-]hardening of [chromium or manganese] alloy steels.**

H. SUTTON, A. J. SIDERY, and B. EVANS (B.P. 345,659, 19.12.29, 23.6. and 25.8.30).—The surface of the metal is coated with copper, silver, platinum, cobalt, molybdenum, or arsenic by electroplating, spraying, or sherardising methods, and the metal is then heated at 450–650° for 4–90 hrs. in a current of ammonia. A. R. POWELL.

**[Reagent for the] concentration of [copper] ores by flotation.**

E. K. BOLTON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,000, 28.10.30. Appl.,

30.11.25).—A mercaptothiazole, especially 1-mercapto-3-methylbenzthiazole or  $\alpha$ -naphthathiazole, is claimed.

A. R. POWELL.

**Selective agent for concentrating ores.** R. LUCKENBACH (U.S.P. 1,780,024, 28.10.30. Appl., 21.12.28).—A mixture of 7 pts. of a heavy petroleum oil of an asphalt base and 2 pts. of horse-grease oil is claimed as a flotation agent.

A. R. POWELL.

**Obtaining metals from sulphide ores.** C. GOETZ (B.P. 345,351, 23.10.29).—The coarsely-ground ore is mixed with finely-divided bituminous coal and a catalyst, e.g., lime, hæmatite, or ferrous sulphate, and the mixture is briquetted with the aid of oil or tar. After drying, the briquettes are broken into coarse pieces which are heated at 600–1000° for several hrs., whereby the sulphides are reduced to metals with the evolution of organic sulphides, hydrogen sulphide, and sulphur. The metals are then recovered from the mass by gravity concentration or magnetic separation. (Cf. B.P. 342,293; B., 1931, 353.)

A. R. POWELL.

**Recovery of metals from metalliferous materials, e.g., ores.** MEYER MINERAL SEPARATION CO., ASSEES. OF R. F. MEYER (B.P. 344,492, 3.12.29. U.S., 28.12.28).—Roasted sulphide ores of zinc, copper, cobalt, or nickel or roasted manganese ores are treated in a damp, pulverulent condition with sulphur dioxide and/or chlorine to convert the above metals into compounds soluble in water, which are then leached out in the usual way. The residue, if desired, is in a suitable condition for treatment with cyanide for the extraction of gold and silver.

A. R. POWELL.

**Manufacture of metal [coarsely crystalline copper].** J. G. DONALDSON and H. L. COLES, ASSRS. TO GUARDIAN METALS CO. (U.S.P. 1,775,159, 9.9.30. Appl., 21.12.26).—Molten copper is deoxidised with 20% of cuprosilicon, and 0.125–0.25% of finely-ground "alberene stone" (an aluminium magnesium silicate) is thoroughly incorporated with the molten metal, which is then allowed to solidify at such a rate that a coarsely crystalline structure is obtained with the silicate deposited in the amorphous material between the grains.

A. R. POWELL.

**Production of feathery copper powder.** W. KOEHLER, ASSR. TO KOEHLER CHEM. CO. (U.S.P. 1,777,371, 7.10.30. Appl., 22.1.30).—A solution containing 0.5–6% Cu, 0.5–6% of a metal more electropositive than copper, and 0.5–10% of free acid is electrolysed at 25–50° with a cathode current density of 40–240 amp./ft.<sup>2</sup>, and the deposited, voluminous, feathery deposit of copper is removed by brushing at frequent intervals. The preferred bath contains 2% Cu and 2% Na or Zn as sulphates with 6% of free sulphuric acid, and is operated at 30° with a cathode current density of 70 amp./ft.<sup>2</sup>

A. R. POWELL.

**[Copper-nickel] alloy.** R. WELLESLEY (B.P. 345,445, 24.2.30).—An alloy made by melting 25–46 oz. of copper, 16–30 oz. of nickel, 6–14 oz. of zinc, 3.5–7.5 oz. of 20% manganese-chromium, 1.5–4 oz. of 20% manganese-copper, 1–1.5 oz. of 15% molybdenum-nickel, and 1–2.5 oz. of an alloy containing 45.5% Sn, 32% Al, 4.5% Cd, 13.5% Mg, and 4.5% Bi is claimed.

A small quantity of vanadium, silicon, and/or boron may also be added.

A. R. POWELL.

**Production of coked metalliferous agglomerates [zinc oxide briquettes].** NEW JERSEY ZINC CO., ASSEES. OF E. H. BUNCE (B.P. 344,569 and 344,570, 23.12.29. U.S., [A] 30.7.29, [B] 11.9.29).—(A) Zinc oxide material and bituminous coal are formed into briquettes containing 6–12% of moisture, and these are brought into contact with a stream of hot gases, so that the surface layers are rapidly heated to about 800°, at which temperature they are promptly converted into a shell of coke; heating is continued until coking is completed throughout the briquette. (B) Heating is effected by large volumes of hot non-oxidising gases passed through the briquettes in a relatively narrow shaft furnace or in a relatively thin bed in a coking chamber, gas and briquettes moving in either case countercurrent to one another.

A. R. POWELL.

**Refining of zinc.** F. R. KEMMER, ASSR. TO AMER. CYANAMID CO. (U.S.P. 1,779,973, 28.10.30. Appl., 2.5.28).—Electrothermal zinc is heated at 700° in an electric resistance furnace, whereby it separates into a lower layer of lead, a middle layer of iron-zinc alloy, and an upper layer of relatively pure zinc. This zinc is tapped off and cooled to 450° to allow more iron-zinc alloy to separate, and the purified zinc is then redistilled in an electric furnace. The iron alloy crusts are returned to the electrothermal reduction furnace and the residues from the redistillation of the zinc are worked up for the recovery of gold, silver, and copper.

A. R. POWELL.

**Annealing and zinc-coating ferrous wires, strip, etc.** W. H. POTTER (B.P. 345,598, 19.7.30).—The wire or strip is passed through a molten mixture of 10% of calcium cyanamide, 35% of sodium carbonate, and 55% of sodium chloride floating above a bath of lead at 800°, thence through an acid pickling bath, a water rinsing bath, an alkaline flux, and finally through zinc at 450°.

A. R. POWELL.

**Aluminium alloy.** METAL CASTINGS, LTD., and A. H. NICHOLSON (B.P. 345,823, 1.4.30).—The alloy contains 3.5–4.5% Cu, 0.8–1.3% Ni, 1.2–1.7% Mg, 0.25–1% W, and 0.25–1% V, the sum of the nickel, tungsten, and vanadium contents being 1.8–2.3%.

A. R. POWELL.

**Casting of magnesium and its alloys.** H. KELLEY, ASSR. TO DOW CHEM. CO. (U.S.P. 1,772,490, 12.8.30. Appl., 13.1.26).—The mould cores are saturated with sulphur dioxide by allowing sulphur to burn in the core-baking oven. The presence of this gas in the mould prevents burning of magnesium or its alloys in the sprue or on the surface of the casting.

A. R. POWELL.

**Provision of insulating coatings on aluminium and its alloys.** A. R. DUNTON, A. A. POLLITT, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 345,437, 17.2.30).—The metal is boiled in a 0.5% solution of barium hydroxide containing in suspension 0.5% of barium, calcium, or strontium sulphate, whereby it becomes coated with an adherent film which can be varnished or impregnated with oil to improve its resistance to corrosion and electrical insulating properties.

A. R. POWELL.

**Recovery of tin from scrap tinned metal and like materials carrying recoverable tin.** H. WADE, J. W. HINCHLEY, and J. D. PARSONS (B.P. 344,899, 13.12.29. Addn. to B.P. 327,997; B., 1930, 618).—A solution of lead hydroxide in hot sodium hydroxide solution is used as the detinning solution. The deposited lead is redissolved in acetic acid and the solution treated at 70–95°, with sodium hydroxide to obtain lead hydroxide for use again. The sodium stannate solution is treated with lime to regenerate sodium hydroxide for use as a precipitant for the lead acetate solution.

A. R. POWELL.

**Refining of bismuth.** T. H. DONAHUE, Assr. to CERRO DE PASCO COPPER CORP. (U.S.P. 1,778,292, 14.10.30. Appl., 8.7.29).—A stream of chlorine is passed through molten bismuth, whereby lead and other impurities are converted into a chloride slag. When clouds of bismuth chloride vapours begin to be evolved the chlorine is stopped, the slag removed, and the metal stirred with molten caustic soda to remove adsorbed chlorine.

A. R. POWELL.

**Lead alloy, particularly for cable sheathings.** M. THIELERS (U.S.P. 1,779,784, 28.10.30. Appl., 15.3.28. Swed., 18.3.26).—An alloy of lead with 1% Sb and up to 0.5% (preferably 0.2%) Bi is claimed to be resistant to chemical action and intercrystalline fatigue.

A. R. POWELL.

**Recovery of vanadium from ores or compounds.** B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,779,856, 28.10.30. Appl., 29.10.27).—The ore (etc.) is ground finely and mixed with water and the pulp treated with chlorine or sulphur chloride to dissolve the vanadium.

A. R. POWELL.

**Manufacture of [precious metal] catalysts.** G. SIEBERT GES.M.B.H. (B.P. 345,890, 6.6.30. Ger., 7.6.29).—Nets, gauzes, or plaited work made of platinum, silver, gold, nickel, copper, or alloy wires or strips plated or otherwise coated with rhodium are claimed.

A. R. POWELL.

**Production of tarnish-resisting silver and silver-plate.** D. GRAY, R. O. BAILEY, and W. S. MURRAY, Assrs. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,779,809, 28.10.30. Appl., 5.11.23).—The article is electroplated with alternate layers of silver and a metal which when alloyed with silver increases its resistance to tarnishing, then annealed in a non-oxidising atmosphere at 220° to produce diffusion of the metals into one another, and finally worked hot or cold. Alternatively, the alloying metal may be caused to diffuse into the silver by a process similar to the sherardising process.

A. R. POWELL.

**[Palladium] alloys for electrical contacts.** E. F. KINGSBURY, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,779,602—3, 28.10.30. Appl., [A] 16.5.24, [B] 2.3.26).—The alloy comprises (A) 60% Pd and 40% Cu, or (B) 70% Pd and 30% W. A small part of the palladium may be replaced by (A) zinc or (B) nickel.

A. R. POWELL.

**Preparation of chromium-plated ferrous articles.** V. M. VERTUCCI (U.S.P. 1,780,213, 4.11.30. Appl., 7.10.26).—The article is heated to dull redness, allowed to cool slowly, pickled in 20% hydrochloric acid at 85°

washed, rinsed in sodium hydroxide solution, cleaned cathodically in 10% sulphuric acid with 150 amp./ft.<sup>2</sup> for 5 min., and plated directly in the usual chromium bath.

A. R. POWELL.

**Treatment of cast iron. Ferrous metal or alloy.** A. F. BURGESS. From LINK-BELT Co. (B.P. 338,924—5, 31.5.29).—See U.S.P. 1,760,239 and 1,760,240; B., 1931, 119.

**Production of non-corrodible [manganese-copper-nickel] alloys.** C. CONTAL (B.P. 346,226, 20.6.30. Fr., 21.6.29).—See U.S.P. 1,771,773; B., 1931, 207.

**Mercury boilers. [Crushing surfaces of] mills. Discs for centrifuges.**—See I. Hydrogenation catalysts.—See II. Salt mixtures from lead refining. Reduction of iron ores. Phosphorus.—See VII. Mirror glass.—See VIII.

## XI.—ELECTROTECHNICS.

**Measurement of  $p_H$  of nickel-plating baths.** J. BARBAUDY, A. GUÉRILLOT, H. MIACHON, and R. SIMON (Compt. rend., 1931, 192, 739–741).—Liquid from the nickel-plating bath is passed continuously through a cooling arrangement and then, after saturation with quinhydrone, past a (gold) quinhydrone electrode and the porous pot containing solid potassium chloride and a calomel electrode. Actual recording is by means of a Leeds and Northrup potentiometer sensitive to 1 millivolt.

C. A. SILBERRAD.

**Boiler feed-water.**—See I. Hydrocyanic acid.—See VII. White tin. Hardness of electrodeposits.—See X. Phenol-formaldehyde resins.—See XIII. Determination of  $p_H$  of soils.—See XVI. Purity of syrups.—See XVII. Photographic plates. Images on photoelectric cells.—See XXI.

## PATENTS.

**Electric furnaces comprising a hearth and resistances arranged above the charge.** E. F. RUSS (B.P. 344,662, 4.3.30. Ger., 4.3.29).—Hot air is forced from the region of the electric heaters to that of the charge, in a direction parallel to the surface of the charge, by an agitator arranged in an enlarged channel placed outside the furnace chamber.

J. S. G. THOMAS.

**[Charging of] electric furnaces.** P. L. J. MIGUET and M. P. PERRON (B.P. 344,493, 4.12.29. Fr., 6.12.28).—Various raw materials are separately fed through feed columns arranged radially with respect to a central upper electrode co-operating with a hearth electrode.

J. S. G. THOMAS.

**Polarised electrical [electrochemical] couple.** P. E. EDELMAN, Assr. to E. BANNING (U.S.P. 1,773,665, 19.8.30. Appl., 12.4.28).—The couple comprises an aluminium electrode coated with coloured dielectric material prepared by treatment of the electrode with a solution of a compound of a molybdate of ammonium, sodium, or potassium, and a solution of a bitartrate compound; a co-operating electrode, e.g., of nickel; and electrolyte, e.g., a dilute solution of ammonium bitartrate containing gum arabic.

J. S. G. THOMAS.





**Determination of rancidity in oils and fats.**

A. TAFFEL and C. REVIS (J.S.C.I., 1931, 50, 87—91 T).—A modified form of the Kreis test is described. A simple and rapid quantitative method for determining the degree of rancidity in oils and fats has been worked out. It depends on the fact that peroxides liberate free iodine quantitatively from barium iodide in glacial acetic acid solution, and that the iodine set free by peroxides present in rancid oils is prevented, by the presence of barium iodide, from attacking the double linkings in the oil molecule. By the use of this method it has been shown that oils such as groundnut oil, which have become rancid at temperatures up to at least 60°, contain a form of peroxide which is rapidly reduced by hydriodic acid. Oils which have become oxidised at fairly elevated temperatures, e.g., 120°, contain a mixture of these easily reducible peroxides together with some other form of active oxide which is reduced only with difficulty, whilst oils which have been air-blown at 170° contain practically only the latter type and very little of the former type. The intensity of the Kreis reaction is correlated with the amount of easily reducible peroxides in an oil. The difficultly reducible peroxides have no connexion with the Kreis reaction. The Kreis reaction is not an infallible test for rancidity, whilst the new methods appear to be of universal application.

**Solubility of fats in various solvents. Solubility of (III) camellia oil, (IV) rape oil, in isopropyl alcohol of various concentrations.** K. HASHI (J. Soc. Chem. Ind., Japan, 1931, 34, 104—105 B, 105—106 B).—The solubilities in aqueous isopropyl alcohol solutions were determined as before (cf. B., 1931, 500). The results are similar to those recorded previously, but here the curves of clouding point concentration of the alcohol (*b*) for various oil concentrations are convex to the abscissa (*b*). In solutions containing over 80% of camellia oil turbidity sets in slowly and concordant results are not always obtained on repetition. For solutions in aqueous isopropyl alcohol of concentration (wt.-%) 100, 95, 90, 87.5, and 84.99, the critical temperatures are: (III) 25°, 48°, 83.5°, >111.5°, >139°; and (IV) 41.3°, 65.5°, 100.3°, —, >148°, respectively; and the critical concentrations (oil-%): (III) 39.0, 42.0, 64.5, >80, >80; and (IV) 38%, 40.3, 58.3, —, >73.4. E. LEWKOWITSCH.

**Antioxidants and the autoxidation of fats.** H. A. MATTILL (J. Biol. Chem., 1931, 90, 141—151).—By a modification of the method of Greenbank and Holm (B., 1925, 556) measurement has been made of the rate of oxygen uptake by a standard mixture of lard and cod-liver oil in presence of various compounds. Monohydric phenols were only very slightly active in increasing the induction period, but polyhydric phenols exhibited very considerable activity when they contained hydroxyl groups in the *ortho* or *para* position.  $\alpha$ -Naphthol was also active, although  $\beta$ -naphthol exhibited only slight activity. Benzoquinone and  $\beta$ -naphthoquinone show a marked inhibiting action, but  $\alpha$ -naphthoquinone is ineffective. Inositol and cholesterol and other naturally occurring sterols were without action, as were also all compounds which did not contain a free phenolic hydroxyl group. W. O. KERMAK.

**Unaponifiable matter and so-called isooleic acids contained in toilet soaps.** S. UENO, G. INAGAKI, and H. TSUCHIKAWA (J. Soc. Chem. Ind., Japan, 1931, 34, 90—92 B).—The amounts of unaponifiable matter found in 12 samples of commercial toilet soaps did not correspond with the amounts of hardened oils present, as deduced from determination of the isooleic acid contents (Richardson's method, B., 1924, 564). It is concluded that the former content of unaponifiable matter is an unreliable guide for the detection of hardened oils (cf. Wittka, B., 1927, 944).

E. LEWKOWITSCH.

**Manganese soaps. II.** H. WAGNER and G. HOFFMANN (Farben-Ztg., 1931, 36, 1214—1216; cf. B., 1931, 211).—Manganese hydroxide and raw and burnt umbers were ground in linseed oil and films were allowed to dry on glass and metal plates for 14 days, when their water absorptions after 1, 3, 5, and 15 days were noted and graphed. Manganese hydroxide had the most pronounced tendency to swelling, whilst umbers were less affected, although in all cases after swelling films deficient in cohesion and elasticity resulted. The addition of 5% and 10% of manganese hydroxide to Mars yellow (containing 53% Fe<sub>2</sub>O<sub>3</sub>, 20% CaCO<sub>3</sub>, and 17% CaSO<sub>4</sub>) causes decreased drying time but increased swelling of paints made therefrom. At concentrations of manganese hydroxide above 5%, the film properties are, in general, adversely affected; the use of pigments rich in manganese hydroxide for rust-prevention is not recommended. Photomicrographs illustrating the appearance of the different films after swelling are included. S. S. WOOLF.

**Migration of sodium chloride in soap cakes.** J. MIKUMO (J. Soc. Chem. Ind., Japan, 1931, 34, 116 B).—Kristen's observations (Seifensieder-Ztg., 1927, 54, 665) of the migration of salt in boiled and settled, or even in milled, soaps have been confirmed. The direction of the migration is determined chiefly by the difference of vapour tension between the inside and outside of the cake; if kept in a very moist atmosphere, salt may concentrate in the outer layers. Probably during cooling and storage curd soaps become more fibrous and release part of the water of hydration as free water (syneresis). E. LEWKOWITSCH.

**Hydrogenation of fatty oils by the so-called "wet process." I. Hydrogenation in presence of nickel acetate under atmospheric pressure.** S. UENO and T. YUKIMORI (J. Soc. Chem. Ind., Japan, 1931, 34, 109—111 B).—Using a nickel acetate-kieselguhr catalyst, the rate of decrease of iodine value is not always proportional to the amount of catalyst used. In all cases this catalyst is inferior in activity to a nickel-kieselguhr catalyst which has been reduced prior to admixture with the oil. E. LEWKOWITSCH.

**Hydrogenation of [fatty] oils at high temperatures and under high pressures. I. Hydrogenation with nickel under constant high pressures.** S. UENO, T. YUKIMORI, H. TSUCHIKAWA, and S. UEDA (J. Soc. Chem. Ind., Japan, 1931, 34, 111—115 B).—Hydrogenation of a refined sardine oil (for 1 hr.) with a reduced nickel-kieselguhr catalyst has been conducted

under pressures of from 50 to 10 atm. over the temperature range 150—230°. (Analytical details of the samples are given.) The amount of isooleic acid produced is very small (especially at high pressures) compared with that obtained at ordinary pressure. The temperature exerts a marked influence on the velocity of the hydrogenation, the reaction being most rapid at 180—200°. E. LEWKOWITSCH.

**Absorption of oxygen by unsaturated [fatty] oils. I. Influence of the solvent on the rate of absorption.** S. UENO, Z. OKAMURA, and T. SAIDA (J. Soc. Chem. Ind., Japan, 1931, 34, 106—108 B).—The oil containing cobalt linoleate as drier was tested in various solvents and the rates at which oxygen was absorbed were compared. The greatest acceleration of the oxidation was obtained in solution in glacial acetic acid; some other solvents fall into the (decreasing) order (for linseed oil) decalin, ethyl acetate, turpentine, chloroform, acetone. For sardine oil the order of the last four is slightly different. E. LEWKOWITSCH.

**The silver-foil or "coin" test for sulphured [fatty] oils.** R. MARCILLE (Ann. Falsif., 1930, 23, 527—530).—The "coin" test is modified so that it will detect as little as 5% of sulphured oil in a pressed oil. It is shown also that pressed olive oils will give a positive reaction, if they are allowed to remain in contact with "foots" containing soluble sulphates, the latter being reduced to sulphides by micro-organisms. The positive "coin" test must therefore be confirmed by other reactions characteristic of sulphured oils.

E. B. HUGHES.

**Twitchell reagent. III.** K. NISHIZAWA and Y. MATUKI (Chem. Umschau, 1931, 38, 73—78).—A fuller account is given of work already noted (B., 1930, 870).

E. LEWKOWITSCH.

**Composition of linseed oils obtained in various ways and stored under various conditions.** K. H. BAUER and A. FREIBURG (Chem. Umschau, 1931, 38, 78—80).—Oils obtained by (a) cold pressing, (b) hot pressing, and (c) extraction with light petroleum from the same sample of Calcutta linseed were analysed. The ordinary constants for these samples were much alike: the drying curves revealed slightly greater differences, the hot-pressed oil giving the worst curve; the fatty acid compositions, determined by the Eibner method, however, showed considerable differences. Samples of the oils were stored for 1 year in filled, stoppered, glass bottles in daylight: the iodine value of the oils rose 2—9 units, and the saponif. values decreased a few points; the illuminated oils all dried in about one third the time of the original samples. Most "astounding" changes, however, had apparently occurred in the fatty acid composition, as is shown by the following figures (%) for the oils (a), (b), (c), respectively (figures for the illuminated specimens in parentheses):  $\alpha$ -linolenic acid 15.88 (29.1), 20.3 (19.99), 18.81 (26.71);  $\beta$ -linolenic acid 3.96 (2.02), 0.21 (0.5), 3.89 (1.53);  $\alpha$ -linoleic acid 19.31 (22.01), 15.09 (8.7), 18.13 (8.86);  $\beta$ -linoleic acid 36.82 (1.8), 40.76 (8.38), 35.52 (15.76); oleic acid 6.65 (31.3), 9.07 (50.14), 8.91 (33.0). Analyses were attempted by applying the thiocyanogen method to the liquid acids separated by the thallium and

lead salt methods; extraordinary differences in the apparent composition of the fresh and stored oils again was found, but the discrepancies between the figures so found and those given by the Eibner method were very large and irregular both in sense and magnitude. The authors can offer no explanation of these "remarkable" results, beyond the uncertain nature of available analytical methods. E. LEWKOWITSCH.

**Formation of drying oil films.** C. P. A. KAPPELMEIER (Chem. Weekblad, 1931, 28, 174—183).—An account of earlier and recent knowledge and theories. Drying is to be regarded as caused by chemical and colloidal factors acting together. S. I. LEVY.

**Factors of quality in copra.** F. C. COOKE (Malay. Agric. J., 1931, 19, 128—136).—The deterioration of copra under mould action is discussed in relation to the moisture content and storage conditions; 4—6% of water represents average stable conditions. A badly-rotted copra will contain a high percentage (e.g., 70%) of ether-extractable fatty matter, but this contains a high proportion of free fatty acid. A mouldy copra of high free fatty acid content, if stored in good conditions of free ventilation, becomes clean and dry (moulds falling off as dust) and the acidity diminishes. Compression into bales under a pressure of 1.5 cwt./in.<sup>2</sup> for transport is satisfactory. E. LEWKOWITSCH.

**Reactions of cod-liver oil and castor oil.** L. EKKERT (Pharm. Zentr., 1931, 72, 209—212).—One drop of cod-liver oil dissolved in 1 c.c. of absolute alcohol is mixed with 3 drops of a 1% alcoholic solution of an aldehyde, and poured cautiously on to 1 c.c. of concentrated sulphuric acid: a coloured ring is produced at the junction of the liquids; on shaking, the liquids become coloured throughout, and are further examined in the light of the quartz lamp. The various shades (browns to violet-reds) obtained by the use of various aldehydes are detailed. Similar colours are obtained with castor oil. E. LEWKOWITSCH.

**Oil from the seeds of *Asteriastigma macrocarpa*.** D. H. PEACOCK and C. THOUNG (J.S.C.I., 1931, 50, 7—8 T).—The results of André (B., 1926, 98) were confirmed. Oleic acid isolated by the lead salt method amounted to about 7% on the fatty acids obtained. By fractional distillation of the ethyl esters followed by crystallisation of the acids from light petroleum chaulmoogric acid was isolated. Hydnocarpic acid could not be isolated. The optically active fatty acids amounted to about 75% at least of the fatty acids. A saturated fatty acid of equivalent weight equal to that of palmitic acid but of m.p. 50° was also isolated.

**Determination of inorganic impurities in sulphonated oils.** R. HART (Chem. Umschau, 1931, 38, 81—83; cf. Nishizawa and Winokuti, B., 1931, 307).—The derivation of the formulæ deduced in previous papers (B., 1921, 226 A, 438 A) is re-stated. Titration of the oils (for combined sulphate) may be facilitated by adding a small measured amount of acid to the salt and ether mixture; after settling, more indicator is added to the lower layer and titration is completed. For greater accuracy the brine washings of the ether layer should be added. The average acid content of

a number of commercial sulphonated castor oils, washed twice with Glauber's salt solution, was found to be equivalent to about 3 mg. KOH per g., *i.e.*, at least 90% (and probably more) of the combined  $\text{SO}_3$  had been neutralised in the washing. E. LEWKOWITSCH.

**Fatty acid components of the oil from (A) Malayan gaviol *Fomistoma Schlegli*, and some properties of the oil from (B) *Pecten (Patinopecten) Yossensis*, Jay. S. UENO and N. KUZEI (J. Soc. Chem. Ind., Japan, 1931, 34, 92—93 B).**—The oil from (A) was a pale yellow liquid (at 18°), and gave no blue colour with antimony trichloride or acid earth. It had  $d_4^{15}$  0.9166,  $n_D^{20}$  1.4623, iodine value (Wijs) 77.4, saponif. value 197.6, acid value 19.4, Reichert-Meissl value 0.54, acetyl value (Cook) 22.4, unsaponifiable matter 0.44%, ether-insoluble bromides trace. The mixed fatty acids (m.p. 33.9—35.2°, iodine value 76.5, neutralisation value 202.3) yielded 64.3% of liquid acids (lead salt-ether method) having iodine value 92.8, neutralisation value 201.0, ether-insoluble bromides 0.052% (consisting of oleic acid with traces of linoleic, linolenic, and more unsaturated acids), and solid acids having iodine value 6, neutralisation value 214.0, m.p. 51.2—52.1°. The saturated acids consisted chiefly of palmitic acid with a small amount of stearic acid: cholesterol, but no hydrocarbon, was present in the unsaponifiable matter.

The deep brown odorous oil from (B) showed a weak green colour reaction with acid earth. The refined oil had  $d_4^{100}$  0.8784,  $n_D^{20}$  1.4781, saponif. value about 187.7, Reichert-Meissl value 1.2, acid value 1.2, iodine value (Wijs) 204.0. The mixed acids [m.p. 30.9°, iodine value 199.8 (!)] yielded 50.46% of ether-insoluble bromides containing 70.34% Br. E. LEWKOWITSCH.

**Burmese species of plants yielding chaulmoogra oils.** D. H. PEACOCK and G. K. AIYAR (Burma Forest Bull., 1930, No. 21, 11—13).—The seeds of four species were examined. *Taraktogenos Kurzii*: The kernels, about 58—70% of the whole fruit, contained about 50% of oil, saponif. value 183—203 (av. 197), iodine (Wijs) value 94—113 (av. 110);  $[\alpha]_D^{25-30}$  in chloroform +38° to +55° (av. +47°). *Asteriastigma macrocarpa*: The seeds contained 36.4—56.9% of oil having  $d_4^{25}$  about 0.9501, iodine value 103—127 (av. 112), saponif. value 192—204 (av. 195), unsaponifiable matter about 3%;  $[\alpha]_D$  +38.8° to +55.6° (av. +53°),  $n_D^{20}$  about 1.4790. Inactive oleic acid content about 7%. Corresponding values for *Hydnocarpus verrucosa* and *H. dawnensis* are: 10.9%, 35.0%; 0.8519, 0.8531; 81.1, 91.4; 202.0, 182.3; 1.54%, 6.2%; +43.6°, +38.8°; 1.4752, 1.4760. T. MCLACHLAN.

**Phthaleinoscope.**—See I. Viscosity of oils.—See II. Wood staining.—See IX. Fat of sow's milk.—See XIX.

#### PATENTS.

**Condensation products or intermediate compounds from fish-liver oils.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, and W. A. SEXTON (B.P. 345,734, 22.1.30).—Alkali-soluble compounds suitable for use in the textile industry are prepared by condensing the halogen derivatives of squalene (*e.g.*, the dodecyl bromide) with a phenol in the presence of aluminium chloride. The compounds are precipitable by acetic

acid, fuse between 120° and 130°, and yield highly coloured azo compounds with diazonium salts.

E. LEWKOWITSCH.

**Filter device [for fish oils etc.]. Turbine lubrication.**—See I. Sizing of textiles. Oiling of fibres.—See VI.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**“Chalking” [of paint films]. IV. Practical trials of chalking of oil paints. I.** R. KEMPF (Farben-Ztg., 1931, 36, 1173—1176, 1216—1217, 1256—1259; cf. B., 1931, 169).—An intermediate report is given on exposure tests on paints based on (a) individual pigments, *i.e.*, basic carbonate white lead, titanium white, titanium dioxide, and lithopone, and (b) mixtures of zinc oxide, basic carbonate and basic sulphate white leads, and lead chromate, each with three types of lithopone, in different vehicles, *e.g.*, boiled linseed oil, tung oil, and stand oils (linseed and tung oils), other variables being locality and aspect of exposure, and nature of priming paint. Full details are given of compositions and degree of chalking after 3, 5, and 10 months, as measured by the author's “stamping method” (B., 1930, 156), the results being illustrated graphically and photographically. The development of cracks and wrinkling is also recorded. The results are discussed at considerable length and the pigments are found to behave similarly (as to order of chalking) in the various vehicles tested. White lead carbonate and lithopone-lead chromate were found to be the more free from chalking tendency of the two groups. The suggestion that cracking and chalking are inversely proportional is not upheld. S. S. WOOLF.

**Paint testing.** P. NETTMANN (Farben-Chem., 1931, 2, 22—23).—Accelerated weathering tests are considered to fail to produce significant results owing to our incomplete knowledge of the processes of film destruction under natural weathering. The disruptive forces acting in a finishing coat-undercoat system on iron are discussed hypothetically and illustrated by diagrams, and the thermodynamics of the system are outlined.

S. S. WOOLF.

**Antimony yellow [pigment].** J. MILBAUER (Chem.-Ztg., 1931, 55, 222).—When antimonite is treated with concentrated caustic soda in a current of air in a porcelain ball mill, it changes gradually into a bright yellow pigment, a solid solution of antimony trisulphide in antimony oxides. Its exact composition varies according to the amount of soda used; if no iron is present the best yellow (6% S) is obtained by working for 3 hrs. with ingredients in the proportions of 1 pt. of the trisulphide to 6 pts. of 25% aqueous caustic soda. This pigment resembles Indian yellow and pale ochre, has  $d$  2.43, covering power 320  $\text{cm}^2$ , is stable up to 200° and towards atmospheric conditions, but is not compatible with metallic pigments which react with sulphides.

E. LEWKOWITSCH.

**Sources of error in the colorimetric determination of iron in red lead.** J. F. SACHER (Farben-Chem., 1931, 2, 120—122).—The work of various investigators on the determination of quantities of the order of 0.0005—0.01% Fe in red lead is summarised. A source

of error in the usual colorimetric method is the hydrolytic decomposition of ferric thiocyanate to an insoluble, brown, basic salt, this effect depending jointly on the concentration of the solution, the acidity, and the amount of thiocyanate added. A further error arises owing to the light-sensitivity of the ferric thiocyanate.

S. S. WOOLF.

**Determination of sp. gr. of pigments.** W. ESCH (Farben-Chem., 1931, 2, 154—156).—The following method, originally intended for use on pigments for the rubber industry, is capable, with limitations, of application to paint pigments. A standard rubber mixing is prepared from "1a latex pale crêpe rubber" (100 pts.), lead-free zinc oxide (7 pts.), mineral rubber (6 pts.), double-pressed stearic acid (3 pts.), accelerator "Captax" (1 pt.), and "velvet" sulphur (3 pts.). This mixing has  $d$  1 before or after vulcanising. A suitable proportion of the pigment to be tested is incorporated (e.g., 1 of carbon black : 3 of "mixing") and the whole is vulcanised. The sp. gr. of the product may be determined simply, and that of the pigment arrived at by calculation.

S. S. WOOLF.

**"Water-spotting" of oil and nitrocellulose lacquers.** F. KOLKE (Farben-Chem., 1931, 2, 106—108).—The occurrence and probable causes of water-spotting are discussed, such factors as moisture, heat, rain, water-soluble constituents in the paint, deposition of limestone dust, etc. accounting for the phenomenon in the case of oil enamels. The function of linseed oil as a "water accumulator" is mentioned, and, where spotting occurs with clear oil varnishes, it is probably due to swelling owing to incorrectly bodied oil, the superiority of tung oil and tung oil enamels in this respect being significant. Nitrocellulose lacquers are treated separately, the hardness of the lacquer film and the nature of the resin constituent here being the most important considerations, but the use of certain red and black aniline dyes may be a contributory cause of the defect. Zinc oxide is known to favour the formation of water spots in oil and cellulose enamels, but it is shown that the pigment portion of a lacquer cannot entirely account for the trouble.

S. S. WOOLF.

**Butyl and amyl lactates as solvents for [nitro]-cotton lacquers.** O. GERHARDT (Chem.-Ztg., 1931, 55, 222).—Butyl lactate (b.p. 177°/760 mm.) is equal and amyl lactate (b.p. 115°/56 mm.) superior in solvent power to ethyl lactate, and their tolerance for diluents (cf. Wolff, B., 1928, 519) is much greater. A 10% nitro-cotton (WAS 10) can be diluted without turbidity by 11 pts. (by wt.) of benzene; it dries to a clear film with 9 pts. (or 10 pts., with a low-viscosity cotton). The corresponding dilution figures for amyl and ethyl lactates are 1:16 or, for clear drying, 1:14—15; and 1:7 or 1:5—5.5, respectively. Lacquers with these solvents, especially the amyl ester, flow well and give good films.

E. LEWKOWITSCH.

**Resins. VIII. Their identification.** E. STOCK (Farben-Ztg., 1931, 36, 1176—1177).—A comprehensive range of resins was tested by Donath's method (cf. B., 1930, 957). The finely-powdered resin (1 g.) is boiled for 1 min. with 5 c.c. of nitric acid ( $d$  1.32—1.33), the

cooled liquor diluted (1:1) with distilled water, and ammonia then added in excess. The colours developed immediately and after 5 min., 1 hr., and 1 day are tabulated for the resins tested; a progressive intensification of the coloration is reported, in all cases excepting that of "Albertol" resins. Rosin gives an immediate orange coloration that deepens in 1 day to the "deep brown-red" described by Donath. In particular groups of resins, distinctions are pointed out in the results at one or other of the time periods observed, but it is apparent that this reaction will not distinguish rosin from other resins with surety, nor detect it in mixtures. Nevertheless it shows the advantage over the Liebermann-Storch reaction that the colorations produced are not fugitive.

S. S. WOOLF.

**Phenol- and cresol-formaldehyde resins.** H. STÄGER (Helv. Chim. Acta, 1931, 14, 285—304).—A series of synthetic resins of phenol-, cresol-, and mixed phenol-cresol-formaldehyde type (prepared by condensation under alkaline conditions) is examined in the crude state by a variety of physical and some chemical methods. The latter, in general, including the loss in weight on thermal hardening, the content of volatile products, and the bromine absorption and mol. wt. of the benzene-soluble fraction of these volatile products, are found insufficient to characterise the resin. The progressive decrease in alcohol-solubility that accompanies thermal hardening is much less in the case of the cresol resins; this does not hold true for alkali-solubility, however, thus confirming Koebner's hypothesis of the formation of insoluble sodium salts. The initial decrease in the "cementing coefficient" (cf. B., 1929, 520) with increasing thermal hardening is slower the higher the phenol content of the resin: the effect on this property of heat-treatment in the case of shellac contrasts markedly with the behaviour of these synthetic resins, a continuous increase in strength being observed. The electrical properties of the synthetic resins vary consistently in the various classes, and show a decided break at a phenol content of about 30%. The breakdown voltage increases with increasing phenol content, and in the pure cresol resins increases with decreasing *m*-cresol content. The dielectric constant also varies directly with the phenol content and, in a simple cresol resin, inversely with the *m*-cresol content; it increases in every case with progressive thermal hardening, but the interrelationships between the various types of resin remain unchanged. Dielectric loss is related to composition in the same way as the dielectric constant, but in the case of resins containing more than 30% of phenol increases very rapidly with the potential gradient, and cannot be measured when the latter exceeds 5 kilovolts/cm.; the values are very low for the pure cresol resins and less dependent on potential gradient. It is concluded that there is an essential difference between the phenol- and cresol-formaldehyde resins in that the degree of polymerisation attained under similar conditions is less, and the polar characteristics of the products are consequently more pronounced in the former. *m*-Cresol is the most reactive of the cresols.

H. A. PIGGOTT.

**Urban glue.**—See XV. **Blood-albumin.**—See XIX.

## PATENTS.

**Separation of anethole from pine oil.** L. T. SMITH, ASSR. to HERCULES POWDER CO. (U.S.P. 1,777,704, 7.10.30. Appl., 11.8.27).—Pine oil is fractionated and the fraction, b.p. 220—250°, is refractionated to give a distillate, b.p. 232—237°, from which anethole is obtained by crystallisation. E. H. SHARPLES.

**Manufacture of surface-decorated [floor-covering] materials [e.g., linoleum].** ARMSTRONG CORK CO., ASSEES. of H. R. WOERNER (B.P. 344,660, 3.3.30. U.S., 7.3.29).—A masking material for use in the process of B.P. 319,655 comprises a filler, e.g., whiting, suspended in a medium of sufficient viscosity to maintain the filler in suspension and of sufficiently low surface tension to spread readily. Glycerin or glycol in admixture with soap, or aqueous gum (tragacanth) solution containing soap and potassium carbonate, is suitable. D. J. NORMAN.

**Manufacture of inlaid linoleum or the like.** ARMSTRONG CORK CO., ASSEES. of J. E. WILEY (B.P. 346,211, 21.5.30. U.S., 24.5.29).

**Coating for writing tablets etc.**—See V. **Balloon fabric.**—See VI. **Barium sulphide solutions.**—See VII. **Reinforced glass etc.**—See VIII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Röntgenographic study of chicle.** C. W. STILLWELL and G. L. CLARK (Kautschuk, 1931, 7, 62—66).—Röntgenographic investigation shows raw chicle to consist of at least three crystalline substances and one or more amorphous compounds; the crystalline constituents are chicle-gutta, which is identical with the hydrocarbon of gutta-percha or of balata, a resin, and calcium oxalate monohydrate, the last-named forming the crystalline portion of the benzene-insoluble fraction. Refined chicle has a similar structure to that of the raw material, except that its gutta is more highly dispersed, the high temperature involved in the purification process converting it into an amorphous condition which is rendered stable by the presence of the other constituents. D. F. TWISS.

**Chemistry of [rubber] latex.** II. P. SCHOLZ and K. KLORZ (Kautschuk, 1931, 7, 66—68).—The dry solid content of latex is determined by carefully evaporating approx. 2 g. in a flat nickel dish (diam. 10 cm.) over a flame or hot plate with avoidance of bubbles, until a clear film remains free from opaque spots. The dry film while still warm is rolled up so as to reduce its free surface and is then removed and weighed. The process takes 6—10 min. and gives results accurate to  $\pm 0.1\%$ . The rubber crêpe yield of latex is measured by weighing about 25 g. of the latex into a Petri dish (diam. 10 cm.). Coagulation is then effected by adding 10—20 c.c. of 10% sulphuric acid by a pipette, beginning at the edge and proceeding spirally to the centre. After a rest of 10 min. the resulting cake of coagulum is removed without pressure and washed with running water at 50° for 2 hrs. until on contact it no longer reddens litmus paper. The coagulum is then pressed between cloth and dried at 70—85° until a clear amber colour throughout. The direct estimation of the rubber crêpe yield may be obviated by approximate calcula-

tion from the total solids figure (*T.S.*); the crêpe yield in g. per 100 c.c. is given by the formula  $0.9T.S.$ , and the percentage by wt. by  $0.9T.S./d$  (where  $d =$  sp. gr.) or  $0.92T.S.$  assuming an average sp. gr. of 0.98. D. F. TWISS.

**Application of accelerators and anti-agers [in rubber manufacture].** A. H. SMITH (Kautschuk, 1931, 7, 70—75).—Conditions are reviewed for the satisfactory technical use of various well-known accelerators of vulcanisation and anti-agers. For the best results generally it is advisable to use the minimum of sulphur, and in order to ensure good flexing and ageing care must be taken to avoid overheating. Hardness must be increased by suitable compounding and not by increased use of sulphur. Excessive use of softeners frequently detracts from good ageing. Vulcanisation should be effected at the lowest possible temperature. D. F. TWISS.

**Sp. gr. of pigments.**—See XIII.

## PATENTS.

**Treatment of rubber. [Dispersing rubber in water.]** W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,780,657, 4.11.30. Appl., 23.8.22).—An aqueous dispersion of rubber, or of an analogous hydrocarbon such as gutta, is obtained by first dissolving it in benzene or similar solvent and then mixing the solution with water and a colloidal dispersing agent, the rubber solvent being finally removed. The first portion of the water is preferably added as an aqueous solution of ammonia, and the colloid dispersing agent, such as soap, is introduced with or after the remainder of the water, but before removal of the solvent by distillation under reduced pressure or in any other way. D. F. TWISS.

**Vulcanisation of rubber.** W. SCOTT, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,780,604, 4.11.30. Appl., 28.11.27).—*s*-Diaryl-substituted guanidines in which one of the aryl substituents contains a piperidyl group *para* to the imino-group, e.g., phenyl-*p*-piperidyl-phenylguanidine, are effective accelerators of vulcanisation. D. F. TWISS.

**Balloon fabric. Rubbed threads.**—See VI.

## XV.—LEATHER; GLUE.

**Microscopical study of the effects of cold temperatures on [raw] hides and skins.** F. O'FLAHERTY and W. T. RODDY (J. Amer. Leather Chem. Assoc., 1931, 26, 172—180).—Sections of freshly slaughtered hides and skins were examined microscopically before and after the skins had been subjected to low temperatures and other treatment. No structural damage was observed, though slight dehydration had taken place, in skins which had been subjected to 4 weeks' treatment in a refrigerator at 0°, but some tissues were ruptured by folding such skins. No damage was found in the product obtained when the frozen skins were kept for 2—4 hrs. at room temperature before soaking, and least change was noticed in such skins if they were then soaked in dilute salt solution. Better results were obtained in skins which had been salt-cured and frozen than in those subjected to either treatment alone. No difference in structure was observed after liming between frozen and unfrozen skins. D. WOODROFFE.

**Moisture determination and its application to leather.** A. COLIN-RUSS (*J. Soc. Leather Trades' Chem.*, 1931, **15**, 113—126, 166—182).—A weighed quantity of leather is placed in a charging tube, which rests on the end of a release mechanism fitting into the upper side tube of a long, thick-walled reaction tube closed at its lower end and containing some calcium carbide. About the middle of the reaction tube is a lower side tube, with a 0.5-cm. dip containing calcium carbide, and leading to a manometer for measuring volume at constant pressure. The reaction tube is provided with a vapour jacket for maintaining it at any definite temperature. The moisture in the sample is calculated from the volume of acetylene generated when the sample falls on to the calcium carbide and has been left until a constant reading has been obtained. This method has been applied to determine the moisture in the crust skiver, heavily-tanned leather, and certain heavily greased leathers. The apparatus was standardised by means of ammonium oxalate monohydrate in presence of currying grease. It was shown that the agreement between the results obtained by the carbide and desiccation methods was good for all the above leathers except the crust skiver. The result by the carbide method agreed with that by the oven method for the sumac-tanned leather, and was higher than that given by the desiccation method. D. WOODROFFE.

**Evaluation of hide- and bone-glue.** E. GOEBEL (*Farben-Chem.*, 1931, **2**, 174—176).—The determination of water in glues is discussed in detail. Direct drying of powdered samples, in one or more stages, tends to give high results, since in the grinding process moisture is absorbed from the air, whilst correct sampling also offers difficulty. More exact methods comprise the conversion of relatively large quantities of the glue into a homogeneous gel, from which reliable small samples can be withdrawn and dehydrated, the amount of water in the original glue being obtained by calculation. Distillation with xylene, tetrachloroethane, etc. offers a rapid method of obtaining approximate results.

S. S. WOOLF.

**Collagen, gelatin, and glue. Röntgen spectrographic studies.** J. R. KATZ and O. GERNGROSS (*Collegium*, 1931, 67—79).—The well-defined crystal-interference lines and the wide indefinite interference of liquids and amorphous substances are shown on the röntgen spectrograph of pure, practically ash-free, isoelectric gelatin. From further studies it is inferred that dry gelatin consists largely, if not entirely, of long crystals with the long axis parallel to that of the fibres. The crystal interference was destroyed by heating a gelatin solution until it would no longer set on cooling. The adhesive power, gel-forming properties, and elasticity of a gelatin can be measured by the amount of its crystal interference of X-rays. The elastic nature of gelatin is due partly to the parallel orientation of the fibre-forming elements and the subsequent arrangement of the fibres parallel to each other. The interferences observed in gelatin and collagen fibres were practically the same. Evidence of a slightly spiral arrangement of the long collagen elements was obtained.

D. WOODROFFE.

**Colloid-chemical nature of Urban glue.** A. EIBNER and E. ROSSMANN (*Z. angew. Chem.*, 1930, **43**, 943—947).—An account of Urban's process and its application to painting is given and the properties of the product are discussed from the colloid-chemical point of view. E. S. HEDGES.

**Blood-albumin.**—See XIX.

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**Filter device [for glue].**—See I. **Foils from gelatin.**—See V. **Mordanting hair.**—See VI.

## XVI.—AGRICULTURE.

**Temperature correction in the hydrometer method of mechanical analysis of soils.** C. RICHTER (*Soil Sci.*, 1931, **31**, 85—92).—Uniform corrections for all temperatures and concentrations of the soil suspension are not applicable. A sliding scale of correction factors is suggested. A. G. POLLARD.

**Laboratory study of the field percolation rates of soils.** C. S. SLATER and H. G. BYERS (*U.S. Dept. Agric., Tech. Bull.*, 1931, No. 232, 23 pp.).—Following the development of a soil-core auger which satisfactorily cuts cores of undisturbed soil, a procedure has been developed for evaluating soil-percolation rates. The field-percolation rate of soil is governed more by the water passageways it contains, such as root channels and structural cleavages, than it is by the character or volume of the pore space of the soil mass. The percolation rates of cores serve as a means of studying field permeability, but they vary too much for cores of the same soil to fix rates by this means alone, unless largely replicated. The ratios for erosion and permeability of soils are inversely proportional in terms of the factors of the percolation ratio. E. HOLMES.

**Determination of sulphur in soil.** M. JARACH (*Giorn. Chim. Ind. Appl.*, 1931, **13**, 73).—The frothing and danger of projection encountered when soil is fused with sodium peroxide are avoided as follows. A mixture of 10 g. of the sieved (1 mm.) soil with 26 g. of anhydrous sodium carbonate and 13 g. of potassium nitrate is fused at 900° in a 100-c.c. porcelain crucible in an electric furnace. The cold crucible is transferred to a beaker and covered with water containing a few drops of alcohol, and then left for a day. When the whole contents of the crucible have been extracted, the liquid is filtered through asbestos by suction, the filtrate being evaporated to dryness on a steam-bath and the residue heated at 150° for 5 hrs. The mass is then treated with hydrochloric acid, heated again at 150° to render the silica insoluble, dissolved in dilute hydrochloric acid, and filtered. The sulphate is precipitated as barium sulphate in the usual way. T. H. POPE.

**Interaction between ammonia and soils as a new method of characterising soil colloids.** A. N. PURI (*Soil Sci.*, 1931, **31**, 93—97).—The reaction between soils and bases is slow unless excess of base is present. The base-exchange capacity is determined by treating soil with dilute acid to remove all bases and subsequently saturating with ammonia. The ammonia adsorbed is equivalent to the base capacity of the soil. Soils

containing free calcium carbonate have approximately the same  $p_H$  after the above treatment as they had in the natural condition. (Cf. B., 1931, 36, 79.)

A. G. POLLARD.

**Laws of soil colloidal behaviour. IV. Isoelectric precipitates.** S. MATTSON (Soil Sci., 1931, 31, 57—77; cf. B., 1931, 173).—From sodium humate purified by electrodialysis, humates of iron and aluminium are prepared by precipitation from the respective chlorides. In the presence of phosphates or silicates, phospho- or silico-humates are formed. Humates are differentiated from phosphates and silicates by being precipitated quantitatively by ferric or aluminium chloride, by exhibiting an electropositive maximum on the acid side of the isoelectric point, and where much humus is present, in having a second isoelectric point. Ferric "humates" are isoelectric at a lower  $p_H$  than that of the corresponding aluminium compounds. The "humate" ion displaces the phosphate ion and, to a greater extent, the silicate ion from combination with iron or aluminium. The nature of the precipitation of aluminium salts in mixed phosphate-silicate solutions is examined. The phosphate ion displaces the silicate ion, but the reverse action is not apparent, the phosphate content of the complex precipitate being somewhat increased in the presence of silicate. The mineral soil colloids react with iron and aluminium salts in the same way as does the humus complex, forming isoelectric precipitates.

A. G. POLLARD.

**Biochemistry of arable soil.** A. A. J. VANDE VELDE, A. VERBELEN, and L. DEKOKER (Compt. rend., 1931, 192, 766—767; cf. B., 1930, 1082).—The retention of acids or alkalis by soil after shaking with the requisite solutions is measured. The proportions of hydrochloric and sulphuric acids adsorbed by individual soils are approximately the same, but those of acetic acid are much smaller. The differences between these values is a measure of the adsorptive power of soils ("volumetric adsorptive value"). Variations in the latter value in numerous soils were closely parallel with those obtained by the use of dyes and of centrifuged milk.

A. G. POLLARD.

**Fungus flora of the soil.** H. L. JENSEN (Soil Sci., 1931, 31, 123—158).—The fungi present in numerous Danish soils of varying type and  $p_H$  are examined and characterised. Fungal numbers are not related to soil type or reaction except that heavy soils usually contain few fungi. The liming of acid soils increases the numbers of bacteria and *Actinomyces*, but has little effect on the fungi. Manurial treatment (notably with farmyard manure) increases the number of fungi, bacteria, and *Actinomyces* in soils. In soils of  $p_H > 6.0$  there is a definite correlation between the numbers of fungi and of bacteria plus *Actinomyces*. The majority of soil fungi decompose cellulose, and their development in both acid and alkaline soils is stimulated by additions of cellulosic material. The proteolytic power of many soil fungi is considerable.

A. G. POLLARD.

**Lime studies. Significance of buffering in practical liming questions; the rule of the base-fixing adsorption zone of soils; buffer grouping, and a new classification of soils in relation to**

**buffering.** S. Goy [with O. Roos] (Z. Pflanz. Düng., 1931, 20A, 1—43; cf. B., 1931, 454).—New methods of grouping soils according to their buffer values are described. Within the base-fixing area of the buffer curves of soils there is a zone corresponding to  $p_H > 5.5$  in which definite plant injury occurs, and a zone of possible injury the characteristic  $p_H$  values of which vary with the buffer values, being high in poorly buffered soils and *vice versa*. The relative importance of  $p_H$  values of total and exchange acidity in respect of lime-requirement determinations is discussed.

A. G. POLLARD.

**Method of oxidising and dissolving soil for the determination of total and filterable manganese and phosphorus.** E. M. EMMERT (Soil Sci., 1931, 31, 175—182).—The soil sample is prepared for analysis of total manganese and phosphorus by treatment with sulphuric acid and sodium chlorate. The available manganese and phosphorus is determined by shaking soil with water (2 c.c. per g. of soil) for 10 min. and filtering through a No. 2 Whatman filter paper. Colloidal matter present in the filtrate is assumed to be available to the plant.

A. G. POLLARD.

**Nature of soil acidity as affected by the silica: sesquioxide ratio.** L. D. BAVER and G. D. SCARSETH (Soil Sci., 1931, 31, 159—173).—The total base-exchange capacity,  $p_H$ , and physico-chemical constants of the acid of the adsorptive complex ("soil acid") for a number of soils are recorded. Soil acids of different soils are dissimilar and in weathered soils are characteristic of the type of weathering rather than of the parent rock. The colloids from weathered soils have high silica: sesquioxide ratios, buffer capacities, and base-exchange capacities and exhibit the greater acidity. The buffer capacity of the colloids depends mainly on the nature of the soil acid. The exchange complex of soils may be formed by the removal of certain constituents from natural aluminosilicates, by mutual flocculation of the colloidal oxides of iron, aluminium, and silica, or by direct precipitation. There is no uniform relationship between the nature of soil acids and the silica: sesquioxide ratio, although in certain groupings of soils direct relationships are indicated. The chemical composition of soil colloids may be represented with advantage in a form in which the free oxides of iron, aluminium, and silica are excluded.

A. G. POLLARD.

**Direct determination of  $p_H$  of soils in their natural moist condition.** A. V. TROFOMOV (Z. Pflanz. Düng., 1931, 20A, 74—111).—The  $p_H$  values of soils, determined by the quinhydrone electrode, vary with the moisture content and are affected by the oxidation-reduction potential of the soil. Methods for determining  $p_H$  values of soils at their natural moisture contents are described, in which a benzoquinone-quinhydrone electrode is used in soils which oxidise quinhydrone and a quinol-quinhydrone electrode for soils reducing quinhydrone. The drying out of soils causes a steady increase in acidity and *vice versa*. The closest relationship between moisture content and  $p_H$  occurs in moisture ranges below maximum capacity. Within a given range of moisture variation the greatest increases in acidity during drying occur in soils of high hygroscopicity.

A. G. POLLARD.



**Determination of the percentage base saturation of soils and its value in different soils at definite  $p_H$  values.** W. H. PIERRE and G. D. SCARSETH (Soil Sci., 1931, 31, 99—114).—A comparison of methods for determining the exchangeable base status of soils is recorded. The percentage base saturation is most satisfactorily calculated from determinations of the total exchange capacity by the barium acetate-ammonium chloride method and of the exchangeable hydrogen by the barium acetate method. It is suggested that the liming of soils results in the formation of an additional adsorptive complex. In soils of the same  $p_H$ , the percentage base saturation varies considerably, the more highly weathered soils showing the lower values. The percentage base saturation of soils of similar  $p_H$  is not related to the nature of the bases present, nor to the content of organic matter, but shows a general correlation with the silica : sesquioxide ratio and the total exchange capacity of the extracted colloids. The avidity or strength of acids present in soils of the same  $p_H$  differs widely, highly weathered soils, in general, containing the weaker acids. Good correlation was observed between the avidities of soil acids and the percentage base saturation at  $p_H$  4.80.

A. G. POLLARD.

**Determination of the phosphate requirement of soils by the Dirks and Scheffer method as compared with the Mitscherlich pot-culture method.** LAMBERG (Superphosphat, 1931, 7, 61—62).—Comparative examination of numerous soils by the two methods showed very close agreement. The Dirks and Scheffer method (B., 1930, 876) is simple, rapid, and eminently suitable for routine work.

A. G. POLLARD.

**Colorimetric determination of phosphate in relation to soil examination.** E. PFUHL (Superphosphat, 1931, 7, 67—69).—Comparative analyses of soil phosphates are recorded and the advantages of the colorimetric method discussed in regard to speed and simplicity of working without undue loss of accuracy.

A. G. POLLARD.

**Mobility of phosphoric acid in soils.** L. SCHMITT (Superphosphat, 1931, 7, 54—58).—Existing data are quoted to show that no appreciable leaching of phosphate occurs in soils after application of superphosphate under normal cultural conditions.

A. G. POLLARD.

**Changes in the availability of phosphorus in irrigated rice soils.** R. P. BARTHOLOMEW (Soil Sci., 1931, 31, 209—218).—Irrigation of rice lands with water containing appreciable amounts of iron, aluminium, and calcium reduced the water-solubility of the soil phosphate. The increased water-soluble organic phosphorus content of the soil, apparent three months after irrigation, is attributed to anaerobic bacterial activity.

A. G. POLLARD.

**Effect of phosphoric acid in accelerating maturity [of sugar beet].** C. BONNE (Superphosphat, 1931, 7, 27—31).—Neither the yield nor the sugar content of sugar beet is reduced by heavy dressings of superphosphate. The ripening of different strains of sugar beet is accelerated to different extents by phosphate fertilisers, which may produce this effect on soils already having a sufficiency of phosphate for the growth of the crop.

A. G. POLLARD.

**Effect of increasing applications of superphosphate on early cabbage.** H. SCHWARZE (Superphosphat, 1931, 7, 63).—Field trials are recorded in which the use of superphosphate in spring and as a top dressing in early summer increased the total yield and materially hastened the maturing of cabbage.

**Influence of combined nitrogen on growth and nitrogen fixation by *Azotobacter*.** J. E. FULLER and L. F. RETGER (Soil Sci., 1931, 31, 219—234).—Fixation of considerable amounts of nitrogen by *Azotobacter* occurred in media and in an atmosphere freed from combined nitrogen. Nucleic acid, tryptophan, tyrosine, and guanine slightly reduced nitrogen fixation without affecting the growth of the organism. Peptone stimulated the growth of the organism, but definitely reduced nitrogen fixation. *Azotobacter* utilised glutamic and aspartic acids, cystine hydrochloride, and glycine. Increased growth resulted, but nitrogen fixation was depressed and in some cases actual losses of original nitrogen occurred. Creatine, creatinine, and urea accelerated growth, but practically inhibited fixation. Indole and skatole in relatively small concentrations were toxic to *Azotobacter*. The sulphate, carbonate, nitrate, and chloride of ammonium and the nitrates of sodium and potassium depressed nitrogen fixation to varying extents, but increased the growth of the organisms.

A. G. POLLARD.

**Influence of various non-nitrogenous compounds on the growth of certain bacteria in soils of low productivity.** H. J. CONN and M. A. DARROW (N.Y. State Agric. Exp. Sta., Tech. Bull., 1930, No. 172, 40 pp.).—The absence of certain commonly occurring non-sporing bacteria (notably *B. globiforme*, Conn) from two soils, and their failure to develop in these soils after inoculation, are examined. Addition to the soils of ammonium salts, nitrates, certain organic nitrogenous substances, or the sulphates, phosphates, and hydroxides of calcium, magnesium, sodium, and potassium enabled the organisms to develop. The effect of the alkali salts was more pronounced in soil of high colloidal content. Non-development in the original soils is ascribed to the deficiency of nitrogen in a form suitable for the utilisation of the bacteria, although the total nitrogen content of the soils was relatively high. The nature of the stimulative effect of the alkali salts is discussed and the possibility of utilising the organisms for determining the nitrogen availability in soils is noted.

A. G. POLLARD.

**Plant nutrient content of soils calculated from the Neubauer, the static, and the Mitscherlich methods.** CLAUSEN (Z. Pflanz. Düng., 1931, 10B, 145—155).—In a comparison of the three processes, the value of the static method is emphasised. In general, the Mitscherlich method yields results in closer conformity with those of field trials than does the Neubauer seedling method.

A. G. POLLARD.

**Composition and quality of Pennsylvania cigar-leaf tobacco as related to fertiliser treatment.** D. E. HALEY, J. B. LONGENECKER, and O. OLSON (Plant Physiol., 1931, 6, 177—182).—Fertiliser treatment of tobacco plants did not affect the K : Ca ratio in the cured leaves. The quantity of sulphur in the leaves was not

increased by considerable dressings of potassium sulphate. During the fermentation process of leaf curing there was an apparent increase in the soluble ash constituents of the leaves, a narrowing of the N : C ratio, and little or no loss of nitrogen. A. G. POLLARD.

**Manuring of the principal cereals and their mean yields in Nederling.** J. WEIGERT (Z. Pflanz. Düng., 1931, 10B, 136—142).—Records of ten-year manurial trials are presented and discussed. A. G. POLLARD.

**Nitrogen losses and action of cold- and hot-fermented manures.** D. MEYER [with P. OBST, F. WILCZEWSKI, and W. DIETRICH] (Z. Pflanz. Düng., 1931, 10B, 121—136).—During the cold-fermentation of manure losses of dry matter were higher and of nitrogen were lower than during hot-fermentation (Krantz). In pot cultures the average percentage utilisation of nitrogen from cold-fermented manure was higher than from the hot-fermented product except when drainage from the latter had been prevented during preparation. In field trials hot-fermented manure showed no superiority in crop production or percentage utilisation over the cold-fermented product. A. G. POLLARD.

**Decomposition in, and effect of straw manures on, soils.** H. ENGEL (Z. Pflanz. Düng., 1931, 20A, 43—73).—Relationships between the C : N ratio of straw manures and their ratio of decomposition in soils are examined and discussed in relation to the significance of carbon dioxide and nitrate production during decomposition. High carbon dioxide production and good humification cannot take place simultaneously. The value of straw manures lies mainly in the physico-chemical properties of the humus produced. With narrowing C : N ratio in manures, the carbon dioxide produced during decomposition decreases and nitrification increases. With a C : N ratio in the manure of 20 : 1, fixation of nitrogen appears to cease and a slow nitrification begins. With a wider ratio no nitrification takes place, but with C : N = 12 : 1 16% (approx.) of the total nitrogen appears as nitrate. The ease of decomposition of the organic matter of straw manures decreases with advancing humification. The effect of changes of C : N ratio on the nitrification process in manures varies with the type or organic matter present. No relationship exists between the ammonia-nitrogen content of manure and its action in soil (cf. Barthel, B., 1930, 73). Of the ammonia-free nitrogen content of manure 16—30% was nitrified in soil. A. G. POLLARD.

**Hydrogen-ion and aluminium concentrations in the soil solution, and percentage base saturation as factors affecting plant growth on acid soils.** W. H. PIERRE (Soil Sci., 1931, 31, 183—207).—Pot-culture experiments are described in which the  $p_H$  of a number of soils was artificially varied. The  $p_H$  at which plant injury first appeared in acidified soils varied with the soil type. The concentration of aluminium in the displaced soil solution is not directly related to the  $p_H$  of the soil, nor is it a primary cause of acid injury to plants. The concentrations of calcium and manganese in the soil solution are not related to plant injury. The ratio K : Ca in the soil solution decreases with increasing soil acidity and is connected with plant injury. In soils of similar  $p_H$  the percentage base saturation varies con-

siderably. The latter factor is closely related to plant injury in acid soils. When the aluminium concentration of the soil solution is high, plant growth is affected in soils of higher percentage base saturation than when aluminium is absent or present only in small proportion. A. G. POLLARD.

**Chibnall's method of extraction for investigation of the winter hardiness of plants.** W. E. TOTTINGHAM, R. G. SHANDS, and E. D. DELWICHE (Plant Physiol., 1931, 6, 167—176).—Analyses of the nitrogenous constituents and sugars in the protoplasmic extracts of wheat and lucerne indicates relationships between variations in composition and winter hardiness. A. G. POLLARD.

**Influence of frequency of cutting on the productivity, botanical and chemical composition, and the nutritive value of "natural" pastures in Southern Australia.** J. G. DAVIES and A. H. SIM (Counc. Sci. Ind. Res., Australia, 1931, Pamphlet 18, 28 pp.).—Maximum removal of calcium from soil by herbage occurred when the latter was cut at intervals of 6—8 weeks. The phosphate content of herbage cut at 2- and 4-week intervals was practically identical, but decreased with less frequent cutting, reaching a minimum when the grass was allowed to reach maturity. With fortnightly cutting the phosphate content was maintained throughout the season. The crude protein content of herbage was greatest when cut at 2- and 4-weekly periods, the value being more than double that of the mature grass. The crude fibre content increased as the frequency of cutting diminished, the minimum being attained with fortnightly cutting. A. G. POLLARD.

**Food reserves in relation to other factors limiting the growth of grasses.** L. F. GABER (Plant Physiol., 1931, 6, 43—71).—The effects of manuring and of the frequency of cutting on the yield and quality of grasses are examined. Productive capacity depends not only on adequate manuring and moisture supply, but also on the food reserves of the plants themselves. Too frequent or too close cutting may result in slow recovery and poorer yields. Frequent cutting results in heavy withdrawals of available nitrogen from the soil, and nitrogen deficiency becomes the chief factor in growth limitation. Where adequate supplies of nitrogen are present in soils, the consumption of carbohydrate reserves within the plant is rapid and slow replenishment may restrict further growth. A. G. POLLARD.

**Preservation of [green] feeding-stuffs.** A. KROZEK (Z. Zuckerind. Czechoslov., 1930, 55, 79—85).—In the silage of green fodder, a much nearer approach to pure lactic fermentation can be attained in concrete silos than in earth trenches. In the former the anaerobic conditions in the closely packed material repress acetic organisms, and respiration is too restricted to produce the rise in temperature necessary for active butyric fermentation, except with very woody material which, even when finely chopped, retains a considerable amount of air. Very moist or sappy material, especially if rich in proteins, suffers badly from soil bacteria which produce ammonia. The only sure safeguard against these is partial withering before silage. Where this is not practicable the material may be rendered more

absorbent, and therefore less wet, by very fine chopping or by admixture of absorbent materials; or a proportion of beet leaves or molasses may be incorporated, the sugar in which favours lactic fermentation. Tabulated data for stored green fodder, in most cases beet leaves, show a much smaller production of acetic and butyric acids in concrete silos than in earth trenches, lactic acid representing 70–80% of the total free acids in the former, as compared with 40% on the average in silage from earth trenches. J. H. LANE.

**Rôle of nitrogen in the intensive working of meadows.** C. MATIGNON (Chim. et Ind., 1931, 25, 271–275).—A description is given of the intensive grazing systems advocated in England and of the recent adaptation of the principle to the growth of cereals for cattle feeding in the United States. A. G. POLLARD.

**Measurement of the nitrate-assimilating power of soils.** R. A. PENDLETON and F. B. SMITH (Proc. Iowa Acad. Sci., 1929, 36, 75–78).—Soils have a definite nitrate-assimilating power which may be favoured by addition of organic matter. CHEMICAL ABSTRACTS.

**Effect of nitrogen fertilisers on nitrification.** R. A. PENDLETON, P. E. BROWN, and F. B. SMITH (Proc. Iowa Acad. Sci., 1929, 36, 99–103).—The fertilisers, particularly sodium nitrate, increased the nitrate content and stimulated the nitrifying power of the soil. CHEMICAL ABSTRACTS.

**Nitrogen fixation by *Rhizobium meliloti* and *R. japonicum*.** G. G. POHLMAN (J. Amer. Soc. Agron., 1931, 23, 22–27).—Under suitable cultural conditions certain species of *Rhizobium* may be able to fix nitrogen in absence of the host plant. CHEMICAL ABSTRACTS.

**Spontaneous culture method for studying the non-symbiotic nitrogen-fixing bacteria of the soil.** R. H. WALKER and J. L. SULLIVAN (Proc. Iowa Acad. Sci., 1929, 36, 53–61).—A discussion. The test may serve as a biological comparison of the fertility of soils. CHEMICAL ABSTRACTS.

**Variations in soil reaction affect nitrification.** P. E. BROWN and G. V. C. HOUGHLAND (Proc. Iowa Acad. Sci., 1929, 36, 93–97).—When differences in  $p_H$  are not great, the influence of greater acidity may be important; where wide variations occur, the effects of reaction may overshadow those of treatment. High acidity retards the nitrification process. CHEMICAL ABSTRACTS.

**Nitrogenous fertiliser prepared by the Muhlert process.** F. GIESECKE and F. KLANDER (J. Landw., 1931, 79, 69–86; cf. Muhlert, B., 1930, 129).—Losses of nitrogen from the material (essentially ammonium sulphate and calcium carbonate) are relatively small. In pot trials the fertiliser proved as efficient as ammonium sulphate and should be valued on its ammonia content. A. G. POLLARD.

**Soil-reaction effects on *Phymatotrichum* root rot.** W. N. EZEKIEL, J. J. TAUBENHAUS, and E. C. CARLYLE (Phytopath., 1930, 20, 803–815).—Cotton plants were grown in soil artificially acidified to varying extents and infected with *Phymatotrichum*. With increasing acidity there was a marked diminution in root rot at about  $p_H$  6.0, but none appeared at  $p_H$  5.0. Soil treatment with sulphur decreased root rot, but the

acidity produced did not extend to a sufficient depth to ensure complete control even when the acidity of the surface soil was sufficient to cause plant injury.

A. G. POLLARD.

**Factors influencing the severity of root-rot troubles of sugar cane.** H. H. FLOR (Louisiana Agric. Exp. Sta. Bull., 1930, No. 212, 40 pp.).—No evidence was obtained indicating that inorganic salts or soluble toxins were concerned in the incidence of root rot. Washing the soil did not affect the growth of sugar cane, but steaming the soil induced a more rapid growth. *Pythium* did not grow in culture solutions of  $p_H < 5.2$  or  $> 10.0$ , and was more tolerant to alkalinity than to acidity. Environmental and biological factors are considered. A. G. POLLARD.

**Preliminary survey of some of the soils in Kenya.** D. S. GRACIE (Dept. Agric., Kenya, Bull., 1930, No. 1, 131 pp.).

**Phthaleinoscope.**—See I. Blood-albumin.—See XIX. Nicotine in insecticides.—See XX.

#### PATENTS.

**Granulation of fertilisers by spraying molten or liquid materials.** W. K. HALL, F. HEYWOOD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 345,658, 19.12.29).—A whirling motion is imparted by a rotating paddle wheel to a supply of molten ammonium nitrate etc. in a steam-jacketed vertical cylinder so that the material issues through a relatively large orifice (e.g., 1 in. in diam.) at the bottom in the form of a conical curtain which breaks up into drops before solidification. The shaft bearing the paddle wheel may also be provided with inclined vanes to impart a downward thrust to the material. L. A. COLES.

**Spray oils [for plants etc.].** W. J. YATES, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 1,778,239–40, 14.10.30. Appl., 15.5.29).—A petroleum oil-water emulsion spray contains (A) up to 1% (calc. on the weight of oil) of a naphthylamine, (B) quinol or a chloro-substitution product thereof. L. A. COLES.

**[Seed and plant] disinfectant composition.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,008, 28.10.30. Appl., 19.5.28).—The composition comprises a mercury alkyl halide (mercury ethyl chloride), a soluble thiosulphate, sulphite, sulphide, thiocyanate, or xanthate (preferably sodium thiosulphate), and an inert powder, e.g., infusorial earth. L. A. COLES.

**Drying of grass etc.**—See I. Fertiliser materials.—See IX. Alkaloids from cocoa products.—See XX. Sewage sludge.—See XXIII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Factors influencing the ash content and sugar recovery.** J. G. THIEME (Archief, 1930, 38, 713–727).—During 1929 some factories in Java produced molasses of a very high purity, notwithstanding their modern equipment. This was traced to an abnormal composition of the non-sugars, especially in inorganic matter. It appears that ever since 1915 the ash factor of the juice of the cane has been gradually increasing, with a sudden rise during 1926–1929, which period coincides with the introduction of the POJ 2878 cane, viz., from

20.8 to 24.2. It would seem that a high ratio of ash to non-sugars is a characteristic of the modern canes, resulting in lower yields in the factory.

J. P. OGILVIE.

**Imbibition [in sugar factories].** N. M. RYDLEWSKI (Internat. Sugar J., 1931, 33, 40—41).—Experiments carried out in a Cuban factory using two mill tandems operating under conditions generally as similar as possible, except that in the first the imbibition water was applied to the bagasse entering the mills, and in the second on emerging, gave figures clearly in favour of the latter procedure.

J. P. OGILVIE.

**Carbonatation [of beet juices]. II. Influence of the reaction medium on the hydration of carbon dioxide in alkaline solutions.** J. DEDEK and J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1930, 55, 43—51).—The interaction between sodium hydroxide and dissolved carbon dioxide, which takes a measurable time in dilute aqueous solutions, is retarded in presence of sucrose or dextrose. Sucrose exerts its maximum effect, 5-fold retardation, at about 15% concentration, which is its concentration in raw beet juice; above this concentration the retardation is less, and above 50% it changes to acceleration. The retarding effect depends on the absolute concentration of the sugar, and not on the ratio of sugar to reacting substances. It is therefore due to an effect on the medium, but the important factor is not viscosity, for glycerol causes acceleration, and starch paste and gum arabic produce a retardation very slight in proportion to their effect on viscosity. The retarding or accelerating effects of different substances present together are not strictly additive.

J. H. LANE.

**Course of the first carbonatation [of beet juices]. II. Factory tests.** J. DEDEK and T. NIELSEN (Z. Zuckerind. Czechoslov., 1931, 55, 312—323; cf. B., 1930, 1125).—Alkalinity determinations made during the first carbonatation, using phenolphthalein and methyl-red, showed a distinct difference between the two readings, both running parallel at the beginning, but the latter subsequently remaining at a constant value much longer than the first. Observations were also made of the manner in which the dry substance, polarisation, purity, and electric conductivity fall during the course of carbonatation.

J. P. OGILVIE.

**Adsorption of the calcium salts of fatty acids in carbonatation [of beet juice].** T. NEMES (Z. Zuckerind. Czechoslov., 1930, 55, 159—162).—Salts of the fatty acids are adsorbed by the calcium carbonate formed during the carbonatation of raw beet juice, and the more readily the more complex is the fatty acid concerned.

J. P. OGILVIE.

**Influence of products of dextran fermentation (*Leuconostoc mesenteroides*) on the liming and carbonatation [of beet] juice.** W. KONN (Z. Zuckerind. Czechoslov., 1930, 55, 131—139).—Gelatinous matter formed by *Leuconostoc mesenteroides* was obtained from the pulp-catchers of a beet-sugar factory, and washed free from soluble matters. Suspensions containing 0.5% of dry matter, in water and in 15% sucrose solutions, were heated at 85° with 2% of pure lime for various periods and carbonated. The substance dis-

solved more or less during liming, according to the duration of heating, but it was almost entirely eliminated by subsequent carbonatation, whether the liquids were filtered after liming or not. Beet juice containing such matters is best heated to 85° before addition of lime, maintained at this temperature for 5—10 min. after liming, and then carbonated as usual, preferably with a further addition of lime before the second carbonatation.

J. H. LANE.

**Conductometric study of over-carbonatation [of sugar solutions].** K. ŠANDERA and V. PREININGER (Z. Zuckerind. Czechoslov., 1930, 55, 115—119; cf. B., 1930, 582).—In the carbonatation of sucrose solutions containing pure lime, the conductivity curve, plotted against diminishing alkalinity, showed a sharply defined minimum at the neutral point to phenolphthalein. In presence of 0.2% of potassium hydroxide the minimum occurred at an alkalinity of 0.04% CaO. In presence of 0.04% of ammonium hydroxide, which is only slightly more than beet juice contains after liming, the minimum occurred at an alkalinity of 0.02% or 0.032% CaO, according as pure lime or technical lime containing 3% of magnesia was used. On saturating beyond the minimum, the increase in conductivity for a given fall in alkalinity was much greater with the technical than with the pure lime, owing mainly to interaction of the ammonia with the magnesia in the lime. Among alkalis, ammonia has thus an exceptional influence on the optimum end-point of carbonatation, as judged by the elimination of electrolytes, especially where impure lime is used, and in this case it also increases the amount of electrolytes redissolved when carbonatation is carried beyond the optimum point.

J. H. LANE.

**Evaporation of [beet] juices in the presence of carbonatation scums, sodium sulphite, and active carbons.** V. STANĚK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1931, 55, 339—348; cf. B., 1929, 572).—Evaporation of clarified beet juice in the presence of some of the last carbonatation scums gave a lighter juice than when the clear juice was so treated. Similarly when 0.0025—0.2% of sodium sulphite was used there was a striking improvement in the colour, though additions above 0.2% failed to increase the effect. Very small additions of active carbon ("Norit") gave a similar result.

J. P. OGILVIE.

**Filtration of the whole juice in sulphitation.** W. C. NIEBOER (Archief, 1930, 38, 499—505; Internat. Sugar J., 1931, 33, 72—73).—In order in the sulphitation method of clarification to filter the whole of the juice, an increase of 50% of the filter-press area compared with that previously used for the mud alone should suffice. A greater addition of lime is not advised to assist the filtration, but the presses should be pre-coated with kieselguhr. Whole-juice filtration is not only more rapid and economical, but improves the quality of the white sugar obtained.

J. P. OGILVIE.

**Adsorption [by carbons] from sugar solutions. II. Adsorption of water.** J. VAŠÁTKO and J. VÁCHA (Z. Zuckerind. Czechoslov., 1931, 55, 183—192).—Different carbons used for decolorising in the sugar industry can be divided into two groups according to their power of adsorbing water from aqueous solutions

of sucrose: (a) Standard-Norit, Polycarbon, and Supra-Norit; (b) Carboraffin, Supra-Norit 5X, and Radit, this latter group adsorbing much more water from sucrose solutions than the first, being characterised by great porosity. Between the two groups lies Supra-Norit 3X. J. P. OGILVIE.

**Decomposition of the reducing sugars in alkaline medium during [cane] sugar manufacture.** K. D. DEKKER (Internat. Sugar J., 1930, 32, 641—642).—Decomposition of the reducing sugars occurs as soon as cane juice becomes slightly alkaline, a mixture of dextrose, lævulose, and mannose being formed in equilibrium with the decrease of the cupric reducing power and an increase of colour, total acidity, and calcium salts. Among the acids produced are saccharinic and lactic. Glucose is also formed, finally to accumulate in the molasses to the extent of 5—12%. J. P. OGILVIE.

**Determination of calcium salts in cane juices and syrups.** M. VAN DER KREKE and K. D. DEKKER (Meded. Proefstat., Java, 1930, 1—11; Internat. Sugar J., 1931, 33, 73).—The modification of the soap test elaborated by Spengler and Brendel (Internat. Sugar J., 1929, 91) is recommended, being found to give results agreeing closely with those found by oxalate precipitation. J. P. OGILVIE.

**Determination of natural alkalinity [of beet juice].** I. B. MINTZ and others (Nauk. Zapiska, 1930, 9, 246).—A modification of the Duwell and Solon method (cf. Internat. Sugar J., 1930, 366) is recommended, in which the sample is carbonatated until the pink obtained by the addition of phenolphthalein has disappeared, after which the liquid is boiled, restored to its original volume, and finally titrated with 0.2N-hydrochloric acid. J. P. OGILVIE.

**Rapid determination of the true purity of syrups, using the conductivity depression method.** J. H. ZISCH (Facts about Sugar, 1930, 25, 741—745).—A quantity representing 2 g. of the syrup with 10 c.c. of dilute hydrochloric acid are made up to 100 c.c., mixed, and the resistance of the solution is read in ohms (*A*), the conductivity of the acid used being also ascertained (*B*); then *B—A* gives the conductivity depression, values for which corresponding to different true purity values for syrups have been compiled. J. P. OGILVIE.

**Dilution [of sugar juice] in relation to comparative purities.** N. DEERR (Internat. Sugar J., 1930, 32, 616—617).—Mathematical evidence is given in support of the following statement given in the "Methods" of the Hawaiian Sugar Technologists' Association: "To be strictly comparable, gravity solids determination should be made in solutions of the same non-sugar concentration." J. P. OGILVIE.

**Determination of unknown losses in juice purification.** J. BOERS (Archief, 1930, 38, 745—754).—After experiments on the determination of sucrose by different double-polarisation methods, the author concludes that in normal cases the unknown losses are not due to chemical losses during the process of juice clarification, or are due only to a small extent. Rather should they be attributed to mechanical losses at the filter-press station and to entrainment in the last body of the

evaporator or the pans, though during these boiling processes it seems certain that some chemical losses do take place. J. P. OGILVIE.

**Determination of small proportions of invert sugar in raw sugars.** L. EYNON and J. H. LANE (J.S.C.I., 1931, 50, 85—86 T).—A method is described for the volumetric determination of small proportions of invert sugar (less than 0.3%) in raw sugars with Fehling's solution in presence of methylene-blue as internal indicator. The procedure is the same as that previously described (*ibid.*, 1923, 42, 32 T) save that the determination is carried out in presence of a known quantity of added invert sugar.

**Action of non-sugars of refined sugars on the caramelisation test.** J. PUCHERNA (Z. Zuckerind. Czechoslov., 1930, 55, 143—151).—Various non-sugar substances were mixed with fine granulated and *pilé* sugars in amounts from 0.002 to 0.2%, and after drying at 100° 6.5 g. of the mixture were heated at 170° for 15 min. Substances without effect on this heating test included: sodium, potassium, and barium chlorides, potassium sulphate, sodium oxalate, sodium carbonate, and sodium and ammonium acetates, *i.e.*, salts reacting neutral or alkaline in aqueous solution. Substances causing the formation of colour comprised the amino-acid salts; whilst those inducing inversion, and colour formation after heating with 2N-alkali, were ammonium chloride, sulphate, and oxalate, potassium bisulphate, calcium chloride, ferrous sulphate, and ferrous ammonium sulphate. J. P. OGILVIE.

**Hygroscopicity of [beet] sugars.** M. GARINO (Ind. Sacc. Ital., 1930, 23, 483—486).—Of the impurities which normally accompany beet sugar, only caramelan is distributed in any notable quantity in the body of the crystal, caramelan and the ammonium, calcium, and potassium salts of glucic and apoglucic acids being found in traces only in the crystal network. Caramelan is the impurity which chiefly confers hygroscopicity on the sugar, although this defect in the case of white beet sugar may be due to a very thin coating of calcium chloride. J. P. OGILVIE.

**Deterioration of Philippine sugar at varying degrees of humidity.** Q. D. RENDON (Philippine Agriculturist, 1930, 19, 383—396).—Small bags of raw and refined sugars were stored in large desiccators, in which humidities ranging from 50 to 100% were maintained, analyses being made at the end of 60, 90, and 100 days. Although the data showed some variations, in general at humidities above 66% the sugars attracted moisture and deteriorated, the loss of sucrose thus caused increasing with the humidity. J. P. OGILVIE.

**Determination of starch in barley and in malt.** IV. A. R. LING (J. Inst. Brew., 1931, 37, 216—224; cf. B., 1924, 991).—The determination is first made of the percentages of "apparent maltose" produced when malts, the diastatic powers of which range from 32 to 100° expressed on the Lintner scale, are made to act at 57° under standard conditions on pure wheat starch paste maintained by an acetate buffer at *p*<sub>H</sub> 4.6. For the starch determination in the barley or malt, the latter are ground and, after extraction with alcohol are digested

under the same standard conditions with a malt extract of known diastatic power. The amount of apparent maltose is determined in the mash and corrected for that of the malt extract. The percentage of starch is calculated from the formula  $S = 100M/M_1$ , in which  $S$  is the percentage of starch in the dry barley or malt,  $M$  the percentage of "apparent maltose" produced in the dry pure starch by a malt of definite diastatic power, and  $M_1$  the percentage of "apparent maltose" produced from the dry barley or malt with a malt of the same diastatic power. In the case of barley the results are confirmed by Ewer's method, which depends on acid hydrolysis and polarimetric measurement, but with malt the polarimetric readings must be multiplied by a factor to bring the results into line with those obtained by the diastatic method.

C. RANKEN.

Root rot in sugar cane.—See XVI.

## PATENTS.

Manufacture of pure lævulose from inulin. SCHERING-KAHLBAUM A.-G. (B.P. 345,926, 9.10.30. Ger., 22.10.29).—Lævulose solutions obtained by the acid inversion of inulin, after freeing from acid, are evaporated to dryness *in vacuo* at 50–60°, and the product is stirred with alcohol to dissolve impurities, separated from the liquor, and dried.

L. A. COLES.

Separating solids from liquids. Polarimeter. Extraction of organic substances.—See XX.

## XVIII.—FERMENTATION INDUSTRIES.

Drying of hops. Institute of Brewing Research Scheme. Report on the eighth and ninth seasons' work at the experimental oast, 1928 and 1929. A. H. BURGESS (J. Inst. Brew., 1931, 37, 186–196; cf. B., 1929, 533).—The time required to dry the hops is related to the air speed, temperature, and depth of loading according to the formula:  $T = [1/(V.P. - v.p.)] [(716 \cdot 5L/a^{1.047}) + (6260/a^{0.39})]$ , where  $T$  is the time of drying in min.,  $L$  the loss of weight in oz. per sq. ft. of kiln floor,  $a$  the air speed above hops in ft. per min.,  $V.P.$  the saturated vapour pressure (in. Hg) of water at the temperature of the air, and  $v.p.$  the vapour pressure (in. Hg) due to the moisture originally present in the air. A reduction of air speed towards the end of drying effects an economy of heat, but entails a larger drying period. The amount of  $\alpha$ -acid decreases with increase of drying temperature, whilst the  $\beta$ -fraction is unaffected. The preservative value of the hops, as shown by a biological test, decreases when they are dried above 70°. Hops preserved by sterilisation have a higher preservative value than the same hops dried in the usual way, and the optimum final moisture content of the hops when removed from the kiln is 5–7%.

C. RANKEN.

The antiseptic content of worts and beers in relation to that of the hops. T. K. WALKER and J. J. H. HASTINGS (J. Inst. Brew., 1931, 37, 206–207).—The grading of the relative antiseptic values of hopped worts as determined by the biological method are in close agreement with the preservative powers of the hops as shown by the gravimetric method. On the other hand, the value of the antiseptic powers of beers

made from those worts give the same order of grading as those obtained with the hopped worts, but show different relative values as between individual samples. It is suggested that the varying relative values for the worts and beers is due to the removal during fermentation in each case of an equal quantity of antiseptic power, and not of an amount which bears a definite ratio to that originally present.

C. RANKEN.

Biological significance of nitrates in brewing liquor. W. WINDISCH and F. WINDISCH (Woch. Brau., 1931, 48, 106–112).—As little as 2 g. of potassium nitrate per hectolitre added to wort markedly depresses the degree of fermentation and yeast reproduction, as shown by laboratory fermentations using two bottom, one top, and one distillery yeast. The effect is increased, but to a less proportional extent, by 5 and 20 g./hectolitre. It is caused by reduction of part of the nitrate to nitrite, which is not only poisonous to the yeast, but may impart a chlorine-like flavour to the beer (cf. A., 1930, 1477).

F. E. DAY.

Influence of adsorbents on fermentation. B. LAMPE (Z. Spiritusind., 1931, 54, 75–76).—The use of aeration of the mash to stimulate the rate of fermentation can be replaced by the addition to the fermentation liquid of 0.1 vol.-% of adsorbents, such as animal and wood charcoal, silicic acid, etc. Acceleration of the fermentation is 3–10 times as great as that produced by aeration, and the method has also the advantage that the production of acetaldehyde, which is increased by aeration, is diminished to such an extent that the use of an aldehyde column in the distillation apparatus is rendered unnecessary. The acceleration of the fermentation may be due to the reduction in the concentration in the liquid of the carbon dioxide by the charcoal, or to the adsorption of the yeast cells due to a mechanical attraction.

C. RANKEN.

Removal of head during bottom fermentation. L. SAILER (Woch. Brau., 1931, 48, 129–133).—The apparatus of Hallermann (B., 1931, 362) is criticised as permitting part of the slimy matter to remain in the beer and as allowing the separated material to be continuously extracted by liquid of relatively high alcohol content. An apparatus is described consisting of a grid of vertically flattened tubes, having keeled edges below, which is supported on the surface of the fermenting wort with the lower part of the tubes immersed. The head rises over the grid at first, and as it subsides adheres to the tubes and dries on. The apparatus is provided with lifting tackle for raising the grid during emptying and removing yeast from the fermentation vessel, and with supports for the grid while cleaning. The tubes are arranged to carry the cooling water. It is claimed that by the use of this arrangement the flavour and brilliancy of beer are greatly improved.

F. E. DAY.

Importance of neutralisation and of the reaction of the medium in the lactic-acetic fermentation. V. BOLCATO (Giorn. Chim. Ind. Appl., 1931, 13, 69–73).—The reaction of the medium exerts on the activity of lactic-acetic micro-organisms an influence which results in appreciable displacement of the balance of the two principal acids produced. If the acidity is high,

the formation of acetic acid increases, and *vice versa*, but it is not found possible to make acetic acid the main product.

T. H. POPE.

**Determination of potassium and alkalis in raisin wine and must.** L. SEMICHON, M. FLANZY, and (MLLE.) M. LAMAZOU-BETBEDER (Ann. Falsif., 1930, 23, 517—526).—The official French method based on the precipitation of potassium as the bitartrate gives high results, and in its place the following is recommended. The wine etc. (50 c.c.) is evaporated over a hot plate to the consistency of an extract; 25 c.c. of nitric acid and 2 drops of mercury are added, and heating is continued until the liquid clears. Dilution with 10 c.c. of boiling water and addition of excess of saturated baryta causes the precipitation of sulphates and phosphates. Carbon dioxide is then passed through for 2 min., followed by hydrogen sulphide to complete precipitation of the mercury and traces of iron. The solution is boiled, filtered, and made up to volume (100 c.c.). The potassium content of the filtrate is determined by the perchloric acid method. For sodium the alcohol filtrate from the potassium perchlorate can be converted into sulphate and weighed. The method should be carried out according to the detailed instructions given in the paper.

E. B. HUGHES.

**Iron and copper in white wines.** J. RIBÉREAU-GAYON (Ann. Falsif., 1930, 23, 535—544).—The wines contain (per litre) 5—50 mg. Fe and 0.5—3 mg. Cu in the form of salts. These small amounts of metal do not affect the taste, but they have an appreciable action on the clarity of the wine and also in catalysing oxidation reactions. The iron causes turbidity when in the ferric state and copper when in the cuprous state. Conditions allowing oxidation (*e.g.*, exposure to air, or aeration) favour the former and those favouring reduction produce the latter kind of turbidity. The presence of copper increases the susceptibility of the wine to turbidity by the iron. Both iron and copper catalyse oxidation of the sulphur dioxide in wine. Turbidity either from wine or copper does not readily appear in wine stored in casks, as aeration is then insufficient to oxidise the iron, but enough to maintain the copper in the cupric state.

E. B. HUGHES.

**Acidity of wines with regard to [French] legal definition.** L. FERRÉ (Ann. Falsif., 1931, 24, 75—80).—In definitions of the acidity of wine some standard  $p_H$  for the neutral point must be fixed, as the tannins present affect the end-point in titrations with alkali, using certain indicators. Litmus gives the best results.

E. B. HUGHES.

**Determination of methyl alcohol.**—See III.  
**Dextran fermentation of beet juice. Starch in barley etc.**—See XVII.

#### PATENTS.

**Manufacture of yeast producing high yields.** R. McD. ALLEN and F. E. TIMMER, Assrs. to VITAMIN FOOD Co., Inc. (U.S.P. 1,775,800, 16.9.30. Appl., 14.12.25).—Yeast is grown in an intensely aerated and very dilute wort which contains molasses, ammonium sulphate, tricalcium phosphate, and, if desired, small amounts of potassium and magnesium sulphates.

C. RANKEN.

**Manufacture of denatured alcohol.** G. H. CONDUCT (U.S.P. 1,777,035, 30.9.30. Appl., 21.12.23).—The liquid containing alcohol is sprayed into a chamber heated to above the b.p. of the alcohol by a furnace supplied with solid organic fuel. The products of the incomplete combustion of the fuel are drawn into the chamber through a perforated bottom, and part with their content of substances of a denaturant nature to the volatilised alcohol, which is thereupon withdrawn to a condenser by means of a fan placed at the exit end of the condenser.

C. RANKEN.

**Manufacture of organic acids [by fermentation].** F. J. CAHN (B.P. 345,368, 2.1.30. U.S., 4.1.29).—Organic acids, such as citric acid, are produced by growing acid-producing fungus on slices of plants containing lævulose or carbohydrates yielding lævulose. The fermentative action is improved by powdering the plant sections with an alkaline material, and by breaking down superficially the cellular tissue of the sections by successive freezing and thawing. If necessary, the plant sections may be intermixed with inert materials, such as stoneware, Raschig rings, etc.

C. RANKEN.

**Increasing the potassium salt content of distiller's waste.** K. CUKER (U.S.P. 1,778,381, 14.10.30. Appl., 25.3.27. Czechoslov., 30.3.26).—The distiller's wash, which is clarified by centrifuging, is used instead of water to prepare the next molasses mash, and the process is repeated until the concentration of the salts in the centrifuged liquor is so high as to interfere with the fermentation.

C. RANKEN.

#### XIX.—FOODS.

**Physico-chemical constitution of spray-dried milk powder.** L. H. LAMPITT and J. H. BUSHILL (J.S.C.I., 1931, 50, 45—54 T).—A characteristic difference between full-cream roller-process milk powders and full-cream spray-process milk powders, which is accounted for by the difference in manufacture, is that in the former the fat is readily extractable in the cold by solvents, whereas in the latter only a small proportion of the fat can be so removed. It is shown that the fat of the spray-process powder becomes "free" when the powder has absorbed a certain minimum amount of moisture ("critical moisture content"), viz., 8.6—9.2% for ordinary full-cream powder (26% of fat). In the case of freshly made spray powders the fat becomes "free" at the lower critical limit only after a certain time (up to 4 days), whereas with older powders less time is required. This "freeing" of the fat is shown to be associated with crystallisation of the lactose of the milk powder. The change by which the fat becomes "free" is irreversible. A standard method for the determination of the "free" fat of milk powder is described.

E. B. HUGHES.

**Nutritional value of raw and pasteurised milk.** S. BARTLETT (J. Min. Agric., 1931, 38, 60—64).—The results obtained by Leighton and McKinlay in their report on an investigation in Lanarkshire schools (*cf.* "Milk Consumption and the Growth of School Children," Dept. Health, Scotland) have been recalculated, using larger groupings. The reclassification shows that the consumption of raw milk produces greater increase in weight and height than does pasteurised milk.

E. B. HUGHES.

**Fat of sow's milk.** O. LAXA (Ann. Falsif., 1931, 24, 87—88).—A complete analysis is given.

E. B. HUGHES.

**Determination of milk proteins. IV. (A) Combined, (B) separate, determination of albumin and globulin.** G. M. MOIR (Analyst, 1931, 56, 228—235).—(A) For the combined determination trichloroacetic acid is added, to a concentration of 4%, to the filtrate after removal of the casein (cf. B., 1931, 512), the mixture is heated for  $\frac{1}{2}$  hr. at the b.p., filtered, and the precipitate washed with 1% trichloroacetic acid. (B) After separation of the casein the filtrate may be neutralised to phenolphthalein and saturated with magnesium or sodium sulphate, when the globulin is filtered off, or alternatively, the casein and globulin may be precipitated together from the milk, after neutralisation, and the nitrogen due to casein deducted to give the figure for globulin. The total protein-nitrogen minus that due to casein and globulin will give albumin-nitrogen. T. McLACHLAN.

**The diphenylamine test for nitrates in milk as a means of detecting added water, and the effect of drenching cows with "nitre."** D. R. WOOD, E. T. ILLING, and A. E. FLETCHER (Analyst, 1931, 56, 248—249).—Lerrigo's test (cf. B., 1930, 837) is capable of detecting 5% of added water containing 0.5 pt. per 100,000 of nitrogen as nitrates. Maximum doses of "nitre" fed to cows over a period of a week do not cause nitrate to occur in unadulterated milk. Filter papers may contain nitrates, and in consequence give fictitious results.

T. McLACHLAN.

**Manufacture and analysis of chocolates.** R. LECOQ (Ann. Falsif., 1931, 24, 11—22, 96—104).—Typical manufacturing formulæ and analyses of various types of chocolates are given. It is shown that reducing sugars are formed from the sucrose due to caramelisation according to the degree of heat-treatment of the chocolate in manufacture. The method of calculation from analysis of the approximate composition of chocolate, including added shell and germ, milk, starch, coffee, or nut (praline), is given.

E. B. HUGHES.

**Investigation and evaluation of honey.** K. BRAUNSDORF (Z. Unters. Lebensm., 1930, 60, 575—588).—In the author's version of Gothe's test (B., 1914, 1217; 1915, 1024), 5 c.c. of fresh 20% honey solution and 1 c.c. of a fresh, filtered 1% solution of starch are maintained for 1 hr. at 40—45°, cooled in ice, and a drop of 0.1N-iodine solution is added. A blue colour corresponds with a diastatic power of 0—1 of Gothe's scale, violet to brown-red 2.5—8.3, brown to green-brown 10.5—13.9, olive >17.9. Forty genuine or adulterated German and other honeys of known history were tested in this way and by the Ley, Lund (tannin), Auzinger, Fiehe, and Fiehe-Kordatzki tests (cf. B., 1929, 146, 375, 955). It is concluded that genuine German bee-honeys have a diastatic power of >17.9, and yet may show traces of hydroxymethylfurfuraldehyde and give a doubtful Fiehe test. A value of 10—17.9 with a hydroxymethylfurfuraldehyde reaction and a normal Lund test indicate genuine honey which has been heated, but which, according to German regulations based on the diastatic power, is suitable only for cooking purposes. Values of <10.9 indicate adulteration. Fiehe's test

(*loc. cit.*) is improved by addition to the residue from an ether extract from 10 g. of honey, of 0.2 c.c. of a 1% solution of resorcinol in hydrochloric acid (*d* 1.19). The faint red precipitate indicating a positive reaction is associated with a positive hydroxymethylfurfuraldehyde test and a diastatic power of 10—20. Apparently anomalous results may be due to unequal degrees of heating of inner and outer layers of the sample. It is suggested that genuine honeys with abnormally low diastatic powers are analogous to genuine milks low in fat.

J. GRANT.

**Determination of sand in vegetable feeding-stuffs.** I. RÜB (Chem.-Ztg., 1931, 55, 221—222).—5 G. of the air-dried sifted meal are treated with carbon tetrachloride for 1 hr. (with occasional stirring) in a Spaeth sedimentation cylinder. Foreign mineral matter detaches itself readily from the material and sinks to the bottom. The upper layer is decanted and the sediment rinsed with carbon tetrachloride into a platinum dish, evaporated, and gently ignited to burn off adherent fat before weighing. In the isolated case of rice meal the silicious epidermal hairs collect in the sediment, which must therefore be warmed (on the water-bath) with 20 c.c. of 3% caustic soda for 15 min., filtered, washed, and ignited.

E. LEWKOWITSCH.

**Blood-albumin.** H. STADLINGER (Farben-Chem., 1931, 2, 125—127, 172—174).—Details are given of the preparation of blood serum from coagulated blood and the subsequent isolation of albumin of different grades (cf. B., 1928, 158), leaving a residue of blood meal. The products are tested for colour, odour, consistency, solubility, calcium and water contents. Distinction is drawn between blood-albumin and "defibrinated dried blood," an inferior product. The chief applications of blood-albumin are as plywood adhesive, and in the leather, paper, and textile industries for various coatings etc., replacing similar products in which casein, egg-albumin, etc. are used. "Blood meal" is used for fodder and manure.

S. S. WOOLF.

**Detection of vitamin-A in medicinal food preparations.** H. MATTIS and E. NOLTE (Arch. Pharm., 1931, 269, 22—24).—Addition of approx. 0.017 g. of a lipid extract, prepared from the medicinal foods "Novo-Tropon" and "Ferripan," to a vitamin-A-free daily diet is sufficient to cure keratomalacia and restore normal growth in rats.

H. E. F. NOTTON.

**Determination of methyl alcohol.**—See III. **Preservation of green feeding-stuffs.**—See XVI.

#### PATENTS.

**Production and sterile packing of bread and pastry.** C. MEYER (B.P. 345,885, 30.5.30. Ger., 31.5.29).—Bread is baked in a metal container and transferred to a preheated receptacle without access of air. The receptacle is then sealed and again heated to ensure sterility.

E. B. HUGHES.

**Milk evaporator and deodoriser.** C. M. PETERSON, Assr. to WARD DRY MILK Co. (U.S.P. 1,778,959, 21.10.30. Appl., 28.5.26).—Milk, cream, buttermilk, etc. is forced under pressure through rotating nozzles each connected with a jacketed tube which may be heated. The liquid



is thus partly evaporated, the vapours being drawn off by means of a rotary blower. A vacuum may also be produced in the apparatus. It is claimed that deodorisation also takes place. E. B. HUGHES.

**Preservation of confectionery, fruit, and other edible substances.** A. F. MACKENZIE (B.P. 345,904, 3.7.30).—A couverture for sweetmeats contains a mixture of illipé butter, sugar and dry flour; colouring and flavouring matter, milk solids, and nuts may be added if desired. E. B. HUGHES.

**Manufacture of compositions for colouring or flavouring foodstuffs.** R. E. ELLIS. From H. KOHNSTAMM & Co., Inc. (B.P. 345,363, 30.12.29).—A mixture of approx. 20% of colouring matter, 30% of colour solvent or diffusion material such as glycerin, 48% of lactose or other sugars, and 2% of a colloidal suspension of a vegetable gum (gum arabic) in water is claimed. The solid preparation is finished in slab form. E. B. HUGHES.

**Egg products [for use in mayonnaise etc.].** A. E. WHITE. From EMULSOL CORP. (B.P. 346,031, 1.1.30).—See U.S.P. 1,744,575; B., 1930, 530.

**Ovens for the heat treatment of edibles.** A. S. JOHN (B.P. 346,479, 21.1.30).

**Drying apparatus [for milk etc.]. Centrifugal machine for milk. Filter device [for fish offal etc.]. Filtration process.**—See I. Yeast.—See XVIII. Alkaloids from cocoa. Extraction of organic substances.—See XX.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Ammonia content of cigar smoke.** D. E. HALEY, C. O. JENSEN, and O. OLSON (Plant Physiol., 1931, 6, 183—187).—Apparatus for the examination of the products from the intermittent smoking of cigars is described. In incompletely fermented tobacco samples, no relation between manuring and the ammonia content of the smoke was apparent. A. G. POLLARD.

**Determination of nicotine in solution and in insecticides.** A. SABATIÉ (Ann. Falsif., 1930, 23, 544—547).—A combination of three methods is suggested: (1) Steam-distillation of the solution made alkaline with magnesia or caustic soda and examination of each fraction of the distillate separately in a 5-dm. tube by means of a polarimeter, the proportion of nicotine being calculated by means of Biot's formula. (2) Titration with 0.1*N*-acid, using methyl-red as indicator. (3) Precipitation as the silicotungstate by Bertrand's method. The substitution of pyridine for nicotine gives results in (2) and (3) higher than in (1), and of ammonia a result in (2) higher than those in (1) and (3). E. B. HUGHES.

**Determination of morphine in opium.** C. G. VAN ARKEL and P. VAN DER WIELEN (Pharm. Weekblad, 1931, 68, 309—316).—The method of the Dutch Pharmacopœia is very unsatisfactory when gum has been added to the opium. The tartaric acid-alcohol method of van der Wielen gives much more uniform results, but when the opium content is high the results are too low.

By using larger quantities of the acid and alcohol, and by partial neutralisation with sodium hydroxide before evaporation, accurate results are obtained in all cases.

S. I. LEVY.

**Determination of morphine. II. Determination in presence of other opium alkaloids.** H. BAGGESGAARD-RASMUSSEN and F. REIMERS (Dansk Tids. Farm., 1931, 5, 21—34).—The official Danish preparation, tetrapon, which contains morphine, narcotine, codeine, and papaverine hydrochlorides, and similar mixtures (e.g., pantopon) may be analysed by a modification of the authors' method (A., 1931, 371). Tetrapon (0.4 g.) in water (20 c.c.) with 2*N*-sodium hydroxide (4 c.c.) is twice extracted with a mixture of chloroform (10 c.c.) and ether (30 c.c.) and once with chloroform (10 c.c.). The combined extracts contain the colouring matter, chryso-idine, and all the alkaloids, except morphine. Addition of ammonium chloride (1 g.) to the aqueous layer liberates the morphine, which is extracted with isopropyl alcohol-chloroform and determined as described previously. H. E. F. NOTTON.

**Essential oils of *Metrosideros*.** R. GARDNER (J.S.C.I., 1931, 50, 141—144 *t*).—The essential oils of the following species of *Metrosideros* (family *Myrtaceæ*), occurring in New Zealand, are described: *M. scandens*, *Parkinsonii*, *diffusa*, *Colensoi*, *excelsa*, *collina*, *umbellata*, *robusta*, and *perforata*. The oils in all cases contain, and in the first six species mentioned consist mainly of, a sesquiterpene similar in physical constants to cadinene and yielding cadinene dihydrochloride but not cadalene. The sesquiterpene from *M. scandens* is levorotatory but yields *d*-cadinene hydrochloride; all other species give *l*-cadinene hydrochloride. The oils of *M. umbellata*, *robusta*, and *perforata* contain also terpenes (mainly dipentene with less  $\alpha$ - and  $\beta$ -pinene) and some esters.

**Essential oil of leaves of *Chamæcyparis obtusa*, Sieb. et zucc., f. *formosana*, Hayata, or Arisan "Hinoki."** I. K. KAFUKU, T. NOZOE, and C. HATA (Bull. Chem. Soc. Japan, 1931, 6, 40—53. Cf. Uchida, B., 1928, 768).—The leaves on steam-distillation yielded 0.3% of oil having  $d_4^{25}$  0.8988,  $n_D^{25}$  1.4878,  $\alpha_D^{25}$   $-5.83^\circ$ , acid value 0.96, ester value 12.63, after acetylation 54.89. Constituents identified were  $\alpha$ -pinene, *d*-sabinene [ $\alpha$ ] $_{17}^{25}$  +86.4°, (?)  $\alpha$ -thujene, *p*-cymene,  $\alpha$ - and  $\gamma$ -terpinenes, dipentene, a substance, *chamene*, considered to be a new terpene, b.p. 86—88°/50 mm., 168—170°/760 mm.,  $d_4^{25}$  0.8228,  $n_D^{25}$  1.4686,  $\alpha_D^{25}$  +35.0°, an octenol, C<sub>8</sub>H<sub>16</sub>O,  $d_4^{30}$  0.8454,  $n_D^{30}$  1.4441,  $\alpha_D^{30}$   $-10.62^\circ$ , *d*- $\Delta^1$ -terpinen-4-ol, *l*-linalool, acetic acid, an acid, C<sub>10</sub>H<sub>16</sub>O<sub>2</sub>, b.p. 133—135°/2 mm.,  $d_4^{20}$  0.9544,  $n_D^{20}$  1.4732,  $\alpha_D^{20}$  0°, and (?) hinokic acid (cf. B., 1928, 768). *Chamene* gives a liquid hydrochloride, b.p. 80—90°/11 mm.,  $d_4^{25}$  1.0196,  $n_D^{25}$  1.4783. Treated with 30% sulphuric it gives *isochamene*, C<sub>10</sub>H<sub>16</sub>, b.p. 88—90°/50 mm.,  $d_4^{25}$  0.8222,  $n_D^{25}$  1.4726,  $\alpha_D^{25}$   $-0.27^\circ$  (which gives  $\alpha$ -terpinene nitrosite), whilst with alcoholic sulphuric acid *chamene* gives *disochamene* b.p. 155—156°/4mm.,  $d_4^{30}$  0.9150,  $n_D^{30}$  1.5134,  $\alpha_D^{30}$   $-0.7^\circ$ . It is suggested that *chamene* is a cyclopentane derivative.

A. A. LEVI.

**Colloidal aluminium hydroxide.**—See VII. **Cigar-leaf tobacco.**—See XVI. **Medicinal foods.**—See XIX. **Chloroform poisoning.**—See XXIII.

## PATENTS.

**Production of pharmaceutical preparations from plants of the genus *Allium*.** "AGLIONAT" GES.M.B.H. (B.P. 345,600, 23.7.30. Ger., 5.2.30).—Ground plants of this genus, particularly garlic, are heated with water under pressure and a current of air or steam is passed through the hot pulp to remove the odour.

E. H. SHARPLES.

**Recovery and purification of alkaloids from cocoa products.** H. E. POTTS. From MONSANTO CHEM. WORKS (B.P. 345,250, 7.10.29).—To a suspension of the cocoa products a mineral acid is added, followed by an alkaline substance (alkaline-earth oxide or hydroxide), preferably in excess of the amount necessary to neutralise the acid. After separation of the liquid, theobromine is obtained therefrom either by crystallisation, extraction, neutralisation, or by removal of the alkaline earth with carbon dioxide, with or without the addition of a soluble carbonate. It may be purified either by acidification of a filtered, aqueous solution of its alkaline-earth salt or by converting this salt into an alkali salt by addition of alkali carbonate and filtering off the precipitated alkaline-earth salt before acidification. From the extracted cocoa product and the mother-liquors an alkaloid-free product of food and fertiliser value may be obtained by neutralisation of free alkaline-earth oxide with flue gases.

E. H. SHARPLES.

**Extraction of organic substances [of high mol. wt.].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 345,249, 12.9.29).—Alkaloids, bitter principles, sugars, saponins, etc. are extracted from dried vegetable or animal materials containing such substances with anhydrous, or practically anhydrous, liquid ammonia. Extractions of tobacco leaves, quillaia bark, sugar beet, lupin or coffee beans, dry Spanish flies (*Cantharis vesicatoria*), dry milk, etc. are given as examples.

E. H. SHARPLES.

Polarimeter.—See I.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Sensitive photographic plates for the red and infra-red.** G. HAFT and W. HANLE (Z. wiss. Phot., 1931, 28, 374—376).—Five Agfa plates and one Kodak plate have been tested by photographing the spectra of a carbon arc (with a step-wedge screen) and a neon Geissler tube. The general sensitivity (over the range 4500—9000 Å.) of the Agfa plates was considerably the greater; the Kodak plate was sensitive to 8200 Å. and the Agfa plates, on the average, to 8000 Å., one plate being sensitive to 8950 Å.

J. LEWKOWITSCH.

**Formation of photographic images on cathodes of alkali-metal photoelectric cells.** A. R. OLPIN and G. R. STILWELL (J. Opt. Soc. Amer., 1931, 21, 177—181; cf. A., 1930, 1230).—When very small amounts of sensitising materials are admitted to an illuminated surface of freshly distilled metal of a photoelectric cell cathode till a maximum current is recorded, the surface develops the power of recording clear negative photographic images of the source of light. If the sensitive stage is passed, more metal may be distilled on to the surface to restore it. The image is "fixed"

by a slight excess of sensitiser, and by continued alternating treatment a positive image may form. As sensitising agents sulphur vapour, oxygen and hydrogen in 9:1 ratio, hydrofluoric acid, and bromine have been found effective. While the image is forming, the photoelectric sensitivity falls by about 30%, but recovers when the image is fixed.

J. LEWKOWITSCH.

## PATENT.

[Taking pairs of complementary colour graduation value pictures for] colour photography. W. CHAPMAN (B.P. 346,010, 1.1.30).

## XXII.—EXPLOSIVES; MATCHES.

**Velocity of phenomena produced by detonation of solid explosives.** P. LAFFITTE and M. PATRY (Compt. rend., 1931, 192, 744—746. Cf. B., 1931, 224; Payman, B., 1926, 30).—The velocities of detonation and of the consequent shock-wave and luminous gases—these latter at different distances of from 1 to 67 cm. from the column of explosive—have been determined for three densities of loading of each of No. 1 dynamite and tetryl. The results clearly show the greater power of tetryl, and how the velocity of the luminous gases falls off more quickly than that of the shock wave, the distances at which separation of the two occurs increasing with increased density of loading.

C. A. SILBERRAD.

1:3:5-Trinitrobenzene.—See III. Cotton for nitration. Nitrated cellulose.—See V.

## PATENTS.

**Production of explosives.** W. FRIEDERICH (B.P. 345,859, 30.4.30. Ger., 10.10.29).—The cyclic keto-alcohols tetramethylolcyclopentanone and -hexanone and octamethylolcyclohexanedione, and the corresponding alcohols tetramethylolcyclopentanol and -hexanol and octamethylolcyclohexanediol, when nitrated, give explosives having a velocity of detonation of 8000 m./sec. or above and of great stability, their low m.p. enabling them to be cast.

W. J. WRIGHT.

**Heating compositions for blasting cartridges.** D. HODGE and W. ESCHBACH (B.P. 345,284, 11.11.29).—In blasting cartridges containing solid or liquid carbon dioxide, a composition, which is more combustible than the heating composition, is interposed between the latter and the match head. A suitable composition consists of a mixture of lead thiocyanate and potassium perchlorate.

W. J. WRIGHT.

Pulp for explosives.—See V.

## XXIII.—SANITATION; WATER PURIFICATION.

**Mechanism of the activated sludge process of sewage disposal.** E. C. C. BALY (J.S.C.I., 1931, 50, 22—26 T).—The isoelectric point of the colloids in sewage has been determined by the methods of flocculation and cataphoresis. In the absence of electrolytes the isoelectric point is at  $p_H$  4.6, in the presence of 0.17% NaCl it is at  $p_H$  6.5, and in the presence of 3% NaCl it is at  $p_H$  8.3. The most attractive theory of the activated sludge process—namely, the mutual coagulation of colloids of opposite charge—would thus seem to be negatived, since in fresh sewage with  $p_H$  7.4 the colloids

are electronegative as well as the activated sludge particles. No attention, however, has been paid to the electronegative charge on the bacteria and its variation with their activity. When 3% NaCl is added to fresh sewage and the mixture is fully aerated, the bacteria are flocculated by the electropositive colloids within four days. In fresh sewage containing 3% NaCl there exist two types of particle, namely, colloid particles with a larger electropositive charge and bacteria-colloid complexes with a smaller electropositive charge. The essential condition for the successful operation of the activated sludge process is the maximum bacterial activity of the sludge particles. This causes these particles to develop an increased electronegative charge, and it is suggested that the activated sludge process consists in the mutual coagulation of the bacteria-colloid complexes by the activated sludge particles, caused by the difference in charge of the two. It follows that an improvement would be effected by operating the process at  $p_H$  5.8–6, when the whole of the colloids would be electropositive. Considerable benefit would be gained in the preparation of the first quantity of activated sludge by adding 3% NaCl to the sewage, followed by aeration for 4 days, or, still better, by the addition of a cream of fine silt or clay to the saline sewage, followed by aeration for one hour.

**Standard methods for the examination of sewage and sewage sludge.** Symposium. I. J. J. HINMAN, JUN. II. S. E. COBURN. III. A. J. FISCHER. IV. L. H. ENSLOW. V. W. RUDOLFS and H. HEUKELEKIAN. VI. M. M. COHEN. VII. T. C. SCHAETZLE. VIII. E. J. THERIAULT. IX. C. C. HOMMON. X. C. K. CALVERT. XI. W. D. HATFIELD. XII. F. W. MOHLMAN. XIII. A. M. BUSWELL and G. E. SYMONS. XIV. M. LEVINE. XV. W. S. MAHLIE (*Sewage Works' J.*, 1930, 3, 349–352, 353, 353–356, 356–357, 357–360, 360–363, 363–365, 365–367, 367–368, 368–370, 370–374, 374–378, 378–384, 384, 385–386).—III. A method for the determination of putrescible solids in grit samples is described.

VIII. In the determination of dissolved oxygen the use of permanganate is unnecessary in the absence of nitrites or iron. In presence of much organic matter permanganate gives lower results than Winkler's method. Permanganate gives untrustworthy results in sulphite waste liquors. More accurate results are obtained by using chlorine or hypochlorites, the excess of oxidant being removed with iodine. For the determination of biochemical oxygen demand buffered water is recommended.

XIII. A method for determining the quantity of settling solids is described.

XIV. A method for the determination of nitrate in sewage is described.

CHEMICAL ABSTRACTS.

**Function of ripe [sewage] sludge.** H. HEUKELEKIAN (*Sewage Works' J.*, 1930, 3, 313–337).—The age of the sludge affects the rate of decomposition and the gasification of the solids added to it; decomposition should be recently complete. Regulation of  $p_H$  with ammonium hydrogen carbonate and carbonate and potassium hydrogen phosphate compares favourably with maintenance of  $p_H$  by repeated addition of lime.

CHEMICAL ABSTRACTS.

**Rôle of protozoa in [sewage purification by] activated sludge.** R. CRAMER (*Ind. Eng. Chem.*, 1931, 23, 309–313).—In presence of sodium chlorate (0.3%) sewage clarifies in 2–4 days, producing sludge which is indistinguishable from well-conditioned activated sludge and a supernatant liquor free from suspended matter and bacteria. This fact was used as the basis of parallel laboratory experiments which showed that the essential factors in the clarification of sewage by the activated-sludge process are the presence of live protozoa, oxygen in solution, and aerobic bacterial life, and that the rate of clarification is considerably improved by increasing the temperature from 10° to 20°. C. JEPSON.

**Influence of seeding material on [sewage] sludge digestion.** C. E. KEEFER and H. KRATZ, JUN. (*Eng. News-Rec.*, 1931, 106, 474–478).—Seeding sludges, varying in age from 10 days to 8 years, were given daily additions of fresh sludge in amounts equivalent to 2% and 6% of their dry matter content. As measured by the rate of gas evolution, a seeding sludge about 10 weeks old in which digestion was just commencing gave the best result in the early stages, but after 60 days' operation all sludges were giving similar results except the freshest one, which did not commence to digest till after 80 days. A rapid resolution of ether-soluble matter was observed in the first 10 days, but there was also a portion of this material which was only digested with difficulty. Approximately 600 c.c. of methane or 800 c.c. of total gas were obtained per g. of fresh organic matter added or 750–800 c.c. of methane and 1200–1300 c.c. of total gas per g. of organic matter destroyed. C. JEPSON.

**Reclamation of treated sewage.** R. F. GOUDEY (*J. Amer. Water Works' Assoc.*, 1931, 23, 230–240).—In Southern California the lowering of the general level of the ground water by excessive pumping for potable and industrial purposes is causing serious infiltration of sea-water. By suitable treatment it is thought that reclaimed sewage may be used to meet such deficiencies and for many industrial purposes. An experimental plant has been installed at Los Angeles to investigate the possibility of the reclamation of sewage, to determine costs of installation and operation, and to work out refinements in the methods adopted. In this plant the effluent from an activated-sludge plant treating preclarified sewage is superchlorinated to remove phenols and odours, coagulated with ferric chloride, filtered through sand, and finally through activated charcoal before disposal on sand beds. Tests in and around the disposal area show that the 200,000 gals. reclaimed daily have no deleterious effect on the condition of the ground water, and chemical tests indicate that it is definitely superior thereto. C. JEPSON.

**Valuation of carbolic powder.** C. E. COULTHARD (*Analyst*, 1931, 56, 251).—The usual specification, that powders must contain not less than 15% of "cresylic acids," should be amended, since many powders, e.g., those containing spent gas-lime, may be more efficient, but are condemned under the present regulations. T. McLACHLAN.

**Testing of disinfectants.** E. K. IDEAL and A. SCIVER (*Analyst*, 1931, 56, 249–250).—A criticism of the technique of the Patterson-Frederick (cf. B., 1931,

419) and of the Martin-Chick tests. The authors claim an accuracy of  $\pm 5\%$  for the Rideal-Walker test, and that it is equal, or superior, to any other method suggested. T. MCLACHLAN.

**Elimination of taste and odour in the water supply of Lancaster, Pa.** E. D. RUTH (J. Amer. Water Works' Assoc., 1931, 23, 396—399).—Prior to April, 1928, the inadequacy of the purification plant treating water from the Conestoga River was responsible for very offensive tastes and the continuous presence of at least an odour of chlorine in the finished water. In that year the feeding of anhydrous ammonia (0.18 p.p.m.) to the raw water by means of a chlorinating machine enabled the post-chlorine dosage to be reduced from 0.45—3.0 p.p.m. to 0.18 p.p.m., with improved bacterial removal. Prechlorination was shown to have no beneficial result. Excessive vegetable growths in summer and autumn were destroyed by copper sulphate (maximum dose 1.0 p.p.m.), which was added with the coagulant, and the dead organic matter was oxidised by potassium permanganate (0.63—5.0 p.p.m.) during autumn to prevent the clogging of the sand filters. C. JEPSON.

**Superchlorination treatment [of water] for taste prevention at Toronto, Ontario.** N. J. HOWARD (J. Amer. Water Works' Assoc., 1931, 23, 387—395).—The difficulties encountered and overcome during the experimental stages of chlorination at Toronto are described. Since 1927 the whole of the supply has been treated at a cost of \$1.81/million gals., of which \$0.72 is due to the cost of superchlorination and dechlorination during periods of probable taste. In this case pollution is accompanied by a high ammonia content, and is subject to rapid variations, hence strict control is necessary to ensure an adequate residual of chlorine. The cost of treatment has been reduced by the use of 1-ton containers of chlorine, and the possibility of a further saving by a reduction in chlorine dosage and subsequent treatment with activated charcoal is being considered. Attention is drawn to the necessity for the study of the effect of temperature, degree and nature of pollution, and the time of contact for each individual water, to secure the best result. C. JEPSON.

**Preammoniation of the filtered water supply of Cleveland, Ohio.** J. W. ELLMS (J. Amer. Water Works' Assoc., 1931, 23, 400—407).—Preammoniation was recommended for the prevention of chlorophenolic tastes in water drawn from Lake Erie in preference to treatment by activated carbon. Anhydrous ammonia (0.096—0.2 p.p.m.) is added to the filtered water before chlorinating, and has successfully prevented objectionable tastes when up to 1.0 p.p.m. of phenol has been present. The cost of treatment is \$0.30 per million gals., and no adverse effect on the bacterial efficiency of the plant has been observed. C. JEPSON.

**Successful superchlorination and dechlorination for medicinal taste of a well supply, Jamaica, N.Y.** F. E. HALE (J. Amer. Water Works' Assoc., 1931, 23, 373—386).—Offensive tastes, due to the effect of chlorine on products introduced into a portion of the ground water by a leakage of petrol at a garage situated  $\frac{3}{4}$  mile away from the nearest supply point,

were prevented by superchlorination, and subsequent dechlorination by sulphur dioxide. The treatment enabled the whole supply to be used for potable purposes and increased the available normal volume by 40% and the peak-load volume by 80%, the cost of chemicals being 92 cents per million gals. C. JEPSON.

**Softening a well-water supply.** N. T. VEATCH, JUN., and B. L. ULRICH (J. Amer. Water Works' Assoc., 1931, 23, 272—275).—Well water at Manhattan, Kansas, with a total hardness of 463 p.p.m., of which only 15 p.p.m. are non-carbonate hardness, and an iron content of 12 p.p.m. is freed from iron by aeration, softened with lime, and then recarbonated with carbon dioxide obtained by the combustion of natural gas; the final water has a total hardness of 124 p.p.m. C. JEPSON.

**Boiler feed-water.**—See I.

#### PATENTS.

**Treatment of sewage sludges.** H. G. GILLILAND (B.P. 345,194, 17.9.29).—Sludge of high organic matter content, obtained by the action of yeast on activated or precipitation tank sludges, is mixed with powdered carbonaceous material, an acid or alkali, and an oxide, hydroxide, or salt of calcium. The mixture is agitated at 26—93°, filtered while hot, and the resultant cake dried and crushed for use as a fertiliser. C. JEPSON.

**Appliances for purifying [removing water vapour and carbon monoxide from] respirable gases.** L. A. LEVY, D. W. WEST, and R. H. DAVIS (B.P. 345,672, 21.12.29 and 30.6.30).—The impure air is drawn through a canister containing successive layers of a dehydrating agent, e.g., silica gel, of material for indicating by a colour change (observable through a window) when the dehydrating agent is exhausted, e.g., pumice granules impregnated with an anhydrous cobalt or copper salt, and of material for the catalytic oxidation of the carbon monoxide. L. A. COLES.

**Removal of chlorine or hypochlorite compounds from water.** E. BERL (B.P. 344,363, 3.12.29).—The water is treated with lignin or material containing it, e.g., wood which preferably has been treated with steam and partly freed from resin; when the chlorine content of the water is high, the latter should be treated with coke, fuller's earth, silica, etc. to remove any lignin chloride formed. L. A. COLES.

**Composition for repelling insects.** STANDARD OIL DEVELOPMENT Co., Asses. of D. H. GRANT (B.P. 345,993, 28.12.29. U.S., 29.12.28).—See U.S.P. 1,755,178; B., 1931, 46.

**Apparatus for adjusting and regulating the purifying and softening of water.** N.V. NECKAR WATERREINIGER MAATS., and A. H. M. TROUSSELOT (B.P. 346,134, 18.3.30).

**Apparatus for softening water.** C. P. EISENHAEUER (B.P. 346,287, 4.1.30. U.S., 11.3.29).

**[Pressure filter for] filtration of water.** FILTRATION & WATER SOFTENING PROPRIETARY, LTD. (B.P. 346,376, 28.10.29. Austral., 29.10.28).

**Filtration process. Dialysing liquids. Vaporisation of liquids.**—See I. **Fumigation. Gas-protection appliances.**—See VII