# BRITISH CHEMICAL ABSTRACTS

B.-APPLIED CHEMISTRY

SEPT. 30 and OCT. 7, 1932.\*

# I.—GENERAL; PLANT; MACHINERY.

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Forced-draught heating in air heaters. A. I. MILLER (Domez, 1931, No. 7, 48–67).—The efficiency of the stove is increased by using a smaller chequerbrick spacing. CH. ABS.

Optical method for measuring temperature distribution and convective heat transfer. R. B. KENNARD (Bur. Stand. J. Res., 1932, 8, 787-805).— The temp. in the region of heated surfaces (verticalplate, horizontal cylinders) have been determined by measuring (with photographs) the displacement of fringes in an interferometer. The temp. distributions do not conform to the requirements of the film theory.

Portable Junker calorimeter. W. WUNSCH and H. SEEBAUM (Gas- u. Wasserfach, 1932, 75, 644-647).-Unlike the ordinary Junker calorimeter, this instrument works discontinuously. It consists of upper and lower H<sub>2</sub>O reservoirs and a gas reservoir. The latter is initially filled with H<sub>2</sub>O, the latter displaced with gas, and the H<sub>2</sub>O returned to the top reservoir. It is then allowed to flow from this through the calorimeter proper into the gas chamber while the displaced gas is burned. Temp. readings are taken as usual. The same H<sub>2</sub>O is retained so that it is always saturated with gas. The vols. of gas and H<sub>2</sub>O are the same. A fixed correction is made for radiation etc. Comparative tests with the standard instrument on H2 and town's gas show an average variation of about 1%. Variations in room temp. between 11° and 24° affect the results very little.

Determination of alkalinity in boiler waters. A comparison of methods. F. G. STRAUB (Ind. Eng. Chem. [Anal.], 1932, 4, 290-294).-Comparative determinations on boiler concentrates and synthetic solutions indicate that (1) the A.P.H.A. and Winkler methods give reliable vals. for OH' in absence of much SiO<sub>2</sub> or org. matter. The Winkler method is the more trustworthy in presence of org. matter; (2) determination of OH' from  $p_{\rm H}$  vals. leads to erroneous results; (3) CO<sub>3</sub>" determined by the A.P.H.A. method is accurate when org. matter is absent, but is not reliable when the  $Na_2CO_3$  content is < 50 p.p.m.; (4) determination of CO3" by the short equilibrium method gives results close to those obtained by the A.P.H.A. method; (5)  $CO_3''$  is determined with greatest accuracy by the E. S. H.  $CO_2$  evolution method.

Determination of hydroxide and carbonate in boiler waters. I. Methods. II. Apparatus for carbonate determination by evolution and absorption in barium hydroxide. E. P. PARTRIDGE and W. C. SCHROEDER. III. Plant tests of various methods of determining ratios for boiler-water control. L. F. Collins and W. C. SCHROEDER (Ind. Eng. Chem. [Anal.], 1932, 4, 271–273, 274–278, 278–283).—I. Available methods are reviewed.

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II. Apparatus and procedure are described, which give accurate results for total  $CO_2$  even at very low concess. Titration in presence of phenolphthalein and Me-orange or by the Winkler BaCl<sub>2</sub> method gives sufficiently accurate results for OH' in boiler H<sub>2</sub>O, but not for  $CO_3''$ .

III. A Cu-screen filter is described for the purpose of obtaining filtered samples of boiler  $H_2O$  at the boiler temp. More accurate results are thus obtained than with the cooled sample. Standard methods for determining  $CO_3''$ , OH', and  $SO_4''$  in boiler  $H_2O$  are compared and sources of inaccuracy indicated. E. S. H.

Cementing constituents of boiler scale, especially silicates. F. HUNDESHAGEN (Chem.-Ztg., 1932, 56, 521-524, 542-544).—The constituents of boiler feed H<sub>2</sub>O which cause the formation of hard crusts are CaSO<sub>4</sub>,2H<sub>2</sub>O, CaSO<sub>4</sub>,0.5H<sub>2</sub>O, Ca(OH)<sub>2</sub>, Mg(OH)<sub>2</sub>, and colloidal SiO<sub>2</sub>. The removal of these substances from the H<sub>2</sub>O is discussed with reference to recent literature, and some experiments on the removal of SiO<sub>2</sub> are described. Freshly pptd. Mg(OH)<sub>2</sub>, especially in the presence of alkali, is more effective than Ca(OH)<sub>2</sub> for the pptn. of SiO<sub>2</sub>. Softening with Na<sub>3</sub>PO<sub>4</sub> should be used only for the removal of the last traces of Ca and Mg after pptn. of the greater part of these elements as CaCO<sub>3</sub> and Mg(OH)<sub>2</sub>, respectively. Phosphate treatment of zeolite-softened H<sub>2</sub>O is unnecessary. A. R. P.

Effect of the removal of gases from steamheated apparatus on steam consumption and capacity. E. KIRSCHBAUM (Chem. Fabr., 1932, 281-283, 290-292).-The relation between steam loss, gas content in steam, and gas content in gas removed fromthe heater is shown by curves. Loss in heat-exchange capacity arises from the reduction of the partial pressure of steam in the steam-gas mixture below the total pressure, and curves show the relation of this loss to the gas content of the withdrawn gas. It is therefore possible to balance the steam loss against the capacity loss, as required in any particular case, by regulating the composition of the withdrawn steam-gas mixture. The condensation temp. for a given total pressure falls with gas content and therefore varies through the heating system to a max. at the point of gas withdrawal if the latter is placed correctly. The temp. of the condensate removed as against the theoretical temp. calc. from the pressure gives an indication as to whether gas removal is satisfactory. Of gases found in steam, NH<sub>3</sub> is lighter than the former, but air, CO<sub>2</sub>, and H<sub>2</sub>S

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

are heavier. The highest point of the vessel is not necessarily the right place for the gas release. The gas concn. is greatest and the temp. lowest at the actual heating surface. The point is discussed in detail and it is concluded that the gas exit should be as far as possible from the steam inlet and that the steam should be directed at right angles to the tubes of the heater.

C. I.

Conditions under which fine-grained solid materials and water can be compressed into blocks. HULLEN (Braunkohle, 1931, 30, 1069–1076; Chem. Zentr., 1932, i; 1317–1318).—The surface tension of the H<sub>2</sub>O present (e.g., in brown coal) is responsible for the briquette formation; the solidity depends on the H<sub>2</sub>O content, fineness of the pores, and the form and arrangement of the individual particles. On the basis of cubic and spherical particles the solidity of material of various H<sub>2</sub>O content has been calc.; the results accord with those encountered in practice. The rôle of air and other gases and the effect of grain size are discussed. A. A. E.

Selective fractionation. C. B. FAUGHT (Refiner Nat. Gas. Mfr., 1932, 11, No. 4, 272).—Specification fractions are obtained directly from a tower designed so that a portion of the vapours from each tray is used to wash down the tower. The continuous washing decreases polymerisation. CH. Abs.

Mixing and dilution calculations. H. MENZEL (Chem. Fabr., 1932, 289—290).—m kg. of solution of  $p_1\%$  concn. are mixed with n kg. of  $p_2\%$  concn. to give (m+n) kg. of  $p_3\%$  concn. Then  $mp_1 + np_2 =$  $(m+n)p_3$ . Vals. for m, n are plotted horizontally and for  $p_1$ ,  $p_2$ ,  $p_3$  vertically, so that the products in the equation are represented by rectangles. The diagram affords a ready means for solving for one unknown, and in a simplified form covers the case of dilution  $(p_2 = 0)$ . C. I.

**Preventatives of freezing for automobile radiators.** TYPKE (Petroleum, 1932, 28, Motorenbetr., 5, No. 6, 6—7).—The f.p. of aq. solutions (of various concns.) of EtOH, glycerin, Dixol (approx. 82% glycerin, coloured red), and Glysantin (tech. glycol) are compared in conjunction with those of pure alcohols. Economic considerations are advanced to show that D.A.B. VI glycerin (87% tech. glycerol) is the most convenient preventative of freezing of cooling-H<sub>2</sub>O. H. E. B.

Determination of the degree of cleanliness of bottles and other containers. E. VOLLHASE (Pharm. Zentr., 1932, 73, 497—503).—The bottles are rinsed with hot distilled  $H_2O$ , and the washings titrated with 0.01N-KMnO<sub>4</sub>. Visible impurities cause much reduction. The results are less accurate with bottles containing < 50 c.c. Allowance must be made for the reducing power of the  $H_2O$  used. R. S. C.

Removal of oil, light petroleum, and benzene residues from condenser water by active charcoal. M. JAENICKE (Chem.-Ztg., 1932, 56, 630-632).

Turbine blowers for coke ovens. Fractionating equipment.—See II. Colour analyser.—See VI. Ammoniates for refrigerators.—See VII. Turbidity measurements.—See XX.

See also A., Aug., 814, Passivity of Fe [in boilers].

# PATENTS.

Dryer. L. G. ROWAND and J. S. PELLETT, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,835,530, 8.12.31. Appl., 3.6.29).—The material is caught in cells formed both inside and outside the rims of wheels of which there are a no. arranged in intercalating tiers in a tower. B. M. V.

Drying. G. C. OXER, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,834,497, 1.12.31. Appl., 7.4.23).— Drying gases are passed in horizontal zig-zags between grates which are composed of dumping bars, alternate grates being operated simultaneously. B. M. V.

Drying of materials by spraying. K. LÖFFL (B.P. 376,288, 27.3.31).—The material is sprayed into a primary stream of drying gas and, without allowing any deposition in the dryer, the partly dried material passes into a secondary drying gas. The latter may be re-used without condensation and the primary gas separated from fine dust in a cyclone and re-used after condensation. B. M. V.

Effecting [exothermic] synthesis. S. K. VARNES, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,835,827, 8.12.31. Appl., 18.11.25).—The catalyst-cooling conduit, carrying the incoming gases, is formed into a series of flat coils, the no. of turns in each one being proportional to the amount of heat to be removed from its locality. B. M. V.

Catalytic apparatus. I. HECKENBLEIKNER and N. TITLESTAD, ASSTS. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,836,166, 15.12.31. Appl., 9.2.29).—In exothermic reactions the catalyst is contained in a no. of tubes the outsides of which are cooled by the incoming gases deflected first downwards, then upwards in contact with them, by means of bells which are adjustable in height so that the dead spaces around the lower parts of the tubes may be varied according to the amount of catalyst it is desired not to cool. B. M. V.

Process for performing endothermic reactions in blast [sintering] apparatus. METALLGES. A.-G. (B.P. 375,950, 19.5.31. Ger., 19.5.30).—The charge is diluted with returned sinter or other inert matter in order to keep the fuel content down to a suitable proportion; by this means CaO may be burned and Portland cement clinkered in an apparatus of the Dwight-Lloyd type. B. M. V.

Means of promoting chemical reactions. H. FRISCHER (U.S.P. 1,834,693, 1.12.31. Appl., 11.6.27. Ger., 4.12.26).—A stationary, horizontal drum is heated by an axial flue or steam conduit around (or with) which is rotated a series of spiders between the spokes of which are loosely lodged a no. of heavy metal bars to break up scale on the heating surfaces; contact with the wall of the drum is prevented by rings attached to the spiders. B. M. V.

Chemical heating composition and receptacle for use therewith. S. L. GOLDBERG (B.P. 376,622, 28.11.31).—The composition consists of Fe particles 84,  $MnO_2$  10, and NaCl 6%. It is contained in a canvas bag and then in a watertight envelope. B. M. V.

Pyrometric device. L. de FLOREZ (U.S.P. 1,837,853, 22.12.31. Appl., 19.10.27).—An odd no. of thermocouples

in the zone to be controlled and a cold junction are connected in series. The thermocouples are divided into hot junctions and intermediate junctions, the latter being connected in opposition to the former and one less in no., and embedded in insulating material causing a delay at any change of temp. The effect of the combination is that the indication of any change from the standard conditions is temporarily much exaggerated unless the change is very slow. B. M. V.

Foam stabiliser [for fire extinguishers]. T. J. FAIRLEY, ASST. to W. J. and M. P. HUNTER (U.S.P. 1,832,109, 17.11.31. Appl., 21.5.28).—Acetates, particularly Fe(OAc)<sub>2</sub>, are claimed. A. R. P.

Foam abatement and chemical recovery. W. T. WEBSTER and C. L. WAGNER (U.S.P. 1,836,563, 15.12.31. Appl., 25.3.30).—The foam is admitted tangentially into a container supplied with highly heated gases. After mixing with the gases, the remaining foam travels upwards among conical baffles and, if desired, tower filling, the process being aided by horizontal and downwardly directed sprays. B. M. V.

**Refrigeration oil separation.** G. W. WATTS, ASST. to STANDARD OIL CO. (INDIANA) (U.S.P. 1,830,863, 10.11.31. Appl., 25.9.30).—In a refrigeration system, oil which may be entrained in  $NH_3$  leaving a booster reciprocating compressor is removed by scrubbing with a suitable liquid, preferably dil. aq.  $NH_3$ . D. K. M.

Apparatus for cooling oils or other liquids. H. W. ELLIS and R. W. BOTTOMLEY (B.P. 374,941, 18.3.31).—The liquid (e.g., lubricating oil) to be cooled flows in a thin (e.g., about  $\frac{1}{24}$ -in.) layer between the periphery of a water-cooled revolving drum and the inner surface of a water-cooled fixed cylindrical casing. The casing and drum are provided with scrapers to remove oil from the relatively moving surfaces. H. E. B.

Prevention of corrosion in apparatus containing circulating liquid applicable to absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEB. (B.P. 376,013 and Addn. B.P. 376,113, [A] 8.7.31, [B] 9.10.31, Ger., [A] 8.7.30, [B] 20.10.30).—In (A) a reserve supply of corrosion-preventing compound, e.g., a chromate in the case of liquid  $NH_3$ , is placed in a pocket communicating with the circulating system by one conduit only, so that it dissolves by diffusion. In (B) the reserve supply has a different base from that originally in solution, e.g.,  $NH_4$  and Na, respectively. B. M. V.

Heat-insulating filler. N. ISHIDA (U.S.P. 1,832,710, 17.11.31. Appl., 4.1.28).—The space between the walls of a refrigerator is filled with a plate made from the plastic mass produced by boiling a 50:50 mixture of paraffin wax and waste cotton or wool until the capillaries of the latter are filled with the wax. A. R. P.

Crushing mill. G. VILLA (U.S.P. 1,835,192, 8.12.31. 18.4.29).—Rolls, for crushing, *e.g.*, sugar cane, are provided with stationary shafts and dust- and moistureproof bearings within the rolls. B. M. V.

Pulveriser and separator. G. H. FRASER (U.S.P. 1,834,094, 1.12.31. Appl., 12.9.24. Renewed 25.9.30).— 80 claims are made for various features of a pulveriser of the horizontal ring and roll type with air separator above. B. M. V.

Mixing of materials. V. and W. BOOTH (B.P. 376,601, 5.11.31).—The mixing vessel is closed when charged and rotated about both vertical and horizontal axes, the latter being near one end of the vessel.

B. M. V.

Mixing apparatus. W. W. TRIGGS. From MASCH-INENFABR. AUGSBURG-NÜRNBERG A.-G. (B.P. 376,684, 10.3.32).—In a vessel of liquid is provided a vertical, rotating shaft carrying upper impellors producing a circulation up the centre and down the sides, and a lower impellor causing a strong current along the bottom of the vessel from the centre outwards.

B. M. V.

Thermostatic mixing valves.HALL ELECTRICHEATING Co., INC. (B.P. 375,910, 14.4.31.U.S., 15.4.30).--A device suitable for incorporation in an ordinaryT-piece is described.B. M. V.

Screening apparatus. W. Ross (B.P. 376,273, 26.2.31).—Screening rolls are described. B. M. V.

Apparatus for separating solids from liquids in which said solids are suspended. W. M. BALDWIN (U.S.P. 1,837,191, 22.12.31. Appl., 22.1.30).—To a funnel-shaped or cylindrical vessel having means for withdrawal of sludge at the bottom and an overflow at the top, the material is admitted through an axial pipe having a rotating foot producing gentle swirling.

B. M. V.

Separation of colloidal and other suspensions from liquids. J. STONE & Co., LTD., and A. W. EMPSON (B.P. 374,915, 9.3. and 1.9.31).—The process, applicable, e.g., to the reconditioning of transformer and lubricating oils, comprises fine atomisation and agitation of a cleansing liquid in the dirty liquid without the employment of even small quantities of assisting solutions, the cleaning liquid being, in the examples named, plain H<sub>2</sub>O. The coagulation and separation may be effected in a single centrifugal apparatus, e.g., that described in B.P. 282,466 (B., 1928, 175). B. M. V.

Apparatus for straining liquids or gases. VICKERS-ARMSTRONGS, LTD., and J. WARDLE (B.P. 376,574, 14.10.31).—The filter comprises a wire wound on a fluted mandrel. B. M. V.

Continuous filter. J. A. McCaskell (U.S.P. 1,835,796, 8.12.31. Appl., 3.5.27).—A rotary leaf filter operating under pressure discharges its cake into a sideways extension of the casing, from the bottom of which it is removed by a worm conveyor pushing the material past a conical valve which is pressed towards the closed position by fluid pressure taken from the main casing. B. M. V.

Filters. J. S. WITHERS. From NEW ENGLAND FILTER CORP. (B.P. 376,680, 29.2.32).—A filter for, e.g., tap water comprises a no. of discs held against seatings by springs followed by a fibrous mass in a separate container. They are cleaned by reverse current. B. M. V.

Filter. A. S. HARRINGTON (U.S.P. 1,836,920, 15.12.31. Appl., 8.2.29).—The filter comprises a centrifugal basket enclosed in a casing so that pump pressure may be added to centrifugal force. The prefilt is

maintained in quiet condition upon the filter surface by means of an inner perforated drum. B. M. V.

Centrifugal bowls. AKTIEB. SEPARATOR (B.P. 376,196, 4.2.32. Swed., 9.2.31).—In a centrifuge of the airtight type having axial discharge of the lighter fluid, means are provided to destroy excess angular velocity of that fluid as it approaches the axis.

B. M. V.

Bowls for centrifugal sludge separators. AKTIEB. SEPARATOR (B.P. 376,123, 20.10.31. Swed., 20.10.30). —A centrifuge utilising an assisting liquid for the discharge of sludge is so arranged that any excess of the former is discharged with the heavier of the two liquid constituents. B. M. V.

Leaching and treating apparatus. L. C. KARRICK and D. GOULD (U.S.P. 1,835,878, 8.12.31. Appl., 6.1.25).—Divided material is allowed to fall through a rising stream of solvent; above the leaching zone the conduit or tower expands, forming a settling zone of lower velocity, and above that is a filter through which the solution is withdrawn for further treatment, *e.g.*, evaporation or pptn. The solids are withdrawn from the bottom of the tower and lifted to above liquid level before discharge. B. M. V.

Apparatus for purifying liquids. C. J. RODMAN and R. P. DUNMIRE, Assrs. to BUCKEYE TWIST DRILL Co. (U.S.P. 1,836,338, 15.12.31. Appl., 30,4.29).—The liquid is sprayed upwards under high pressure into a space maintained under vac., being guided upwards by an inverted conical baffle to impinge on baffles depending from the roof. The purified liquid falls through the annular space surrounding the cone, tumbling over other baffles as it falls. B. M. V.

**Evaporation of liquids.** INDUSTRIKEMISKA AKTIEB. (B.P. 376,430, 21.5.31. Swed., 22.5.30).—Evaporation of a heated liquid in countercurrent contact with a cold gas-vapour mixture is effected in at least two stages; the ratio of the quantity of liquid to the quantity of gas is greater when the temp. of the gas is rising than when it is falling. B. M. V.

Evaporation of sea-water or other liquids from which water or other liquid is to be removed. J. O. NYGAARD (B.P. 376,619, 26.11.31).—The liquid is heated while under a pressure > the v.p. of the liquid at the temp. of the heat-transmitting wall, whereby even incipient bubbles and consequent crystals are prevented from forming on the heating surface. Evaporation is effected on release of the pressure.

B. M. V.

Apparatus for maintaining a constant flow of variable liquid residue from vaporisers. W. A. PETERS, jun., Assr. to E. B. BADGER & SONS Co. (U.S.P. 1,837,834, 22.12.31. Appl., 19.4.27).—A preliminary vaporiser (A) is arranged abreast of a main vaporiser (B), the latter being situated in the base of a rectifier. Cross-connexions for both vapour and bottoms are provided between A and B, so that, although the circulating pump usually withdraws bottoms from A and delivers them, after reheating, to B, any deficiency can be made up from B.

Syrup evaporator. W. B. MARTIN and P. S. EVANS, Assrs. to MOBILE STEEL Co. (U.S.P. 1,835,606, 8.12.31. Appl., 8.8.29).—A direct-heated evaporating pan constructed of rolled steel is described. The liquor is caused to flow in a zig-zag manner over the bottom. B. M. V.

Vacuum pans. A. L. WEBRE, Assr. to UNITED STATES PIPE & FOUNDRY Co. (U.S.P. 1,835,250 and 1,835,620—1, 8.12.31. Appl., 28.5.30).—Evaporators each having a steam heater in the form of a belt of vertical tubes for the upflow of liquid and a large axial space for downflow are described. In (A) the heater serves also as a cooler by partially filling it with  $H_2O$  and subjecting it to a progressive vac. In (B) upper and lower mixing propellers are provided. In (c) the const.-speed motor is provided with an ammeter which serves also as a viscosimeter. B. M. V.

Selective crystallisation. W. E. BURKE, C. F. RITCHIE, and R. B. PEET, Assrs. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,835,271, 8.12.31. Appl., 3.3.30).—A solution of two or more salts is brought to a metastable supersaturated condition of both principal salts and is passed upwards through a bed of comparatively large seed crystals of one of the salts, e.g., Na<sub>3</sub>PO<sub>4</sub>, NaBO<sub>2</sub>, while the other salt, e.g., borax, forms fine crystals which are borne away in the current of mother-liquor and are separated elsewhere.

B. M. V.

Fractionating tower. J. B. WEAVER (U.S.P. 1,835,683, 8.12.31. Appl., 17.12.27).—A tower is divided by vertical partitions into a no, of substantially independent units. B. M. V.

Diffusion apparatus. McG. CLINE, ASST. to WOOD PROCESS CO., INC. (U.S.P. 1,834,577, 1.12.31. Appl., 17.6.26. Renewed 21.12.28).—A strictly horizontal drum is divided into compartments by vertical partitions having central apertures through which passes a continuous helix rotated independently of the drum to advance the material. The liquid is advanced in the opposite direction by the presence of overflow openings in the partitions, arranged at radii from the axis which increase in the desired direction of flow. B. M. V.

Apparatus for use in the [spectrum] analysis of volatile liquids. V. HENRI, ASST. to URBAIN CORP. (U.S.P. 1,835,985, 8.12.31. Appl., 7.6.29. Fr., 11.6.28).— A tube with transparent ends is provided with a side branch; both branches are surrounded by electric heaters, the main tube being maintained at a temp. above condensation and the temp. of the side branch being gradually raised to volatilise different constituents in succession. B. M. V.

Liquid and gas separator. C. O. LEE, Assr. to FREEPORT SULPHUR Co. (U.S.P. 1,834,065, 1.12.31. Appl., 1.11.29).—A device for the reception of the broken column of gas and S emerging from the mine pump is described; it embodies a drainable seal of liquid S. B. M. V.

Steam or gas dryer and separator or a gas and water heating apparatus. T. G. THOMAS (B.P. 375,811, 31.3.31).—A device on the deflection principle for insertion in the steam space of a boiler is described. B. M. V.

Appliances for centrifugally purifying steam, gases, and vapours. W. ALEXANDER (B.P. 376,555,

24.9.31).—In a cyclone separator an upper forced vortex, produced by the tangential inlet of the gas, is separated from a free vortex by a conical baffle; another conical baffle guards the cleaned gas outlet from the dust-collecting zone in the conical bottom.

B. M. V.

**Removing oxygen from steam.** A. B. LAMB (U.S.P. 1,832,401, 17.11.31. Appl., 5.8.25).—Steam, preferably generated from an electric boiler, is passed through a vessel containing the product obtained by the action of steam on reduced granular magnetite. This substance may be replaced by Cu, Ni, or oxides of these metals, and small quantities of  $H_2$  may be added to the steam to prevent complete oxidation of the catalyst.

A. R. P.

Filtering [dust from a] blow chamber. E. R. POWELL, ASST. to BANNER ROCK CORP. (U.S.P. 1,837,836, 22.12.31. Appl., 20.7.28).—A gas vent for a chamber in which slag wool is collected is described. B. M. V.

Removal of dust and sulphur compounds from flue gases by washing. G. H. C. CORNER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 376,383, 14.4.31). -Vertical, irrigated baffles are composed of half-round troughs arranged (convex) back to back and (concave) face to face, in the direction of flow of the gases, their adjacent edges being spaced further apart and the intercalating edges nearer together so that gradually a greater proportion of the zig-zagging streams of gas is cut off and allowed to pass to the spaces between the backs of the troughs which are comparatively dead. The curvature of the troughs may also be progressively increased to maintain centrifugal force, except at the final outlet where the curvature is reduced to avoid B. M. V. entrainment of moisture.

Viscosimeter. F. S. STICKNEY, ASST. to WESTING-HOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,836,995, 15.12.31. Appl., 7.6.26).—A drum rotating at const. speed is surrounded by a drum which changes its angular position under the drag of the liquid acting against a spring or other resisting torque. The whole is surrounded by a perforated guard cylinder and the rotating drum is provided with apertures giving a slight pumping action to renew the sample of the liquid in which the drums are immersed. B. M. V.

Viscosimeters. W. J. ALBERSHEIM and H. S. KONHEIM (B.P. 376,253, 23.3.31. U.S., 22.3.30).-In a viscosimeter of the type in which a fluid, e.g., lubricating oil, is passed at a const. rate (determined by a bellows valve) through an orifice in which the pressure drop is determined by  $v^2 = 2gh$  and then through a jet where the pressure drop is determined by friction, the reading of the pressure gauge is proportional to the viscosity. In this invention the same pressure gauge is enabled to cover a larger range of viscosities, while still retaining the same open scale at the dangerous "thin" end, by forming the first orifice so that the pressure is partly determined by  $v^2 = 2gh$  and partly by friction, the effect being that gauge pressure for very high viscosities does not rise so high. B. M. V.

Producing fire-extinguishing foam. F. L. BOYD (B.P. 377,864, 6.11.31).

[Filling of containers with de-fibrated fibre during] manufacture of smoke and fog filters. B. DRÄGER, Assee. of DRÄGERWERK H. U. B. DRÄGER (B.P. 375,203, 11.12.31. Ger., 12.3.31).

# II.—FUEL; GAS; TAR; MINERAL OILS.

The Northumberland and Durham coalfield. Northumberland area : main seam. ANON. (Dept. Sci. Ind. Res., Fuel Res., Phys. and Chem. Survey of the Nat. Coal Resources, No. 22, 1932, 60 pp.).—The results of a comprehensive and detailed examination, from floor to roof, of 18 sections of the main seam, taken at different points distributed over its area, are given. The following properties of the seam at its different points have been determined : proximate and ultimate analyses, calorific vals., ash m.p., and Gray-King assays. C. B. M.

Utilisation of New Zealand coals. J. A. C. BAYNE, F. W. J. BENTON, and W. DONOVAN (New Zealand J. Sci. Tech., 1932, 13, 353—370).—Recommendations for various uses of coal, directly as a fuel, and in the manufacture of road-surfacing pitch and benzol for internalcombustion engines are recorded. A. G. P.

Physical properties of Pennsylvania anthracite and some related materials. J. L. MEYER (Amer. Inst. Min. Met. Eng., Tech. Publ., 1932, No. 482, 19 pp.).—Methods for the determination of sp. heat, electrical resistivity, and thermal expansion are described and the results obtained for typical Pennsylvania anthracites, bituminous coal, lampblack, graphite, Iceland spar, quartz, sand, charcoal, and metallurgical coke are tabulated. C. B. M.

Application of selenium as a catalyst to the Kjeldahl method for determination of nitrogen in coal and coke. H. E. CROSSLEY (J.S.C.I., 1932, 51, 237-238 T; cf. Beet, B., 1932, 630).—Using a Se catalyst (10 g. of a mixture of 98 pts. of  $K_2SO_4$  and 2 pts. of Se) the time taken for the digest to clear of char was approx. 40 min. as compared with 87 min. necessary with a HgO catalyst (10 g. of a mixture of 89 pts. of  $K_2SO_4$  and 11 pts. of HgO). In both cases a further period of heating of 90—120 min. was necessary to give a steady max. val. for N. Se has the advantages of not requiring subsequent addition of Na<sub>2</sub>S, of giving a sharp titration end-point, and of lower cost than the HgO. A. B. M.

Influence of heat and oxidation on properties of coking coals. H. A. J. PIETERS and H. KOOPMANS (Brennstoff-Chem., 1932, 13, 261–264).—The swelling and caking power, composition, and the course of volatile matter evolution during carbonisation have been determined for a coking coal of high vitrain content before and after heating at 100–385° in N<sub>2</sub> or air. The coal was very sensitive to oxidation and on heating in air above 160° rapidly lost its swelling and caking properties. These properties were also adversely affected on heating in N<sub>2</sub> for 1 hr. at 250°, at which temp. an oily substance was distilled from the coal. Oxidation of the coal in air below 250° had no effect on the softening point. The bitumen was apparently little affected by oxidation, whereas the remainder of the coal

lost its power of being caked with the bitumen. On heating in air part of the coal is oxidised to  $CO_2$  and  $H_2O$ , and at the same time  $O_2$  is absorbed; the coal may lose its coking properties entirely with little or no accompanying change in its ultimate composition.

A. B. M.

Determination of combustibility of coke ; combustibility of some grades of Donetz coke. V. S. ABRAMOV (Domez, 1931, No. 8, 72–92).—Kreulen's method was modified. Vals. for Donetz coke were  $33\cdot1-46\cdot5\%$  (charcoal 100%). For determination of combustibility in CO<sub>2</sub> Koppers' method was modified ; vals. of (CO × 100)/(CO<sub>2</sub> + CO) were  $67\cdot3-126\cdot9$ .

CH. ABS.

Determination of thermal efficiency of a coke oven. V. V. YUSHIN (Domez., 1931, No. 4-5, 66-76). -(a) l = (750b/2)Z, where l = kg. of coke produced per hr. per sq. m. of wall surface of oven, 750 is the wt. in kg. of 1 cu. m. of coal, b is the mean width of the oven in m., and Z is the coking period. For a normally operating oven lined with SiO<sub>2</sub> brick  $l = 7 \cdot 8 - 9 \cdot 5$ ; for one lined with firebrick  $l = 5 \cdot 9 - 6 \cdot 7$ . (b)  $\eta = (Q - V - W)/Q$ , where  $\eta$  is % efficiency, Q = heat entering oven, V = heat leaving oven, and W = heat lost by conduction. CH. ABS.

Efficiency of coke ovens. A. A. AGROSKIN and E. V. VIROZUB (Domez, 1931, No. 12, 48-62).—Methods of determination are discussed; formulæ and tabulated data are given. CH. ABS.

Turbine blowers in coke-oven works. O. KREBS (Brennstoff-Chem., 1932, 13, 249—252).—Data relating to the performance of turbine gas blowers are discussed. A. B. M.

Fuel and smoke. A. NAITO (J. Fuel Soc., Japan, 1932, 11, 51-52).—The causes of smoke are discussed. It is suggested that the addition of a small amount of coal of high moisture content (50%) to the normal coal used is an efficient method for preventing smoke.

C. B. M.

Ignition and combustion process in the coaldust engine. W. WENTZEL (Forschungsheft 343, V.D.I. Publ., 1931; Fuel, 1932, 11, 177-196, 222-228). -Calculation shows that the energy developed in an oil engine is about 2.5 times dearer than that produced by a coal-dust engine. An apparatus has been designed for investigating the speed of combustion of coal dustair mixtures under pressure. It consists of a spherical steel bomb of 40 cm. internal diam., provided with the necessary valves, pressure-recording devices, etc., and is immersed in a water-bath. The bomb is filled with air heated to the required temp. by the combustion therein of the requisite amount of H2, and adjusted to give the desired initial pressure ; the coal dust is then injected into the bomb by means of compressed air. The presence of H<sub>2</sub>O vapour had no marked effect on the ignition and combustion processes. The ignition lag was independent of the amount of excess air and almost independent of the pressure of the injection air; it increased with falling temp. of the combustion air. The ignition lag and the ignition limit, *i.e.*, the temp. below which ignition no longer occurred, fell as the density of the combustion air was increased. Other things

being equal, the ignition lag was determined by the size of the smallest particles present ; as the finer particles were removed the ignition lag and the ignition limit rose rapidly. The ignition limit and ignition lag depended to a great extent on the ignition point of the coal used ; the richer the coal in gas the lower was the ignition limit. For temp. of the combustion air near to the ignition limit the vals. for the ignition lag in the case of prepared dust lay between 0.01 and 0.03 sec. Combustion time was independent of the temp. of the combustion air as long as this was not too near the ignition limit. On the contrary there was a strong dependency on excess air and on ignition pressure. The density of the combustion air had no effect on combustion. The combustion time was greatly dependent on the grainsize composition of the dust. With sufficient excess air and good ignition the vals. for combustion time of prepared dust lay between 0.15 and 0.35 sec. according to the grain-size composition and the kind of coal. The experimental vals. accord well with a new formula for ignition lag and with Nusselt's formula for combustion time. The application of the results to the coal-dust engine is discussed. A. B. M.

Formation of nitro-hydrocarbons in the [explosion] motor. E. GRAEFE (Petroleum, 1932, 28; Motorenbetr., 5, No. 6, 5—6).—Several used motor oils (chiefly from Diesel engines) are shown to contain  $NO_2$ -compounds which partly decompose at about 130° and are formed probably by chemical reaction between air and fuel or lubricating oil during the explosion in the cylinder. H. E. B.

Hydrogenation of American coals. Preliminary investigation. W. L. BEUSCHLEIN, B. E. CHRISTENSEN, and C. C. WRIGHT (Ind. Eng. Chem., 1932, 24, 747— 751).—PhOH being adopted as the dispersion medium, initial experiments were made on the hydrogenation of PhOH in the presence of small amounts of coal ash, which, however, exerted no catalytic effect at 400°. Qual. and quant. data are given for the H<sub>2</sub> absorption of 2 bituminous American coals. Optimum conversion into PhOH-sol. constituents was obtained at 300—350°; the yields obtained for these coals compare favourably with those reported for European coals. C. B. M.

Determination of hydrogen cyanide in coaldistillation gases. K. VOITURET (Brennstoff-Chem., 1932, 13, 264-265).—Gluud's method gives consistent results for the HCN content of coal gas if the following procedure is used. The HCN is absorbed from the gas in 3% aq.  $(NH_4)_2S_x$ , and dil.  $H_2SO_4$  is added until the solution remains just faintly yellow; the solution is boiled, filtered from the pptd. S, cooled, the thiocyanate is pptd. in known manner with  $CuSO_4$  and  $H_2SO_3$ , and may be determined either volumetrically or gravimetrically. In the presence of  $CS_2$  the method gives high vals. due to the reactions:  $(NH_4)_2S + CS_2 \rightarrow$  $(NH_4)_2CS_3 \rightarrow NH_4CNS + H_2S$ . In such cases Feld's method, in which the HCN is absorbed in aq. KOH and Fe(OH)<sub>2</sub>, is to be preferred. A. B. M.

Position of the wet-purification process for blast-furnace gas. R. WALTER (Stahl u. Eisen, 1932, 52, 625-631).—The Theisen, Zschocke, and Dingler plants for the removal of dust from flue gas by spraying with  $H_2O$  are illustrated and their operation is discussed. The advantages claimed for these processes are: low capital cost, small space occupied, and efficient operation under varying conditions of temp., dust content, moisture, and rate of flow of gas. A. R. P.

Combustion cartridges for the industrial analysis of gases. G. DUNKEL (Chem. Listy, 1932, 26, 236— 237).—The contraction consequent on combustion of  $CH_4$  or  $H_2$  in a capillary tube is measured. R. T.

Determining the most favourable design of gas burners. J. H. EISEMAN, E. R. WEAVER, and F. A. SMITH (Bur. Stand. J. Res., 1932, 8, 669–709).—The limiting conditions of "flashing back," and incomplete ("carbonising") combustion, CO and CO<sub>2</sub> contents of the combustion products, heating efficiency, etc. have been determined in relation to burner design. J. L.

Isolation of s-xylenol from crude [coal-]tar acids. E. B. KESTER (Ind. Eng. Chem., 1932, 24, 770-771).—s-Xylenol, m.p.  $63-64^{\circ}$ , b.p.  $220 \cdot 2^{\circ}/1$  atm., may be separated from the acids by fractional distillation, followed by crystallisation from light petroleum, by formation and crystallisation of its Na salt, by preferential sulphonation of accompanying phenols, or by a combination of these methods. H. A. P.

Toxic action of coal-tar creosote. Existence of a barren, non-toxic oil. H. SCHMITZ and S. BUCKMAN (Ind. Eng. Chem., 1932, 24, 772—777).—The fraction of coal-tar creosote having b.p.  $< 285^{\circ}$  is exceedingly toxic to *Fomes annosus* and *Trametes serialis*; its toxicity is not markedly altered by repeated alternate extraction with aq. acids and alkalis, or by subsequent successive treatment with HgCl<sub>2</sub> in MeOH and Na. If, after the former treatment, this fraction is cooled, a solid, mainly  $C_{10}H_8$ , is deposited, and has the same toxicity as the mother-liquor. The fractions of higher b.p. do not completely prevent growth at concns. > 20% and yet considerably retard it at much lower concns. The "barren oils" cannot therefore be regarded as definitely non-toxic, and must aid in wood preservation.

H. A. P. Modern [petroleum] refining practice. P. M. E. SCHMITZ (Petroleum, 1932, 28, No. 26, 1-4).—The risk of explosion in petroleum storage tanks is discussed and the methods for the stabilisation of gasoline (removal of  $CH_4$ ,  $C_2H_6$ ,  $C_3H_8$ , and *iso*butane to prevent "vapour locks " during use in pipes) and reclamation of gasoline from refinery gases (*e.g.*, by controlled pressure condensation) are discussed with reference to the efficiency of the refinery process. The permanent gases removed during stabilisation or reclamation processes may be used to provide inert gas atm. in storage tanks.

#### H. E. B.

Application of aluminium chloride in the petroleum industry. DUBROV, LAVROVSKI, GOLDSTEIN, FISH, and MIKHNOVSKAYA (Neft. Choz., 1932, 22, 19–25).— Cracking in presence of  $AlCl_3$  is accompanied by hydrogenation; intermediate mol. compounds are decomposed at higher temp. into  $AlCl_3$ , gas, and unsaturated residue,  $H_2$  then formed saturating the unsaturated fragments. Polymerisation and carbonisation take place simultaneously with hydrogenation. Paraffin is more stable in presence of AlCl<sub>3</sub> than are petroleum products containing paraffin. Surakhani fuel oil gave 72% of gasoline (aromatic 5.28, naphthenes 41.52, paraffins 38.71%); Bibi-Eibat fuel oil gave 60-62%, Balakhani 60%, and Ramani 50-65% of naphtha; Grozni mixed-base fuel oil gave practically no gasoline. Residues from cracking in Vickers units yielded up to 19% of gasoline and oils containing paraffin. AlCl<sub>3</sub> is not recoverable; the AlCl<sub>3</sub>-petroleum mixture gave satisfactory results, but a continuous process failed owing to clogging of pipes. CH. ABS.

Manufacture of bright stocks from paraffinic residues of petroleum from Grozni oilfield. L. G. ZHERDEVA (Azerbaid. Neft. Choz., 1932, No. 1, 65—76). —Complete separation of acid sludge decreases consumption of clay and improves the colour of the oil by contact filtration; natural clays are less efficient than treated clays. Neutralisation of the acid oil with NaOH decreases clay consumption. Two refining schemes are recommended, and the characteristics of the finished stock are recorded. CH. ABS.

Preparing bright stocks from Baku fuel oil. L. G. ZHERDEVA (Rep. Lubr. Oil Comm., U.S.S.R., 1932, 2, 16-27).—The procedure involves distillation; treatment of bottoms (a) with  $H_2SO_4$ , (b) with adsorbent; dewaxing of the treated residue by (a) dilution with naphtha, (b) chilling, (c) centrifuging; filtration through an adsorbent; recovery of solvent. The average yields are: bright stocks 50-60, ceresins 10-12, petrolatum 3-5%. CH. ABS.

Critical temperatures of petroleum oils. G. L. EATON and C. A. PORTER (Ind. Eng. Chem., 1932, 24, 819—822).—A method is described for determining the crit. temp., in tubes, provision being made for agitation of the oil sample while it is being heated. From the experimental data, an empirical relationship between crit. temp. and b.p. has been developed. C. B. M.

Vacuum distillation; its effect on lubricating [oil] stocks. M. B. MILLER (Refiner Nat. Gas Mfr., 1931, 10, No. 10, 95).—Use of vac. reduces the acid required by 50% and sludge losses by 66%. Heavier fractions are more easily dewaxed. CH. Abs.

Dewaxing lubricating oils with methylene chloride. P. J. CARLISLE and A. A. LEVINE (Ind. Eng. Chem., 1932, 24, 384-387).-Lubricating oils are miscible with CH<sub>2</sub>Cl<sub>2</sub> in all proportions at temp. down to  $-45 \cdot 6^{\circ}$ , whilst at the usual chilling temp. wax is only very slightly sol., thereby enabling an oil to be produced with a pour point only slightly above the chilling temp. At room temp. CH<sub>2</sub>Cl<sub>2</sub> is miscible with an oil containing 27% of wax. Simple steam-distillation or distillation in a current of air completely removes all trace of solvent from the wax or oil. The wax crystallises from the CH2Cl2-oil mixture and may be removed readily by filtration or centrifuging. CH<sub>2</sub>Cl<sub>2</sub> has no damaging effect on wax or oil; it is only slightly sol. in  $H_2O$  and  $H_2O$  is insol. therein. By adding an excess of CH2Cl2 to the oil to be dewaxed and reducing the pressure, the CH<sub>2</sub>Cl<sub>2</sub> may be made to perform functions of both refrigerant and dewaxing solvent. H. S. G.

a 4

Continuous acid treating. E. PETTY (Refiner Nat. Gas Mfr., 1932, 11, No. 1, 13).—Acid-treating reactions comprise (1) dissolution of asphaltic matter, and (2) interaction with asphaltic matter affording  $SO_2$ . When the temp. is too high or the acid too conc., acid gas is dissolved or entrained by the oil and "pepper" formation occurs. CH. ABS.

Clay volume and vapour velocity govern colour and gum. R. E. GOODE (Refiner Nat. Gas Mfr., 1931, 10, No. 12, 79).—As the charge of clay in a gray tower was reduced the throughput per ton of clay fell considerably; the colour was not so good and the gum content increased. CH. ABS.

Elements of high-pressure stabilisation. I. N. BEALL (Refiner Nat. Gas Mfr., 1931, 10, No. 8, 83; No. 9, 97).—The operation is discussed. CH. Abs.

Composition of straight-run Pennsylvania gasoline. I. Design of fractionating equipment. M. R. FENSKE, D. QUIGGLE, and C. O. TONGBERG (Ing. Eng. Chem., 1932, 24, 408-417).-The construction of two packed metal fractionating columns, (a) 27 ft. long by 3 in. diam., capacity 13 gals., and (b) 52 ft. long by <sup>3</sup>/<sub>4</sub> in. diam., capacity 1.5 gals., is described. The essential features in design are adiabatic operation, controlled reflux, high efficiency with a large throughput, and min. liquid holdup in the column combined with simplicity in operation. To test the efficiency of the columns, diisobutylene, known to consists of two isomeric olefines, was fractionated, and no difficulty was experienced in separating the two isomerides, boiling 3.3° apart, in one fractionation. H. S. G.

Composition of straight-run Pennsylvania gasoline. II. Fractionation and knock-rating. M. R. FENSKE, D. QUIGGLE, and C. O. TONGBERG (Ind. Eng. Chem., 1932, 24, 542—546; cf. preceding abstract).— The gasoline was separated by one fractionation into fractions of alternate high and low knock-rating in which straight-chain paraffins and aromatic and naphthenic hydrocarbons were conc. Any one normal paraffin is present in this gasoline to the extent of 2—5%, yet although such paraffins constitute only about 20% they are largely responsible for the knock. By refractionation these paraffins may largely be removed, the product in some cases being nearly pure. H. I.

Composition of straight-run Pennsylvania gasoline. III. Isolation of pure hydrocarbons. C. O. TONGBERG and M. R. FENSKE (Ind. Eng. Chem., 1932, 24, 314—318, cf. preceding abstracts).—Straightrun Pennsylvania gasoline, previously fractionated in a column 27 ft. by 3 in., was refractionated in 8.5-ft. and 52-ft. columns. The following hydrocarbons were isolated:  $\beta$ -methylpentane, b.p.  $60.3^{\circ}$ ; *n*-heptane, b.p. 98.4°, f.p. —91.2°; methylcyclohexane, b.p. 100.8—  $100.9^{\circ}$ , f.p. —126.7°; and *n*-octane, b.p. 125.2°. *n*-Heptane and methylcyclohexane did not form a const.boiling mixture; that of C<sub>6</sub>H<sub>6</sub> and *n*-hexane was found to be of widely different composition from the accepted val. C. B. M.

Vapour-phase cracking of gasoline. Optimum conditions for production of unsaturated gases from gasoline. H. A. CASSAR (Ind. Eng. Chem., 1932, 24, 802-804).—Starting with an untreated straightrun distillate made from crude Pennsylvania oil, the optimum conditions were determined by passing the gasoline through a heated Fe pipe packed with pumice. The olefines in the cracked gas reached a max. of 47%at about 600°; aromatics in the distillate increased rapidly with temp. and at 700° were 60% of the distillate. The olefine content of the distillate rose from 3% at 500° to 35% at 700°. The gas formed per unit time is proportional to the quantity of gas already formed, and the rate of reaction doubles for every 70° between 500° and 700°. Numerous metal oxides were tried as catalysts, but no marked effects were produced. C. B. M.

Highly cracked gasoline. C. R. PAYNE and A. Lowy (Ind. Eng. Chem., 1932, 24, 432-435).-In order to determine the changes in composition effected when a highly cracked gasoline is subjected to high temp., a highly cracked gasoline distillate was fractionated into  $5 \cdot 6^{\circ}$  fractions and the physical and chemical properties of each were determined. A quantity of the same distillate was treated at 300°/68 atm. for 6 hr. in a steel bomb. The treated material was then fractionated in the same manner as before and the changes in chemical and physical properties were noted. From the data general conclusions are drawn as to the types of hydrocarbons present in the most important fractions. In all fractions isolated the % of unsaturated hydrocarbons had been decreased by the treatment with increase in the % of naphthenes. H. S. G.

Formation of aromatic and hydroaromatic hydrocarbons from unsaturated compounds in the hydrogenation of a cracked benzine. J. VARGA and I. MAKRAY (Brennstoff-Chem., 1932, 13, 248-249). -Towne's method (B., 1931, 469) proved more suitable than that of Riesenfeld and Bandte for the analysis of a benzine (unsaturated hydrocarbons 50%, aromatic hydrocarbons 28%) obtained by cracking a brown-coal tar. In the latter method some of the unsaturated hydrocarbons appear as aromatics. The results of the two methods agree better the lower is the proportion of unsaturated hydrocarbons in the benzine. During hydrogenation of the benzine the unsaturated compounds are converted not only into aliphatic but also into hydroaromatic compounds. On hydrogenation at 380° and 440° the abs. amount of the aromatic hydrocarbons decreased, whilst that of the hydroaromatic hydrocarbons increased. A. B. M.

Determination of olefines by bromine titration. J. C. MORRELL and I. M. LEVINE (Ind. Eng. Chem. [Anal.], 1932, 4, 319—321).—Experimental procedure is described for the titration of olefines with Br, which is standardised against octylene. Special directions are given for mixtures. E. S. H.

**Evaporation tests for mineral oils.** J. J. SHANK (Ind. Eng. Chem. [Anal.], 1932, 4, 335).—The relative volatilities of oils are determined under standard conditions by mixing equal wts. of the oils with Pb shot in a porcelain dish and weighing after evaporating for a given time at a definite temp. E. S. H.

Determination of sulphur compounds in petroleum gases and liquids. A. W. TRUSTY (Refiner Nat. Gas Mfr., 1931, 10, No. 7, 91).— $H_2S$  in natural gas is determined by absorption in I solution and titration with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>;  $H_2S$  in refinery gas containing unsaturated hydrocarbons which react with I is pptd. as PbS from Pb(NO<sub>3</sub>)<sub>2</sub> solution, the excess of which is titrated with NH<sub>4</sub> molybdate. Vals. for H<sub>2</sub>S and mercaptans are recorded for various products.

# CH. ABS.

Erratum. On p. 7, col. 2, line 25, for Pt read Pb.

Junker calorimeter. Compressed solids. Fractionation. Oil etc. from condenser  $H_2O.$ —See I. Determination of olefines etc.—See III. SiO<sub>2</sub> bricks for coke ovens.—See VIII. Liquid-fuel containers.—See X. Mineral oil sprays.—See XVI. Poisoning by gas-heater.—See XXIII.

See also A., Aug., 808, Water-gas equilibrium. 841, Thermal behaviour of phenols.

#### PATENTS.

Separation of coal and shale. HUMBOLDT-DEUTZ-MOTOREN A.-G. (B.P. 373,808, 22.6.31. Ger., 20.6.30).— The raw material is graded into coarse and small coal, e.g., > and < 10 mm., respectively. The former is fed directly on to a perforated vibrating table, where the coal and shale are separated by upward air currents. The small coal is dried in a tubular apparatus through which hot gases are passed, and is then fed on to a second separating table. A. B. M.

Production of solid fuel. F. T. SNYDER (U.S.P. 1,822,383, 8.9.31. Appl., 8.6.25).—Coal, wood, etc. is distilled in a current of hot gas produced by recirculating part of the distillation gases after the separation of the condensable constituents. The recirculated gases are reheated by using them as the cooling medium in the first condenser. The higher-boiling condensates are injected by means of compressed air into the distillation kiln, their partial combustion, combined with the exothermic heat of decomp. of the material, supplying the heat required for the process. The injected oil which is not burned is cracked in the kiln with the deposition of solid carbonaceous material on the distilled product. A. B. M.

Fuels for internal-combustion engines. D. T. JONES, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 374,481, 12.2.31. Cf. B.P. 294,129; B., 1928, 703).— Fuels for Diesel-type engines are produced by adding to a heavy fuel oil, *e.g.*, creosote oils or oils from the hydrogenation of coal, a small % of an alkyl nitrate having  $C_{2-4}$  in the mol., *e.g.*, EtNO<sub>3</sub>. A. B. M.

Removal of carbon deposits [from engine cylinders]. W. G. LOVELL and T. A. BOYD, ASSTS. to GEN. MOTORS RES. CORP. (U.S.P. 1,833,429, 24.11.31. Appl., 28.8.29).—C deposits are removed from the cylinders of internal-combustion engines by heating and applying an oxazine which removes varnish, e.g., morpholine, either alone or in admixture with  $C_6H_6$  and EtOH etc. A. B. M.

Destructive distillation of carbonaceous materials. A. J. MEIER (B.P. 374,838, 12.12.30).—The coal travels under gravity through an inclined, frusto-conical, rotary retort, the lower end of which communicates with a chamber wherein a pile of coke is formed which supports the wt. of the charge in the retort. The coal, preferably preheated by a current of hot gas, is charged into the retort and, simultaneously, coke is withdrawn from the pile by means of screw conveyors. Distillation is effected by recirculating through the charge of coal and coke a part of the distillation gas which has been superheated by the combustion of another part of the gas, A. B. M.

Distillation of solid fuels. Soc. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 374,923, 12.3.31. Ger., 4.4.30).—Coal or lignite in the form of briquettes, agglomerates, or solid pieces is distilled in chambers through which a gaseous heating medium is passed at a sufficiently high velocity, e.g., 20—40 m./sec., to bring about rapid heating of the solid. Several chambers may be used in series, the direction of flow being periodically reversed in the hottest chambers while being maintained upwards in the coolest.

A. B. M.

Distillation of solid fuels. H. DUPUY (B.P. 375,250, 2.4.32. Fr., 3.4.31).—Distillation is effected by the recirculation of part of the distillation gas which is heated by the partial combustion of the hydrocarbons therein with hot or cold air injected into the circuit. The apparatus is brought into operation initially by using a combustible gas from an outside source; if necessary this gas may be used to supplement the distillation gas whenever the latter becomes deficient in hydrocarbons. A. B. M.

Shale distillation. M. J. TRUMBLE, ASST. to COALS & CHEMICALS, LTD. (U.S.P. 1,836,051, 15.12.31. Appl., 24.11.24).—The shale is charged into a no. of retorts and is distilled by passing superheated steam through them in series. Interposed between the retorts are superheaters wherein the mixture of steam and hydrocarbon vapours is raised to a temp. suitable for vapour-phase cracking of the hydrocarbons before admission to the succeeding retort. The superheaters are heated by the combustion of the C in the spent shale. A. B. M.

Treatment of carbonaceous articles. M. J. MARCIN, ASST. to D. MACKENZIE (U.S.P. 1,804,361, 5.5.31. Appl., 29.8.30).—Electric resistors, crucibles, etc. made of graphite or other carbonaceous material are given a hard surface layer by embedding them in a mixture of finely-divided  $SiO_2$  (e.g., flint) and C, and heating them in an induction furnace to a temp. sufficiently high, e.g., 1750—3300°, to form an adherent and uniform surface of SiC thereon. A. B. M.

Carbonising and coking process. C. B. WISNER, Assr. to COAL PROCESS CORP. (U.S.P. 1,835,128, 8.12.31. Appl., 13.7.28. Cf. U.S.P. 1,490,357; B., 1924, 548).— Coal, preferably crushed to  $< \frac{1}{4}$  in., is passed through an externally-heated rotary preheater, wherein it is maintained for approx. 45 min. above 175° but below the point at which thermal decomp. begins, and is then carbonised in known manner. A. B. M.

Coking retort ovens. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 374,515, 12.3.31. U.S., 12.3.30). —In one partition wall of each vertical flue of the oven a narrow passageway is provided which communicates

at its top and bottom with the top and bottom, respectively, of the parallel portion of the flue. This brings about a recirculation of part of the combustion gases with a consequent lengthening of the flame and more uniform heating of the oven chambers. A. B. M.

Coke ovens. G. HILGER (B.P. 374,962—3, 19.2.31. Addns. to B.P. 373,257; B., 1932, 761).—(A) The method of charging the ovens described in the prior patent is modified. (B) Chimney flues connected to the delivery hoppers communicate with a fan for leading off and burning gases evolved from the coking chambers during the charging operation. A. B. M.

Coke ovens. Soc. GÉN. DE FOURS À COKE, SYSTÈMES LECOCQ (B.P. 375,128, 20.8.31. Ger., 15.7.31).—In an oven for the manufacture of semi-coke the horizontal carbonising chambers are separated from the combustion flues by double walls so that the walls of the chambers do not come in direct contact with the combustion flames. The intermediate chambers formed in this way between the carbonising chambers and the flues are connected with the latter in such a manner that the carbonising chambers may be heated by radiation from the walls of the combustion flues and/or by convection currents of combustion gases passing through the intermediate chambers. A. B. M.

Apparatus for wood distillation. F. BRASS, N. OTTEN, and G. C. TOLAND (U.S.P. 1,836,305, 15.12.31. Appl., 15.3.27).—A furnace structure with a horizontal base and an arched top wall contains a horizontal, cylindrical retort of diam. substantially equal to the height of the furnace, which is thereby divided into two combustion chambers; these have lateral openings for the supply of fuel and air, and a common flue for the discharge of waste gases. The wood is loaded on to a car which can be run along a track into the retort, which is then closed. The volatile distillation products are withdrawn through a pipe leading from the bottom of the retort to a condensing system. A. B. M.

Temperature treatment [of wood etc.]. J. C. SCHAFFER, Assr. to AMER. PINE CHEMICALS, INC. (U.S.P. 1,836,510, 15.12.31. Appl., 23.2.27).—The apparatus consists of a chamber having a multiple V-shaped floor, which is water-cooled, and an arched top and sides, which are heated by the combustion gases from fire pots. The wood is stacked inside the apparatus and is distilled for the production of rosin or tar, most of which is withdrawn from troughs in the floor, and the remainder condensed in a water-cooled chamber with which the vapour offtake communicates. A. B. M.

Production of carbon black. R. B. DAY (U.S.P. 1,804,249, 5.5.31. Appl., 21.12.26).—Natural gas, or other hydrocarbon gas, is thermally decomposed by admixture with a current of heated gases free from  $O_2$ , the products are cooled, and the C black is separated. The heated gases are produced by admixing combustion gases with cooler gases in an amount regulated to give the mixture of optimum temp. for the production of C black by the method used. A suitable retort is described. A. B. M.

Catalytic hydrogenation of carbonaceous materials. CHEM. REACTIONS, LTD. From DEUTS.

GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 374,567, 10.4.31) .- Coals, tars, oils, etc. are hydrogenated at high temp. and pressure in the presence of Fe, Co, or Ni, or compounds thereof, and of H<sub>2</sub>S, which may be added in the free state or formed in situ, e.g., from S. The amount of H<sub>o</sub>S used is regulated to be in excess of that necessary to convert the metals or their compounds into sulphides under the operating conditions. In the hydrogenation of C<sub>10</sub>H<sub>8</sub> a crit. temp., usually between 480° and 500°, is observed at which the reaction becomes endothermic, and if it is desired to produce benzene hydrocarbons from C<sub>10</sub>H<sub>8</sub> the reaction is carried out 30-50° above this crit. temp.; below this temp. tetra- and deca-hydronaphthalene are formed. A. B. M.

Gas producer. L. N. BURNSIDE, ASST. to DELAWARE, LACKAWANNA & WESTERN COAL Co. (U.S.P. 1,832,092, 17.11.31. Appl., 20.11.26).—A producer suitable for the continuous production of gas from finely-divided solid fuel contains a fuel bed supported on a travelling endless grate inclined so as to cause the fuel at the surface of the bed to roll back upon itself with the assistance of the streams of air and steam which are passed through the bed. Fuel is fed continuously into the chamber at a point above the grate. A part of the air supplied to the grate is caused to blow back on to the bed any particles of unburned fuel mixed with the clinker. A. B. M.

Manufacture of mixed water-gas and oil gas. C. W. ANDREWS and H. A. BRASSERT, Assrs. to H. A. BRASSERT & Co. (U.S.P. 1,821,050, 1.9.31. Appl., 18.8.26).-The apparatus comprises two generators (A, B), each containing an ignited bed of solid fuel, and a superheater (C), and is operated in the following cycle of steps: (i) A and B are up-blasted with air, the blast gases being burned with secondary air in C: (ii) A is down-blasted with air and part of the blast gases are passed up through B, the remainder being burned with secondary air in the lower part of C; (iii) steam is passed up through A and the water-gas formed is passed over the fuel bed in B, which is simultaneously sprayed with heavy oil or tar, and the enriched gas is then passed through C; during this step some air may be supplied to B if desired; (iv) the uprun (iii) may be followed by a down-run in A, during which all or part of the water-gas formed is passed through B and C in succession. The cycle is then repeated. Both generators are provided with means for agitating the lower parts of the fuel beds to prevent clogging. A. B. M.

Purification of gas. S. P. COLEMAN and B. MEAD, Assrs. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,832,448, 17.11.31. Appl., 21.9.28).—Gases are freed from  $H_2S$  by washing with an aq. solution of  $SO_2$ . Washing is effected in at least two stages in the first and last of which the  $H_2S$  and the  $SO_2$ , respectively, is in excess. S is separated from the spent solution, to which salt may be added to prevent the formation of colloidal S. A. B. M.

**Purification of combustion gases.** R. LESSING (B.P. 374,467, 10.3.31).—The gases are freed from oxides of S by washing them with an aq. suspension of  $Ca(OH)_2$  or  $CaCO_3$ , withdrawing the suspension from contact with the gases before the normal sulphite has been converted into bisulphite, allowing the insol. Ca salts to settle, and using the liquor to prepare another suspension of  $Ca(OH)_2$  or  $CaCO_3$ , which is then recirculated. A. B. M.

Treatment of waste liquors from gas purification. H.McCormack, Assr. to NORTH SHORE COKE & CHEM. Co. (U.S.P. 1,831,968, 17.11.31. Appl., 5.3.31). —Waste liquor from the purification of coal gas etc. with alkaline solutions containing As compounds is treated with  $SO_2$ , heated, and pptd.  $As_2S_3$  filtered off; the filtrate is made alkaline with  $Na_2CO_3$ , aërated, and filtered from the pptd. Fe compounds, and this filtrate is conc., cooled to separate cryst.  $Na_2S_2O_3$ , and further conc. to separate cryst. NaCNS. A. B. M.

Retorts for the drying and/or distillation of bituminous and other similar materials. NESFIELD RETORTS, LTD., and A. G. NESFIELD (B.P. 373,644 and 373,929, [A] 14.2.31, [B] 14.11.31).—(A) A vertical retort, preferably rectangular in cross-section, contains a series of sloping grates arranged in zig-zag fashion. The material is fed in at the top of the retort and is periodically transferred from each grate to the one below by means of reciprocating rams, being eventually discharged at the bottom of the retort. (B) The retort is divided into two compartments, the upper for drying and the lower for distillation. The grates are arranged horizontally and are given a reciprocating motion in order to transfer the material from each grate to the one below. The discharge openings from each grate to the next below are so constructed that the material forms a seal therein, thus compelling the heating medium to pass through the grates and the superposed layer of material. A briquetting device may be interposed between two of the grates at a point where the plasticity of the material is suitable. If desired, the pitch from the process may be sprayed on to the material at a suitable point in the retort to be cracked to coke and lighter hydrocarbons. A. B. M.

Oven or retort for treating tarry matter and the like. A. S. KNOWLES, Assr. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,832,867, 24.11.31. Appl., 26.5.26). -A substantially rectangular retort having a floor of refractory material is heated from below by means of parallel transverse flues, provided with burners at both ends so that the flow of the combustion gases can be periodically reversed; below these flues is another set so arranged as to act as regenerators for heating the air for combustion. The chamber is lined with steel and has trackways for steel containers. The containers, the bottoms of which may be perforated, are filled with anthracite or coke, on to which the tar, oil, pitch, etc. to be distilled is sprayed from suitably arranged feed pipes. When desired, the containers can be removed from the retort through end doors for recharging. Steam can be injected into the space between the floor of the retort and the bottoms of the containers. A. B. M.

Conversion of tars rich in creosote into pitch, benzine, and phenols. MAGYAR KIRALYI ALLAMI VAS-ACÉLÉS GÉPGYÁRAK, and J. KÁRPÁTI (B.P. 374,024, 26.2.31).—The crude tar is heated under  $\leq 20$  atm. pressure at 400—450° for > 1 min., and the more volatile products are then distilled off by diminution of the pressure. The residual pitch is practically free from phenols and paraffin wax. The distillate, which is rich in phenols, is then cracked at 450—600° under pressure, the low-boiling hydrocarbons formed being separated from the unattacked phenols by fractional distillation. If the initial material or the first distillate is poor in phenols the phenol content may be raised to at least 40% by the addition of suitable oil fractions. A. B. M.

Separation of phenols from coal tar and tar distillates. C. STILL (B.P. 374,386, 19.1.32. Ger., 19.1.31). —Separation is effected by extraction with 77—84 wt.-% of EtOH. The process is particularly applicable to tars obtained by withdrawing the distillation gases from chamber ovens or retorts through ducts formed in the charge (cf., B.P. 369,089; B., 1932, 489). A. B. M.

**Treatment of phenol-bearing liquors.** J. A. SHAW, ASST. to KOPPERS Co. (U.S.P. 1,832,140, 17.11.31. Appl., 27.5.26).—The liquor, preferably after removal of the  $\mathrm{NH}_3$  by distillation, is treated with a petroleum distillate, *e.g.*, kerosene, from which the PhOH is subsequently recovered by extraction with aq. NaOH and treatment of the aq. NaOPh with CO<sub>2</sub>. A. B. M.

Production of bituminous emulsions. L. KIRSCH-BRAUN, ASST. to FLINTKOTE Co. (U.S.P. 1,832,987, 24.11.31. Appl., 24.7.29).—An emulsifying agent for the production of aq. emulsions of bitumen, pitch, etc. is made by mixing tannic acid with finely-divided hard bitumen, asbestine, lampblack, or other solid which is more readily wetted by bitumen than by  $H_2O$ , and forming the mixture into a pasty mass with  $H_2O$ .

A. B. M.

Manufacture of aqueous dispersions of bitumen. J. A. MONTGOMERIE (B.P. 374,111, 4.3.31. Cf. B.P. 226,032; B., 1925, 91).—A quick-breaking, aq. bitumen emulsion is first produced by mixing molten bitumen with hot, dil., aq. alkali. This is then stabilised by the addition of a small amount of an alkali caseinate. The emulsion may be used for impregnating paper pulp, cement, sand, or other solid or comminuted material.

A. B. M.

**Bituminous composition.** R. ARNOT (B.P. 374,987, 2.4.31).—A small proportion ( $\geq 10\%$ ) of a fatty pitch, *e.g.*, stearine pitch, is dispersed in low-temp. tar, and an alkali, *e.g.*, Ca(OH)<sub>2</sub>, is added to accelerate hardening. The fatty pitch may be dispersed in a suitable solvent, *e.g.*, shale oil, by heating it therewith under pressure, before addition to the tar. A. B. M.

Apparatus for cracking mineral oil. A. E. PEW, JUN., and H. THOMAS, ASSTS. to SUN OIL CO. (U.S.P. 1,831,424, 10.11.31. Appl., 26.6.29).—Preheated raw oil flows through 3 coils in series each of which is heated by Hg vapour condensing at a predetermined pressure different for each coil. Safety devices prevent serious rise in pressure in the Hg-vapour system, contamination of Hg with oil, etc. in the event of tube failure.

D. K. M.

Apparatus for cracking hydrocarbon oil. G. EGLOFF, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,832,970, 24.11.31. Appl., 4.1.29. Cf. U.S.P. 1,703,617; B., 1929, 465).—A battery of stills, each still having its

own condenser and receiver, is connected by a common gas header (A) receiving the incondensable gas through a check valve which prevents back-flow of gas from Ato each receiver and still. A delivers the gas through a pump to a pressure reservoir from which gas may be supplied to any or all of the still heating coils. H. E. B.

Cracking of hydrocarbon oils. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,832,927, 24.11.31. Appl., 20.8.20).—Heavy or residual oils (e.g., petroleum fuel oils) are continuously passed under pressure through a heating zone, A, e.g., 4 in. diam. pipe coils at  $> 430^{\circ}$ , into a separating chamber B (e.g., 10 in. diam. pipes at slightly  $< 430^{\circ}$ ), whence the vaporised fractions pass through C and scale traps to a reflux condenser where the heavy fractions are condensed and the lighter fractions pass to a final condenser. The residual oil containing suspended C is continuously removed from B and the reflux condensate is returned to A with the charging stock. H. E. B.

Cracking of hydrocarbon oils. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,832,928, 24.11.31. Appl., 26.8.21).—The oils are cracked in a primary still (A) and the vapours superheated in a secondary chamber adjacent to the still and passed to a reflux condenser (B). The uncondensed vapours are condensed in a final condenser and the reflux condensate, mixed with raw charging stock, is returned to A. The rate of condensation in B is adjusted to give excess of reflux condensate in the charging oil, and the cracked residue is removed from one end of A, the C removed by baffles and settling, and the residual oil fed to the superheated vessel. H. E. B.

Treatment of hydrocarbon oil. J. R. SCHONBERG, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,831,265, 10.11.31. Appl., 18.7.29).—A mixture of hydrocarbon oil, oleum, and/or  $H_2SO_4$  and  $SO_2$  (liquid or under pressure) is expanded through a nozzle into a compartment of a tank through the liquid in which  $SO_2$  is bubbled, and thence it passes into the other compartment in which settlement of the sludge takes place. The oil and more acid and  $SO_2$  are similarly treated in several tanks and nozzles. The gases from the tanks (after scrubbing with oil) and the  $SO_2$  obtained by warming the oil leaving the last tank are re-used. D. K. M.

Conversion of high-boiling hydrocarbon oil. W. E. WARWICK, ASST. to STANDARD OIL CO. (U.S.P. 1,830,972, 10.11.31. Appl., 9.11.25).—Oil from a cracking coil flows into two vaporising chambers in parallel, each with its own dephlegmator and each capable of independent control. D. K. M.

Apparatus for treating [hydrocarbon] oil with aluminium chloride. F. W. HALL, Assr. to TEXAS Co. (U.S.P. 1,832,629, 17.11.31. Appl., 25.5.27).— AlCl<sub>3</sub> vapours are generated in a retort and conducted to the upper chamber (A) of a reaction apparatus into which the liquid undergoing treatment is sprayed over a conical distributor. The resultant mixture comprising treated oil and condensed AlCl<sub>3</sub> is collected in a lower chamber (B) where the more liquid portion separates and is withdrawn from an intermediate point. The less liquid portions are withdrawn from the bottom of B, whilst heating means are provided for maintaining a given temp. at any portion of the apparatus. Outlet means are provided in A for withdrawing vapours. H. S. G.

Thermal treatment of hydrocarbon gases. ANGLO-PERSIAN OIL CO., LTD., and G. H. COXON (B.P. 374,017, 25.2.31).—Aromatic hydrocarbons are produced by the thermal treatment of hydrocarbon gases (of. B.P. 309,455; B., 1929, 509), the heating being effected by mixing the gases with another gas which has been heated to a suitable temp. and thus serves as a vehicle of heat. The mixed gases are passed through a reactor, cooled, and the condensable hydrocarbons are separated. A. B. M.

Manufacture of hydrogen and carbon monoxide from hydrocarbon gases. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of M. DE SIMS (B.P. 373,701, 20.3.31. U.S., 24.3.30).—Hydrocarbon gases, e.g., CH<sub>4</sub> or natural gas, are passed over, or otherwise treated with, an easily reducible metal oxide, preferably ZnO, at about 950—1000° (CH<sub>4</sub> + ZnO  $\rightarrow$  CO + 2H<sub>2</sub> + Zn). The metal is reoxidised and returned to the process. A. B. M.

Removing hydrogen sulphide [from petroleum products]. L. ROSENSTEIN (U.S.P. 1,832,325, 17.11.31. Appl., 16.7.28).—The gas or liquid is washed with aq.  $Na_2S_4O_6$ , whereby S is pptd. and  $Na_2S_2O_3$  formed. The latter is reconverted into  $Na_2S_4O_6$  by anodic oxidation or by agitating with air in the presence of a catalyst, *e.g.*, pptd. NiS, and NaHCO<sub>3</sub> or NaH<sub>2</sub>PO<sub>4</sub>. A. R. P.

Refining of hydrocarbon liquids under high vacuum. W. W. GRIMM (U.S.P. 1,832,202, 17.11.31. Appl., 25.6.26).—Petroleum is heated in a pipe still and the lower-boiling oils are removed at approx. atm. pressure. The oil is thereafter submitted to a single, substantially uniform, high vac., without additional heat, to evolve a complex distillate containing lubricating oils. The residual, unvaporised, tarry residue is immediately conducted away and the vapours are fractionally condensed to produce lubricating oils of other grades. H. S. G.

Dehydration and purification of oil. A. E. PEW, JUN., ASST. to SUN OIL CO. (U.S.P. 1,833,618—9, 24.11.31. Appl., [A] 14.6.27, [B] 4.4.29).—(A) Crude oil is heated to a temp. at which its viscosity is  $\gg 35$  sec. (Saybolt) and under pressure to prevent vaporisation and settled, whereby H<sub>2</sub>O, NaCl, etc. separate out. (B) The process is worked continuously and the oil heated by condensing the vapours produced by the distillation of the purified oil. D. K. M.

Refining of oils. J. C. BLACK, ASST. to CONTACT FILTRATION CO. (U.S.P. 1,832,892, 24.11.31. Appl., 26.10.26).—The oil, mixed with a solid adsorbent material (e.g., SiO<sub>2</sub> gel, charcoal, fuller's earth), is circulated under reduced pressure through a reaction zone at  $< 120^{\circ}$  and the liberated air and H<sub>2</sub>O vapour are removed. The vac. is then increased and the temp. raised to  $> 120^{\circ}$ , any vaporised fractions being condensed and separated, and after circulation the mixture is cooled and the refined oil separated from the adsorbent. H. E. B,

**Reactivation of adsorbents.** GAS LIGHT & COKE Co., R. H. GRIFFITH, and R. S. CHAPLIN (B.P. 374,597, 12.5. and 22.12.31).—Active charcoal,  $SiO_2$  gel, or other adsorbent which has become fouled, *e.g.*, by resinous material formed by the polymerisation of unsaturated hydrocarbons, is reactivated by hydrogenating the impurities at high temp. and pressure in the presence of a catalyst, *e.g.*, a compound of V, Cr, W, Fe, or U. A. B. M.

**Regeneration of catalysts.** A. JOSEPH, ASST. to COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (U.S.P. 1,832,217, 17.11.31. Appl., 11.7.29. Fr., 13.7.28).—Carbonaceous and S impurities are removed by submitting the spent catalyst to the simultaneous action of steam and air or  $O_2$ , the  $O_2$  supply being controlled to prevent the development of excessive temp. and reduction of the catalyst. H. S. G.

Preparation of crude oil for distillation into lubricating oils. A. E. PEW, JUN., Assr. to SUN OIL Co. (U.S.P. 1,833,691, 24.11.31. Appl., 22.12.26).-Crude oil is heated in reflux condensers (A and B) by the vapours from towers (C and D), respectively, and in heat exchangers by the effluent from the bottom of D, to 154° approx. and then flows through a baffled tank under 100 lb. per sq. in. pressure at a velocity  $\geq$  25 ft. per hr., salt H<sub>2</sub>O being separated. The oil is then heated to 232° approx. and passed with reduction of pressure to atm. into C about half-way up. The effluent from the bottom of C is heated to about  $310^{\circ}$ and passed into D (as in C). The condensate from A(a light gasoline) and from B, the upper part of C, and from D (an intermediate, light, and heavy gas oil, respectively) are withdrawn. The effluent from the bottom of D is worked up according to U.S.P. 1,761,153 D. K. M. (B., 1931, 384).

Reclaiming of contaminated [lubricating] oils. H. H. MORETON, ASST. to O. B. ENGLISCH and C. F. CRAIG (U.S.P. 1,831,875—6, 17.11.31. Appl., 2.2.28).— The used oil is electrically heated to about 126° in a chamber fitted with a hood to carry away the vapours and thermostatically controlled, and then mixed with powdered material, e.g., gypsum (5—10%), settled, and, after further heating the clear liquid, it is filtered. The settling of the gypsum may be facilitated by adding  $H_2SO_4$  (2%). D. K. M.

Prediluted lubricating oil. R. E. WILSON, Assr. to STANDARD OLL CO. (U.S.P. 1,831,053, 10.11.31. Appl., 2.7.28).—A mixture of pressed paraffin-base oil (36 pts.) and heavy naphtha (10 pts.) is cooled to -23°, wax is removed, and the product blended with filtered cylinder stock (18 pts.) and low-cold-test naphthenic-base oil (36 pts.). D. K. M.

Mineral oil composition. W. W. EVANS, Assr. to R. T. VANDERBILT Co., INC. (U.S.P. 1,832,929, 24.11.31. Appl., 18.8.28).—A composition for electrical insulation purposes is prepared by dispersing about 3% of rubber latex in a mineral oil, dissolving and driving off H<sub>2</sub>O by heating to about 120°, and adding small amounts (e.g., 0.75%) of a "nitrogenous modifier" (e.g., diphenylethylenediamine or aldehyde-amine condensation products). H. E. B. Manufacture of candles. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 373,806, 20.6.31, 13.1. and 4.3.32).—Candles are cast or drawn in the usual manner with non-fibrous, org., combustible substances, e.g., paraffin wax, beeswax, stearine, etc., mixed with 2% or more of solid, preferably saturated, aliphatic alcohols, e.g., myricyl or octadecyl alcohols, or alcohols formed by the reduction of the acids obtained on oxidising montan wax (cf. B.P. 305,552; B., 1929, 273), or those formed by the process of B.P. 356,731 (B., 1932, 94). A. B. M.

[Radiating] gas burners. H. A. GILL. From WESTDEUTSCHER INDUSTRIE-OFENBAU M.B.H. (B.P. 377,741, 29.4.31).

Powdered - fuel burners. A. DOCKING and J. MCCLEARY (B.P. 377,682, 23.4.31, Addn. to B.P. 352,516).

Liquid-fuel burners. E. BRILLIÉ (B.P. 377,765, 8.5.31. Fr., 14.5.30).

Liquid-fuel burners or atomisers. C. ZULVER, and SWINNEY BROS., LTD. (B.P. 377,821, 11.8.31).

Separating suspensions. Cooling oils etc. Purifying liquids [oil etc.]. Viscosimeters.—See I. Washing etc. agents.—See III, Hormone-like substances from coal.—See XX.

# III.—ORGANIC INTERMEDIATES.

Approximate determination of olefine and aromatic hydrocarbons. J. C. MORRELL and I. M. LEVINE (Ind. Eng. Chem. [Anal.], 1932, 4, 321—322).—The sum of the olefines and aromatic hydrocarbons is determined by a slight modification of the method described previously (B., 1930, 359). Empirical formulæ are developed for calculating the olefine concn. from the wt. of residue remaining after distilling the oil obtained by treatment with 91% H<sub>2</sub>SO<sub>4</sub>. The aromatic hydrocarbon content is determined by difference. E. S. H.

Determination of small amounts of methyl chloride in air. F. A. PATTY, H. H. SCHRENK, and W. P. YANT (Ind. Eng. Chem. [Anal.], 1932, 4, 259—262).—The air containing MeCl is mixed with natural gas and burned in a micro-burner. The halogen products combine with NH<sub>3</sub> obtained from  $(NH_4)_2CO_3$  cubes placed around the burner; they are collected in an absorption tower and determined by the Volhard method. Concns. of 0.02-0.16 vol.-% can be determined satisfactorily. The error is < 1% for a sample containing 30.74 mg. MeCl. The method has also been used to determine 2% CHCl<sub>2</sub>F and 20% CCl<sub>2</sub>F<sub>2</sub> and is applicable to other org. halides. E. S. H.

rapidly decomposed by Fe, less rapidly by Cu. A continuous process for dehydration (from 92.5% to 99%) of aq. EtOH is described. H. A. P.

Berginisation of carbazole. E. I. PROKOPETZ and I. I. IERU (Ukrain. Chem. J., 1931, 6, [Tech.], 244-252). —Carbazole is converted in the presence of  $(NH_4)_2MOO_4$ successively into indole and cyclohexane, whilst in the presence of  $MOS_3$  the successive products are dicyclohexane, 3:3-dimethyl- and methyl-cyclopentane.

R. T.

 $NO_2$ -compounds in explosion motors. s-Xylenol from tar acids. Pure hydrocarbons from gasoline. Hydrocarbons from benzine.—See II.  $\mu$ -Polychloroprene.—See XIV. Citric fermentation.—See XVIII.

See also A., Aug., 819, Reduction of NO<sub>2</sub>-compounds. 830, Prep. of pure octene. 831, Synthesis of aliphatic alcohols. Prep. of mixed ethers. 832, Oxidation of aliphatic dicarboxylic acids. 833, Prep. of NH<sub>4</sub> *l*-tartrate. 842, Prep. of new ethylnaphthol. 843, Preps. of Ph<sub>2</sub> derivatives and of  $\beta$ -arylethylamines from  $\alpha$ -cyano- $\beta$ -arylacrylic acids. 846, Prep. of *tert*.-Bu phenylacetate. 847, Fluoranthene and its derivatives. 851, Prep. of s-trinitrobenzene. 853, Prep. of ketochloroimines. 854, Identification of nitriles.

# PATENTS.

Transforming aliphatic hydrocarbons into higher hydrocarbons. RUHRCHEMIE A.-G. (B.P. 373,249 and Addn. B.P. 373,822 and 374,055, [A] 14.2.31, [B] 4.7.31, [c] 25.2.31. Ger., [A] 14.2.30, [B] 5.7.30, [C] 26.2.30).-(A)  $CH_4$  is heated at > 1600° for < 0.001 sec. for partial conversion into C<sub>2</sub>H<sub>2</sub>, and the gases are then cooled to  $450-1000^{\circ}$  for a longer period, whereby  $C_{e}H_{e}$ hydrocarbons are produced. A catalyst (Al<sub>2</sub>O<sub>3</sub> or  $Al_2O_3 + 10\% K_2CO_3$  and suitable heat-exchange means may be employed. Other aliphatic hydrocarbons  $(C_2H_6, C_2H_4), H_2, N_2$ , etc. may be present. 100 g. of  $C_6H_6$  are obtained per cu. m. of  $CH_4$ . (B) In (A), the first stage may be conducted at 1120-1600° and for periods up to 0.5 sec., preferably at reduced pressure, e.g., 0.05 atm., which may then be raised to, say, 10 atm. during pyrolysis of the C2H2 formed. E.g., CH4, led at 130 litres/hr. at 50 mm. pressure through a 10-mm. tube heated for 80 cm. at 1400° (reaction time 0.018 sec.), gives 250 litres/hr. of gas containing 15.8% C<sub>2</sub>H<sub>2</sub>; this is pyrolysed in an outer tube at 950° carrying a Ag inner tube through which cold H<sub>2</sub>O flows. The yield per hr. is 27 g. of tar containing 60% of  $C_6H_6$  hydrocarbons, b.p. up to 160°. (c) In the second (pyrolysis) stage of (B), conductors are placed between the inner cooling tube and the walls of the outer tube. Catalyst may be deposited on the conductor. C. H.

Purification of ethylene and production of ethyl alcohol therefrom. G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 374,510, 12.3.31).—Crude olefines are passed over a metal phosphate  $[Cd_3(PO_4)_2]$ at 150—200°, preferably under pressure (50 atm.), whereby propylene and higher olefines are condensed to liquid hydrocarbons, and the exit gas, now richer in  $C_2H_4$ , is hydrated by known means to give EtOH. The

exit gas may be washed with kerosene to remove residual propylene etc.

Manufacture of conversion products of higher paraffin hydrocarbons containing sulphur. I. G. FARBENIND. A.-G., Assees. of DEUTS. HYDRIERWERKE A.-G. (B.P. 374,380, 23.12.31. Ger., 23.12.30. Addn. to B.P. 360,993; B., 1932, 93).—In the process of the prior patent, mineral acid esters (except halides) of aliphatic mono- or poly-hydric alcohols above  $C_7$  having at least 1 primary OH group are used as starting materials. *E.g.*, alcohols from coconut oil acids are sulphated with ClSO<sub>3</sub>H, and the neutralised product is heated at 150° with K<sub>2</sub>S<sub>3</sub>. The oil may be oxidised to a sulphonic acid with 20% HNO<sub>3</sub>. C. H.

Manufacture of acetaldehyde, acetone, and acetic acid [from acetylene]. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 373,893, 23.9.31. Ger., 27.9.30).—  $C_2H_2$  and steam are passed through molten ZnCl<sub>2</sub>, with or without solid catalyst (BaSO<sub>4</sub> + ZnO, or a mixture of CeO<sub>2</sub>, ZnO, Cr<sub>2</sub>O<sub>3</sub>, MnO, BaO, TiO<sub>2</sub>, KOH, and Na<sub>2</sub>SiO<sub>3</sub>), at 300—450°. Incrustation of basic salts on the supply pipe may be avoided by adding HCl gas continuously or periodically. The exit vapours may be passed over a solid catalyst to promote formation of MeCHO or of COMe<sub>2</sub>. C. H.

Manufacture of acrylic acid. Röнм & HAAS A.-G. (B.P. 373,326, 20.3.31. Ger., 21.3.30).—Acraldehyde is oxidised with air or  $O_2$  in  $C_6H_6$  or other org. solvent in absence of  $H_2O$ , reaction being stopped before 80% of the acraldehyde has been oxidised, whereby polymerisation is avoided. 50% of acraldehyde in  $C_6H_6$  kept for 14 hr. at 50° under  $O_2$  at 10 atm. gives 73% of acrylic acid. A catalyst may be used. C. H.

Manufacture of cyclic alcohols and [aliphatic] ketones. W. A. LAZIER, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,829,869, 3.11.31. Appl., 23.4.30).—A sec. aliphatic alcohol is dehydrogenated by heating with a phenol and a catalyst (Ni + NiO) at about 200°. CHMe<sub>2</sub>·OH and PhOH give COMe<sub>2</sub> and cyclohexanol. C. H.

Manufacture of cyclic ethers. H. DREYFUS (B.P. 374,134, 5.3.31).—A dialkyl ether containing halogen in each alkyl group is heated with metal oxide (HgO, Ag<sub>2</sub>O, PbO, CuO).  $\beta\beta'$ -Dichlorodiethyl ether gives dioxan; chloromethyl  $\beta$ -chloroethyl ether gives glycol methylene ether. C. H.

Manufacture of wetting, emulsifying, washing, and like agents. J. Y. JOHNSON. From I. G. FARB-ENIND. A.-G. (B.P. 373,642, 26.1.31).—Oxidised hydrocarbons above  $C_8$ , containing up to 60% of alcohols (preferably about 30%), with or without addition of cetyl, lauryl, or other high-mol. alcohols, are sulphonated. Suitable starting materials are oxidised paraffin wax, Russian petroleum, etc. [Stat. ref.] C. H.

Manufacture and use of washing, cleansing, wetting-out, and emulsifying agents. DEUTS. HYDRIERWERKE A.-G. (B.P. 373,315, 10.3.31. Ger., 18.6.30).—The products (dihydrodioxols) obtained by condensing polyhydric alcohols (glycerol) with ketones or aldehydes (cyclopentanone, cyclohexanone) are used in

conjunction with soaps or soap-like compounds (K oleate, curd soap, Turkey-red oil). C. H.

Manufacture of  $\beta$ -di(arylmethlyl)aminoethanols. I. G. FARBENIND. A.-G. (B.P. 373,440, 10.6.31. Ger., 10.6.30).— $\beta$ -Aminoethyl alcohol is condensed, preferably in presence of acid-binder (K<sub>2</sub>CO<sub>3</sub>), with 2 mols. of an arylmethyl halide, e.g., CH<sub>2</sub>PhCl (product, m.p. 46—47°, b.p. 175°/3 mm.; B,HCl, m.p. 192°), p-chlorobenzyl chloride (b.p. 212°/3 mm.), or 1-naphthylmethyl chloride (m.p. 80—81°, b.p. 283°/3 mm.; B,HCl, m.p. 211—212°). C. H.

Manufacture [purification] of coumarin. MON-SANTO CHEM. WORKS, Assees. of E. CLEMMENSEN (B.P. 373,490, 11.8.31. U.S., 2.9.30).—Crude coumarin is heated with aq. NaHSO<sub>3</sub>, filtered from resins, conc. to 110° to volatilise salicylaldehyde, phenols, etc., steamed at 110° if necessary, and coumarin recovered by heating with  $H_2SO_4$  followed by dilution. C. H.

Resolution [separation] of mixtures of phenolic substances into their components. W. BLYTHE & Co., LTD., W. H. BENTLEY, and B. CATLOW (B.P. 374,010, 24.2.31).-Mixtures of phenols (P) are separated by means of their compounds with aromatic NH2-compounds (B). Compounds of the type P2B are formed from PhOH with benzidine (m.p. 140°); o-, m-, and p-cresols with benzidine (m.p. 97°, 95°, 140°), tolidine (m.p. -, 97°, 63°), dianisidine (m.p. -, 82°, 84°), p-C6H4(NH2)2 (m.p. -, 59°, 109°); benzidine and tolidine with 6-chloro-m-cresol (m.p. 110°, 96°), 3-chlorop-cresol (m.p. 89°, 69°), p-hydroxyethylbenzene (m.p. 138°, 57°), m-5-xylenol (m.p. 68°, 85°), p-xylenol (m.p. 84°, 82°), guaiacol (m.p. 85°, 80°); benzidine with creosol (m.p. 60°), resorcinol (m.p. 145°). Compounds PB are formed from *p*-cresol with 2:4-tolylenediamine  $(50^{\circ})$ ; 2:7-dihydroxynaphthalene with p-toluidine (m.p. 140°), NPhMe<sub>2</sub> (m.p. 150°), N-(op-diamino-phenyl)benzidine (m.p. 145—150°), p-aminodimethyl-aniline (m.p. 88—90°);  $\beta$ -naphthol with N-(op-diam-inophenylbenzibing (m.p. 115°). N inophenyl)benzidine (m.p. 110-115°). No compound is formed from *m*-cresol with 2:4-tolylenediamine or 4:4'-diamino-2:2'-dimethyldiphenylmethane; p-cresol with 4:4'-diamino-2:2'-dimethyldiphenylmethane; creosol with tolidine; Me salicylate with benzidine or tolidine; PhOH or cresols with 3:5-diaminobenzamide. For the separation of m- and p-cresols the mixture is melted with benzidine, cooled, and pressed; the oil is distilled, and the distillate is melted with tolidine, cooled, and pressed. The press-cakes yield p- and m-cresol, respectively, on distillation in vac. Other separations described are: p-hydroxyethylbenzene, m-5-xylenol, and p-xylenol; guaiacol and creosol; 6-chloro-m-cresol from chlorinated m-cresol; 3:4dinitrophenol from PhOH and mononitrophenols.

C. H.

Manufacture of nitroaminodiphenyl derivatives. IMPERIAL CHEM. INDUSTRIES, LTD., S. COFFEY, and K. W. PALMER (B.P. 373,664, 2.3.31).—3:3'-Dinitro-4:4'-dimethoxy- and -diethoxy-diphenyls are reduced with 10% excess of 1 equiv. of Na<sub>2</sub>S, NaHS, or polysulphide in boiling EtOH, whereby only 1 NO<sub>2</sub> group is reduced; the nitroamines have m.p. 172° and 182—183°, respectively. C. H.

Manufacture of hydroxythionaphthens [thioindoxyls] of the naphthalene series, and of dyes derived therefrom. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 373,601, 17.2. and 20.4.31). -Dialkoxy- or halogenoalkoxy-naphthalenesulphonyl chlorides are reduced with Zn or Sn and acid, the resulting thiols are condensed with CH2Cl·CO2H, and the thioglycollyl chloride is converted, e.g., by AlCla in CS2, C6H6, or PhCl, into naphthathioindoxyls. Examples are: 5:7-dimethoxy-β-naphthathioindoxyl (I) m.p. 194°, from 2:8-dimethoxynaphthalene-6sulphonyl chloride, via the thioglycollic acid, m.p. 129°; the 5:8-isomeride (II), m.p. 165°, from the 2:5:7-sulphonyl chloride (thioglycollic acid, m.p. 174°); 8-chloro-5-methoxy-β-naphthathioindoxyl (III), m.p. 242°, from 2-chloro-5-methoxynaphthalene-7sulphonyl chloride (thioglycollic acid, m.p. 134°); the 7-chloro-5-methoxy-isomeride (IV), m.p. 209°. from the 2:8:6-sulphonyl chloride (thioglycollic acid, m.p. 135°). Vat dyes are obtained from : (I) with 5:7-dichloroisatin (yellow-brown), 5-bromoisatin  $\alpha$ -chloride (currant), or 6-chlorothioisatin a-pdimethylaminoanil (red-brown); (II) with \beta-naphthathioisatin 2-p-dimethylaminoanil (brown); (IV) with β-naphthathioisatin 2-anil (red-brown), or with 5:7-dichloroisatin (yellow-brown); with 5:7-dichloroisatin α-chloride (currant). [Stat. ref.] C. H.

Manufacture of anthraquinone derivatives. J. F. THORPE and A. A. GOLDBERG (B.P. 373,127, 9.2. and 18.6.31).-1-Chloroanthraquinone is sulphonated with 20% oleum at 155-160° in absence of Hg compounds to give 6- and 7-sulphonic acids, and thence 2:6- and 2:7-disulphonic acids; or in presence of HgO to give the 5-sulphonic and thence the 4:5disulphonic acid. The acid mixtures are separated by fractional crystallisation of Na salts, and converted into di- and tri-chloroanthraquinones or into amino- or alkylamino-anthraquinone-mono- and -di-sulphonic acids. The chloroanthraquinones melt at 203-205°  $(1:6), 212-214^{\circ}, (1:7), 224-225^{\circ}, (1:2:6), 236-238^{\circ}$ (1:4:6),  $221-222^{\circ}$  (1:2:7), and  $258^{\circ}$  (1:4:5), respectively. C. H.

**Preparation of hydroxyanthraquinones [quinizarins].** I. GUBELMANN and H. J. WEILAND (B.P. 373,999, 28.1.31).—Phthalic anhydrides are heated at 200° with 4-chlorobenzenediazonium sulphate (or a 3-substituted derivative) in presence of  $H_2SO_4$  and  $H_3BO_3$ . E.g.,  $p-C_6H_4Cl\cdot NH_2$  or  $3:4-C_6H_3Cl_2\cdot NH_2$ is diazotised in  $H_2SO_4$  and added with  $H_3BO_3$  to  $H_2SO_4$ at 200° to give quinizarin or 3-chloroquinizarin. C. H.

Manufacture of condensation products and dyes [from methyleneanthrone]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 373,129, 12.2.31).— 9-Methylene-10-anthrones are converted into alkali-sol. products by heating with acid condensing agents (alcoholic HCl,  $H_2SO_4$ , or HCO<sub>2</sub>H, metal halides), or into alkali-insol. products with stronger acid agents (AlCl<sub>8</sub>). The products give vat dyes (or, if sulphonated, acid dyes) by further heating, *e.g.*, in high-boiling solvents (ethylcarbazole,  $\beta$ -naphthol), or with caustic alkali,  $H_2SO_4$ , or AlCl<sub>3</sub>. Methyleneanthrone, when refluxed with MeOH-HCl gives a yellow compound, m.p. 254°, which is converted by AlCl<sub>3</sub> at 140° into an almost colourless product, and by AlCl<sub>3</sub> above 200° into 5:6:11:12-dibenzperylene-4:10-quinone (violet vat dye), C<sub>2</sub>H<sub>4</sub> being evolved. When H<sub>2</sub>SO<sub>4</sub> at 200° is used for the final condensation a violet-blue acid wool dye is formed. 2-Chloro-9-methylene-10-anthrone similarly gives a yellow product, m.p. 284°, and finally a red-violet vat dye. C. H.

Preparation of homologues and derivatives of phenanthridine. G. T. MORGAN and L. P. WALLS (B.P. 372,859, 2.3.31).-A 2-acylaminodiaryl having a free 2'-position is heated with a P halide, e.g., POCl<sub>3</sub> or PCl<sub>5</sub>, to give 6-substituted phenanthridines. The prep. of the following phenanthridines is described : 6-methyl-, m.p. 84°; 6-ethyl-, m.p. 55-56°; 6chloromethyl-, m.p. 135° (converted by NaI into 6iodomethyl compound, m.p. 157°; piperidinomethyl compound, m.p. 94°); 6-phenyl-, m.p.  $105-106^{\circ}$ ; 6-o-nitrophenyl-, m.p.  $122 \cdot 5^{\circ}$  [amine, m.p.  $168 \cdot 5^{\circ}$ (Ac derivative, m.p.  $185^{\circ}$ )]; 6-m-nitrophenyl-, m.p. 173° [amine, m.p. 159-161° (Ac derivative, m.p. 237.5°)]; 6-p-nitrophenyl-, m.p. 191° (amine, m.p. 197-199° (Ac derivative, m.p. 219°)]; 2-nitro-6-methylm.p. 200-201° [amine, m.p. 152° (Ac derivative, m.p. 240°)]; 8-nitro-6-methyl-, m.p. 244°. C. H.

Manufacture of organic compounds containing the sulphhydryl [thiol] group. SCHERING-KAHLBAUM A.-G. (B.P. 373,755, 5.5.31. Ger., 10.5.30).-Carbohydrate derivatives having reactive halogen, e.g., acetobromoglucose, are treated with a salt of a thioacid, and hydrolysed, whereby the halogen of the starting material is replaced by SH. Examples are : tetra-acetobromoglucose with K thioacetate in MeOH, giving penta-acetylthioglucose, m.p. 121°, converted by methyl-alcoholic NH3 into NH4 thioglucosate; acetobromoglyceraldehyde with K thioacetate, giving diacetylthioglyceraldehyde, m.p. 160°, hydrolysed to salts of thioglyceraldehyde; tetra-acetylthioxylose, m.p. 99°, and tetra-acetylthioarabinose, m.p. 78°, similarly prepared and hydrolysed. C. H.

Catalytic hydrogenation. PhOH-bearing liquors. Phenols etc. from tar.—See II. Wetting etc. agents—See VI.

# IV.-DYESTUFFS.

Acylaminoanthraquinone vat dyes from furoyl chloride. R. S. WILDER (J. Colo. Wyo. Acad. Sci., 1931, 1, No. 3, 23).—Light yellow to yellowish-brown furoyl derivatives of 1- and 2-amino-, 1-amino-2-methyl-, and 1:5- and 1:8-diamino-anthraquinone were prepared by boiling for 1 hr. in PhNO<sub>2</sub> or NH<sub>2</sub>Ph; the latter condenses to some extent with the furoyl chloride. CH. ABS.

Volumetric reduction of dyes with sodium hyposulphite. W. C. HOLMES, C. G. MELUN, and A. R. PETERSON (Amer. Dyestuff Rep., 1932, 21, 213). -25 dyes (various) were analysed by Siegmund's method (B., 1913, 16) using a standard solution of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> protected from oxidation by a H<sub>2</sub> gas-seal instead of a layer of paraffin oil, whereby the solution remained unchanged during 1 month; 13 of the dyes yielded results within 1.5% of those obtained by the TiCl<sub>3</sub> method, the best agreement being obtained with

readily reducible quinoneimine derivatives such as Thionine, Methylene Blue, Safranine, and Nile Blue A.  $Na_2S_2O_4$  solutions are less stable than those of TiCl<sub>3</sub>, and have less effective reducing power than has TiCl<sub>3</sub> for difficultly reducible dyes; the TiCl<sub>3</sub> method is preferred. A. J. H.

Oxidation of leuco-bases of CHPh, dyes .-- See XI.

See also A., Aug., 841, Azo dyes from hydroxyquinolines. 847, Fluoranthene derivatives. 861, Indigoid dyes.

#### PATENTS.

Manufacture of basic [polymethine] dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 373,160, 17.2.31).—The methylene base of a heterocyclic  $NH_4$ salt having an  $\alpha$ -Me group is heated at 100—110° with  $CCl_4$  or  $CBr_4$ . Examples are dyes from 2-methylene-1:3:3-trimethylindoline (red-violet), its 6-acetamidocompound, m.p. 146° (blue-violet), 2-methylene-3methylbenzthiazoline (brown-violet), and 2-methylenel-ethyl-1:2-dihydroquinoline (blue-violet). C. H.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 373,175, 18.2.31. Addn. to B.P. 339,267, B., 1931, 338).-An a-aminoanthraquinone (2 mols.) is condensed with a diphenylenedicarboxylic acid, incapable of anhydride formation and carrying a 2:2'-bridge comprising CO, CO · CH<sub>2</sub>, or 1 or 2 hetero-atoms. Examples are : a-aminoanthraquinone with the di-acid chloride of 4:4'-dicarboxydiphenylene oxide (greenish-yellow), of phenazone-3: S-dicarboxylic acid (greenish-yellow), of 5-ethylcarbazole-2:8-dicarboxylic acid (red-yellow), or of fluorenone-2:8-dicarboxylic acid; 1-amino-5benzamidoanthraquinone gives yellow to orange vat dyes, and 1-amino-4-methoxyanthraquinone with 4:4'dicarboxydiphenylene oxide orange. C. H.

[Manufacture of] dye preparations for cotton printing. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 371,848, 27.1.31. Addn. to B.P. 350,963; B., 1931, 1004).—In the process of the prior patent, β-arylsulphonamidoanthraquinones or the corresponding leuco-compounds or anthrones are used in place of the aminoanthraquinone. C. H.

Erratum.—For "B.P. 350,936" in B., 1931, 1004, col. 1, line 27, referred to in the above abstract, read "B.P. 350,963."

[Manufacture of] azo dyes [for leather]. WILLIAMS (HOUNSLOW), LTD., and H. ACKROYD (B.P. 373,689, 13.3. and 26.6.31).—Resorcinol-4-sulphonic acid (R) is coupled with suitable diazotised amines or diamines, or the corresponding resorcinol couplings are sulphonated. Examples are : sulphanilic acid  $\rightarrow$  R (yellow on leather); naphthionic acid  $\rightarrow$  R (red-brown); R  $\leftarrow$ benzidine  $\rightarrow$  H-acid  $\leftarrow$  sulphanilic acid (greenblack); NH<sub>2</sub>Ph  $\rightarrow$  resorcinol, trisulphonated with 20% oleum at 60° (yellow on wool); benzidine  $\rightarrow$ resorcinol (2 mols.), sulphonated (blue-red on leather); NH<sub>2</sub>Ph  $\rightarrow$  R  $\leftarrow$  p-nitroaniline, sulphonated (nutbrown). C. H.

Manufacture of [azo] dyes [containing metal]. Soc. CHEM. IND. IN BASLE (B.P. 374,186, 27.3.31. Switz., 27.3.30. Addn. to B.P. 357,543; B., 1932, 58).— Disazo dyes of the type: o-aminophenol  $\rightarrow$  J-acid  $\leftarrow$  arylamine, having at least 1 NO<sub>2</sub> group, are treated with agents yielding metal. Examples are chromed dyes from 5-nitro-o-aminophenol  $\rightarrow$  J-acid, coupled alkaline with diazotised 5-nitro-o-aminophenol (level blue-green on viscose), NH<sub>2</sub>Ph (violet-black), 4-nitro-oaminophenol-6-sulphonic acid (blue-green), p-nitroaniline (grey), or sulphanilic acid (violet-black). C. H.

Dyes from thioindoxyls. Methyleneanthrone dyes.—See III.

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

VIII. Effect of Moisture relations of cotton. processing. A. R. URQUHART, W. BOSTOCK, and N. ECKERSALL (J. Text. Inst., 1932, 23, 135-149 T; cf. B., 1926, 186).-Vals. for the hygroscopicity under different conditions of atm. humidity are given for samples of cotton cloth corresponding with the various stages in the normal bleaching, mercerising, and dyeing Hygroscopicity decreases slightly but processes. steadily as the duration and temp. of drying are increased, but if a damp sample is treated for a short time at not too high a temp. its subsequent hygroscopicity may be slightly greater than that of the untreated sample. The very great adsorptive capacity possessed by cotton immediately after the mercerisation process is impaired continuously as the material is dried down the primary desorption curve. Chemical attack reduces the hygroscopicity, but the reduction is small except where the attack is so serious as to cause almost complete loss of B. P. R. strength.

Distinguishing between old and new cotton fibres. A. W. WINNE and J. D. DONOVAN (Melliand Text. Month., 1932, 4, 298—299).—Examination under ultra-violet light sometimes enables conclusions to be drawn as to the age of cotton samples, but the method is unreliable if mixed samples of old and new cottons are in question, and other tests for the detection of oxycellulose and damage by heat, fungi, or mechanical means should be made. B. P. R.

Emulsion oiling of wool. J. B. SPEAKMAN and H. FRANKS (J. Text. Inst., 1932, 23, 154—162 T).— Oleine and olive oil wool creams applied to wool immediately before carding give far stronger yarns than do the corresponding "dry" oils under conditions of rapid working. Even when prolonged storage of oiled wool is customary the creams can give no worse results than ordinary oils, whilst under normal conditions of storage for a limited time they give superior results. B. P. R.

Mildewed wool. Comparison of some oil compounds for probable nutrient properties. A. H. GRIMSHAW (Melliand Text. Month., 1932, 4, 253-255, 300-303).—Olive and lard oils appear to act as sources of food supply for mildew obtained from wool, whilst mineral oil does not. B. P. R.

Measurement of fibre length. W. SEVER (J. Text. Inst., 1932, 23, 151–159 P). B. P. R.

Strength of textile fabrics and their satisfactiongiving qualities in conditions of normal use. J. G. WILLIAMS (J. Text. Inst., 1932, 23, 161–167 P).— The importance of the grab test as applied to fabrics and worn or tendered garments is discussed. B. P. R.

Testing of laundered fabrics. R. E. V. HAMPSON (J. Text. Inst., 1932, 23, 182-186 P).—Causes and methods of examination of defects in laundered goods are discussed. B. P. R.

Significance of results in textile testing. F. P. SLATER (J. Text. Inst., 1932, 23, 170-179 P).

B. P. R.

Depolymerisation of alkali cellulose. III. Changes of the cellulose in the viscose process. S. M. LIEPATOV and N. A. KROTOVA (J. Appl. Chem., Russia, 1931, 4, 1030—1035). — The changes are explained by destruction of the micelle structure preceded by gradual weakening of the attraction between individual chains. CH. Abs.

Solubility curves for nitrocellulose. I. SAKURADA and M. TANIGUCHI (J. Soc. Chem. Ind., Japan, 1932, 35. 253-256 B).-Fractional dissolution of nitrocellulose is less simple than that of cellulose acetate, but there is substantially the same relationship between solvent power and the polar nature of the solvent. Dissolution curves again show the characteristic S-form, and are independent of the nature of the nitrocellulose used. In general they are 25-30 vol.-% lower than corresponding curves for cellulose acetate. With a common non-polar solvent the MeOAc curve lies 9 vol.-% higher than that of  $\rm COMe_2$ , whilst the product of the dipole moment ( $\mu$  × 10<sup>18</sup>) and the concn. for 50% solution was const. in each case. These curves give a direct comparison of the solvent power of the polar solvents V. E. Y. present.

Analysis of mixed cellulose esters. H. TATU (Russa, 1932, 7, 115-121, 251-255).-The NHPh, test is satisfactory for the detection of NO3', and the nitrometer method for its determination, provided that a correction is applied for the solubility of NO<sub>2</sub> in conc.  $H_2SO_4$  (3.7 vol.-%). For the detection of Ac, 2 g. of cellulose acetate nitrate are treated for 15 min. with H<sub>2</sub>O (500 c.c.) and H<sub>2</sub>SO<sub>4</sub> (d 1.63-1.67, 5 c.c.); H<sub>2</sub>O (300 c.c.) is then added and the liquid distilled at const. vol. until all volatile acid is removed. The distillate is neutralised with NaOH (litmus) and evaporated to dryness; the abs. EtOH extract is filtered, evaporated to dryness, dissolved in H<sub>2</sub>O (10-15 drops), and tested (microscope) with U formate and HCO<sub>2</sub>H for Na U acetate. Billing and Tinsley's method for the determination of Ac gives high results unless the abovementioned correction is applied. CH. ABS.

Measurement of surface hardness of cellulose derivatives. S. E. SHEPPARD and J. J. SCHMIDT (Ind. Eng. Chem. [Anal.], 1932, 4, 302—304).—A scratch dynamometer and apparatus for observing the scratch are described. Results are given, illustrating the effect of plasticisers on the scratch susceptibility of a lowviscosity nitrocellulose. The characteristic effect of camphor is well shown. E. S. H.

Viscose. XLIII. Viscosity of viscose. S. IWABAKI and E. SUGINO (J. Soc. Chem. Ind., Japan, 1932, 35, 244-247 B).—Small additions of glucose or biosan caused considerable fall in the viscosity of viscose, but with when biosan was added.

V. E. Y.

Spinning of viscose silk. Incomplete coagulation. I—III. S. HASE (J. Soc. Chem. Ind., Japan, 1932, 35, 218—220 B).—The characteristic properties of a viscose thread incompletely coagulated in a bath containing  $H_2SO_4$ ,  $Na_2SO_4$ ,  $MgSO_4$ , and  $ZnSO_4$  are enumerated, and a method for estimating the degree of incompleteness of the coagulation is given. The various causes of incomplete coagulation are discussed. V. E. Y.

Physical test of viscose silk. Z. KAWATA (J. Soc. Chem. Ind., Japan, 1932, 35, 220—221 B).—The strength and elongation of viscose silk were recorded on the "Seligraph." The effect of acid treatment is shown graphically. When a sample is tested under various conditions of temp. and humidity, the area enclosed by the curve (work done) is const. The work done per denier (area/denier), and the ratio of work done in dry and wet states, are also const. for a given sample under sp. conditions. A sample is good when two factors are large. Stretch-spinning increases the fibre strength, but decreases elongation and work done. After desulphurisation with Na<sub>2</sub>S, the elongation increases, the work done decreases slightly, but the strength shows little change. V. E. Y.

Swelling of acetate silk in aqueous solutions. L. MEUNIER and G. RÉVILLON (Chim. et Ind., 1932, 27, 251-262).-Although the H<sub>2</sub>O absorption of commercial cellulose acetate is higher than that of the triacetate, it rarely exceeds 25% at 15° compared with 70-80% for viscose. The swelling of acetate silk in H<sub>2</sub>O results from the attraction between the H<sub>2</sub>O and the free OH groups in the cellulose ester, and differs from that observed in the case of conc. aq. solutions, which definitely disperse the ester. The absorption of both weakly and strongly basic substances by Rhodiaseta 100-denier silk (54% OAc) was investigated. Saturated aq. NH,Ph did not hydrolyse the silk, whilst at concns. up to 2.4% the partition coeff. between silk and solution remained const. at 18.2%. Aq. pyridine up to 28% concn. effected no chemical change in the fibre; above this concn. saponification was observed. Aq. HCO<sub>2</sub>H (0-7N) reduced the OAc content from  $54 \cdot 23$ to  $50.30^{\circ}$ , according to the concn. of the acid used, after 15 days' treatment at 25°. In aq. COMe<sub>2</sub> swelling increases with increase in COMe2 content up to 35%, at which point dispersion of the fibre commences; the amount of COMe2 absorbed under equal conditions decreases with progressive hydrolysis of the fibre. Max, swelling in NH<sub>4</sub>CNS was observed with a 40% solution. In aq. solutions of N(C2H4.OH)3 acetate silk suffers no change after 1 hr.: the change observed after prolonged treatment depends also on the temp. and concn. of the solution. Naphthol AS dissolved in N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub> is absorbed by the fibre. In aq. EtOH max. swelling was recorded with 75% EtOH. V. E. Y.

[French] Colonial woods. P. MARMASSE (Assoc. col. sci. Comité nat. bois col., 1931, 71 pp.).—Vals. of d,

ash, cold-H<sub>2</sub>O extract, COMe<sub>2</sub>-sol. matter, pentosans (Brown), cellulose (Cross and Bevan), lignin (Willstätter), and C and H in lignin are recorded for wood of *Khaya* 

ivorensis, A. Chev.; Dicorynia paraensis, Benth.; Turreanthus africanus, Pellegr.; Lophira procera, A. Chev.; Piptadenia africana, Hook; Dialium sp.; Vitex pachyphylla, Baker; Chlorophora excelsa, Bentham et Hooker; Mimusops Heckeli, H. Lee; Symphonia globulifera, L.f.; and Tectona grandis, L.f. CH. ABS.

Botanical classification of cellulose sources for paper-making. J. STRACHAN (Paper-Maker, 1932, 84, 241-243 T.S.).—Some 20 botanical families are classified, all of which are characterised by the presence in appreciable quantity of fibrous cellulose, derived from bark, bast, stem, or leaf, and of satisfactory length and strength for paper-making. H. A. H.

Synthetic lignin. L. F. HAWLEY and E. E. HARRIS (Ind. Eng. Chem., 1932, 24, 873—875; cf. B., 1931, 387).—Purified wood cellulose heated in sealed tubes for several days at 135° yields material ("synthetic lignin") which, as regards its resistance to hydrolysis, chlorination-sulphite reaction, ultra-violet absorption spectrum, and reducing val., resembles natural lignin very closely. B. P. R.

Determination of hemicelluloses by oxidation with potassium dichromate. A. JÄGER (Chem.-Ztg., 1932, 56, 570—571).—Variation in results when hemicelluloses in artificial-silk press liquors are determined by  $K_2Cr_2O_7$  oxidation at varying temp. and periods of heating is shown to be due to imperfect oxidation at lower temp. Even glucose is not completely oxidised in 5 min. at 100—107°. It is recommended that 5-c.c. samples be heated with a mixture of 10 c.c. of 8%  $K_2Cr_2O_7$  solution and 15 c.c. of conc.  $H_2SO_4$  for 5 min. at 125—135°, followed by dilution and titration. Heating of more dil. reaction mixtures must be longer, and of mixtures kept at 100° must be very much longer. E. W. W.

Corrosion in a groundwood pulp mill. I. H. ANDREWS (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 34—35).—Corrosion-resistant metals were found to be satisfactory in service. Wrought Fe is superior to wrought steel. It is not practicable to reduce corrosion by neutralising the acids in the pulp with alkali, owing to the darkening in colour, even when the  $p_{\rm H}$  is kept below 5.5. H. A. H.

Maintaining uniform cooking acid conditions [for sulphite pulp]. J. P. V. FAGAN (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 27).—Recent advances in materials and methods employed for sulphite-pulp manufacture, especially on the Pacific Coast of U.S.A., are reviewed. H. A. H.

Development of a rational cooking system for sulphite[-pulp] digesters. W. H. SWANSON, L. LANG, and B. F. SMITH (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 19—24).—A system is described which permits of accurate control during cooking. Numerous advantages are claimed. H. A. H.

Process improvements and controls in sulphite [-pulp] cooking practice. W. N. KELLY (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 28–29).—The different variables existing during its manufacture are described and methods of control indicated. H. A. H.

Chlorination of wood pulp. R. B. HANSEN (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 30-32).—In presence of lignin HOCl acts as a chlorinating agent and yields the same reaction products as does  $Cl_2$  alone.  $Cl_2$  causes oxidation of purified cellulose since lignin and similar material are no longer present. The removal of lignin by  $Cl_2$  results in a more uniform wood pulp in greater yield than that produced by bleach liquor. The batch system of chlorination is superior to the continuous system; reasons are given. H. A. H.

Pitch problems in sulphate [pulp and paper] mills. W. F. GILLESPIE (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 37—39).—Factors affecting pitch production are considered, the most important being correct adjustment of  $p_{\rm H}$ . H. A. H.

Concentration of waste sulphite liquor by submerged combustion. K. A. KOBE (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 33-34).—Preliminary experiments indicate that many difficulties arising from other methods of concn. may be overcome by this method. H. A. H.

Use of glucose in the paper industry. H. WREDE (Papier-Fabr., 1932, 30, 463-464).—Addition of glucose to the stuff in the beater induces more rapid "hydration." Surface application by spraying increases pliability and resistance to folding and twisting; surface-parchmentised papers so treated are less subject to curling. Glucose is of great val. in the case of papers for sacks to be filled with hot cement, these remaining more pliable. T. T. P.

Effect of white-water on [paper-]sheet properties. E. R. SCHAFER (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 16–18).—The presence of white-H<sub>2</sub>O fibre in book paper up to 2% and in newsprint paper up to 5% has no effect on the physical properties; neither has clay introduced into book paper from white-H<sub>2</sub>O. An increase in strength occurs if white-H<sub>2</sub>O is replaced by fresh H<sub>2</sub>O, owing to the reduced retention of filler, but if the same amount of filler is furnished to the stock to compensate for such reduction, no appreciable change in strength results. H. A. H.

Ink-resistance of printing papers. B. L. WEHMOFF (Paper Trade J., 1932, 94, No. 13, 39–42).—Increase in the  $SO_3''$  content of newsprint increases the tensile and bursting strengths, the basis wt. for a given thickness, and the gloss, but decreases the ash content and opacity; differences between bleached and unbleached sulphite are recorded. The higher is the groundwood content, the more rapidly is ink absorbed. Newsprint with bleached sulphite is more absorbent than that with unbleached sulphite. Penetration is least with 100% groundwood paper, and increases with increase in sulphite content. CH. ABS.

Estimating stability of paper by heating. R. H. RASCH and G. O. STONE (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 40-42).—In conducting accelerated ageing tests on paper by determining the loss in foldingendurance after heat-treatment, the accuracy is affected by the lack of homogeneity of paper, variations between individual folding instruments, non-uniformity in drying conditions, and variations in R.H. during testing. If these are carefully controlled, satisfactory duplication may be expected. H. A. H.

Microscopical investigation of the structure of paper. K. ALBRECHT (Papier-Fabr., 1932, 30, 473— 475).—The structure and texture of paper are best examined microscopically when the surface is illuminated at a very small angle of incidence. The Busch illuminator, giving dark-ground illumination, is described. The usual type of vertical illuminator is of use in certain cases, but reflexion from the paper surface may render structure difficult to see. Photomicrographs are given. T. T. P.

See A., Aug., 836, Vanillin as colour reagent. Higher fatty acid esters of cellulose. 852, Fir lignin. Conifers. 888, Lignins from cereal straws.

#### PATENTS.

**Carbonisation of rags.** M. GESSNER (B.P. 374,796, 1.1.32. Ger., 2.1.31).—The carbonising acid, e.g., 8-10% of HCl ( $d \ 1\cdot 18$ ) on the wt. of dry rags, is atomised into a rotating drum through a nozzle located in one of the axles. Carbonisation is effected at  $> 140^{\circ}$  and is complete in  $\frac{3}{4}-1\frac{1}{4}$  hr. D. J. N.

Working up waste liquors of the cuprammonium silk stretch-spinning process. BRIT. BEMBERG, LTD. (B.P. 374,556, 31.3.31. Ger., 12.4.30. Addn. to B.P. 361,240; B., 1932, 98).—A tervalent metal salt solution, e.g., aq.  $Fe_2(SO_4)_3$ , is added to react with free NH<sub>3</sub> and give a ppt. of  $Fe(OH)_3$  which carries down  $Cu(OH)_2$ and the colloidal cellulose associated therewith. The sludge is separated and treated with sufficient dil.  $H_2SO_4$ to dissolve the  $Cu(OH)_2$ , leaving an insol. residue of  $Fe(OH)_3$ , basic Fe sulphate, and cellulose, from which  $Fe_2(SO_4)_3$  is regenerated by extraction with a further quantity of dil.  $H_2SO_4$ . The acid used in each case is preferably that obtained as waste during the hardening of the filaments. D. J. N.

Manufacture of cellulose acetate film. A. F. SULZER, ASST. to EASTMAN KODAK CO. (U.S.P. 1,833,136, 24,11,31. Appl., 8.4.29).—If in the hydrolysis of primary cellulose acetate to an COMe<sub>2</sub>-sol. product the reaction is stopped when 90% or more of the sample ppts. in a 40 : 60 (by vol.) H<sub>2</sub>O-COMe<sub>2</sub> mixture at 20°, the resulting cellulose acetate gives clear, flexible, H<sub>2</sub>O-resistant films which lie flat and are particularly suitable for photographic purposes. A method of determining this point in the hydrolysis is described. D. J. N.

Digester system [for wood pulp]. W. H. SWANSON and L. LANG, ASSIS. to PAPER PATENTS CO. (U.S.P. 1,831,206, 10.11.31. Appl., 6.3.31).—A heating and circulating system is described in which the liquor is drawn off at a no. of points near the top of the digester, passed upwardly through a vertical heat exchanger, and injected through a series of jets into the bottom of the digester. Provision is made for automatic control of temp. and pressure according to any predetermined time-temp. schedule. D. J. N.

Preparation of  $\alpha$ -cellulose pulp. C. A. BLODGETT and H. H. HANSON, Assrs. to EASTERN MANUFG. Co.

(U.S.P. 1,831,435, 10.11.31. Appl., 12.2.26).—An undercooked (" prime strong ") sulphite pulp is digested in a rotary boiler with about 15 times its wt. of a liquor containing 1.5% each of NaOH and Na<sub>2</sub>SO<sub>3</sub> at a temp. rising, e.g., to 133° in  $2\frac{3}{4}$  hr. D. J. N.

Manufacture of low-viscosity cellulose fibre. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,831,197, 10.11.31. Appl., 20.1.28).—Cellulosic material, in bulk or in sheet form, is impregnated with about twice its wt. of an alkaline solution of < mercerising conc., e.g., 1-5% aq. NaOH, and exposed to air or other O<sub>2</sub>-containing gas in admixture with H<sub>2</sub>O vapour at room temp. or above until depolymerisation of the cellulose has reached the desired degree. For high- $\alpha$ -cellulose wood pulp impregnated with 3% aq. NaOH, exposure to a current of moist air at 100° for 30 min. is sufficient. Alternatively, a moist atm. containing 50% each of air and NH<sub>3</sub> may be employed. D. J. N.

Hydrolysis of cellulosic substances [for production of fermentable liquid]. J. LEFRANC, ASST. to W. E. CELESTIN (U.S.P. 1,831,419, 10.11.31. Appl., 25.1.27. Fr., 20.5.26).—Sawdust is mixed with an equal wt. of 10% aq. H<sub>2</sub>SO<sub>4</sub>, dried to a H<sub>2</sub>O content of 50—60% at 100—120°, and heated in a digester at 120—130° for 2—3 hr., a fluoride, *e.g.*, CaF<sub>2</sub>, being added to replace the whole or a part of the free H<sub>2</sub>SO<sub>4</sub> by HF. When hydrolysis is complete the acid is neutralised and pptd. by the addition of Ca(OH)<sub>2</sub> or sugar skimmings (CaCO<sub>3</sub>), after which the sugars are recovered in a form suitable for fermentation by lixiviation and filtration. D. J. N.

Preservation of vegetable material in storage. T. B. MUNROE and E. C. LATHROP, Assrs. to CELOTEX Co. (U.S.P. 1,833,493, 24.11.31. Appl., 26.12.28).— Vegetable material containing sufficient  $H_2O$  to support the growth of micro-organisms which generate heat, *e.g.*, raw bagasse fibres containing 55—60% of  $H_2O$ , is treated with 1—1.25% (on the dry wt.) of  $H_3BO_3$ , pressed into bales, and suitably stacked into piles to prevent excessive rise of temp. at any point. During the initial stages of storage, fermentation sets in and the resulting rise in temp. causes loss of  $H_2O$  and hence concn. of the  $H_3BO_3$  to the point where it inhibits further fermentation which might deleteriously affect the cellulose.  $H_3BO_3$  may be replaced by an equiv. (as regards toxicity) quantity of other antiseptics.

# D. J. N.

Manufacture of paper-making material. K. WATANABE (U.S.P. 1,833,804-5, 24.11.31. Appl., 1.11.29. Jap., 23.2.29).—Old printed paper is kneaded with aq. NaOH, the ink is removed by means of an emulsion of (A) turpentine oil, (B) decalin or tetralin, and the washed pulp is used again. B. P. R.

Manufacture of paper-making material. N. OSAWA, ASST. to K. WATANABE (U.S.P. 1,833,852, 24.11.31. Appl., 1.11.29. Jap., 23.1.29).—Old printed paper is treated with aq. NaOH, an aq. emulsion of camphor oil and soap is added to remove the ink and size, the whole is kneaded, the liquor separated off, and the white pulp remaining is used for making paper. B. P. R.

Cellulose sheet. M. GENTILE, Assr. to S. W. WEIS, G. PICK, and N. SCHWARTZ (U.S.P. 1,832,978, 24.11.31. Appl., 22.4.29).—A flexible,  $H_2O$ - and grease-proof material suitable for wrapping foodstuffs is obtained by treating any type of paper or cardboard with a transparent nitrocellulose lacquer containing a softener, *e.g.*, tolyl phosphate. D. J. N.

Decalcomania [transfer] paper for offset printing. F. B. DEHN. From DECORATIVE DEVELOPMENT, INC. (B.P. 374,519, 13.3.31).—The body paper is coated first with a H<sub>2</sub>O-sol. coating (starch, gum, etc.) and then with a H<sub>2</sub>O-insol. coating (cellulose acetate or nitrate) containing a drier (Co or Mn linoleate) and a finelydivided filler. Suitable (lacquer-type) formulæ are given for the H<sub>2</sub>O-insol, coating. D. J. N.

Treatment of residual liquors [from sulphitepulp processes]. L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,833,313, 24.11.31. Appl., 14.1.24).—Spent liquors from digestions in which Na<sub>2</sub>SO<sub>3</sub> either alone or in admixture with NaOH or NaHSO<sub>3</sub> is used are conc., incinerated to recover volatile by-products, and smelted to give a mixture of Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>S. This product is dissolved in H<sub>2</sub>O and treated with CO<sub>2</sub> to convert Na<sub>2</sub>SO into Na<sub>2</sub>CO<sub>3</sub>, the H<sub>2</sub>S thus set free being burned to SO<sub>2</sub> which is subsequently used to convert Na<sub>2</sub>CO<sub>3</sub> into Na<sub>2</sub>SO<sub>3</sub> with recovery of CO<sub>2</sub>. Losses of Na compounds and S are made good by adding, *e.g.*, Na<sub>2</sub>SO<sub>4</sub> before smelting and burning S to SO<sub>2</sub>. CO<sub>2</sub> is produced in excess. By causticising part of the Na<sub>2</sub>CO<sub>3</sub>, and NaHSO<sub>3</sub> in any desired proportion may be obtained. D. J. N.

[Slag wool] blow chamber.—See I.

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

[Uses of] ammonium sulphate [in dyeing]. H. E. MILLSON (Melliand Text. Month., 1932, 4, 42-44, 121-124, 189-191, 251-252).-Chemical analysis and  $p_{\rm H}$  measurements show that during the boiling of aq.  $(NH_4)_2SO_4$  decomp. with consequent escape of  $NH_3$ does not occur (only free NH<sub>3</sub> in the salt is evolved); hence the explanation, that the levelling and exhausting powers of  $(NH_4)_2SO_4$  on the dyeing of wool with acid dyes, particularly the Sulphon Cyanines, are due to escape of NH<sub>3</sub> and consequent acidification of the dye liquor, is not accepted. Similarly, it is shown that loss of NH<sub>3</sub> is not responsible for the exhausting power of NH<sub>4</sub>OAc when present in dye liquors containing Buff F Wool Blue B; equal exhaustions are obtained with 6% of  $(NH_4)_2SO_4 + 0.5\%$  of  $NH_4OAc$ , 8% of  $NH_4OAc$ , and 2.5% of AcOH (56%). A further series of tests were made using F. Crimson GR in baths containing  $(NH_4)_2SO_4$  and  $H_2SO_4$ .  $(NH_4)_2SO_4$  is preferred to  $Na_2SO_4$  for exhausting in dyeing cellulose acetate silk with Cellit dyes, and is useful for securing good penetration in the dyeing of hard spun wool yarn with acid dyes, and wool-cotton mixtures with union dyes. Formulæ are given for fireproofing textile materials, using  $(NH_4)_2SO_4$ . A. J. H.

Dyeing of wool. GURLT (Rev. Gén. Mat. Col., 1932, 36, 241—246).—The selection of dyes for satisfying the various requirements as regards fastness in processing and in wear for different types of wool materials are discussed. A. J. H. Specification of colour on dyed fabrics by spectro-analysis. E. M. SHELTON and R. L. EMERSON (Ind. Eng. Chem. [Anal.], 1932, 4, 248—253).—The colours of samples of dyed fabrics can be recorded in terms of spectrophotometric curves, using a modified form of the General Electric Co.'s colour analyser. Technique is described for the recording of colour of individual dyes, standard colour lines, commercial matches, and loss of colour during fastness tests. Wider applications are suggested : *e.g.*, the tarnishing effect of velvet on Ag may be determined by examining the colour of a Ag disc before and after standard exposure.

E. S. H.

Practical notes on dyeing aniline black. A. J. HALL (Amer. Dyestuff Rep., 1932, 21, 299–302, 319– 321). A. J. H.

Overcoming shady dyeings on hosiery. N. D. WHITE (Melliand Text. Month., 1932, 4, 101–102).— Various causes of uneven dyeing of real and artificial silk hose are discussed. The addition of  $Na_2HPO_4$  to the dye bath is claimed to overcome the defect. A. J. H.

# PATENTS.

Treatment [steaming and moistening] of yarns and the like. MASCHINEN- & APPARATE-BAUANSTALT G.M.B.H. (B.P. 375,223, 8.1.32. Ger., 4.2.31).—Yarn wound on bobbins is successively steamed and moistened while being carried along conveyor belts through a 2-compartment chamber, which is described. A. J. H.

Treating [scouring] textile fabrics. L. MELLERSH-JACKSON. From H. W. BUTTERWORTH & SONS CO. (B.P. 375,156, 29.9.31).—Fabric is treated with a boiling alkaline liquor while passing continuously through a J-shaped tubular box, inclined at 30° to the vertical, contained within a kier. A. J. H.

Vat dyeing. F. L. REMLEIN, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,832,425, 17.11.31. Appl., 21.9.27).—Cotton fabric is padded with a solution containing the vat dye in its leuco-form together with glue (as a dispersing agent) and anthraquinone (antioxidant), then dried on drying cylinders for development of the fully oxidised shade, and afterwards soaped at the boil. A. J. H.

Transferring water-soluble dyes to fabrics and porous surfaces. H. S. SADTLER (U.S.P. 1,832,661, 17.11.31. Appl., 21.9.27).—The surface to be decorated (fabric or tile) is coated with a solution of gum tragacanth or gum karaya containing NaOBz as an antiseptic and while moist is pressed in contact with a transfer bearing a design in  $H_2O$ -sol. dyes. A. J. H.

Coloration of textile materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 373,628, 20.2.31).—Thiocarbamide is applied before, during, or after dyeing and retained in the material. Dyeings fast to light and acid are thus obtained on acetate silk with 1:4-di(methylamino)anthraquinone and other aminoanthraquinones. C. H.

Production of [discharge] effects on materials made of or containing organic derivatives of cellulose. BRIT. CELANESE, LTD., J. ALLAN, and G. H. ELLIS (B.P. 373,653, 19.2.31).—The discharge, especially with  $CH_2O$ -sulphoxylates, is more complete on steaming when the discharge paste contains an org. or inorg. ester, *e.g.*, triacetin, ethylene chlorohydrin, glycerol mono- or di-chlorohydrin, Et tartrate, KEtSO<sub>4</sub>, etc. C. H.

Cotton printing by means of sulphur dyes. A. CARPMAEL, and I. G. FARBENIND. A.-G. (B.P. 373,212, 12.2.31).—A sulphide dye is pasted with glycerol, glycol, thiodiglycol, or thiodiglycerol, and a hydrotropic agent (carbamide, thiocarbamide, Na benzylaniline-*p*sulphonate, Na tetrahydronaphthalene-2-sulphonate, etc.). Sulphide colours such as Hydron Blue and Indocarbon CL may thus be printed like vat dyes. C. H.

Manufacture of softening agents for textile goods containing artificial silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 374,029, and Addn. B.P. 374,043, 27.2.31).—(A) The handle of the goods is improved by treatment with a mixture of a H<sub>2</sub>O-insol. fatty acid amide (except N-hydroxylalkyl derivatives) and a sulphonated alcohol  $C_{12}$ — $C_{30}$ . Examples are : palmitamide with sulphonated dichlorohydroxy-hard paraffin  $C_{24}$ — $C_{27}$ ; stearamide with lauryl H sulphate; palmitamide with sulphonated cetyl and oleyl alcohols. (B) The sulphonated alcohol is replaced by a sulphonated oleamide or N-substituted derivative. C. H.

[Treatment of] textile materials [with wetting agents]. A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 372,855, 27.2.31).—Esters (above  $C_2$ ) of sulphoaromatic dicarboxylic acids (cf. B.P. 371,144; B., 1932, 763) are used as wetting agents etc. in carbonising, mercerising, dyeing, and cleaning of textiles.

C. H.

Treatment of natural and artificial cellulosic fibres with alkali [wetting agents for mercerising liquors]. CHEM. FABR. VORM. SANDOZ (B.P. 374,214, 23.4.31. Ger., 23.4.30).—To the phenolic wetting agents for mercerising liquors there is added a sulphonated fat, fatty oil, fatty acid, or aromatic hydrocarbon. Examples are : tech. xylenol, diglycol mono-*n*-butyl ether, and sulphonated ricinoleic acid; tech. cresol, methylcyclohexanol, and sulphonated ricinoleic acid. C. H.

Treatment [increasing the heat-resistance] of [artificial silk] textile materials. BRIT. CELANESE, LTD., G. H. ELLIS, and R. C. STOREY (B.P. 374,049, 20.2.31).—The safe ironing temp. of cellulose ester and ether fibres, and the heat-resistance of cellulose and partly hydrolysed cellulose ester fibres, are increased by deposition therein of a comparatively small quantity of an insol. metal compound by double decomp., with or without further impregnation with fireproofing substances such as mixtures of  $(NH_4)_2B_4O_7$  or  $(NH_4)_3PO_4$  with NH<sub>4</sub>Br. Formation of the insol. compound is effected at a high temp.  $(75-95^\circ)$  so that it is present in the fibres in a voluminous form ; salts of Sn, Al, Ca, Ba, Fe, Mg, and Cr are used for this purpose. A. J. H.

Treatment [coating] of fabrics [with pyroxylin]. M. GOLDBERG (B.P. 374,983, 30.3.31).—Fabrics are backed or cemented together by means of a composition consisting of pyroxylin (8 pts.), a softening agent such as castor oil (1 pt.), and an ester of phthalic acid (1 pt.) in a solvent (e.g., Et phthalate) dissolved in MeOH and  $C_6H_8$ . A. J. H.

Washing etc. agents.—See III. Cotton printing.—See IV. Gum solutions.—See XVII.

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

Manufacture of sulphuric acid and its salts by means of magnesium carbonate. V. SCHKATELOV (J. Appl. Chem., Russia, 1931, 4, 51–54).—In the manufacture of  $H_2SO_4$  by means of the reactions:  $CaSO_4 \rightarrow CaS$  and  $CaS + MgCl_2 + H_2O \rightarrow CaCl_2 + H_2S$ + MgO and catalytic oxidation of the  $H_3S$ , MgCl\_2 is obtained from dolomite by the reaction:  $CaCl_2 + H_2O$ + MgO +  $2CO_2 \rightarrow Ca(HCO_3)_2 + MgCl_2$ , under pressure. By continuing the reactions  $MgSO_4 + 2NaCl + MnO_2$  $\rightarrow Na_2SO_4 + Cl_2 + MgO + MnO$  and  $CaSO_4 + H_2O +$ MgO +  $2CO_2 \rightarrow Ca(HCO_3)_2 + MgSO_4$ , a saturated solution of  $MgSO_4$  is obtained. Then  $MgSO_4 + 2NaCl$  $\rightarrow MgCl_2 + Na_2SO_4$ . HCl and  $HNO_3$  may be obtained by interaction of  $MgSO_4, H_2O$  with NaCl or NaNO<sub>3</sub>, affording MgO and the acid. CH. ABS.

Chamber process. XXIII. Physical and mechanical tests of lead sheets. XXIV. Corrosion of lead sheet by sulphuric acid. M. MATSUI and H. KATO (J. Soc. Chem. Ind., Japan, 1932, 35, 304—306B, 307— 308B).—XXIII. The Brinell hardness (measured in a special device designed for testing very soft material), tensile strength, elongation, and m.p. of 7 samples of Pb, Pb–Sb, and Pb–Bi–Cu sheet are recorded. Annealing at 100° increases both the tensile strength and elongation. The flash temp. of the specimens, *i.e.*, the temp. at which the sheets dissolved suddenly when immersed in conc.  $H_2SO_4$  of rising temp., ranged from 252° for a 99.98% Pb sheet to 300° for a sample containing 4—5% Bi and 1% Cu; for the alloy Pb 95, Sb 5% the flash temp. was 270°.

XXIV. The comparative rates of corrosion of the samples in  $H_2SO_4$ , in  $NO \cdot SO_2 \cdot OH$ , and in the gases passing from the last chamber to the Gay-Lussac tower have been determined. Annealing the sheets at 100° markedly increases the rate of corrosion. The Pb-Sb alloy is less resistant than are the other samples to  $H_2SO_4$  and gases containing NO. H. F. G.

Synthetic production of nitric acid of high concentration. G. FAUSER (Giorn. Chim. Ind. Appl., 1932, 14, 348-355).-HNO3 obtained by absorbing nitrous gases in H<sub>2</sub>O, even when pressure or a low temp. is used, is at most 60-68% and requires a dehydrating agent for its concn. The reaction  $2H_2O + 2N_2O_4 + O_2 =$  $4HNO_3 + 18.8$  cal., which occurs under pressure and requires liquid  $N_2O_4$ , occupies 12 hr. at 15° or 4 hr. at 70°, higher temp. involving plant difficulties. If a slight excess of  $N_2O_4$  is used, 97–98% acid is obtainable. Denitration is effected at atm. pressure, the nitrous acid passing down a Raschig ring-filled rectifying column surmounted by a reflux condenser; the liberated N<sub>2</sub>O<sub>4</sub> is condensed and returned to process. The H<sub>2</sub>O used in the reaction may be replaced by dil. HNO3. This process has not been worked on a large scale, as the prep. of pure N<sub>2</sub>O<sub>4</sub> has not been satisfactorily accomplished. The best results are obtained by oxidising NH<sub>3</sub> to NO and, before much further oxidation to N2O4 occurs, rapidly cooling the gases so as to condense part of the  $H_2O$  formed without appreciable loss of  $HNO_3$ ; this procedure is possible only at atm. pressure. A diagram shows the layout of the plant (of Cr-steel), and the energy required for compressing the nitrous gases and for cooling the gases is cal. The cost per ton of  $HNO_3$ ( $1H_2O$ ) by this direct synthesis of the conc. acid is 207 lire, the figure for the older process, in which the dil.  $HNO_3$  is conc. with  $H_2SO_4$ , being 370 lire. T. H. P.

Thermal properties of ammoniates and similar compounds and their use in refrigerators. R. PLANK and L. VAHL (Forsch. Gebiet Ingenieurwes., 1931, A,2, 11—18; Chem. Zentr., 1932, i, 1403).— Conditions for the use of solid absorbents for NH<sub>3</sub> are discussed. Log p - 1/T curves for CaCl<sub>2</sub>,8NH<sub>3</sub> and 4NH<sub>3</sub> are given, and partial heats of formation are tabulated for 0—100°. Advantages of NH<sub>2</sub>Me are considered. NH<sub>2</sub>Me has sp. heat 0.77 at 20°; at —10° the latent heat of evaporation is two thirds of that of NH<sub>3</sub>. In absence of O<sub>2</sub> and H<sub>2</sub>O, NH<sub>2</sub>Me does not attack Cu. The compound CaCl<sub>2</sub>,6NH<sub>2</sub>Me and the LiCl methylaminates were studied. A. A. E.

Rapid determination of free sulphuric acid in ammonium sulphate. E. KÖNIG (Brennstoff-Chem., 1932, 13, 265–266).—A 10-g. sample of the salt is taken from the centrifuge, dissolved in 100 c.c. of H<sub>2</sub>O, and titrated with 0.1N-NaOH, using a dropping-bottle calibrated to deliver drops of 0.05 c.c. and as indicator a Me-red-methylene-blue mixture. The colour change is red-violet  $\rightarrow$  dirty green  $\rightarrow$  bright green.

A. B. M.

Determination of chlorate in nitrates. K. LESNIČENKO (Chem. Listy, 1932, 26, 260–263).—2 c.c. of the solution, containing  $\gg 30\%$  NH<sub>4</sub>NO<sub>3</sub> or 40% NaNO<sub>3</sub>, are added to 6 c.c. of a solution of 5 g. of NH<sub>2</sub>Ph and 0.05 g. of AgNO<sub>3</sub> in 100 c.c. of HCl (d 1.12) and the concn. of ClO<sub>3</sub>' is determined colorimetrically after 60 min. This method is applicable in the presence of ClO' and of BrO<sub>3</sub>', but not of IO<sub>3</sub>'. The optimal concn. of KClO<sub>3</sub> is 0.1% but  $\ll 0.0001\%$  solutions may be taken. R. T.

Electrochemical production of sodium hydrosulphite. I. M. A. RABINOVITSCH and A. S. FOKIN. II. P. B. SHIVOTINSKI, A. P. MASCHOVETZ, and A. S. FOKIN. III. P. B. SHIVOTINSKI (Ukrain. Chem., J. 1931, 6, [Tech.], 191–204, 205–211, 212–219).—I. SO<sub>2</sub> is reduced at a Hg-Na cathode, in which Na is renewed electrolytically. The yield of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> is 70–80% of theory, and any desired concn. can be obtained. The chief source of loss is due to the reaction :  $2Na_2S_2O_4 \rightarrow$ Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> + Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>. Aq. Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> is stabilised by Na<sub>2</sub>SiO<sub>3</sub>.

II. A process for the industrial production of  $Na_2S_2O_4$ on the above principle is described, giving a yield of 55—60% on the current used. Variation of temp., c.d., and agitation does not appreciably affect the results, the most important factor being regulation of the SO<sub>2</sub> supply.

III. The above process can be carried out in apparatus of the Solvay type. R. T.

Calcium gluconate. H. PINCASS (Pharm. Zentr., 1932, 73, 469–470).—Details are given for its commercial prep. (cf. A., 1932, 602). R. S. C. Distribution of sulphur-containing waste gases in air. W. LIESEGANG (Gesundheitsing., 1931, 54, 705-709; Chem. Zentr., 1932, i, 1401).—A discussion. L. S. T.

Automatic apparatus for determination of small concentrations of sulphur dioxide in air. III. M. D. THOMAS (Ind. Eng. Chem. [Anal.], 1932, 4, 253— 256; cf. B., 1929, 282).—An improved form of the earlier apparatus is described, which is available for the continuous recording of (1) small concess. of SO<sub>2</sub> in air (0.01-7.0 p.p.m.), (2) higher concess. up to 6% in flue gases, which are low in H<sub>2</sub>SO<sub>4</sub>, but contain appreciable amounts of CO<sub>2</sub>, (3) the absorption of SO<sub>2</sub> by plants in fumigation experiments (range < 0.1— 50 p.p.m.). E. S. H.

Oxidation rates of silicon carbide and graphite powders. A. H. BALLARD and R. R. RIDGWAY (Trans. Electrochem. Soc., 1932, 61, 397-408).-Powdered samples of SiC and graphite were heated in a regulated stream of  $O_2$  at various const. temp. (600-1250°) and the  $CO_2$  evolved was measured at intervals. Percentage oxidation (as CO2) is plotted against time. Extrapolation of the curves for impure SiC at temp. around 950° permits calculation of free C content, and such results agree well with those derived from the increase in wt. of the sample. Experiments with specially purified SiC show that at 1250° the rate of oxidation is notably affected by grain size. At the temp. used, Fe oxide does not accelerate the oxidation of SiC. The above process serves for the comparison of commercial grades of SiC. H. J. T. E.

Rapid determination of boron. Evaluation of boron-bearing ores for boron content. W. W. Scorr *et al.* (Ind. Eng. Chem. [Anal.], 1932, 4, 306–308).—NaOH is preferable to Na<sub>2</sub>CO<sub>3</sub> for pptg. Fe<sup>TT</sup> and Al<sup>TT</sup> in H<sub>3</sub>BO<sub>3</sub> solutions, using Sofnol-red or *p*-nitrophenol as indicator.  $\text{NH}_3$  leads to high results and a poor end-point. The error with NaOH is <0.25%. The procedure is applicable to B-bearing ores, such as crude borax, kernite, tincalconite, ulexite, colemanite, and boracite. E. S. H.

Junker calorimeter.—See I. Rocking arc furnaces. SiC furnaces.—See XI. "Gas red" for paints. Luminous paints.—See XIII. Analysis of tanning materials.—See XV. Spray fluids. Determination of  $PO_4$ ".—See XVI.

See also A., Aug., 793, Phosphorescent sulphides. 805, Au sols. 808, Flame temp. of  $NH_3$  and its products of dissociation. 820, Electrodes for fixation of  $N_2$ . 823, Prep. of IC1. 824, Detection and determination of halogens. 825, Determination of Na and Li. 826, Spectrum analysis of Pb. 833, Prep. of  $NH_4$  *l*-tartrate.

#### PATENTS.

Conversion of atmospheric nitrogen into a compound containing combined nitrogen [ammonia]. F. W. DE JAHN, ASST. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,832,102, 17.11.31. Appl., 3.11.17. Renewed 20.5.21).—Air and steam are passed through red-hot coke to obtain a gas mixture containing 1 vol. of N<sub>2</sub> and 3 vols. of H<sub>2</sub> + CO, which is washed with H<sub>2</sub>O, passed over FeO(OH) to remove H<sub>2</sub>S, mixed

with steam, and passed over  $Fe_2O_3$  at 500° to convert the CO into  $CO_2$ . The resulting 3:1 mixture of  $H_2$ and  $N_2$  is compressed to 1400 lb. per sq. in. and washed with  $H_2O$  to remove  $CO_2$ ; further purification is effected by treatment with 20% aq. NaOH, a solution of  $Cu_2CO_3$  in aq. NH<sub>3</sub>, soda-lime towers, and NaNH<sub>2</sub>. NH<sub>3</sub> is obtained by passing the purified gases over a catalyst comprising Fe impregnated with NaNH<sub>2</sub>.

A. R. P.

Purification of zinc solutions [from chloridising roasting of pyrites]. W. SEGUINE, JUN., Assr. to KREES PIGMENT & COLOR CORP. (U.S.P. 1,832,329, 17.11.31. Appl., 21.9.29).—Pyrites residues after roasting with NaCl are extracted with  $H_2O$  or dil. HCl or  $H_2SO_4$  in two stages to obtain a solution rich in Zn and poor in Cu and a second solution rich in Cu and poor in Zn. The second solution is treated with scrap Fe to ppt. Cu and part of this ppt. is agitated with the first solution to remove most of its Cu content as  $Cu_2Cl_2$ , which is passed back to the Fe-treatment vats; the filtrate from the  $Cu_2Cl_2$  is agitated with scrap Zn to remove Cu, Ni, Ag, Cd, Pb, and Co and the purified solution is employed in the manufacture of lithopone. A. R. P.

Manufacture of barium aluminate. F. ROTHE and H. BRENEK, ASSTS. to RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,831,824, 17.11.31. Appl., 22.7.27. Ger., 30.7.26).—A mixture of bauxite with sufficient BaSO<sub>4</sub> to give BaO:  $Al_2O_3 = 1:1$  and BaO:  $(TiO_2 + SiO_2) = 1-3:1$  is heated at  $<1100^{\circ}$ in a non-reducing atm. in the presence of steam. Lixiviation of the product affords a solution of BaAl<sub>2</sub>O<sub>4</sub> and a residue of Ba compounds of SiO<sub>2</sub> and TiO<sub>2</sub> which may be utilised in the manufacture of Ba salts. A. R. P.

Sintering apparatus [for CaO etc.]. Crystallisation. Liquid and gas separator [for S]. S compounds from flue gases.—See I.  $H_2$ , CO, and S from gases. Gas-purification waste liquors.  $H_2$ S from petroleum products.—See II. Sulphitepulp liquors.—See V.

#### VIII.—GLASS; CERAMICS.

Use of electrochemical by-products in glass making. I. Arsenic, selenium, and tellurium. A. SILVERMAN (Trans. Electrochem. Soc., 1932, 61, 419–430).—The history of the use of As, Se, and Te in the glass industry is outlined, and the recent literature of the subject is reviewed and discussed. H. J. T. E.

Removal of lead from earthenware vessels. A. GRONOVER and E. WOHNLICH (Z. Unters. Lebensm., 1932, 63, 623–633).—Reliable results are obtained only if the vessel is first washed and heated in boiling  $H_2O$ , and then filled with boiling 4% AcOH which, after 30 min. on the  $H_2O$  bath, is cooled, diluted to the vol. of the container, and Pb determined on an aliquot portion. Vessels > 3 litres in capacity should be  $\frac{3}{4}$  filled. Results are calc. as mg. per litre of vessel content, and if high the extraction is repeated for 24 hr. J. G.

Use of unburnt bricks for the linings of induction furnaces. J. H. CHESTERS and W. J. REES (Trans. Ceram. Soc., 1932, 31, 243-252).—A method of lining induction furnaces with unburnt bricks is described. The results of 3 tests on different types of brick tend to indicate that an unburnt lining is superior to a burnt lining of the same material. H. H. M.

Magnesite bricks insensitive to temperature changes. K. ENDELL (Stahl u. Eisen, 1932, 52, 759-763) .- Five different types of magnesite bricks were tested for their sensitivity to temp, change by repeated heating at 950° followed by cooling in a blast of cold air. Ordinary MgO bricks broke into pieces after 1-2 repetitions, bricks made from electrically fused MgO after 12-25, and special heat-resistant Austrian bricks after > 30. Bending tests on these bricks at 20-900° showed that the temp.-sensitivity is inversely proportional to the max. bending and directly to the coeff. of expansion. The max. bending is conditional on the formation of cleavage, and possibly also of slip and twinning, planes; these planes are entirely absent from the sensitive bricks, but are very pronounced in the insensitive. The relatively high bending angle of the best quality bricks is attributed to the plasticity of periclase at high temp. A. R. P.

Silica bricks for coke ovens. P. B. ROBINSON (Trans. Ceram. Soc., 1932, 31, 253–272).—The physical and chemical properties of  $SiO_2$  and  $semi-SiO_2$  bricks are compared and discussed in the light of practical experience. The former are preferred. H. H. M.

Cleanliness of bottles etc.—See I. SiC.—See VII. Rocking arc furnaces. SiC furnaces. Electrolytic cell.—See XI.

See also A., Aug., 806, Colloidal clay. 811, Reactions of CO, with silicates. 822, Ca aluminates.

#### PATENTS.

Manufacture of a [sillimanite] composition for ceramic uses. T. S. CURTIS, ASST. to PACIFIC-SOUTH-WEST TRUST & SAVINGS BANK (U.S.P. 1,830,934, 10.11.31. Appl., 5.9.25).—Pyrophillite is heated at 925° under neutral or slightly reducing conditions for 1 hr., then allowed to cool to 500° during 4—6 hr. while a current of natural gas is passed through the furnace. The product is crushed through 8-mesh and material finer than 40-mesh is removed; the coarse material is passed under a powerful electromagnet to remove tourmaline and the residual artificial sillimanite is used in the manufacture of porcelain or refractories. A. R. P.

["Opalised "] protecting glass for miners' lamps. O. SCHNEIDER (B.P. 377,783, 4.6.31. Ger., 29.4.31).

Roofing .--- See IX. control had to to to the

## IX.—BUILDING MATERIALS.

Cement-making materials from Nyasaland. ANON. (Bull. Imp. Inst., 1932, 30, 139-159).—Tests and analyses of numerous samples of clays from the bed of Lake Malombe are detailed. E. L.

Preparation and properties of cement diaphragms. A. P. MASCHOVETZ (Ukrain. Chem. J., 1931, 6, [Tech.], 220–243).—The conductivity of freshly prepared diaphragms is greater than that of used ones, owing to conversion of  $Ca(OH)_2$  into  $CaCO_3$ . The introduction of asbestos and of NaCl into the cement increases conductivity. The diaphragm should be extracted with CO<sub>3</sub>-free H<sub>2</sub>O after prep. R. T.

Visualising concrete economy in terms of strength. I. LYSE (Eng. News-Rec., 1932, 109, 109–110).—For aggregates of a given type and gradation the consistency of concrete remains const. irrespective of the cement content, if the  $H_2O$  content remains const. Thus the cost per cu. yd. of concrete varies directly with the cement content for a given consistency. The cost may also be expressed in terms of the strength, and a curve shows that the cost of concrete per unit of strength decreases rapidly with an increase in the strength. C. A. K.

Conservation of wood posts. HUGRON (Bull. Inst. Pin, 1932, 65-70, 90-94, 115-118).-The 3 preservatives commonly employed are CuSO, HgCl, and creosote. CuSO<sub>4</sub> is injected by the Boucherie pressure-vat method, the vac.-pressure method being unsatisfactory with Abies and Picea. The posts last 13-14 years, but in certain soils they must be renewed annually; the tops must be protected from rain by cutting to a blunt point and painting for about 10 cm. with ochre and Zn white.  $CuSO_4$  is little protection against septic, micellar, or ferruginous soils, and supplementary means, e.g., packing with chalk, metal, or cement sleeves, local injection of "Inject" (hydrocarbons and antiseptics), or a creosote bath are employed. HgCl<sub>2</sub> forms an insol. compound and the penetration is usually 1-5 mm., which may be greatly increased by preliminary steaming (steam and PhOH) or dry heating. Various methods of applying creosote are reviewed. Other preservatives are "Aczol' (CuSO<sub>4</sub>, ZnSO<sub>4</sub>, and phenols with NH<sub>3</sub>), NaF (as effective as HgCl<sub>2</sub>), and "Basilite" (NaF and NH<sub>2</sub>Ph salt of 2:4-dinitrophenol). Specifications of the French P.T.T. C. H. are quoted.

Experience with flooring in industrial buildings. O. C. SPURLING (Eng. News-Rec., 1932, 109, 191–194).

New Zealand coal. Coal-tar creosote.—See II. Glucose for paper [sacks].—See V.

#### PATENTS.

Mineral surfacing and mineralised roofing. A. W. SEWALL, ASST. to BARBER ASPHALT Co. (U.S.P. 1,831,469, 10.11.31. Appl., 18.7.25).—Crushed waste porcelain, stoneware, or tile from which the fines have been removed is coated with a coloured vitreous glaze or enamel which is fired on. The treated granules are then used in surfacing shingles of fabric impregnated with bitumen. A. R. P.

Sintering apparatus.-See I.

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

Temperature conditions in the open-hearth furnace in relation to the calorific value of the fuel. V. A. MOJHAROV (Domez, 1931, No. 2, 1-19).—A discussion and criticism of Russian practice. CH. ABS.

Calculation of charge, slag composition, and deoxidation in the open-hearth furnace. N. A. KOSTULLEV (Domez, 1931, No. 4-5, 1-18).—A discussion. Mn (1.5-1.8%) should be present in the charge. CH. ABS.

Behaviour of the sulphur in the gas and the sulphur balance in the basic open-hearth process. F. EISENSTECKEN and E. H. SCHULZ (Stahl u. Eisen, 1932, 52, 677-686).-In a basic open-hearth furnace fired with a mixture of coke-oven gas and blast-furnace gas the air in passing through the preheating chambers takes up SO2 from the waste gases equiv. to 10-15% of the total S in the gases, and the gas preheating chambers retain about 60% of the S introduced by the mixed gases, the amount taken up depending on the temp. and the quantity of CaO, Fe<sub>2</sub>O<sub>3</sub>, and Fe on the walls of the chamber. Above 1150° the desulphurising action of the chambers is reduced, their greatest efficiency being reached at 1000-1100°, in which range CaS and FeS are formed on the chamber walls. During the period in which waste gases are passed through the gas chambers the sulphides are oxidised to sulphates, which are then decomposed, thereby increasing the SO<sub>2</sub> content of the waste gases. The S content of the gases in the furnace is absorbed by the bath only during the charging and melting-down period, and then only when the atm. is a reducing one; on the other hand, S is removed from the charge in an oxidising atm. and when large quantities of slag have formed irrespective of the atm. above the bath. All the S taken up by the charge is returned to the gases during the boiling and refining stages, hence removal of S from the gases used in firing the furnace is A. R. P. unnecessary.

Effect of burnt and unburnt lime on the production of steel in basic electric furnaces. O. VON KEIL and E. CZERMAK (Stahl u. Eisen, 1932, 52, 749-756) .---CaCO<sub>3</sub> as a flux increases the loss of Mn and C by the Fe during melting in high-Mn-C irons and results in a greater oxidation of the bath with Fe low in Mn and C than is the case with CaO as a flux. The greater refining action of CaCO<sub>3</sub> compared with CaO is also made evident by the closer approximation of the charge to the equilibrium between FeO and MnO in the slag and Mn in the metal. Between the time when the charge is melted and that of deoxidation, loss of Mn by the metal is the same whether CaO or CaCO<sub>3</sub> is used, but loss of C 18 greater with CaCO<sub>3</sub>, which also lengthens the time of melting. The gas content of steel made with CaCO<sub>3</sub> flux is about 50 c.c. per 100 g., whereas that of steel made with CaO is 86 c.c. per 100 g.; consequently there is a tendency in the latter case for the cast ingots to develop porosity in the outer layers. To sum up, it appears that both forms of CaO can be used equally satisfactorily in the manufacture of basic electric furnace steel. A. R. P.

Desulphurisation by modern processes in the production of steel and pig iron. C. BETTENDORF and N. J. WARK (Stahl u. Eisen, 1932, 52, 577–582, 606–610).—Rapid and complete removal of S from the open-hearth charge depends on the maintenance of a high basicity in the slag and a sufficiently high temp. CaO is the chief desulphurising agent, and its action is enhanced by the presence of a suitable solvent, e.g.,  $CaF_2$ ; SiO<sub>2</sub> instead of  $CaF_2$  is effective only when it constitutes a small proportion of the slag. Determination of the S balance during desulphurisation indicates that some S is lost in a volatile form when  $CaF_2$  is present, so that the efficiency of the slag is to this extent improved. Desulphurisation is also accelerated by a high Mn content of the metal bath and by addition of  $SrCO_3$ ,  $K_2CO_3$ , or  $Na_2CO_3$  to the charge; alkali chlorides have the same effect only when the metal bath contains reducing agents. Addition of reducing agents, *e.g.*,  $CaC_2$ , to the slag reduces the S content of the metal to the min. Desulphurisation in the blast furnace is briefly discussed. A. R. P.

Mechanism of carburisation [of iron] in cupolas. K. SIPP and P. TOBIAS (Stahl u. Eisen, 1932, 52, 662— 664).—In melting cast Fe in a cupola, absorption of C is the greater the lower are the C content of the charge and the Si content of the Fe, the greater is the reactivity of the coke, the broader the tuyère zone, and the longer the Fe remains in the furnace sump. The size of the steel scrap is without effect. A. R. P.

Nitriding of iron and its alloys. I. Ammonia dissociation and nitrogen absorption in the nitriding process. A. W. COFFMAN (Ind. Eng. Chem., 1932, 24, 751—754).—NH<sub>3</sub> is almost entirely decomposed at 800° if the rate of flow is low and surface area of steel specimen fairly large. Gain in wt./temp. curves have a max. which corresponds with the temp. of max. rate of change of NH<sub>3</sub> concn. This temp. is 650° for steels containing 0.93% Al with 1.70% Cr or 0.81% Al with 0.80% Mo, but is 700° for a steel containing 2.50% Al and 0.85% Mo. E. H. B.

Case-hardening of steel in molten sodium cyanide. H. N. BEILBY and W. NELSON (J.S.C.I., 1932, 51, 149—159 T).—No advantage results by working at > 60% of cyanide or in the use of more complex mixtures than 1:1 NaCN: Na<sub>2</sub>CO<sub>3</sub>, which may be used at 550—650°, except in the case of low NaCN concens. for surface-hardening. Cyanide is preferable to gaseous or solid cements because hypereutectoid steel is not formed under proper control. The mode of treatment of steel and the control of heating are discussed.

C. A. K.

Chemical control in case-hardening [of steel]. Copper-plating for protected areas. ANON. (Chem. Trade J., 1932, 91, 119—120).—For effective protection of localised areas against case-hardening, reasonably thick deposits of Cu are necessary. For penetrations of 0.005-0.01 in. the Cu should be > 0.001 in. thick, and for deeper cases 0.002 in. should be used. The cyanide method of plating is advised and described. C. A. K.

Corrosion. Use of aluminium for liquid-fuel containers. A. AUDIGÉ (Rev. Pétrolif., 1932, 10—11; Chem. Zentr., 1932, i, 1295).—Corrosion at low temp. is attributed chiefly to  $MgCl_2$  and  $CaCl_2$ , FeS being only a secondary catalyst.  $O_2$  plays an important part. At higher temp. corrosion is favoured more by mechanical or physical than by chemical effects, particularly by the attachment of  $CO_2$  and  $H_2S$  bubbles to the metallic surface. A. A. E.

Corrosion in neutral waters. I. D. VAN GIESEN (J. Amer. Water Works' Assoc., 1932, 24, 984-994).— The electrochemical theory of corrosion is discussed with particular reference to the action of natural waters

on Fe pipes. Maintenance of the  $p_{\rm H}$  between 7.1 and 8.3 is recommended, using Ca(OH)<sub>2</sub> or CO<sub>2</sub> as required in order to permit the formation of only a slight protective scale. C. J.

Galvanic corrosion. Possible galvanic effects in the corrosion of metals in milk. W. A. WESLEY, H. A. TREBLER, and F. L. LAQUE (Trans. Electrochem. Soc., 1932, 61, 267-283) .- In hot, aërated milk appreciable electric currents are generated by coupling Ni with Cu, or Al with Ni, Cu, or a Cr-Ni-Fe alloy, the dissolution of the first-mentioned metal being accelerated in each case. Cr-Ni and Cr-Ni-Fe alloys not only resist the action of the milk, but also do not increase the rate of dissolution of Ni or Cu coupled with them, the currents generated by these couples being negligibly small. Very small currents flow between Ni electrodes exposed to milk at two different temp., the hotter electrode being anodic, but in pasteurising plant such temp. effects are probably of no importance, the influence of differential aëration being notably greater. The single potential of Ni in milk has been measured under various conditions. H. J. T. E.

Beryllium. A. STOCK (Trans. Electrochem. Soc., 1932, 61, 451-468).-The extraction of Be by the Siemens-Halske process and the experimental work of the author and others leading to its development are reviewed. Properties of Be and its alloys are surveyed. A Cu-Be alloy (10% Be) prepared directly by electrolysis is a useful dioxidiser for Cu; the conductivity of ordinary cast Cu may be raised 20% thereby.

H. J. T. E.

Determination of sodium in aluminium. I. Chemical analysis. R. W. BRIDGES and M. F. LEE. II. Spectrographic analysis. A. W. PETREY (Ind. Eng. Chem. [Anal.], 1932, 4, 264-265, 265-267).-I. The fusion-leach method described depends on the fact that when Al is kept at just above its m.p. the Na comes to the surface and forms compounds, which may be leached out with H<sub>2</sub>O after cooling. The alkaline solution is then titrated. These operations are repeated until all the Na is removed. The procedure is not suitable when other alkali or alkaline-earth metals are present, but is applicable to Na alone over the concn. range 0.001-0.05%. The UO2(OAc)2 method cannot be used when the Na content is <0.01%. The results obtained by the fusion-leach method are compared with those obtained by the UO2(OAc)2 and nitrate-crystallisation methods.

II. The spectrum is obtained by an excitation of Al between electrodes of graphite. The metal burns and practically eliminates the continuous spectrum due to graphite. The spectral intensities are measured against those obtained from standard samples. The procedure can be used for determining <0.001-0.04% Na and has the advantage over the chemical method that other alkali metals can be determined simultaneously, if present. E. S. H.

Direct gravimetric determination of sodium in commercial aluminium. E. R. CALEY (Ind. Eng. Chem. [Anal.], 1932, 4, 340-341).-The Mg U acetate method is applicable when the amount of Na in Al is >0.01%. E. S. H.

Determination of silicon in aluminium. L. H. CALLENDAR (Analyst, 1932, 57, 500-511).-Si is best determined by dissolving the metal in 10% NaOH, diluting with H2O, pptg. with 60% H2SO4, and evaporating till fumes are evolved. The SiO, should be heated at 900° for  $1\frac{1}{2}$  hr. After treatment with H<sub>2</sub>SO<sub>4</sub> and HF, the residue should be heated to 1000° to decomp. T. McL. Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.

Determination of hydrochloric acid-soluble aluminium in aluminium alloys. BERGMANN (Chem.-Ztg., 1932, 56, 643) .- The alloy is dissolved in dil. HCl and Cu etc. are removed by H<sub>2</sub>S; preliminary oxidation of the insol. residue and conversion of SiO<sub>2</sub> into the insol. form are not essential. Al(OH)<sub>3</sub> is pptd. with aq. NH<sub>3</sub> under specified conditions and added to the ppt. obtained by passing H<sub>2</sub>S through the alkaline filtrate; to a separate portion of the solution containing Al tartaric acid is added, and Fe, Mn, etc. are pptd. with  $H_2S$ and aq.  $NH_3$ . The Al is calc. by difference. With experience, a method based on pptn. of the Al as phosphate gives satisfactory results, but Ti, if present, is pptd. simultaneously. The 8-hydroxyquinoline method also may be used. All three methods give more reliable results than the usual indirect and difference H. F. G. methods.

Effect of antimony on aluminium alloys. A. W. BONARETTI (Metalli leggeri, 1931, 1, 29-52; Chem. Zentr., 1932, i, 1425).-A commercial alloy containing Al 95-96, Sb 0.2, Si 0.7, Mg 2, and Mn 1.4% shows considerable resistance to corrosion. The stability towards corrosion of alloys with Cu, Zn, Mg, Fe, Si, Mn, Pb, and Sb has been investigated. Cu additions increase corrosion. Alloys with 1.2, 0.5, and 0.6% Sb combine increased strength with resistance to corrosion. L. S. T.

Deoxidation of copper with the metallic deoxidisers calcium, zinc, beryllium, barium, strontium, and lithium. E. E. SCHUMACHER and W. C. ELLIS (Trans. Electrochem. Soc., 1932, 61, 249-257) .- Ca, Zn, or Be deoxidises Cu effectively, and excess of these metals has only a small effect on the mechanical properties. Each 0.05% of Ca (up to 0.2%) remaining in the Cu reduces the conductivity by about 1%, each 0.05% of Zn (up to 0.3%) by 0.8%, and each 0.5% of Be (up to 1%) by about 10%. Ba, Sr, and Li are satisfactory deoxidisers, and their solubility in Cu is so small that excess of them has little effect on the conductivity. Li has the disadvantage of reacting readily with the oxides ordinarily present in refractories, thus producing Al and Si which lower the conductivity. H. J. T. E.

Determination of small amounts of iron in copper. B. PARK (Ind. Eng. Chem. [Anal.], 1932, 4, 247-248).-Errors in determining small amounts of Fe in Cu turnings are due mainly to incidental contamination of the relatively large surface, particularly by the material of the cutting tool. Preliminary washing of the Cu with dil. HCl is recommended.

E. S. H. Copper-tin compound in babbitt [metal]. G. A. NELSON (Metals & Alloys, 1932, 3, 168-170).-The Cu-Sn compound occurs as long hexagonal needles and the irregular forms observed on a polished sample are outcroppings from the main crystals. The no. of SbSn crystals decreases with > 6% Cu, together with an increase in the size of crystal. With < 2.5% Cu, the available Cu is depleted before star-shaped crystals can form, and the result is in the form of long spines. When fully developed stars are found, the CuSn crystals penetrate the matrix for a considerable distance. CuSn is more ductile than SbSn. C. A. K.

Corrosion of bronzes by vinegar. E. M. MRAK and J. C. LE ROUX (Ind. Eng. Chem., 1932, 24, 797— 799).—Cu, Sn, Pb, Cu–Sn, and Cu–Sn with addition of Pb, Pb + Zn, Pb + P, or Fe, or Cu with P + Fe + Al + Mn have been exposed to attack by vinegar ( $4 \cdot 27\%$ AcOH) under 3 sets of conditions: (i) immersion at rest, in a solution, (ii) immersion in an aërated solution, (iii) exposure to spray. All specimens corroded to a marked extent in all tests, and, except in the case of Sn, corrosion was most pronounced in the spray tests and least in test (i). It is regarded as inadvisable to employ any of these bronzes, particularly those containing Pb, in the vinegar industry. E. H. B.

Rapid determination of zinc and other impurities in cadmium. H. G. ISBELL (Ind. Eng. Chem. [Anal.], 1932, 4, 284—286).—The violet ppt. obtained from neutral Zn<sup> $\circ$ </sup> solutions by adding a small amount of CuSO<sub>4</sub> and then HgCl<sub>2</sub> + NH<sub>4</sub>CNS can be used for the rapid determination of small quantities of Zn (0·0001—0·003 g. in 5 c.c.) in presence of Cd. The ppt. is matched both in colour and quantity with a series of standards. Pb, Mn, Al, and Cr do not interfere. Standard methods are selected for determining Pb, Cu, Fe, As, and S in Cd. E. S. H.

White-metal bearing alloys. L. E. GRANT (Metals & Alloys, 1932, 3, 138—145, 152—158).—Work done on the structure and properties of babbitt metals is reviewed. The presence of up to 2% Pb does not appear to be detrimental to Sn-base bearings at normal working temp. For high pressures and high speeds 90% Sn, 7% Sb, and 3% Cu is recommended. Pb-base alloys may be used under conditions of moderate strength and resistance to impact, particularly when supported by an adequate backing. Properties of particular compositions are given. C. A. K.

Melting fine silver in high-frequency induction furnace. D. L. OGDEN (Trans. Electrochem. Soc., 1932, 61, 221–223).—Ag refined electrolytically by the Balbach-Thum process is melted, without preliminary drying, in an Ajax-Northrup high-frequency induction furnace. For a 500-kg, charge poured at 1040°, the energy consumption is 240 kw.-hr.

H. J. T. E. **Tarnish-resistant silver alloys.** K. W. RAY and W. N. BAKER (Ind. Eng. Chem., 1932, 24, 778—781).— Resistance to attack by aq. NH<sub>4</sub> polysulphide and the hardness, malleability, and constitution of some binary, ternary, and complex Ag alloys have been investigated, and an attempt has been made to nitride some alloys. Whilst many of the alloys, e.g., Ag–Zn, Ag–Zn–Al, are more resistant to tarnish than is standard Ag, none was found which was "stainless." Nitriding did not increase resistance to staining. E. H. B. Arsenic. P. M. TYLER and A. V. PETAR (Trans. Electrochem. Soc., 1932, 61, 431–436).—The occurrence and extraction of As are outlined, and uses of the element and its compounds reviewed. H. J. T. E.

Selenium and tellurium. S. SKOWRONSKI and M. A. MOSHER (Trans. Electrochem. Soc., 1932, 61, 285-293).--Methods for recovering Se and Te from Cu-refinery slimes are reviewed and consumption data for the U.S.A. quoted. Uses of Se and Te and their effects on the electrical conductivity of Cu are discussed. H. J. T. E.

Throwing power of plating baths. M. SCHLÖTTER and J. KORPIUN (Trans. Electrochem. Soc., 1932, 62, 95-106).—A plating solution is electrolysed for a few sec. between a Pt-sheet cathode and a centrally placed Pt-point anode. After washing the deposit, the cathode is heated to redness. The spacing of the rings of interference colours thus developed (cf. Nobili, 1826) indicate how the thickness of the metal deposit diminishes with distance from the anode, and serves to demonstrate differences in throwing power of plating baths. Dependence of throwing power on conductivity of solution, cathodic polarisation, c.d., and current efficiency is discussed mathematically, and a relationship deduced which is independent of the shape and spacing of electrodes and the size of the cell. H. J. T. E.

Control of operations in chromium[-plating] baths. A. WOGRINZ (Chem.-Ztg., 1932, 56, 571).-Low vals. are found when H2SO4 in the Cr bath is determined after reduction with MeOH or EtOH. It is found better to dilute the sample, add HCl and, in the hot, BaCl, incinerate the wet ppt. and fuse with Na<sub>2</sub>CO<sub>3</sub>, dissolve in HCl, add and boil off MeOH, and ppt. and weigh  $BaSO_4$ .  $CrO_3$  and  $Cr_2O_3$  are determined (1) by diluting, acidifying with HNO<sub>3</sub>, adding HgNO<sub>3</sub>, and heating the ppt. until converted into Cr2O3, which is fused with  $Na_2O_2$ , dissolved in  $H_2SO_4$ , and heated, after which the  $CrO_4''$  is determined by KI and  $Na_2S_2O_3$ , and (2) by evaporating a sample, fusing with Na2O2, dissolving, evaporating and re-fusing with Na2O2, and determining  $CrO_4''$  as above. Total  $CrO_3$  is given by (1), and total Cr by (2), from which "free"  $CrO_3$  and the Cr as 2Cr<sub>2</sub>O<sub>3</sub>,CrO<sub>3</sub> are calc. E. W. W.

Control of the chromium-plating bath. J. KOR-ECKY (Chem. Obzor, 1931, 6, 174–177, 204–205; Chem. Zentr., 1932, i, 1294–1295).—The  $\text{CrO}_3$  content can be determined rapidly by measurement of d. Determinations of Fe and  $\text{H}_2\text{SO}_4$  are important. A. A. E.

Chromium plating of measuring- and workingtools. Anon. (Chem.-Ztg., 1932, 56, 629-630).—A review of modern practice. E. S. H.

Modern practice in copper electrolysis. G. EGER (Chem.-Ztg., 1932, 56, 449-451, 470-472, 490-491). -A review of modern American and German procedures for the recovery of Cu from its ores by electrolysis and for the electrolytic refining of crude Cu. A. R. P.

Roasting of copper anode slimes in the Nichols-Herreshoff furnace. C. W. EICHRODT and R. W. ROWEN (Trans. Electrochem. Soc., 1932, 61, 469-473).—Slimes containing up to 50% of moisture are

charged directly into an oil-fired furnace consisting of an upper drying hearth and six internal hearths provided with rabbles. After being dried and preheated on the upper hearths, the slimes are roasted at  $260-290^{\circ}$  on the 4 lower hearths. If the slimes tend to ball up, charcoal may be added to the feed. As compared with reverberatory furnaces previously used, there is a decrease in fuel consumption, dust losses, amount of material tied up in the furnace, and amount of Cu rendered insol. The furnace is specially advantageous for slimes rich in Se. H. J. T. E.

Electrometallurgy applied to copper and to complex ores. W. E. GREENAWALT (Trans. Electrochem. Soc., 1932, 61, 359-396).-The future of electrolytic Cu extraction is discussed with particular reference to treatment of mixed oxide-sulphide ores. The process developed by the author (numerous U.S. patents) for mixed ores comprises fine grinding (about 48-mesh), flotation to separate sulphide concentrate from oxidised tailing, roasting the concentrate, leaching, electrolysing the resulting CuSO<sub>4</sub> solution to deposit Cu and regenerate H<sub>2</sub>SO<sub>4</sub>, and using the excess H<sub>2</sub>SO<sub>4</sub> regenerated to leach the oxidised tailing. The sulphide concentrate is separated into high- and low-grade portions which are roasted separately. The latter is then leached under conditions giving highest extraction of Cu, irrespective of the amount of Fe dissolving, and the former added to the resulting solution and agitated with air so as to ppt. most of the Fe. The solution obtained is pure enough to yield Cu at high current efficiency on electrolysis. The technical details and economic advantages of this process are discussed, and modifications are suggested for ores which contain notable amounts of Ag and Au. For complex ores gravity separation or flotation yields Pb, Zn, and Cu concentrates. These are roasted and the last two leached, and the resulting solutions after suitable purification electrolysed for recovery of Zn and Cu. The residues from the leachings are added to the Pb concentrate, which is then smelted and the resulting Pb bullion refined with recovery of precious metals.

H. J. T. E.

Recovery of precious metals from electrolytic copper refining. C. W. CLARK and A. A. HEIMROD (Trans. Electrochem. Soc., 1932, 61, 437-450) .--At the Canadian Copper Refiners' plant, Montreal East, the anode slimes from Cu refinery are roasted, leached with foul electrolyte to remove most of the Cu, and smelted to doré bullion, which is then refined in Moebius cells yielding Ag (> 999 fine). Owing to the high Au content of the bullion, the anodes are removed every 12 hr. and cleaned mechanically from Au crust which otherwise hinders dissolution. The electrolyte contains 30 g. Ag, 20 g. Cu, and 7 g. free HNO<sub>3</sub> per litre; the c.d. is 25 amp./sq. ft. The anode slimes from this process are boiled with conc. H2SO4 to remove traces of Ag, and cast as Au bars. Fumes from roasters and doré furnaces are recovered by electrostatic pptn. and worked up for Ag and Au; much Se and Te is present. H. J. T. E.

Ultra-rapid nickel-plating in France. M. BALLAY (Trans. Electrochem. Soc., 1932, 62, 59-73).— Recent development of Ni-plating in France is outlined with special reference to modern practice for plating at high c.d. on steel. Some features of semi-automatic and completely automatic installations are described. For plating on Al, preliminary pickling in hot acid FeCl<sub>a</sub> solution is favoured. H. J. T. E.

Electrodeposition of cadmium from cadmium sulphate solutions. I. S. WERNICK (Trans. Electrochem. Soc., 1932, 62, 75-86)-Cd electrodeposited on steel or Cu from M-CdSO<sub>4</sub> at  $p_{\rm H}$  5-5.7 is white and finer grained than that from solutions of lower  $p_{\rm H}$ ; at higher  $p_{\rm H}$  darker and less adherent deposits are produced. Since  $p_{\rm H}$  tends to increase during deposition, buffering at about  $p_{\rm H}$  5.6 by H<sub>3</sub>BO<sub>3</sub> and NaCl is adopted. With this solution, increase of c.d. has practically no effect on cathodic and anodic current efficiencies, but notably decreases the grain size and improves adhesion and burnishability. Above 4.5 amp./sq. dm., however, treeing becomes prominent even with a violently agitated solution. The anodic current efficiency is always > 100% owing to chemical dissolution. Up to 36° the current efficiencies are practically const., but at higher temp. there is a small decrease in cathodic and a larger increase in anodic current efficiency. Up to 50° the grain size decreases somewhat but the deposit darkens, whilst at higher temp. an undesirable coarse cryst. structure develops.

Ĥ. J. T. E.

Electrodeposition of tin. J. J. COLLINS (Chem. Trade J., 1932, 91, 77).—Articles are plated with Sn by making them the cathode in molten anhyd.  $SnCl_2$ at 270°, using 75—100 amp./sq. ft.  $SnCl_4$  produced at the C anode boils off and is condensed or absorbed in H<sub>2</sub>O and converted back into SnCl<sub>2</sub>. H. J. T. E.

Repair of worn parts by electrodeposition of iron. T. P. THOMAS (Trans. Amer. Soc. Mech. Eng., 1931, 53, No. 2, 29–30).—The bath should contain  $Fe(NH_4)_2(SO_4)_2$  (2.5 lb. per gal. of  $H_2O$ ),  $FeCO_3$  (freshly pptd. and kept under  $H_2O$ ; 5 oz.), and "a handful" of powdered C. CNS' must be absent. The bath is used at 60°; the c.d. is 60–65 amp. per sq. ft. Electrolytic cleaning prior to plating is recommended.

CH. ABS. Modern works plant and equipment for the hot-working of nickel and nickel alloys. W. R. BARCLAY, G. A. V. RUSSELL, and H. WILLIAMSON (Inst. Metals, Oct., 1932. Advance copy, 19 pp.).

Distortion of wires on passing through a draw plate. G. I. TAYLOR and H. QUINNEY (Inst. Metals, Oct., 1932. Advance copy, 13 pp.).

Blast-furnace gas.—See II. Corrosion in pulp mill.—See V. Colour analyser [for tarnished Ag.]. See VI. Electric furnace for cast Fe. Rocking arc furnaces.—See XI.

See also A., Aug., 800, Cu–Zn, Cu–Ag, and Au–Ag alloys. 801, Cu–Zn, Ag–Zn, Ni–Zn, Mn–Mg, Fe–Co–Mo alloys. 811, Equilibria between CO, CO<sub>2</sub>, and Fe, in reduction etc. processes in Fe, and between metals and slags in melts. 814, Passivity of Fe [in boilers]. 820, Electrodeposition of alloys. 826, Determination of Mg. Spectrum analysis of ZnO. 828, Au electrodes for spectrographs.

#### PATENTS.

[Metal-heating] furnace. C. STEIN, ASST. to SOC. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (U.S.P. 1,835,677, 8.12.31. Appl., 8.3.28. Fr., 21.3.27).—A temp. sufficient for heating billets previous to rolling is attained by the combustion of poor blastfurnace gas and air which has been preheated by the combustion of a separate lot of poor gas used solely for that purpose. B. M. V.

Metal recovering apparatus [for treating galvanised iron scrap]. C. E. BOWERS (U.S.P. 1,831,091, 10.11.31. Appl., 23.8.29).—The apparatus comprises a horizontal rotary drum divided into compartments by partitions fitted with closure plates, each compartment having a diagonally disposed plate adapted to transfer the charge of scrap from one compartment to the next when the drum is rotated. Zn is removed from the scrap by leaching it with dil.  $H_2SO_4$  in the drum, time being allowed for reaction to take place before every revolution of the drum. A. R. P.

Inhibitor [for steel-pickling baths]. J. M. LEAPER, ASST. to E. F. HOUGHTON & Co. (U.S.P. 1,832,781, 17.11.31. Appl., 1.8.30).—Claim is made for the use of compounds of the type R·S·SO<sub>2</sub>·OM, where R is an org. radical and M an alkali metal, *e.g.*, NaEtS<sub>2</sub>O<sub>3</sub>, or Na(CH<sub>2</sub>Ph)S<sub>2</sub>O<sub>3</sub>, with or without the addition of C<sub>5</sub>H<sub>5</sub>N. A. R. P.

Cleaning of metals [e.g., iron or steel]. H. S. GEORGE, ASST. to ELECTRO METALLURG. Co. (U.S.P. 1,832,979, 24.11.31. Appl., 19.6.29).—The surface is coated with  $H_4P_2O_7$ , heated at 160—250°, and rinsed in hot  $H_2O$ . A. R. P.

Alloy steels [for nitriding]. W. R. BREELER (U.S.P. 1,831,946, 17.11.31. Appl., 23.6.30).—The steels contain  $\gg 1$  (0.25)% C, 0.5—2 (1)% Al, 0.25— 2 (1.2)% Mn, 0.15—0.25 (0.2)% S, 0.05—0.1 (0.05)% P, 0.5—3.5 (1.5)% Cr,  $\gg 2(0.2)$ % Si,  $\gg 0.5$  (0.2)% Mo, and up to 0.5% V and 0.2% Zr. A. R. P.

[Aluminium - chromium - iron heat - resisting] alloy. W. E. RUDER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,833,723, 24.11.31. Appl., 7.6.23).—The alloy comprises Fe 65, Cr 25, and Al 10% with 1% of a grainrefining constituent, preferably Ti; it can be drawn into wire for electric resistors, is non-scaling, and has a zero temp. coeff. of resistance throughout its normal operating range. A. R. P.

[Collector for] ore concentration [by flotation]. C. H. KELLER, ASST. to MINERALS SEPARATION NORTH AMER. CORP. (U.S.P. 1,833,464, 24,11.31. Appl., 10.9.29). —Alkali furfurylxanthates are claimed. A. R. P.

Preparing oxidised minerals for concentration by froth flotation. L. W. FERRIS (U.S.P. 1,831,808, 17.11.31. Appl., 31.3.30).—Carbonate, silicate, and oxide ores of Cu and Pb are floated in a pulp containing NaCS<sub>3</sub>, using a frothing oil and a xanthate collector with or without a gangue depressant, e.g., Na<sub>2</sub>SiO<sub>3</sub>.

A. R. P.

Reducing zinciferous materials. F. G. BREYER, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,832,354 and 1,832,356, 17.11.31. Appl., [A] 17.2.27, [B] 22.3.29).— (A) The apparatus comprises a vertical metal retort with a charging device at its upper end and a lateral gas outlet below this device and communicating with a condenser. The retort is supported in a heating chamber, its upper end projecting well above the chamber and being well heat-insulated to prevent condensation of the Zn vapours on the retort walls. (B) Zn is produced from roasted ore by continuously passing a charge of briquettes of the ore and coke through the retort claimed in (A) which is made of a heat-resisting Ni alloy, *e.g.*, nichrome, hybnickel, or duralloy. A. R. P.

Zinc-base alloys and wrought products therefrom. W. M. PEIRCE and E. A. ANDERSON, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,832,653 and 1,832,733, 17.11.31. Appl., 18.3.29).—Alloys having a high resistance to cold flow comprise Zn with (A) 0.05-2 (0.5)% Cd and any or all of the following : 0.005-0.5 (0.005)% Li, 0.005-0.5 (0.01)% Mg, 0.01-2 (0.4)% Mn, 0.05-22% Cu, and 0.05-1 (0.3)% Ni ; or (B) 0.005-0.5(0.05)% Li with any or all of the following : 0.05-2%Cu, 0.005-0.55% Mg, 0.05-2% Mn, and 0.05-1%Ni. A. R. P.

Magnesium-tin-zinc alloys.R. T. Wood, Assr.to AMER. MAGNESIUM CORP. (U.S.P. 1,831,987, 17.11.31.Appl., 31.3.31).—Alloys of Mg with 1—15% Sn and0.1—10% Zn are claimed.A. R. P.

[Aluminium-zinc-copper] alloy. H. J. LORANG, Assr. to F. J. KUNA (U.S.P. 1,832,992, 24.11.31. Appl., 18.7.29).—The alloy is made by melting Zn (28 pts.) with Al (1 pt.) and a 50 : 50 Cu–Zn alloy (2 pts.). A. R. P.

Separation of tin from [white-metal] alloys. A. HANAK (U.S.P. 1,832,386, 17.11.31. Appl., 22.10.30).—  $Cl_2$  is passed through the molten alloy at 350—500° in the presence of  $Cu_2S$  or NiS. All the Sn distils as SnCl<sub>4</sub> before chlorination of Sb commences. A. R. P.

[Palladium-nickel] alloy for electrical contacts. E. F. KINGSBURY, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,832,307, 17.11.31. Appl., 11.7.25).— Alloys of 6—80 at.-% of a metal of the Fe group (70—30% Ni) with 94—6 at.-% of a metal of the Pt group (30—70% Pd) are claimed. A. R. P.

Elimination of phosphorus [from tungsten, molybdenum, or vanadium ores] by smelting. W. C. READ, ASST. to ELECTRO METALLURG. Co. (U.S.P. 1,833,125, 24.11.31. Appl., 3.4.30).—The ore is smelted with Si or a high-grade ferrosilicon, and a CaO slag having a base : acid ratio  $\ll 2:1$  to obtain an alloy containing at least 10% Si; all the P is taken up by the slag. A. R. P.

Treatment of quicksilver [mercury] ores. L. H. DUSCHAK and C. G. DENNIS, ASSTS. to GOLDEN GATE GOLD MINING CO., and HOMESTAKE GOLD MINING CO. (U.S.P. 1,832,006, 17.11.31. Appl., 19.7.27).—Hg ore is passed through a rotary furnace in the same direction as the heating flame and the discharge from the furnace is utilised for preheating the air for the combustion of the fuel. A. R. P.

Cadmium-plating bath. A. W. YOUNG and G. H. STINSON, ASSTS. to J. C. PATTEN (U.S.P. 1,833,450, 24.11.31, Appl., 23.1.28).—The bath contains  $2 \cdot 5$  oz. CdO, 6 oz.  $H_2SO_4$ , 4 oz.  $(NH_4)_2SO_4$ , and 1—10 g. of

animal glue per gal.; it yields dense, lustrous deposits of Cd at c.d. of 6—50 amp. per sq. ft. A. R. P.

Recovering metal values from ores and other metallurgical materials. MEYER MINERAL SEPARA-TION Co., Assees. of R. F. MEYER (B.P. 377,705, 23.4.31. U.S., 8.10.30).—See U.S.P. 1,822,995; B., 1932, 556.

Zn solutions from pyrites .- See VII.

# XI.—ELECTROTECHNICS.

Electric furnace in the cast-iron industry. W. E. MOORE Electrochem. Soc., 1932, 61, (Trans. 409-417).-The development of the electric furnace for the production of grey cast Fe is reviewed. The 3-phase direct-arc furnace with 3 upper vertical electrodes and a spheroidal bottom and roof is now preeminent in this field. The modern type is fed by a substation providing 4 voltages any of which is available for immediate use at the different stages of the heat. Types of melting procedure are outlined and energy consumption data quoted. The advantages of superheating on the properties of electric grey Fe are dis-H. J. T. E. cussed.

Applications of the rocking indirect-arc electric furnace. A. E. RHOADES and C. H. MORKEN (Trans. Soc., 1932, 61, 295-324).-The Electrochem. indirect-arc furnace with two horizontal electrodes and automatic mechanical rocking, giving a controlled stirring of the charge during heating, has special advantages for melting and superheating metals. The applications of this furnace to the production of grey cast Fe of various qualities, alloy irons and steels of various types, malleable and heat-treated irons, and non-ferrous metals, such as Cu, Ni, Al, and their alloys, are discussed at length with examples of operating data and costs. Modifications of procedure for obtaining products with special properties and compositions are indicated. Reference is made to special applications: the melting of glass, production of SrO from SrCO3, and smelting of Co ores. H. J. T. E.

Temperature measurements in commercial silicon carbide furnaces. R. R. RIDGWAY (Trans. Electrochem. Soc., 1932, 61, 225-236).-The temp. at various regions of the reaction mass of a commercial SiC furnace were determined by means of optical pyrometers focussed on the discs forming the ends of graphite tubes inserted in the mass, the interior of the tubes being kept free from fumes by a vac. pump. Temp.-time curves were obtained during operation at 800 kw. The curve for the central core showed a break at 1850-1950°, but in about 17 hr. reached a max. of 2600°, corresponding with active vaporisation of SiC, and then fell off to 1950-2050°. This latter temp. represents the formation and recrystallisation of SiC: the middle of the ingot wall attained it after 26 hr., and the border of the fires and zone approached it after about 43 hr., when the power was shut off. The form and position of the heating curves for the various zones are discussed in relation to the furnace reactions. H. J. T. E.

Electrolytic cell. G. I. WHITLATCH and R. D. BLUE (Ind. Eng. Chem. [Anal.], 1932, 4, 338-339).—The

multiple-cell box described is made of stoneware, with porous diaphragms of the mixture: French flint, Kentucky ball clay, English china clay (1:2:2). The diaphragms are more porous than those in most commercial cells and do not deteriorate even after long service, E. S. H.

Electrolytic oxidation of leuco-bases of the triphenylmethane series of dyestuffs. G. H. WHITE, JUN. [with A. Lowy] (Trans. Electrochem. Soc., 1932, 61, 237-248) .- The leuco-base of malachite-green (insol. in alkalis) is melted, mixed with C, and the mixture pressed on to a Pt-gauze or graphite-disc anode. Electrolysis in a diaphragm cell with aq. Na<sub>2</sub>SO<sub>4</sub> as anolyte, using 0.276 amp./sq. dm. at 0°, yielded the colour base with current efficiencies of 53% and 44%, respectively, for Pt and graphite. Similar conditions can be used for brilliant-green. Leuco-bases containing SO<sub>3</sub>H groups are dissolved in aq. Na<sub>2</sub>CO<sub>3</sub> and the solution is electrolysed at 0° between Pt or graphite electrodes. With Pt electrodes at 0.534 amp./sq. dm. the current efficiencies for guinea-green and brilliant-blue were > 60% and 66% respectively. The effects of varying the conditions of electrolysis have been studied.

H. J. T. E.

 $Na_2S_2O_4$ .—See VII. By-products in glass making. Bricks for induction furnaces.—See VIII. Galvanic corrosion. Be. Melting Ag. Se and Te. Plating baths. Cr-, Ni-, Cd-, and Fe-plate. Cu ores and anode slimes. Sn.—See X. Electric discharge and oils etc.—See XII.

See also A., Aug., 820, Electrodeposition of alloys. Electrodes for fixation of  $N_2$ . 3-kw. furnace using Cu electrodes. 821, [Photovoltaic accumulator for] photochemical reactions. 826, Determination of Mg. 827, Capillary Hg-vapour lamp. 828, Electroanalysis. Continuous  $p_{\rm H}$  recorder. Hg electrodes. 832, Oxidation of aliphatic dicarboxylic acids.

# PATENTS.

**Optical pyrometers.** SIEMENS & HALSKE A.-G. (B.P. 375,575, and 375,662, [A] 17.7.31, [B] 22.10.31. Ger., [A] 3.9.30, [B] 22.11.30. [A] Addn. to B.P. 366,292; B., 1932, 353).—In (A) one of the crossed filaments is in the form of a band with the broad face exposed to the observer. The filaments may be of the same or different materials and one should be cooled, *e.g.*, by heavy supporting leads, so that the temp.-current characteristics of the filaments are different, equal luminosity being attained at only one temp. The use of C, Re, Pt alloy, or W is claimed. In (B) one filament is a band over its whole length, the other a round wire flattened in the middle and has a length in mm. < 200,000 times its cross-section in sq. mm. B. M. V.

Kerr cells. GRAMOPHONE Co., LTD., and W. D. WRIGHT (B.P. 377,871, 25.11.31).

Producing carbon dollies for dry cells and apparatus therefor. H. A. BUMKE GES.M.B.H., and AUTOM-SPEZIAL-MASCHINEN G.M.B.H. (B.P. 377,823, 13.8.31. Ger., 21.1.31).

Carbonaceous articles. Mineral oil composition. —See II. Al-Cr-Fe alloy. Alloy for contacts. Cd-plate.—See X.

# XII.—FATS; OILS; WAXES.

Goat's fats. Determination of solid fatty acids. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1932, 63, 612—619).—The analytical data of 3 samples are tabulated and are similar to those obtained for beef and mutton fats;  $1\cdot1-2\cdot8\%$  of unsaturated solid fatty acid (*iso*oleic or vaccenic) was found by Grossfeld's method. Comparisons of Grossfeld's (B., 1930, 381, 990, 956) and Bertram's methods (A., 1928, 1152) for the determination of fatty acids showed that the former gives results which are low by 20%, the figures found by the authors' slightly modified form of the latter (54-62.9% for goat's fat and 47.5-60.2% for beef and mutton fats) being considered reliable to within  $\pm0.2\%$ . J. G.

Titre points of mixed fatty acids. II. Mixtures of pure fatty acids. G. W. JENNINGS (Ind. Eng. Chem. [Anal.], 1932, 4, 262-263; cf. B., 1931, 595).—Titration curves are given for lauric, myristic, palmitic, and oleic acids and their binary mixtures. No generalisation can be made concerning the effect of mixing these acids. E. S. H.

Reactions for [detecting] the rancidification of fats. P. BRUÈRE and A. FOURMONT (Ann. Falsif., 1932, 25, 91-97).—A recapitulation of existing methods, grouped into (a) reactions depending on the formation of aldehydes and ketones, (b) reactions based on the fixation of  $O_2$  by the fats with the formation of peroxides. H. J. D.

Chemistry of the spoilage [rancidity] of fats. K. TÄUFEL [with J. MÜLLER, F. K. RUSSOW, P. SADLER, and H. THALER] (Chem. Umschau, 1932, **39**, 147–151). —A review, including details of the author's modification of the Kreis test (cf. B., 1931, 1105) and test for "ketonerancidity" (B., 1932, 515). E. L.

Steam-distillation in fat analysis. W. WEISS (Chem. Umschau, 1932, 39, 151-154).—The Dons-Lüft (ef. Z. Unters. Nahr. Genussm., 1908, 16, 705; Arnold, B., 1922, 181A) method of fractional steam-distillation is applied to the quant. analysis of mixtures of higher saturated fatty acids (e.g., of cottonseed oil), the distillation figures (curve) being checked by matching them with those derived from known mixtures. E. L.

"Aniline point" as constant for fats and oils. C. G. KATRAKIS and J. G. MEGALOIKONOMOS (Praktika, 1930, 5, 267—269; Chem. Zentr., 1932, i, 1459).—A measured quantity of liquid fat or oil (filtered) is neutralised with 15% NaOH at 60° and mixed with an equal amount of Kahlbaum's normal benzene. 5 c.c. are mixed with 5 c.c. of NH<sub>2</sub>Ph and n is determined at 40°, whereby various adulterations of olive oil and butter fat can be detected. A. A. E.

Determination of hydroxyl number of fats, oils, and waxes. W. L. ROBERTS and H. A. SCHUETTE (Ind. Eng. Chem. [Anal.], 1932, 4, 257–259).—Determination of the Ac val. is replaced by that of the OH val., which is defined as the no. of mg. of KOH equiv. to the OH content of 1 g. of fat, oil, or wax. The substance is acetylated in a sealed tube by  $Ac_2O$ , and excess  $Ac_2O$ is hydrolysed and determined as AcOH by titration with 0.5N-KOH. Filtration is not necessary before titration, unless the sample contains free sol. acids. Results for a no. of oils and waxes show that OH vals. and Ac vals. are practically the same below 20. E. S. H.

Fixed oil from the nuts of Calophyllum inophyllum (dilo oil). K. W. R. GLASGOW (J.S.C.I., 1932, 51, 172—1747).—The semi-solid oil, best extracted with hot  $\text{Et}_2O$  (up to 58% yield), m.p. 50°, is unaffected by air, but partly oxidised by  $O_2$  at 125° without increase in free acid content, which is high and similar in composition to the acids obtained from the glycerides. The oil contains (%) palmitic (14·1), stearic (11·0), (?) erucic (3·0), oleic (48·0), linoleic (14·3), and resin acids (9·7) [(?) abietic acid], and unsaponifiable matter (0·25) containing sitosterol. A sample of oil obtained from native sources was discoloured and had different properties. A. A. L.

Determination of oil content of pecans. Sulphuric acid digestion method. R. D. LEWIS (Ind. Eng. Chem. [Anal.], 1932, 4, 296—297).—The oil is extracted by treatment of the finely-ground nuts with  $H_2SO_4$  $(d\ 1.5)$  at 65° and its vol. is measured directly. The results agree satisfactorily with those obtained by extracting the oil with light petroleum. The oil obtained by the  $H_2SO_4$  digestion method has the same d and n, but a higher I val. and sap. val., and is probably a purer product. E. S. H.

Improvement of Fiji copra. ANON. (Bull. Imp. Inst., 1932, 30, 125—199).—Treating copra with S fumes (3 hr.) yields a whiter product, more resistant to attack by moulds and insects than ordinary copra, provided that the sulphured copra is kept covered at night and is protected from rain ; such copra is less brittle than an ordinary, good, sun-dried sample. The best copra was obtained by washing the fresh nuts in aq. Na<sub>2</sub>CO<sub>3</sub>, sulphuring while still wet, and drying with covering at night and during rain ; this copra yielded oil of very low acidity. E. L.

Investigations on soap and Igepon solutions. K. BOEDEKER (Textilber., 1932, 13, 436-438).-Warm 3% solutions of Na stearate and oleate contain colloidal particles visible under the ultra-microscope, but on cooling to 20° the Na stearate solution gels and then contains closely interlocked needle crystals which gradually disperse when the solution is again heated; a 20% solution of Na oleate gels at 20° and then shows a similar structure to the Na stearate solution. 3% solutions of Igepon-A and -T (cf. B., 1932, 144) contain no colloidal particles and behave as true mol. solutions. The formation of insol. Ca soaps of stearic and oleic acids in hard H<sub>2</sub>O is prevented by the presence of about 16% of Igepon-A (calc. on the wt. of Na stearate or oleate). A. J. H.

Material losses [volatile products] in the drying process of linseed oil : chemical processes in atmospheric oxidation. P. SLANSKY (Chem. Umschau, 1932, 39, 155—156; cf. B., 1932, 391).—No appreciable amount of volatile products (which include  $H_2O$ ) is formed during the early stages of the oxidation; these appear only after the bulk of the  $O_2$  has been absorbed, *i.e.*, near the gelation point of the oil. Hence it is suggested that oxidation proceeds first by saturation of

the double linkings of the unsaturated acids by O, and that then the oxidised triglycerides catalyse an addition of O to the saturated C atoms, finally liberating volatile derivatives from them. Driers (e.g., Co oleate), as also higher temp., accelerate the formation of volatile products. E. L.

Cotton seed and cotton-seed oil at various stages of maturity. E. LONZINGER and R. RASKINA (Maslob. Zhir. Delo, 1931, No. 2—3, 57—60).—The oil content of cotton seed increases regularly and rapidly up to 50 days, and thereafter more slowly. The H<sub>2</sub>O-sol. portion decreases rapidly up to 35 days, and is then const.; the crude fibre increases rapidly during 40 days, and is then almost const. The ash content increases up to 35 days, is then const. to 50 days, and then decreases. The *n* and *d* of the oil increase with age of seed; the viscosity increases up to 60 days, and thereafter more slowly. The *n* and I val. of the fatty acids increase up to 50 days and then remain const. The sap. and acid vals. decrease with increase in age of seed.

CH. ABS.

Reactions of tung oil and elæostearic acid. J. MARCUSSON (Farben-Ztg., 1932, 37, 1354).-Addition of conc. H<sub>2</sub>SO<sub>4</sub> to tung oil in benzine solution produces a mixed isomeride-polymeride mass capable of separation. If  $HNO_3$  be used (1 c.c. of  $HNO_3$ , d 1.48, to 10 c.c. of tung oil and 20 c.c. of benzine) an identification of tung oil is afforded since linseed and other fatty oils give no solid separation ; 10% of tung oil is detectable in this way. The action of H<sub>2</sub>O<sub>2</sub> on tung oil or elæostearic acid in slightly alkaline solution is shown to produce the  $\beta$ -isomeride, whilst if excess of  $H_2O_2$  be used polymerisation occurs. Parallel instances of the same agent producing isomeride or polymeride according to conditions are given by the behaviour of light, S, and I. S. S. W.

Composition of palm oil. A: HEIDUSCHKA and A. ENDLER (Pharm. Zentr., 1932, 73, 481-483).— Hydrolysis of Lagos palm oil gives 47.65% of saturated (I) and 51.68% of unsaturated (II) acids. (I) contains 97.9% of palmitic and 2.1% of stearic acid, (II) 95.98%of oleic and 2.02% of linoleic acid. Daturic acid is a mixture. The oil contains tripalmitin, tristearin, and a highly unsaturated colouring matter. R. S. C.

Sesamé oil from the Far East. RUDAKOV and BELOPOLSKI (Maslob. Zhir. Delo, 1931, No. 2-3, 60).— The hand-pressed oil had  $d \ 0.9234$ ,  $n^{20} \ 1.4740$ , acid val. 0.98, sap. val. 191.01, unsaponifiable matter 0.29%, Hehner val. 94.85, I val. 113.21, CNS val. 72-74, saturated fatty acids (Bertram) 14.76%. The composition (by Kaufmann's formula) is: unsaturated glycerides 15.77, oleic acid glycerides 37.50, linoleic acid glycerides 46.7%. CH. ABS.

Properties of vegetable oils and resins subjected to silent electric discharge. P. SLANSKY [with W. Görz] (Farben-Ztg., 1932, 37, 1419—1420).— Previous work in this field is summarised and an apparatus for submitting oils to silent electric discharge while bubbling gases through them is described and illustrated. Linseed oil submitted to this treatment shows increase in viscosity and solid fatty acid content and decrease in I val. By increasing the c.d., the undesirable fatty

acids are eliminated and the production of stand oils of valuable properties in relatively short periods is made possible. Resins, *e.g.*, ester gum, rosin, are improved (for varnish manufacture) by this treatment.

S. S. W.

Sea-perch (Sebastes marinus) oil. I. LEVANDOV (Maslob. Zhir. Delo, 1931, No. 2—3, 30—32).— Sea-perch (uterus, liver) oil (average 21.2%) has  $d^{15}$ 0.920-0.9257, acid val. 0.46-6.11, sap. val. 183.3-195, I val. 120-133.44,  $n^{20}$  1.475-1.4751. The fatty acids have neutralisation val. 283.65, saturated acids 13.58% (I val. 6.65), unsaturated acids 85.41%(I val. 128.85). The antirachitic vitamin content is high. CH. ABS.

Acidimetric titration of glycerol in fatty oils. H. BULL (Tidskr. Kjemi, 1932, 12, 112).—Formulæ given in an earlier paper (cf. B., 1932, 686) are corrected.

Rapid determination of fat [fatty matter] in sulphonated oils. R. HART (Chem. Umschau, 1932, 39, 156—159.—Total fatty matter (F) is determined by calculation from the relation F = 100 — [organicallycombined SO<sub>3</sub> (a) + Na combined as soap (b) + NH<sub>3</sub> combined as soap (c) + inorg. impurities (d) + H<sub>2</sub>O (f)]. (a), (b), and (f) are determined as usual, together with "fixed alkali" and "total alkali" from which (c) is calc.; (d) is calc. from the alkali and ash as shown previously (B., 1931, 551). E. L.

Emulsification of carnauba wax. H. BENNETT (Melliand Text. Month., 1932, 4, 297).—Failure to make good stable emulsions is due either to improper stirring or to too low temp. High-speed and not hand-stirring is essential; the temp. should be kept above  $82-85^{\circ}$ , the m.p. of the wax. Directions are given for the prep. of polishes by the emulsification of wax in H<sub>2</sub>O containing org. linoleate or stearate. B. P. R.

Determination of m.p. of special waxes. C. S. GLICKMAN (Ind. Eng. Chem. [Anal.], 1932, 4, 304).— The wax is allowed to solidify in a glass tube (0.58 in. bore) open at both ends, which is then immersed upright in a H<sub>2</sub>O-bath. At the softening point the wax becomes transparent; at the m.p. the wax rises in the tube. Comparisons with the drop method and capillary-tube method are given; greater accuracy is claimed for the new method. E. S. H.

"Aniline point" as constant for waxes. C. G. KATRAKIS and J. G. MEGALOIKONOMOS (Praktika, 1930, 5, 311—314; Chem. Zentr., 1932, i, 1459).—Vals. are: beeswax 62—65, carnauba wax 78, Japan wax 36—37, white wax 61, paraffin 115—117. A. A. E.

Copra deterioration.—See XVI. Fat in milk powder. Salting of butter etc. See XIX.

See also A., Aug., 831, Prep. of branched-chain fatty acids. 832, Fatty acids from cassava starch. Et linoleate. 870, Wild-animal fats. 886, Vitamins in cod-liver oil. 887, Vitamin-D of fat of sea perch, porpoise, and seal. 888, Sterols of grapeseed oil.

## PATENTS.

Candles.-See II. Washing etc. agents.-See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Blistering of paint. H. CORTNUM (Farbe u. Lack, 1932, 389—391).—The various causes and types of blistering are discussed and a particular case of delayed blistering of an entire 7-coat paint system from a foundation of boiled oil is illustrated and described.

S. S. W.

"Gas red "—a new rust-inhibitive iron oxide pigment. H. A. BAHR and M. BRÄUTIGAM (Farbe u. Lack, 1932, 392).—A protected process is described whereby S-free Fe ores are treated with CO-containing gases at 475°, the ore being reduced and converted into carbides which are subsequently oxidised. A closelypacked, finely-divided pigment of high  $Fe_2O_3$  content is obtained, which is rust-resistant in paints, even in moist fumes. S. S. W.

Effect of white pigments on light-fastness of coal-tar dyes. H. WAGNER and M. ZIPFEL (Farben-Ztg., 1932, 37, 1480—1482).—Paint films comprising mixtures of a range of dyes with Ti whites of various grades, ZnO, lithopone, white lead, etc., ground in vehicles of the linseed oil, tung oil varnish, and dextrin types, were tested for bleaching in natural and artificial light over a period of 8 months. The results, which are graphed, show that TiO<sub>2</sub> definitely accelerates the fading, whilst ZnO retards it, regardless of vehicle and light source. The photochemical stabilising action of ZnO is attributed to its high ultra-violet absorption, and its advantageous effects in this sense are observed in mixtures with lithopone, white lead, etc. S. S. W.

Analysis of Prussian-blue. C. P. A. KAPPELMEIER (Chem. Weekblad, 1932, 29, 482—484).—The sample, (0.5 g.) is heated with 10 c.c. of 2N-KOH for 5—10 min. and the solid residue washed and dissolved in dil. HCl. The Fe is completely converted into Fe<sup>...</sup> by Br-H<sub>2</sub>O and determined iodometrically. The filtrate from the first operation is neutralised (phenolphthalein) with dil. HCl and boiled for 10 min. with 2—3 g. of yellow HgO. A clear solution is obtained by adding HCl, and the Feis oxidised with Br-H<sub>2</sub>O, determined iodometrically, and calc. to Fe(CN)<sub>6</sub>; the Hg(CN)<sub>2</sub> present, and org. matter, do not interfere. H. F. G.

Vapour tension of mixtures of terebenthene and colophony. (MLLE.) M. H. BARRAUD (Bull. Inst. Pin, 1932, 133—137).—Steam is passed very slowly into the mixture maintained at the desired temp. by a vapour jacket and the v.p. of the terebenthene is calc. from the wts. of  $H_2O$  and terebenthene distilling over. Results for a mixture initially containing 30% of terebenthene at 107°, 118—122°, 136—138°, 155—157°, and 172—175° are tabulated. C. H.

Comparative study of lac hosts with special reference to Acacia catechu and Cassia florida. A. K. THAKUR (Indian Lac Res. Inst., Bull. No. 9, 1932, 8 pp.).—The  $p_{\rm H}$  and d of the sap influence the suitability of a host for lac infection. S. M.

Effects of temperature and humidity on oviposition, incubation, and emergence in the lac insect, *Laccifer (Tachardia) lacca*, Kerr (*Coccidæ*), and on the resulting lac crop. P. M. GLOVER, P. S. NEGI, M. P. MISRA, and S. N. GUPTA (Indian Lac Res. Inst., Bull. No. 6, 1932, 18 pp.). S. M. Bleaching shellac. N. N. MURTY and R. W. ALDIS (Indian Lac Res. Inst., Res. Note No. 1, June, 1932, 1 p.).—The concn. of the Na<sub>2</sub>CO<sub>3</sub> solution used to dissolve the shellac should be  $\geq 2\%$ , otherwise larger quantities of hypochlorite are necessary. S. M.

Iodine value of shellac. R. W. ALDIS (Indian Lac Res. Inst., Bull. No. 8, 1932, 5 pp.).—Over 70 pure sticklacs showed I vals. between 12.6 and 15.5, excepting those from Assam and Burma, which gave higher figures. The I val. does not increase after storage for 2 years or after heating for 3 hr. at 100—110°. The accepted max. of 18 for the I val. of pure shellac is too high ; rosin determinations are accordingly low. S. M.

Orpiment and the iodine value of shellac. M. RANGASWAMI and R. W. ALDIS (Indian Lac Res. Inst., Bull. No. 7, 1932, 4 pp.).—The determination of rosin in commercial shellac by the I val. method is seriously affected by the fact that Wijs' solution attacks both orpiment and pure  $As_2S_3$ ; the extent varies with the particle size and relative quantity present. The orpiment can be separated by extracting the sample with glacial AcOH or with EtOH. S. M.

Swelling of shellac. R. W. ALDIS (Indian Lac Res. Inst., Res. Note No. 3, June, 1932, 1 p.; cf. B., 1932, 314).—Shellac flakes swell markedly when immersed for 6 months in  $H_2O$  containing a trace of  $Na_2CO_3$ ; in distilled  $H_2O$  microscopical swelling takes place with development of opacity; in  $H_2O$  which contains a trace of HCl no swelling and only slight loss of gloss ensue. S. M.

Determination of shellac fluidity. R. W. ALDIS (Indian Lac Res. Inst., Res. Note No. 2, June, 1932, 1 p.). —The fluidity is considerably affected by the presence of moisture and rosin and by laboratory fumes. Drying is effected by heating the sample, ground to 60-mesh, for 6 hr. at 35—40° and desiccating for 18 hr. S. M.

Humidity and storage of button lac. R. W. ALDIS (Indian Lac Res. Inst., Bull. No. 5, 1932, 4 pp.).— Cracking of button lac is due to shrinking consequent on loss of  $H_2O$ ; storage in a moist atm. is therefore recommended. S. M.

Synthetic resins and their application in the paint and varnish industry. E. A. BEVAN and F. J. SIDDLE (J. Oil Col. Chem. Assoc., 1932, 15, 177—190).— A general survey is given of the following types of synthetic resins : coumarone polymerisation products, vinyl ester polymerides, monocyclic ketone condensation products, urea resins, PhOH-CH<sub>2</sub>O condensation products, and "glyptals." S. S. W.

Oil bleaching. Resins subjected to electric discharge.—See XII.

See also A., Aug., 836, Vanillin as colour reagent. 841, Diazo resins.

#### PATENTS.

Manufacture of [mixed] polymerisation products [from vinyl ethers]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 373,643, 11.2.31).—Rubber-like or celluloid-like masses are obtained by interpolymerisation by the usual agents of aq. neutral to alkaline emulsions of mixtures of vinyl ethers with other unsaturated, polymerisable compounds. Examples are : vinyl Et C. H.

or Bu ether and acrylonitrile; glycol vinyl Et ether

and Me acrylate or acrylonitrile. [Stat. ref.] Arrangement of colour pigments [in paint boxes] for paint-mixing purposes. W. GRUMBACHER (B.P. 377,787, 10.6.31).

Zn solutions.-See VII.

# XIV.—INDIA-RUBBER : GUTTA-PERCHA.

Purification of rubber and properties of the purified rubber. A. T. MCPHERSON (Bur. Stand. J. Res., 1932, 8, 751-758).-Crude rubber was digested with  $H_2O$  at 190° in an autoclave, then extracted with H<sub>2</sub>O and EtOH, and dried in N<sub>2</sub>; 99.5% pure rubber hydrocarbon was obtained, having  $d^{25}$  0.9060,  $n_{\rm p}^{25}$ 1.5184, dielectric const. (average of 14 samples) 2.37, power factor 0.0015 (av.), and conductivity (after 1 min.)  $2 \cdot 2 \times 10^{-7}$  mho (av.). The electrical properties are apparently independent of the botanical source or kind of rubber. J. L.

Recent views on the elasticity of rubber. A. NIELSEN (Kautschuk, 1932, 8, 122-125).-A review of recent investigations and theories bearing on the physical changes in rubber accompanying its extension.

# D. F. T.

Röntgenographic investigation of µ-polychloroprene. P. GARBSCH and G. VON SUSICH (Kautschuk, 1932, 8, 122).—Polymerised  $\beta$ -chloro- $\Delta^{\alpha\gamma}$ -butadiene (B., 1932, 156) resembles natural rubber in exhibiting a marked fibre diagram when stretched, but an amorphous diagram in the unstretched condition; the fibre period is  $4.81 \pm 0.03$  Å. (natural rubber  $8.39 \pm$ 0.12 Å.). It has not yet been possible to crystallise µ-polychloroprene by cooling. D. F. T.

# PATENTS.

Rubber vulcanisation and manufacture of accelerators therefor. GOODYEAR TIRE & RUBBER CO. (B.P. 374,459, 4.3.31. U.S., 8.4.30).-Aldehydes intended for condensation with amines for accelerators are treated, while freshly prepared, with anti-ageing amines. The stabilised aldehydes yield condensation products having consistent accelerator val. Crotonaldehyde, e.g., is stabilised with 2% of \$\$-dinaphthylamine. C. H.

Products from vinyl ethers.-See XIII.

# XV.-LEATHER; GLUE.

Tanning plants of Italian Somaliland and Trans-Juba. G. A. BRAVO (Boll. Uff. R. Staz. sperim. Ind. Pelli, 1932, 10, 189-199).-Acacia Adansonii contains in the leaves 10.12 and in the branch bark 13.5% of tannins; A. Bussei (Harms) bark 20.46 (filter method) or 17.0% (Baldracco-Darmstadt method); A. Lebbek (Willd) leaves and bark of small branches 4.0%; Cæsalpinia pulcherrima, Sw., leaves 7.0, wood 2.32%; Cassia siamea (or florida), Lam., leaves 6.2, branch bark 2.92%; Avicennia marina (Forsk), Vierapp., officinalis, A. Rich, leaves 6.15, branch bark 2.22%; Terminalia catappa, L., leaves 12.88, branch bark 5.88; Rhizophora mucronata, Lam. (mangrove), bark 43.16 (filter method) or 40.2 (Baldracco-Darmstadt) %. T. H. P.

Tannin data on hairy Chinese chestnut trees [Castanea mollissima] grown in the United States. I. D. CLARKE and R. W. FREY (J. Amer. Leather Chem. Assoc., 1932, 27, 206-215).-The tannin content of the bark and wood of C. mollissima (I) steadily decreases the higher is the point from which it is sampled. Root bark contains 28.4% of tannin. Less tannin is present in root wood than in crown wood, but more in the bark than in the wood. The tannin, non-tans, and sugar contents of (I) and of American chestnut trees [C]. dentata (II)] were practically the same for different parts of the tree respectively except for the bottom of the tree trunk, where a tannin content of 17.6% was found in (I) compared with 24.5% in (II), and the nontans and sugars were slightly higher in all wood sections D. W. of (II).

Differentiation of cutch, gambier, and kino. H. FREYTAG (Collegium, 1932, 506-511).-A 1% aq. solution of the material was treated with K4Fe(CN)6 and NaNO3, strips of filter paper were soaked in the product, drained, exposed to ultra-violet rays for 20 min., and the colour of the oxidation products was observed both in daylight and in filtered ultra-violet light. Characteristic colours were obtained which indicate that anthocyanidin is formed from the catechin in cutch.

D. W.

Analysis of miscellaneous tanning materials. Report of Committee [of the American Leather Chemists' Association]. V. J. MLEJNEK (J. Amer. Leather Chem. Assoc., 1932, 27, 269-277).-Methods have been drafted for the analysis of Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, NaCl, and alum. D. W.

Evaluation of vegetable tannin extracts. J. A. SAGOSCHEN (Collegium, 1932, 500-506).-Aq. solutions of the extract having different concns. were prepared at 24°, and the vol. of sediment ("insol. matter") was noted after 24 hr. A solution was then prepared, of the concn. at which max. insol. matter had been obtained, with boiling H<sub>2</sub>O; this was then cooled to 24°, a piece of pelt immersed therein for 48 hr., and the colour, penetration, swelling, and condition of the fibres of the pelt, and also the amount of sediment (" pit ") when the used extract liquor was allowed to remain for 24 hr., were noted. The two sediments were not identical. More "pit sediment" was obtained with certain extracts free from insol. matter than with some normal extracts; this is attributable to the intense cooling to which they are subjected. Their natural composition is thereby disturbed and greater flocculation caused by their contact with pelt. D. W.

Detection and determination of sulphited [tannin] extracts. M. AUERBACH (Collegium, 1932, 251-254).-The detection of sulphited extracts by the evolution of H<sub>2</sub>S on treatment with dil. HCl and Zn is criticised. The following method is recommended : 50 c.c. of the analytical solution are treated with CH<sub>2</sub>O and HCl, the ppt. is filtered off after adding KOAc, washed, transferred to a Kjeldahl flask, and 0.2 g. of solid Fe(NO<sub>3</sub>)<sub>3</sub> and 5 c.c. of fuming HNO3 are added, followed, at intervals, by 20 c.c. of H<sub>2</sub>O<sub>2</sub> (30%) until all the org. matter is destroyed. After the reaction has subsided, the flask is heated over a small flame to expel O2, the liquor diluted, and the S which was combined with the tannin mol. determined with BaCl<sub>2</sub>. D. W. Detection of sulphites in sulphited [tannin] extracts. II. F. STATHER and R. LAUFFMANN (Collegium, 1932, 511—513).—A reply to Auerbach's criticism (cf. preceeding abstract). D. W.

Accumulation of non-tans in used [vegetable tan] liquors. E. IMMENDÖRFER (Collegium, 1932, 494—499). —Formulæ are given for calculating the non-tan contents of various tan liquors before and after introducing the hides into them. Difficulties arising from the high non-tan content of synthetic tannins and sulphited extracts are thereby avoided. D. W.

Conductometric titration methods in tannery laboratories. T. FASOL and E. UBERBACHER (Collegium, 1932, 232-237).-These methods can be used to detect mineral acids in HCO<sub>2</sub>H or AcOH or for the titration of acids in coloured solutions. 0.1% HCl can be detected in AcOH by determining the conductivity on titration of the AcOH with 0.1N-NaOH. 10 or 20 c.c. of a tan liquor can be titrated with 0.1N-NaOH and the conductivity curve determined. From the point of inflexion the amount of free acid can be calc. In an alternative method, the tannin is pptd. with MgO and the filtrate titrated with NaOH. Alkaline solutions can be titrated similarly with acid and the points of inflexion in the conductivity curves used to determine the  $NH_3$ . The degree of "olation" can be determined from the point of inflexion obtained in the conductivity curves derived on titrating a chrome liquor with N-H2SO4. The "formiato"-radicals in the chrome liquor can be determined by conductometric titration with Hg(ClO4)2, or the oxalato-radicals with D. W. AgNO3.

Analysis of various products used in tanning. ANON. (Boll. Uff. Staz. sperim. Ind. Pelli, 1932, 10, 176-182).—The methods recommended for the analysis of Na<sub>2</sub>S, CaO, MgSO<sub>4</sub>, Solvay Na<sub>2</sub>CO<sub>3</sub>, NaOH, and  $H_2SO_4$  by the commission appointed by the American Leather Chemists Association are given. T. H. P.

Graphical representation of the height of the liquid added to a tanning drum. VON ALADAR and A. VISNYA (Collegium, 1932, 513-517).—Mathematical. D. W.

Oil in relation to leather. A. A. CLAFLIN (J. Amer. Leather Chem. Assoc., 1932, 27, 277–282).—A lecture. D. W.

Effect of ultra-violet rays on [vegetable-tanned] leather and production of patterns on leather thereby. H. FREYTAG (Collegium, 1932, 161-167).-The discoloration produced by exposure to ultra-violet rays for 12-25 min. of vegetable-tanned leathers, which had been covered with a stencil, was intensified sufficiently by after-treatment with dil. FeCl<sub>3</sub> solution to permit of photographic reproduction. Patterns can thus be produced on leather. The collagen-tannin compound in the leather is split up by the ultra-violet rays, the tannin is thereby activated, complex Fe compounds in the leather are dissolved, and the Fe is "demasked." Ultimately the collagen proteins are decomposed, which process is catalysed by different metals and by O3, coloured phenolic substances being D. W. formed.

Effect of width of test-piece on result of determination of the tensile strength of leather. J. P. SYBIN (Collegium, 1932, 17—22).—Lower results were obtained as the width of the test-piece was reduced below 0.5 cm. D. W.

Chrome-leather glue. O. GERNGROSS and H. HEID-RICH (Kunstdüng. u. Leim, 1931, 28, 249–253; Chem. Zentr., 1932, i, 1327–1328).—The glue has viscosity  $3\cdot 2 - 8\cdot 3^{\circ}$  Engler at  $p_{\rm H} 6\cdot 4 - 6\cdot 9$ . The determination of adhesive power is discussed. A. A. E.

Glue and gelatin. O. GERNGROSS (Erste Mitt. Neuen Int. Verb. Materialprüf., 1930, C, 136—144; Chem. Zentr., 1932, i, 1327).—Conditions leading to deterioration of quality, and methods of examination, are discussed. A method for determination of f.p. is described. A. A. E.

Testing of caterpillar glue. F. MENTZEL (Kunstdüng. u. Leim, 1931, 28, 532—535; Chem. Zentr., 1932, i, 1328).—Attempts to evolve satisfactory rapid methods are described. A. A. E.

Determination of strength of glue and gelatin, gels. E. GOEBEL (Farben-Ztg., 1932, 37, 1435—1437 1467—1468, 1501).—Existing methods are summarised and a new apparatus for measuring the extent of penetration of gels by a plunger carrying various loads, together with a thermostat designed for the prep. of the necessary gels, are described and illustrated. Typical results are quoted and the calculation of modulus of elasticity of the gel material is discussed. S. S. W.

Some aspects of the science and art of leather making. H. G. TURLEY (J. Amer. Leather Chem. Assoc., 1932, 27, 316–334).

Application of ultra-violet rays to the leather industry. M. CUCCODORO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1932, 10, 238-250).

#### PATENTS.

Continuous process for moulding gelatin, glue, or like gelatinous substances. A. R. JAHN (B.P. 377,861, 6.11.31).

Azo dyes.-See IV.

# XVI.—AGRICULTURE.

Chemical nature and origin of humus. I. Synthesis of the "humus nucleus." II. Influence of " synthesised " humus compounds and of " natural humus" on soil microbiological processes. S. A. WAKSMAN and K. R. N. IYER (Soil Sci., 1932, 34, 43-69, 71-79).-The decomp. of proteins by microorganisms in liquid media was depressed by additions of lignin. The latter is not a toxic agent, but combines with a portion of the protein to produce a more resistant complex. The "synthetic" prep. is described of a complex ligno-protein having similar chemical, physical. and biological properties to those of natural humus. The existence of this "humus nucleus" (as distinct from "humus," i.e., soil org. matter as a whole) serves to explain the resistance of soil N to microbial decomp. and the tendency of soils to attain a const. C : N ratio. The synthetic humus nucleus combines with various soil bases to yield ligno-proteinates; e.g., at  $p_{\rm H} < 4.8$ the complex is saturated principally with H', Fe"

and Al<sup>···</sup>, at higher  $p_{\rm H}$  H is partly replaced by Ca<sup>··</sup> and Mg<sup>··</sup>, and at  $p_{\rm H} > 9 \cdot 0$  by Na<sup>·</sup>.

II. "Synthesised" humus complexes facilitate the decomp. of glucose by soil organisms, especially in the absence of added combined N. Neither synthesised nor natural humus can be utilised by cellulose-decomposing organisms as a source of N, but both accelerate cellulose decomp. in the presence of available N, without affecting the ratio of cellulose decomposed to N consumed. Lignin did not retard the decomp. of cellulose, and in some cases had a beneficial effect. The decomp. of fungus mycelium and of casein was decreased in the presence of lignin and a portion of the N of the mycelium was subsequently recovered in the form of a lignoprotein complex. Ca ligno-proteinate favoured the decomp. of fungus mycelium, but the corresponding H compound was injurious. Synthesised humus complexes containing Fe markedly increased the fixation of N by Azotobacter. A. G. P.

Carbon and nitrogen cycles in the soil. VII. Nature of the organic nitrogen compounds of "Humic" nitrogen. VIII. "Nonthe soil. humic" nitrogen. R. P. HOBSON and H. J. PAGE (J. Agric. Sci., 1932, 22, 497-515, 516-526; cf. B., 1932, 692) .- VII. The N of purified soil humic acid cannot be eliminated by customary methods for removing simple N impurities. Hydrolysates of humic acid contain N in forms similar to those of protein hydrolysates. The distribution of N from a mixture of albumin and artificial lignin humic acid resembles that of soil humic acid. The greater part of the N in each case is not decomposed by proteolytic enzymes. Soil humic acid may be represented as a close association of non-N humic acid and protein.

VIII. Non-humic N (*i.e.*, N remaining in solution when humic matter is pptd. from alkaline soil extracts by excess of acid) consists of 30-40% of peptides (peptone, proteoses, and polypeptides), 5% of NH<sub>2</sub>-N, and 12% of N, with a balance (40-50%) of which the greater part is pptd. by basic Pb acetate and is nonbasic. The peptides are colloidal and pptd. by basic Pb acetate and phosphotungstic acid. The non-humic N compounds are incorporated in the humic-clay gel, partly uncombined and partly associated with the inorg. colloid as Fe and Al compounds They are sol, only in solvents for humic matter and in proportion to the amount of the latter so dissolved. A. G. P.

Determination of soil type. A. TILL (Fortschr. Landw., 1931, 6, 738—747; Chem. Zentr., 1932, i, 1418).—Linear contraction, resistance to compression, and crushability are measured; tap- $H_2O$  may be used. The consistency is the best criterion for characterisation. A. A. E.

Effect of climatic variations on plasticity of soil. G. W. S. BLAIR and F. YATES (J. Agric. Sci., 1932, 22, 639—646).—Soil plasticity as measured by the flowplasticity method shows seasonal fluctuations depending on soil temp. and  $H_2O$  content. A. G. P.

Field method for determining the consistency of soil. A. TILL (Fortschr. Landw., 1931, 6, 705—707; Chem. Zentr., 1932, i, 1418).—An apparatus is described. A. A. E. Stability of soil structure and effect of rain and irrigation. F. SEKERA (Z. Pflanz. Düng., 1932, 25A, 297—304).—The effect of dilution of the soil solution on the peptisation of the clay is examined as follows. Soil is saturated with  $H_2O$  and the soil solution obtained by pressure. Soil in definite proportions is shaken with soil solution diluted to varying extents and the clay remaining in suspension after a given period is ascertained by coagulation and weighing. A fully peptised control is obtained by the addition of  $Li_2CO_3$  to the suspension. Data showing the effect of CaO and fertilisers, and of seasonal changes, on the dispersion of clays by diluted soil solutions are recorded. A. G. P.

Relationships between the degree of saturation and buffering of a soil and its reaction. H. NIKLAS, A. HOCK, and A. FREY (Z. Pflanz. Düng., 1932, 25A, 330—348).—In the electrometric titration of soils (Goy) alkali added beyond the val.  $p_{\rm H}$  7.0 is in some cases taken up very slowly and vals. obtained in 30-min. reaction periods are unsuitable for extrapolation to the final end-point. The classification of soils on the basis of buffer nos. (Goy) is impracticable. The buffer no. of a soil is not related to the degree of saturation or to the buffer area (Jensen). A. G. P.

Effect of changes in soil moisture on the composition and concentration of the free soil solution. S. M. DRATSCHEV and V. P. ALEXANDROVA (Z. Pflanz. Düng., 1932, 25A, 349—365).—The total electrolyte concn. of the free soil solution (obtained by pressure) is inversely proportional to the total  $H_2O$  content of the soil. This relationship obtains, in general, for the concns. of Ca<sup>''</sup> and NO<sub>3</sub>', with certain exceptions in the case of Ca<sup>''</sup> in cultivated podsols and alkali chernozems. The total dissolved  $PO_4^{'''}$  increased with rising  $H_2O$  content, the effect being most evident in forest loams, alkali chernozems, and heavily fertilised podsols. The solubility product of the phosphate in soil solutions from all except certain podsols lies within the limiting vals. for Ca<sub>3</sub>PO<sub>4</sub>. A. G. P.

Determinations of hygroscopicity and water capacity for soil evaluation. von Nostitz (Z. Pflanz. Düng., 1932, 25A, 275–287; cf. B., 1931, 688). —Hygroscopicity measurements of soils although bearing some relationship to the size distribution of particles cannot replace mechanical analyses in the characterisation of soils. The  $H_2O$  capacity as determined by Wahnschaffe cylinders is too dependent on temporary soil conditions to be of service as a soil characteristic. A. G. P.

Colorimetric determination of phosphoric acid with special reference to Arrhenius' method [for soil extracts]. H. RIEHM (Z. Pflanz. Düng., 1932, 25A, 323—329).—In Arrhenius' Mo-blue method (B., 1929, 787) the presence of citric acid, citrates, or tartrates prevents the formation of blue colour by small amounts of silicates without affecting that produced by  $PO_4^{\prime\prime\prime}$ . The smaller the concn. of molybdate reagent used the greater is the proportion of silicate which may be present without affecting colour production. The intensity of the  $PO_4$ -blue is not affected by reducing the prescribed concn. of the molybdate reagent by one half provided sufficient is present to overcome the action of citric acid in diminishing the colour. In 5 c.c. of a citric extract of soil the presence of 0.2-2.0 mg. of SiO<sub>2</sub> does not vitiate results, but if 10 c.c. of extract are used the influence of citric acid is apparent. A. G. P.

Determination of sodium [in soils] by precipitation as the triple salt sodium uranyl magnesium acetate. C. S. PIPER (J. Agric. Sci., 1932, 22, 676— 687).—Under conditions obtaining in NH<sub>4</sub>Cl extracts of soils, Bray's method for determining Na (B., 1929, 407) gave poor results, but Kahane's method (A., 1930, 726) was very satisfactory. Moderate proportions of Ca, Ba, or NH<sub>4</sub> chlorides and K up to 0.034 g. KCl per test (=  $\geq 5$  mg. Na) did not vitiate results. Sulphates interfere only if Ca is present and the solubility of CaSO<sub>4</sub> is exceeded. A. G. P.

Distribution of potassium in deciduous orchard soils in California. O. LILLELAND (Soil Sci., 1932, 34, 11—18).—The observed decrease in replaceable K contents of these soils with depth is attributed to the withdrawal of K from the lower levels by roots and its return to the surface layers in the form of leaves and surface vegetation. A. G. P.

Behaviour of magnesium in soil. S. ARANY (Mezog. Kutat., 1931, 4, 439–448; Chem. Zentr., 1932, i, 1286).—Alkaline,  $CaCO_3$ -bearing, and acid (org. matter-rich) soils absorb from a dil. solution of  $CaCl_2$  and  $MgCl_2$  no Ca, but Mg, the Ca content of the soil diminishing. Alkali soils poor in Ca absorb Ca, but not so readily as Mg. A. A. E.

Representation of soil analyses by the device of double plotting. W. H. GREEN (J. Agric. Sci., 1932, 22, 548-550).—The soil characteristics shown by customary mechanical analysis are represented by a straight-line graph from which may be calc. the "omitted balance" (*i.e.*, loss on treatment with  $H_2O_2$ and acid). A. G. P.

Distribution, availability, and nature of the phosphates in certain Kentucky soils. M. C. FORD (J. Amer. Soc. Agron., 1932, 24, 395-410).-The relatively available PO4 "" (sol. in carbonated H2O or in 0.002N-H<sub>2</sub>SO<sub>4</sub> buffered to  $p_{\rm H} 3.0$ ) was roughly proportional to the total native PO<sub>4</sub><sup>'''</sup> present. Liming did not increase the availability of PO<sub>4</sub><sup>'''</sup> in these soils. The availability of superphosphate varied with the ability of the soils to fix PO," in insol. forms. Liming reduced the fixation of added PO<sub>4</sub>" and also reduced the rate of dissolution of rock P. The latter effect was greater in soils having a low capacity for fixing  $PO_4'''$ . In soils of high  $PO_4'''$ -fixing power the rate of dissolution of rock P was high, but transition to insol. forms was rapid and a PO<sub>4</sub>" deficiency resulted. Liming reduced the availability of Mn, but there was no correlation between Mn availability and the influence of CaO on the response of crops to rock P. Fixation of P by soils occurred largely in the fine-clay fraction, but also, to an appreciable extent, in the coarse-clay and silt A. G. P. fractions.

Colorimetric methods for determining readily available phosphorus in soils. C. O. ROST and R. M. PINCKNEY (J. Amer. Soc. Agron., 1932, 24, 377395).—In the soils examined, results of the field methods of Bray (B., 1930, 295), of Spurway, and of Truog (B., 1931, 266) showed good general agreement, especially in the case of the last two. Determinations of P deficiency were of the same order when surface samples were used as when subsoils were included also.

A. G. P.

Colorimetric determination of the citrate-soluble phosphate in soils. A. NĚMEC, J. LANÍK, and A. KOPPOVÁ (Z. Pflanz. Düng., 1932, 25A, 365—373).—In Nemec's method (B., 1930, 207), the presence of Fe<sup>···</sup> in proportions > 0.5 mg. per 100 c.c. caused high results for PO<sub>4</sub><sup>···</sup> in solutions containing < 0.075 mg. P<sub>2</sub>O<sub>5</sub> per 100 c.c. The presence of 0.5 mg. SiO<sub>2</sub> per 100 c.c. does not affect the vals. obtained. Warren and Pugh's method (B., 1931, 81) leads to lower vals. for PO<sub>4</sub><sup>···</sup> than does that of the author or the gravimetric method. This is ascribed to the co-pptn. of PO<sub>4</sub><sup>···</sup> with the ferrocyanide ppt. A. G. P.

Removal of added nitrogen from grassland soils. H. L. RICHARDSON (Nature, 1932, 130, 96–97).— The rate of removal of N, whether added as  $NH_4$  or  $NO_3$ , by vigorously growing grassland is unexpectedly high. L. S. T.

High-protein pasture. Rotational or closegrazing system of pasture management. F. T. SHUTT, S. N. HAMILTON, and H. H. SELWYN (J. Agric. Sci., 1932, 22, 647-656; cf. A., 1930, 384).—Grass "cut for hay" produced higher dry-matter yields but lower protein yields than frequently cut pasture. The decline in protein content of grass with age was appreciable but not great during the first 3 weeks of growth. The gradual incursion of clover into frequently cut pasture raised its protein content considerably. The development of clover reached a max. in the second or third year of operation, but declined subsequently.

A. G. P.

Interrelation of factors controlling the production of cotton under irrigation in the Sudan. F. G. GREGORY, F. CROWTHEE, and A. R. LAMBERT (J. Agric. Sci., 1932, 22, 617-638).—The effects of spacing,  $H_2O$ supply, and applications of N are recorded. Optimum yields, with and without N, corresponded with the same sowing date. The efficiency of N fertilisers decreased with the advancing period of sowing. The effect of spacing on yields was not influenced by applications of N. The  $H_2O$  supply with early sowings had little effect without, but considerable effect with, N dressings. A. G. P.

Crop husbandry. I. Effects of seed treatments on germination and yield of sugar beet. F. H. GARNER and H. G. SANDERS (J. Agric. Sci., 1932, 22, 551-559).—Treatment of seed with conc. H<sub>2</sub>SO<sub>4</sub> to remove a portion of the hard coat results in more rapid and more even germination and a greater yield. Milling of seed is approx. as effective as acid treatment.

A. G. P.

Influence of time on the activity of rye seed used in the seedling method. H. NEUBAUER (Z. Pflanz. Düng., 1932, 25A, 373—376).—A reply to Kuke (B., 1932, 697). Discrepancies are ascribed largely to failure to observe prescribed details of the method. A. G. P.

Physiological significance of mineral soil acidity to plants. I. Influence of soluble and insoluble aluminium on the nutrient intake of young rye plants in sand culture with and without silicic acid. M. TRÉNEL and H. J. FREY (Z. Pflanz. Düng., 1932, 25A, 305-323).-The presence of Al salts or Al(OH)<sub>3</sub> in sand cultures with media of  $p_{\rm H} < 5$  retarded the germination of rye and depressed the intake of P by seedlings. The ash content of seedlings is reduced, Mg and Ca being considerably and K only slightly affected. AlCl<sub>3</sub> is more injurious than is  $Al_2(SO_4)_3$ . The toxic action is reduced by the addition of CaSO<sub>4</sub> or hydrated SiO<sub>2</sub> to the media without alteration of its  $p_{\rm H}.~$  Al has a sp. toxicity quite apart from its action in rendering  ${\rm PO}_4^{\prime\prime\prime\prime}$  unavailable to plants. Sol. Al salts show a reduced toxicity in the presence of AlPO4 or Al(OH)<sub>3</sub> and become non-toxic in the presence of mixed gels of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The increased assimilation of PO4''' in the presence of SiO2 depends on the ability of the latter to restrict the fixation of PO4''' by Al2O3 or sol. Al salts. A. G. P.

Ripening and transport of bananas in Australia. W. J. YOUNG, L. S. BAGSTER, E. W. HICKS, and F. E. HUELIN (Counc. Sci. Ind. Res., Australia, Bull., 1932, No. 64, 52 pp.).—Rapid and uniform ripening of bananas was effected by treatment with  $C_2H_4$  or with coal gas (1 in 1000 in summer or 3 in 1000 in winter), provided the ripening process had not already commenced. Recommendations for the storage and transport of fruit are appended. A. G. P.

Influence of continuous aëration on growth of tomato plants in solution cultures. H. E. CLARK and J. W. SHIVE (Soil Sci., 1932, 34, 37-41).—Aëration of culture solutions increased both the total crop and the proportion of tops to roots. A. G. P.

Removal of plant nutrients in coconut cultivation. C. D. V. GEORGI and G. L. TEIK (Malayan Agric. J., 1932, 20, 358—364).—Records of the N, P, K, Ca, and Mg contents of the various organs of the palms are recorded and means of maintaining and improving soil productivity are discussed. The composition of the palms is no criterion of the nutrient content of soils in which they grow. A. G. P.

Copra deterioration. F. S. WARD and F. C. COOKE (Malayan Agric. J., 1932, 20, 351-357).—Continuous and complete drying of copra prevents more than a surface growth of mould, and subsequent insect attacks are not serious. A. G. P.

Effect of soil reaction on the early growth of certain coniferous seedlings. H. L. SUNDLING, A. C. MCINTYRE, and A. L. PATRICK (J. Amer. Soc. Agron., 1932, 24, 341–351).—Optimum  $p_{\rm H}$  vals. in various soils for total growth top growth, length and no. of rootlets are recorded. Vals. vary from soil to soil. A. G. P.

Iron toxicity from liming. A. H. MEYER (Science, 1932, 76, 56).—Heavy liming of acid soils with  $CaCO_3$  increases the contents of Fe and org. matter in the soil solution. The transitory high level of Fe apparently accounts for toxic effects to crops after heavy liming. L. S. T. Influence of potash manuring on resistance [to disease] of cultivated plants. W. ACKER (Z. Pflanz. Düng., 1932, 11B, 313—319).—The effects of K on the mechanical structure and physiological activities of plants are discussed. K increased the negative geotropic movement of the stems of barley plants. A. G. P.

Manurial experiments with hops in pot culture. F. H. BEARD (J. Pomology, 1932, 10, 91-105).-Effects of the omission of various essential nutrients on the growth and physiological activity of hops are recorded. The marked results of the omission of N or P were apparent in stunted plants unable to produce cones. Leaves were pale green in N-starved plants and dull green where P was deficient. K deficiency had little effect on total growth, but tended to prolong the growing period. Leaves showed marginal scorch, slight chlorosis, and purpling. These effects were intensified by increasing the N supply. Ca-deficient plants made good growth, but lower leaves suffered marginal scorching. Growth was not markedly affected by Mg deficiency, but leaves showed angular spotting and scorching; leaf fall was premature and cones failed to develop. A. G. P.

Nitrogen manuring of hops. SEEGER (Z. Pflanz. Düng., 1932, 11B, 306—313).—Profitable crop increases were produced by  $(NH_4)_2SO_4$  on soils rich in CaCO<sub>3</sub>, but not on acid soils. Urea was more effective than  $(NH_4)_2SO_4$  on acid soils and superior to CaCN<sub>2</sub> on neutral and alkaline soils. Best returns on very acid soils were obtained with CaCN<sub>2</sub>, especially in moist seasons. CaCN<sub>2</sub> also showed better effects in the second season than the other fertilisers. Applications of N (in conjunction with P and K) improved both the yield and quality of the hops. A. G. P.

Growing cotton and other crop plants with ammonium nitrogen. V. A. TIEDJENS (Science, 1932, 75, 648).—Mainly a discussion (cf. B., 1931, 215). Cotton plants grow well in sand cultures with  $(NH_4)_2SO_4$ , aq. NH<sub>3</sub>, or Ca(NO<sub>3</sub>)<sub>2</sub>, respectively. Aq. NH<sub>3</sub> supplied in a complete nutrient solution at  $p_{\rm H}$  8 had no injurious effect. The generalisation that NH<sub>3</sub> is toxic to plant growth under good cultural conditions is untenable.

L. S. T.

Soya beans and other green manure crops for sugar-cane plantations. G. ARCENEAUX, N. MCKAIG, JUN., and I. E. STOKES (J. Amer. Soc. Agron., 1932, 24, 354—363).—Soya beans yield max. green matter, dry matter, and N per acre at the full-bloom stage. Analyses of a no. of legumes at various stages of growth are recorded. A. G. P.

Manurial trials with beet molasses. K. MAIWALD (Z. Pflanz. Düng., 1932, 11B, 289–306).—Barley, oats, maize, carrots, and potatoes were unable to utilise the K and N of beet molasses in the year of application and in some cases yields were reduced. Prolonged composting with soil improved the manurial action of the molasses, but returns were uneconomical.

A. G. P.

Determination of rate of decomposition of organic matter under field conditions. M. B. STURGIS (Soil Sci., 1932, 34, 19–23).—The carbohydrate, lignin,

and protein fractions of sugar-cane trash decompose in soil at different rates. Carbohydrates are the most rapidly affected and the addition of N fertilisers increases the rate of their decomp. The lignin fraction is decomposed slowly, but does not tend to accumulate in the soil as does the more resistant protein fraction. Carbohydrate material in soil may be determined as reducing sugars after decomp. by  $H_2SO_4$ -HCl mixtures.

A. G. P.

Toxicity of mineral oil sprays to vegetation. F. TUTIN (J. Pomology, 1932, 10, 65–70).—The I val. (Hübl or Wijs, but not Hübl–Waller) of mineral oils is as good an index of their toxicity to plants as the sulphonation test. Oils having I val. > 1.0 are unsafe for summer use in 3% concn. Considerable differences in plant injury are shown by the same oil when emulsified by different methods. A. G. P.

Fungicidal properties of certain spray fluids. IX. Fungicidal properties of the products of hydrolysis of sulphur. H. MARTIN and E. S. SALMON (J. Agric. Sci., 1932, 22, 595-616).—The fungicidal action of polysulphide washes or of finely-divided elementary S cannot be attributed to the formation or presence of sulphite, hyposulphite, sulphoxylate, thiosulphate, or monosulphide S, but is due to polysulphide-S as such. The fungicidal action of solutions of Na<sub>2</sub>S, NaOH, and Na<sub>2</sub>CO<sub>3</sub> is due to their alkalinity.  $H_2S$  liberated from Na<sub>2</sub>S solutions is without action on the powdery mildew of the hop. A. G. P.

Winter spraying trials in the West Midlands. E. E. EDWARDS (J. Min. Agric., 1932, 39, 411-420). A winter wash containing  $12\frac{1}{2}\%$  of a mixture of 2 pts. of mineral oil emulsion and 1 pt. of modified Long Ashton tar oil gave best results for apples where capsid bug was present. With certain varieties of apples tar oils alone may cause injury to fruit buds. A. G. P.

Wood posts [in soils].—See IX. Sludge as fertiliser.—See XXIII.

See also A., Aug., 890, Nitrate reduction by plants.

# XVII.—SUGARS; STARCHES; GUMS.

Dynamics of drying sugar beets with different sugar contents. V. DENISIVSKI and S. E. SAGANSKA (Nauk. Zapiski Tzuk. Prom., 1931, 13, 787—802).—The rate of evaporation of  $H_2O$  from sugar-beet cossettes depends on the sugar content. During the first period beets with the higher sugar content lose the less  $H_2O$ . For 1% increase in sugar the  $H_2O$  evaporated decreases by 5.6%. Near the end of the drying the conditions are reversed; 1% increase in sugar increases the  $H_2O$ evaporated by 9.3%. CH. ABS.

Determination of sugars in vegetable substances. E. BELTRAN (Ann. Chim. Analyt., 1932, [ii], 14, 58–59).— The clarification of sacchariferous vegetable extracts or org. liquids (e.g., urine, wine, etc.) is effected by flocculation of the hydrosols with colloidal  $Cu_2Fe(CN)_6$ .  $K_4Fe(CN)_6$  is added to the diluted extract, followed by an approx. equiv. of Cu (OAc)<sub>2</sub>, added drop by drop, the mixture being then centrifuged and the excess of Cu, if any, decolorised by  $Na_2CO_3$ . Sugars may then be determined polarimetrically or by Fehling's method. The results obtained were more accurate than those given by defecation with Pb subacetate. H. J. D.

Determination of reducing sugars in food products. Colorimetric method. C. F. POE and F. G. EDSON (Ind. Eng. Chem. [Anal.], 1932, 4, 300—302).— Two reagents are proposed for the colorimetric determination of reducing sugars. The first is useful for 1-10% sugar solutions and contains Na 2:4-dinitrophenoxide 8 g., 5% NaOH 200 c.c., PhOH 2.5 g., Na K tartrate 100 g., and H<sub>2</sub>O 1000 c.c. The second, used for larger amounts of reducing sugar, is prepared similarly, omitting the PhOH. Results are given for syrups, fruit products, and milk. E. S. H.

Impurities in white sugars. V. Determination of phosphorus and silica. S. BYALL and J. A. AMBLER (Ind. Eng. Chem. [Anal.], 1932, 4, 325—327; cf. B., 1932, 279).—Irregularities in the method for determining P (B., 1931, 648) are due to interference by SiO<sub>2</sub>. The cœruleomolybdate method may be adapted to determine total  $PO_4^{\prime\prime\prime}$  plus SiO<sub>2</sub>. SiO<sub>2</sub> is then removed by dehydration with HNO<sub>3</sub>, and total  $PO_4^{\prime\prime\prime}$  determined colorimetrically in the filtrate; SiO<sub>2</sub> is found by difference. Inorg.  $PO_4^{\prime\prime\prime}$  may be determined directly on solutions of the sugar. The SiO<sub>2</sub> present in white sugars is in a form which does not react in the cœruleomolybdate method until after the ash has been fused with Na<sub>2</sub>CO<sub>3</sub>. E. S. H.

Destruction of sugar on shelves and in wagonettes in sugar refineries. J. G. FELDSHTEIN (Nauk. Zapiski Tsuk. Prom., 1931, 14, 121—125).—The scrapings of the sugar and syrup returned to remelt increase the colour and the yield of invert sugar. CH. ABS.

Biochemical study of yeast candy. C. F. PoE and H. A. FEHLMANN (Univ. Colorado Stud., 1929, 18, 197— 199).—Little or no vitamin-*B* was present.

CH. ABS.

Determination of the quality of starches by a colour reaction. A. P. SCHULZ and G. STEINHOFF (Z. Spiritusind., 1932, 55, 150).—The Pekar test is modified by using a 0.2% solution of pyrocatechol to replace the H<sub>2</sub>O in which the samples of starch in layers of equal thickness are immersed to bring them to the same moisture content prior to their examination by transmitted and reflected light 15—20 min. later. Potato starch is coloured varying shades of blue according to the quality (highest quality, almost unaffected ; inferior, dark blue). Wheat starch shows shades of yellow, whereas maize starch is coloured green. The reddish colours which rye and rice starch yield are probably due to the slight alkalinity of these starches. C. R.

Microscopical analysis of starches by dyes. A. P. SCHULZ and G. STEINHOFF (Z. Spiritusind., 1932, 55, 162).—A tabulated list of staining tests for the identification of potato, wheat, maize, rye, and rice starches is given. The Triacid mixture (Me-orange, fuchsin, and Me-green) and neutral-red are most suitable for the identification of potato starch, which is coloured bright blue by the former and pink by the latter; other starches remain unstained. If the starch is in the form of a paste, or, if the grains are corroded, potato starch is best identified by metachromate-G (Agfa) or by Congo-red. There is no sharp differentiating colour

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test for the starches other than potato, but they can generally be identified by the above dyes augmented by safranine and thionine. C. R.

# Citric fermentation.—See XVIII.

See also A., Aug., 832, Fatty acids from cassava starch. 833, Sugar acids. 835, Determination of fructose. 836, Vanillin as colour reagent.

# PATENTS.

Sugar cane process and apparatus. E. M. CAPP, Assr. to PETREE & DORR ENGINEERS, INC. (U.S.P. 1,824,875, 29.9.31. Appl., 22.11.28).—In a milling train, juice from the crushers and first milling unit is defecated, yielding primary juice which goes to the evaporators, and primary mud which, together with dil. juice from the 3rd and 4th units, is returned to the bagasse leaving the first unit. Juice from the second milling unit is defecated separately, yielding secondary juice which is defecated again with the raw juice from the first unit, and secondary mud which is mixed with  $H_2O$  or dil. juice from the last milling unit and returned to the bagasse leaving the second unit. J. H. L.

Manufacture of sugar. F. B. DEHN. From SCHWIE-GER CHEM. Co. (B.P. 375,078, 23.6.31).—A process resembling that of U.S.P. 1,815,276 (B., 1932, 480) is claimed, adaptable to beet- or cane-sugar production or refining. Beet juice is limed as usual, mixed with 25-50% of green syrup, treated with 65-75 g. of  $(NH_4)_2CO_3$  per 250 cu. ft., agitated for 15 min., carbonated to 0.05-0.15% CaO, filtered, treated as before with  $(NH_4)_2CO_3$ , agitated for 2 min., partly evaporated in 3 stages, carbonated to 0.02-0.04%CaO, filtered, sulphited and filtered, evaporated to syrup, again sulphited and filtered, and boiled for white sugar; 25-50% of the resulting green syrup is mixed with raw juice and the remainder is boiled for second sugar and final molasses. J. H. L.

Manufacture of dextrose [glucose] and dextrose compounds. C. RACH (U.S.P. 1,825,603, 29.9.31. Appl., 17.3.30).—Liquids containing impure glucose, *e.g.*, grape juice, glucose syrups, or converted starch solutions, are treated with 16 pts. of NaCl to 84 pts. of glucose present, conc. to syrup, and crystallised in shallow pans. The *compound*  $(C_6H_{12}O_6)_2$ ,NaCl,H<sub>2</sub>O separates and is drained and washed with conc. glucose solution; it is suitable for cake-making etc. Glucose may be isolated from it by stirring it with half its wt. of cold H<sub>2</sub>O; the mixture first liquefies and then sets again to a stiff magma of cryst. glucose from which the liquor is removed under pressure. J. H. L.

Manufacture of vegetable gum solutions. A. PRISTER (B.P. 375,161, 9.10.31).—Tragacanth, shiraz, or other gums which swell in cold  $H_2O$  are converted into solutions substantially free from foreign matter and colour by allowing them to swell fully in cold  $H_2O$  and then filtering under pressure, *e.g.*, through wire gauze; the filtered gels are converted into solutions suitable for textile or pharmaceutical purposes by heating or other known methods. J. H. L.

Crushing mill. Syrup evaporator.—See I. Hydrolysis of cellulosic substances. Vegetable material in storage.—See V. Sugar-cane plantations.—See XVI.

# XVIII.—FERMENTATION INDUSTRIES.

[Tannins in hops, barley husks, and wort coagulum.] H. LÜERS (Woch. Brau., 1932, 49, 233; cf. B., 1932, 42, 43).—Barley husks contain a true tannin, contrary to the opinion of Heintz who considers that tannins are absent from hops, barley husks, and beer coagulum since none of the fractions extracted from those materials shows the characteristic reactions of tannins. Many true tannins do not conform to Heintz's selected criteria and are insol. in  $Et_2O$  and in  $H_2O$ , do not ppt. protein, or give a ppt. with protein which is easily sol. in hot  $H_2O$ . The discrimination of tannins is best effected from their methods of degradation and by the identification of their degradation products.

HEINTZ, in reply, considers that the term "tannin" is erroneously applied, and that a tannin which has no "tanning" action is a contradiction of terms. In addition, no single degradation product is obtained from, or is characteristic of, all tannins. C. R.

Gravimetric determination of the preservative value of hops. J. S. FORD and A. TAIT (J. Inst. Brew., 1932, 38, 351-352).-50-60 g. of cleaned hops are ground and 10 g. are extracted (Soxhlet) for 1 hr. with pure Et<sub>2</sub>O. The Et<sub>2</sub>O is distilled off on a H<sub>2</sub>O-bath, the last 5-20 c.c. being removed under reduced pressure at approx. 60°. The residue is dissolved in about 30 c.c. of hot, COMe,-free MeOH, and the solution with washings is made up to 75 c.c. with MeOH and filtered. 15 c.c. of this MeOH extract with the addition of 30 c.c. of 1% aq. NaCl are extracted with successive lots of light petroleum (b.p.  $30-40^{\circ}$ ), the filtered petroleum extracts are distilled on a H<sub>2</sub>O-bath, and the residue, which consists of the total  $\alpha$ - and  $\beta$ -resins, is dried to const. wt. in a H<sub>2</sub>O-oven at 98° in a current of air. To determine the  $\alpha$ -resin, 40 c.c. of the MeOH extract are pptd. at 60° with a slight excess of a 1% MeOH solution of Pb(OAc)2. The amount of acetate solution necessary is calc. from a preliminary titration of 10 c.c. of the MeOH extract with the acetate solution, using aq.  $Na_2S$  as indicator. The pptd. solution is heated at  $60^{\circ}$ for 5 min. and after 30 min. is filtered through a Gooch crucible and washed with MeOH. Then wt. of Pb salt  $\times 11 \cdot 8 = \%$  of  $\alpha$ -resin in the hop; the pre-servative val. is expressed as  $10(\alpha + \beta/3)$ . It is essential that the detailed method be rigorously followed.

Industrial production of pure yeast and the history of pure yeast culture in brewing. E. ELION (Chem. Weekblad, 1932, 29, 470–475).—A claim for priority. C. R.

C. R.

Mead fermentation. A. OSTERWALDER (Landw. Jahrb. Schweiz, 1931, 45, 651–659; Chem. Zentr., 1932; i, 1453).—Fermentation was accelerated by addition of Steinberg yeast,  $(NH_4)_2SO_4$ , and tartaric acid (0.5%), but the sugar was not completely decomposed in 186 days. A. A. E.

Fermentation products of Cortinellus Shiitake, P. Hem. K. SAKAGUCHI and K. NAKAO (J. Agric. Chem. Soc. Japan, 1931, 7, 1110–1112).—C. Shiitake

was grown at room temp. for 45 days in malt (5 g.) extract (1 litre) containing glucose 100 g., peptone 5 g.,  $K_2HPO_4 \ 0.3 \text{ g.}$ ,  $KH_2PO_4 \ 0.3 \text{ g.}$ , and  $CaCO_3$ .  $H_2C_2O_4$  (26.21% on the sugar used) and a small quantity of a reducing org. acid were formed. The *C. Shiitake*, a common food, contains 0.195% of  $H_2C_2O_4$ .

CH. ABS.

Citric fermentation. A. CORBELLINI and O. CANONICI (Giorn. Chim. Ind. Appl., 1932, 14, 355— 363).—The max. yield of citric acid obtained in the fermentation of sucrose and glucose by strains of Aspergillus niger isolated from soils of different origins was 32% of the total sugar. The yield was increased by cultivating the organism on malt agar and lemon juice alternately. Certain strains act on sucrose in presence of CaO or CaCO<sub>3</sub>, giving good yields of gluconic acid.

T. H. P.

Protein haze in beer. I. JANENSCH (Woch. Brau., 1932, 49, 234—235).—The haze of an unfiltered beer was diagnosed as being due to protein since no microorganisms were detected by drop-culture tests, whilst the beer became bright on warming and on the addition of a 10% solution of NaOH. Chilling the beer to 0° to  $-1^{\circ}$  increased the haze, but the latter disappeared, leaving the original opalescence when the beer again acquired the room temp. Filtered beer acted similarly when chilled and warmed. The original opalescence of the beer was due to the too high temp. of the lager cellar resulting in insufficient pptn. of protein, and the beer became hazy as soon as it reached a temp. below that of the lager cellar. C. R.

[Beer] foam. J. RAUX (Brass. Malt., 1932, 22, 85; Woch. Brau., 1932, 49, 235-238).-The chief foam-forming substances in beer are those proteins which are present in the beer in the form of colloidal particles of intermediate size, and of which the albumoses are the most important. EtOH, by lowering the surface tension of the beer, favours the formation of foam to a smaller extent. The foam is stabilised by the gums derived from the barley, which are negatively charged and react with the positively charged proteins. Stabilisation is increased by the emulsion derived from the hop resins and by the dextrins and sugars which increase the viscosity of the beer. The small amount of glyerol present increases the elasticity and prevents the coalescence of the gas bubbles. Mineral salts influence the foam in so far as they affect the  $p_{\rm H}$ , the optimal val. of which coincides with the isoelectric point of the coagul-C. R. able proteins.

Colour changes in bilberry wine. W. DIEMAIR and M. HERING (Z. Unters. Lebensm., 1932, 63, 593-611). --Myrtillin has been isolated from the wine as the picrate and is considered to be the principal source of the red colour, the secondary source being Fe<sup>···</sup>-tannin complexes. Myrtillidin, which is formed in the unripe fruit, is not produced from myrtillin by hydrolysis and does not increase in quantity during fermentation. Aq. and dil. alcoholic solutions of the wine decolorise rapidly under the action of heat and sunlight with the formation of a violet ppt., and more slowly in the dark at 20° with the intermediate formation of a cinnabarred colour. These changes are accelerated at low acidities and great dilutions, but are inhibited by salts, and by proteins and tannins which stabilise the colour by the formation of adsorption complexes. Atm. oxidation (e.g., of the Fe<sup>···</sup>-tannin complexes) is also responsible for some colour changes. Small amounts of SO<sub>2</sub> are without effect as they form additive compounds with the dye, but large amounts bleach it.

J. G.

Heavy-metal content of S. African wines and treatment of wine with potassium ferrocyanide. H. L. DE WAAL (Ann. Univ. Stellenbosch, 1932, 10A, 1-50) .- Turbidity of wines is largely due to Fe phosphate or tannate in oxidising media and to Cu salts in reducing medium. Heavy metals arise chiefly from vessels and sprays etc.; little relation was found between the heavy-metal content of grapes and that of the soil. Grapes contained 1-3 and wines 7-23 mg. per litre; turbidity appears at 8 mg. and is objectionable above 15 mg. Experiments for determining the amount of K4Fe(CN)6 required to ppt. the Fe are described. In one case 5 mg. of Cu per litre were present. Other contents (mg. per litre) are : Ca up to 110, Mg 86-112, Al 1.8-7.7, PO4 87-574, Mn 4-10. Usually the Fe content of wine is about 99% of the total heavy-metal content. Treatment of juice with SO2, with subsequent contact with Fe, raises the Fe content considerably. Tinned vessels should be employed, Fe" should be pptd. by aëration, and treatment with SO<sub>2</sub> or high acid content should be avoided. No HCN or other objectionable substances were found. CH. ABS.

Radioactivity of must and wine. E. CANALS and A. MÉDAILLE (J. Pharm. Chim., 1932, [viii], 16, 62-67). -Four musts had a radioactivity of 0.054-0.112 millimicrocurie, and the corresponding wines 0.149-0.218, decreasing to 0.044-0.102 in 4-5 months. R. S. C.

Replacement of the periodic process of rectification of alcohol by a semi-continuous process. K. R. DIETRICH (Z. Spiritusind., 1932, 55, 146).-In the semi-continuous process 80-85 wt.-% EtOH led into a still of 60,000 litres capacity yielded 725 litres per hr. of 94 wt.-% EtOH at the cost of 170 kg. of steam per hectolitre of 94 wt .- % spirit. The level of the liquid in the still was kept const. for approx. 15 days until the concn. of the spirit in the still had reached 15 wt.-%. At this stage the inflow of spirit was stopped, and after the contents of the still were distilled off, the process was again repeated. With the same apparatus and using the periodic process, 550 litres per hr. of 94 wt.-% EtOH were obtained at a cost of 240 kg. of steam per hectolitre of 94 wt.-% spirit. C. R.

Dehydration of alcohols.—See III. Corrosion of bronze by vinegar.—See X. Sugars in vegetable substances.—See XVII.

See also A., Aug., 819, Reactions of EtOH on Ni-Cr catalysts. 882, Yeast and fermentation. Osmophilic yeasts.

### PATENTS.

Elimination of immature odoriferous constituents and the like from beer. HANSENA, A.-G. (B.P. 375,145, 11.9.31. Ger., 2.12.30).—Two superimposed plates are placed at the top of the conical fermentation vessel in use in the Nathan process. The beer is introduced from the centre of the plates and issues in the form of a bell from the gap between them, impinging against and running down the sides of the vessel. At the bottom of the vessel the thin layer of beer is met by a current of cold  $CO_2$ , which checks foam and is drawn off at the top of the vessel carrying the odoriferous constituents with it. The upper part of the vessel is warmed by a hot  $H_2O$  jacket, whilst the lower part is cooled by a second jacket. C. R.

Hydrolysis of cellulosic substances.-See V.

## XIX.—FOODS.

Report of Committee on testing soft wheat flours. M. M. BROOKE (Cereal Chem., 1932, 9, 406– 407).—Suggested procedures are given for baking tests for cake, pie, biscuit, and cookie flours. Bleaching was found to improve cake flours. E. B. H.

Separation of characteristics of variety and effects due to the place of origin as a useful means of wheat evaluation. E. BERLINER (Mühlenlab., 1932, 41-44).—Gluten-quality tests provide a rapid and reliable means of assessing the baking qualities of wheat of different varieties and from different regions. W. J. B.

Separation of diastatic power and essential baking quality in carrying out baking tests. H. JGERGENSEN (Mühlenlab., 1932, 34–38).—With doughs prepared from flours of medium or low diastatic power, even with addition of sugar, a crit. point occurs in the fermentation process at which the rate of evolution of  $CO_2$  suddenly decreases. In testing the baking quality of flour apart from diastatic power, the quantity of yeast used and the fermentation time should be adjusted so that the crit. point is not reached even although the flour is of low diastatic power. For this purpose the addition of 0.9% of yeast and adoption of a fermentation time of 190 min. at 30° are found suitable.

W. J. B.

Proteolytic power of wheaten flour and its importance in the evaluation of baking quality. T. BIÉCHY (Mühlenlab., 1932, 34).—Only when wheat has ripened under abnormal conditions is a determination of the proteolytic power of the flour a useful index of baking quality. W. J. B.

Swelling of wheat kernels. W. KRANZ (Mühlenlab., 1932, 45—46).—Wheat soaked in  $H_2O$  at 20° increases in wt. and vol. rapidly during the first hr. and more slowly thereafter. The vol. increase varies greatly according to quality and variety of the sample. Samples of higher protein content and stronger gluten quality appear, in general, to exhibit greater true swelling (difference between vol. increase and wt. increase). Germinated wheat shows a high wt. increase but minimal true swelling. True swelling increases with rising temp. of the  $H_2O$  used. W. J. B.

Heating of feed grains. H. J. SMITH and J. P. BARTZ (Cereal Chem., 1932, 9, 393-401).—Respiration of stored grain is an exothermic reaction and varies with the temp. and R.H. of the surrounding air. The moisture content for safe storage of cereals should be  $\geq 13.5\%$ . E. B. H. Effects of the amount and kind of bleach used on flour in relation to its ageing. W. H. HANSON (Cereal Chem., 1932, 9, 358–377).—NCl<sub>3</sub> and  $Cl_2$ produce immediate "ageing" of flours. No loss in baking quality was noticed on keeping bleached flours for 9 months. E. B. H.

Sugars, diastatic activity, and "gassing power" of flours. M. J. BLISH, R. M. SANDSTEDT, and G. R. ASTLEFORD (Cereal Chem., 1932, 9, 378–393).—The Rumsey method for determining diastatic activity is modified by increasing the  $H_2SO_4$  concn. and cooling to 0°, thus reducing diastatic activity to the min. The reducing sugar content of flour is nearly const. and about 0.15%. "Diastatic activity" and "gassing power" of a flour are related but not synonymous, owing to differences in the sucrose content. E. B. H.

Some effects of varying sugar concentrations in bread dough on fermentation by-products and fermentation tolerance. M. J. BLISH and R. C. HUGHES (Cereal Chem., 1932, 9, 331–356).—The variation during fermentation for 24 hr. of the  $p_{\rm H}$ , EtOH content, total acid, CO<sub>2</sub>, and non-volatile acid in dough is shown graphically. Fermentation tolerance of flours is found to depend entirely on the maintenance of gas production; gluten degradation is unimportant.

E. B. H.

Detection of powders used as aids to bakery. H. SIMONS (Mühlenlab., 1932, 46—47).—Pap's methods (B., 1932, 574) are criticised and improvements suggested. In the detection of B in flour, 50 g. of sample are treated with 50 c.c. of 2% alcoholic NaOH and after the EtOH has been burnt off the sample is carbonised. The mass is extracted repeatedly with hot  $H_2O$  and the decanted liquid evaporated to dryness. A few c.c. each of pure MeOH and conc.  $H_2SO_4$  are added to the residue and a light is applied to the mixture. A green flame indicates the presence of B. W. J. B.

Report of Committee on methods of analysis [in food chemistry]. C. E. MANGELS (Cereal Chem., 1932, 9, 429--431).—Recommendations of methods for use in connexion with wheat, flour, feeds, sugar, leavening agents, and baking materials are made. E. B. H.

Magnesium acetate-alcohol method for ashing flours. C. F. DAVIS (Cereal Chem., 1932, 9, 431-437). --3 c.c. of a solution of 15 g. of Mg(OAc)<sub>2</sub> in 1000 c.c. of denatured EtOH are added to the flour, which is then ashed at 850°. Ashing is complete in 30-45 min. and a satisfactory degree of accuracy can be obtained. E. B. H.

Rapid determination of ash [of flour]. W. KRANZ (Mühlenlab., 1932, 33–34).—The time of incineration of the flour sample can be lessened considerably by using an EtOH solution of  $Mg(OAc)_2$ , as recommended by Spalding (B., 1930, 584), and incinerating at 800°, the use of  $O_2$  being omitted. W. J. B.

Pasteurisation of milk. O. SKAR (Milch. Zentr., 1932, 61, 173-177, 185-190).—The effect of different methods of short high-temp. (85°), short low-temp. (70-75°), and time-pasteurisation (63° for 30 min.) on milk is discussed. The economy of different processes, their effect on the nutritional val. and palatability of

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milk, and their relative advantages and disadvantages are considered. E. B. H.

Antineuritic vitamin content of boiled milk. J. P. SPRUYT and W. F. DONATH (Med. Dienst Volksgezondh. Ned.-Indië, 1932, [i], 64—70. Reprint).—Milk retains its antineuritic properties on being boiled.

H. D. Determination of citric acid in milk. B. ROGINA (Arh. Hemiju, 1932, 6, 119–137).-2N-H<sub>2</sub>SO<sub>4</sub> is added to complete pptn. of caseinogen to 100 c.c. of milk diluted to 200 c.c., and excess of CaO is added to the filtrate. The solution is again filtered, and 20 g. of Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O, 5 g. of NaNO<sub>8</sub>, and 20 c.c. of Denigès' reagent are added to 40 c.c. of the filtrate, which is boiled and again filtered. The filtrate and washings are made up to 400 c.c., 3-4 g. of MnSO<sub>4</sub> are added, and saturated aq. K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (1 drop per 10 mg. of citric acid) is added drop by drop to the boiling solution. Oxalacetic and acetonedicarboxylic acid, formed by oxidation of citric acid, are pptd. as a double salt with HgSO<sub>4</sub>; citric acid is calc. from the Hg content of the ppt. R. T.

Changes in milk fat due to drying the milk. K. TEICHERT and R. KOTTERER (Milch. Zentr., 1932, 61, 157—158).—The observation that milk powder prepared from whole milk is not so suitable for the manufacture of milk chocolate as that from skim milk has been verified. The unpleasant taste developed by chocolate made from whole-milk powder has been traced to the rancidification of the fat after storage for a few days. The I val., refractometer val. at 40°, and acid val. were, respectively,  $34 \cdot 8$ ,  $43 \cdot 0$ , and  $2 \cdot 13$  for the dried milk fat,  $37 \cdot 3$ ,  $43 \cdot 4$ , and  $0 \cdot 56$  for rancid butter fat, and  $39 \cdot 9$ ,  $43 \cdot 7$ , and  $0 \cdot 56$  for fresh butter fat. H. J. D.

Determination of fat in milk powders. D. FLORENTIN (Ann. Falsif., 1932, 25, 88-91).-Results given by (A) the Dutch (Weibull's) and (B) the French methods are compared. With non-sweetened powders method A consists in treating a 2.5-g. sample for  $\frac{1}{4}$  hr. with 30 c.c. of 8N-HCl and 20 c.c. of H<sub>2</sub>O. After boiling and filtering, the residue is dried and extracted with Et<sub>2</sub>O. In method B a 2-g. sample is extracted direct for 5 hr. with Et<sub>2</sub>O, the solvent being then evaporated and the residue dried and weighed. The results given by method B proved to be higher than those from A although the contrary had been reported. With sweetened milk powders, however, direct extraction is inapplicable as the sugar encases the fat globules and prevents complete extraction. When the samples are in solution the various methods give practically Ĥ. J. D. identical results.

Sterilisation of dairy-farm utensils with dry heat. A. C. DAHLBERG and J. C. MARQUARDT (New York State Agric. Exp. Sta. Bull., 1932, No. 612, 16 pp.). —Drying apparatus using dry air is described. The efficiency compares favourably with steam-sterilisation. Heating at  $\leq 93^{\circ}$  for 10 min. is sufficient. E. B. H.

Bacteriology of common salt. VI. Salting of butter and margarine. W. CLAYTON (Foof Manuf., 1932, 7, 240-241).—A review dealing with work on the inhibition of bacteria and moulds at different salt concns. in butter and margarine. Defects in butter flavour due to bacterial causes are prevented by 2.5%of NaCl, but above 3% it may inhibit desirable lactic acid strains and permit objectionable micro-organisms to develop. (Cf. B., 1932, 784.) E. B. H.

Determination of total solid matter and density of egg-white by refractometer. H. J. ALMQUIST, F. W. LORENZ, and B. R. BURMESTER (Ind. Eng. Chem. [Anal.], 1932, 4, 305-306).—The total solid content and d of egg-white are linear functions of n. Total solids cannot be determined from n for egg-yolk or whole egg. E. S. H.

Quality of potato flakes. E. LÜHDER (Z. Spiritusind., 1932, 55, 149—150).—Potato flakes having moisture content  $\gg 15\%$  remain quite stable under normal conditions. If such flakes deteriorate through the development of fungoid or bacterial growths, the causes must be due to faulty transport, storage, or other external factor, and not to the moisture content.

C. R. Castor seed in feeding-stuffs. F. R. DODD (Analyst, 1932, 57, 488-492).—Seeds which may be mistaken for castor seed include grape and ucuhuba, both of which are harmless, and croton and curcas, which are more poisonous than, and are usually returned as, castor seed. Photomicrographs are given.

T. McL. Vitamin-B in peanut. F. W. SHERWOOD and J. O. HALVERSON (J. Agric. Res., 1932, 44, 849—860).— Heating the peanut at 149° in oil destroys most of the vitamin-B in the seed coats. In the raw kernel the antineuritic vitamin predominates over the antipellagric. H. D.

Preservation problems in the dairy industry. J. H. NAIR (Ind. Eng. Chem., 1932, 24, 671-674).— Fluid milk, conc. milks, butter, cheese, and cream are discussed. Among developments considered are: the use of paper containers for milk and vac. packages for certain products; the storage of butter at  $-17^{\circ}$  or below; elimination of Cu from dairy plant; the use of frozen cream; the prep. of cream with 80% butter fat. \_\_\_\_\_\_E. B. H.

Tomato preserves. C. GRIMME (Pharm. Zentr., 1932, 73, 483-486).—Details are given for the determination of dry wt., Cl, sugar, volatile and non-volatile acids, dyes, and starch in preserved tomatoes.

R. S. C.

Hydrogen-ion concentration in [food-]preservative action. W. V. CRUESS (Ind. Eng. Chem., 1932, 24, 648-649).—Preservative action of NaOBz, Na salicylate, Na<sub>2</sub>SO<sub>3</sub>, and KOAc was many times greater at  $p_{\rm H}$  2-4.5 than at  $p_{\rm H}$  5-9, whilst that of NaCl and CH<sub>2</sub>O was not affected. The tests were on moulds, yeasts, fermentation bacteria, and acid-intolerant bacteria. B. botulinus grew and produced toxin in 0.8% NaOBz solution at neutrality. E. B. H.

Sulphur dioxide as dried fruit preservative. P. F. NICHOLS and W. V. CRUESS (Ind. Eng. Chem., 1932, 24, 649-650).—The amounts of  $SO_2$  permitted in dried fruits in certain countries are considered to be too low since  $SO_2$  has no toxic effect in fruits and is lost by oxidation on storage and also on cooking. Vitamins are preserved by sulphuring before drying. E. B. H.

Preserving orange juice by freezing. M. A. JOSLYN (Ind. Eng. Chem., 1932, 24, 665-668).—A survey of the methods advocated for freezing orange juice is given; the chief reason for deterioration is said to be oxidation, and this can be very largely prevented by de-aëration of the juice and handling under an inert gas. E. B. H.

Lemon juice in foodstuffs. W. E. BAIER and J. W. STEVENS (Food Manuf., 1932, 7, 233–236).—The use of stable conc. lemon juice in the prep. of canned prunes, cherries, and other fruits, tomato juice, and mayonnaise is discussed. Flavour and keeping properties are said to be improved. E. B. H.

Use of hexylresorcinol as a food preservative. H. W. FEHLMANN and C. F. POE (J. Colo. Wyo. Acad. Sci., 1931, 1, No. 3, 21-22).—Low concns. were effective bactericidal agents in milk and tomatoes, but higher concns. (1 in 100-250) were necessary for meat and eggs. In the concns. administered it was not deleterious to rats. CH. ABS.

Sterilising canned foods. G. V. HALLMAN and R. G. STEVENS (Ind. Eng. Chem., 1932, 24, 659-661).— The factors involved in determining the sterilising times and temp. required are discussed. E. B. H.

Stabilising nutrients by canning. E. F. KOHMAN (Ind. Eng. Chem., 1932, 24, 650-654).—A survey is given of the accomplishments of the canning industry. Data show that most nutritional properties of canned foods compare very favourably with those of foods cooked in the normal manner. One difficulty yet to be overcome is the change in colour on canning foods (chiefly those containing chlorophyll). E. B. H.

Microbiology of canning. W. D. BIGELOW and E. J. CAMERON (Ind. Eng. Chem., 1932, 24, 655-658). -A survey of recent work on spoilage by thermophiles is given. This is of three types, viz., flat-souring, sulphide spoilage, and spoilage by gas-forming thermophiles. Non-acid products are most liable to spoilage. Contamination occurs from sugar and wooden equipment. E. B. H.

Micro-organisms as affecting frozen foods. C. A. Macoon (Ind. Eng. Chem., 1932, 24, 669-671).--Moulds and yeasts are most liable to attack foods in a frozen state, and can be controlled by packing under vac. Bacterial spoilage is liable to occur during and after thawing, owing to the physical changes produced. Washing and blanching improve the keeping quality. E. B. H.

Influence of the nature of the containers on the quality of preserved foods [vegetables]. MACHE-BGUF, H. CHEFTEL, and J. BLASS (Ann. Falsif., 1932, 25, 68—80).—The reputed superiority in flavour of canned over bottled vegetables has been examined by tests with green peas, French beans, spinach, and tomatoes. Only in the last case was the expert evidence definitely in favour of the canned product, the remaining canned products being voted slightly superior. In neither type of container could the presence of  $H_2S$  be detected, and there was no difference in the acidities of the pairs of products. Chemical attack of the glass was insignificant. The slightly lower savouriness of the bottled vegetables was ascribed to the slight overcooking due to the simmering which results from the slow rate of cooling after removal from the autoclave. Slow cooling of the glass to avoid fracture also permits the germination at 40—60° of the spores of thermophilic bacteria which are capable of conferring a sour taste without rendering the product harmful to the consumer. H. J. D.

Rapid centrifugal method for pectic acid determination. C. R. FELLERS and C. C. RICE (Ind. Eng. Chem. [Anal.], 1932, 4, 268—271).—The filtered fruit juice or extract is hydrolysed by NaOH in a small, graduated centrifuge tube, the pectic acid formed is pptd. by HCl, flocculated by heating on a H<sub>2</sub>O-bath, cooled, and centrifuged. The vol. of ppt. is measured and compared with a standard result checked by the gravimetric method. The error is about  $5 \cdot 5\%$ . E S. H.

Corrosion of metals in milk.—See X. Reducing sugars in foods.—See XVII.

See also A., Aug., 825, Determination of  $PO_4^{\prime\prime\prime}$ [in egg, milk, etc.]. 836, Vanillin as colour reagent. 871, Composition of *Pleuronectes* and mackerel. 886—7, Vitamins (various).

#### PATENTS.

Cooking and sterilising comestibles by steam or other vapours under pressure and apparatus therefor. R. W. WEBSTER (B.P. 377,890, 5.1.32).

Cellulose sheet [for foods].—See V. Glucose compounds.—See XVII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation and evaluation of tinctures. H. BREDDIN (Pharm. Ztg., 1932, 77, 783—784).—Methods of preparing tinctures are critically reviewed with special reference to the occurrence of turbidity. A technique employing an effective suspension of drug in menstruum followed by percolation is described and claimed to be rapid and efficient. A. C.

Turbidimetric measurements for pharmaceutical preparations. S. CLAMAN, C. J. CARR, and J. C. KRANTZ, jun. (J. Amer. Pharm. Assoc., 1932, 21, 670– 671).—Turbidity standards (*i.e.*, 1 g. of dry, pptd. fuller's earth sifted through 200-mesh into 1 litre of  $H_2O$  has a turbidity of 1000) used with the Baylis turbidimeter (cf. A., 1926, 378) for measuring the degree of clarity of liquids are described. E. H. S.

Physical [constants] study of medicinal comphorated oils. FAURE (J. Pharm. Chim., 1932, [viii], 16, 58—62).—Addition of camphor (I) to olive oil does not greatly affect the  $d, n, \eta$ , or capillarity of the oil, but the lowering of the crit. solution temp. of the oil in EtOH or AcOH by natural (not "artificial") (I) can be used for approx. determination of (I) in commercial oils. Camomile oil can be distinguished from poppy oil by the higher crit. solution temp. of the latter in 95% EtOH. R. S. C.

New numerical characteristics of drugs and galenicals. I. Copper numbers of some drugs. J. A. MÜLLER (Pharm. Zentr., 1932, 73, 450-457).--

The characterisation of a drug by numerical designations of its constituents or reactions (e.g., N val., sap. val., etc.) is discussed. The Cu no. is defined as the equiv. amount (mg.) of Cu reduced when 1 g. of the material is heated with Fehling's solution. A second or "inversion" Cu no. is the corresponding val. after hydrolysis for 15 min. with 20% HCl, the ratio of the two being the Cu-no. quotient. With drugs treated directly with Fehling's solution, or where extraneous ppts. are formed, the Cu<sub>2</sub>O is determined by addition of Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and back-titration of the excess with 0.1N-KMnO<sub>4</sub>. Detailed methods and typical results are given.

F. O. H.

[Colour] reactions of some guaiacol preparations. L. EKKERT (Pharm. Zentr., 1932, 73, 504—505). --Colour reactions of guaiacol, its carbonate, "kreosotum fagi," "kreosotal," K guaiacolsulphonate, and "kreosolsulphonate" with FeCl<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and H<sub>2</sub>SO<sub>4</sub>-CH<sub>2</sub>O are described. R. S. C.

[Colour] reactions of istizin [1:8-dihydroxyanthraquinone] and chrysophanic acid. L. EKKERT (Pharm. Zentr., 1932, 73, 486–487).—Distinguishing colour reactions are described. R. S. C.

[Colour] reactions of veronal, luminal, atophan, and novatophan. L. EKKERT (Pharm. Zentr., 1932, 73, 487–488).—Distinguishing colour reactions are described. R. S. C.

Preparation, differentiation tests, and evaluation of varieties of aloe. K. GOLDNER (J. Amer. Pharm. Assoc., 1932, 21, 658—668).—Present methods for the determination of aloin and evaluation of aloe are criticised. Hydrolysis of aloin with either 3% aq. or alcoholic HCl or KOH is incomplete and the determination of arabinose liberated either by hydrolysis or by treatment with Na<sub>2</sub>O<sub>2</sub> (9% decomp. of aloin) or NaBO<sub>3</sub> (30% decomp.) has no quant. val. The possibility of determination by acetylation is discussed. E. H. S.

Fluid extract of Rhamnus purshiana. C. SCHOU-SEN (Dansk Tidsskr. Farm., 1932, 6, 105-115).-Determinations of the amount of solid residue left on evaporation of an extract of the cortex are not satisfactory as a means of comparing different samples of the material, and the following method is recommended. 2 g. of extract are diluted to 100 c.c. with 50% EtOH, and 5 c.c. of the solution are mixed with 5 c.c. of 12% H2SO4. After leaving overnight, the whole is heated on a waterbath under reflux for 2 hr. The resulting hydroxymethylanthraquinones (emodin) are extracted with Et20. 10 c.c. of the extract are shaken with 10 c.c. of 25% aq. NH3, the ammoniacal layer is separated, and compared colorimetrically with a frangula extract of known emodin content. The method is regarded as a satisfactory means of comparing different commercial preps. In preparing cascara extract too dil. spirit must not be used, and the material should remain for 4 days before H. F. H. percolation commences.

Indian ipecacuanha. R. N. CHOPRA and B. MUK-HERJEE (Indian Med. Gaz., 1932, 67, 88—90).—The chief alkaloids are emetine, cephaeline, and psychotrine; ipecacuanhic acid, methylpsychotrine, emetamine, and much starch are also present. CH. ABS. "Peperina," Bystropogon mollis, Kth. P. J. PREIONI (Rev. farm., 1931, 74, No. 5, 173–183; 1932, 74, No. 6, 1–30).—The air (shadow)-dried drug contains  $H_2O$  11·2, ash 5·71 (Si, S, K, Na, Fe, Al, Mn, Mg, P,  $CO_2$ ), pectins 3·46, extractable colouring matter 6·59, sol. protein 7·72, total protein 11·93, insol. 37·79%. Light petroleum extracts 3·97% of fat, wax, and colouring matter; EtOH extracts 2·56% of a soft aromatic substance. The essential oil (4·5%) has  $d^{15}$  0·9108,  $[\alpha]_D$ —0·93°,  $n^{15}$  1·47, m.p.—24·2°, b.p. 204°, and contains total menthol 46·60, free menthol 43·0, menthone 5·40%. CH. ABS.

Assay of hyoscyamus. N. C. SCHALLER and L. H. BALDINGER (J. Amer. Pharm. Assoc., 1932, 21, 442— 444).—The U.S.P. X method for the assay of both the crude drug and tincture gives higher yields of alkaloid if the solvents are evaporated at room temp. *i.e.*, *in* vacuo, instead of by heat. E. H. S.

Determination of the alkaloids of Peganum harmala. S. M. BOLOTNIKOV (Farm. Zhur., 1931, 362—364).—The powdered root (4 g.) is refluxed for 1 hr. with 80% AcOH (4 g.) and 95% EtOH to 120 g.; the mixture is conc. to 24 g. and the 20-g. aliquot part is left overnight with 10 c.c. of 15% NaOH. The ppt. is washed, dried at 60—70°, and extracted with CHCl<sub>3</sub>; the residue on evaporation is dried at 60—70°. The method is applicable to tinctures. Ch. Abs.

Determination of santonin by means of 2:4dinitrophenylhydrazine. O. FERNANDEZ and L. SOCIAS (J. Pharm. Chim., 1932, viii, 16, 49–54).— Santonin in commercial powders is determined with an accuracy of  $\pm 1\%$  by extraction with C<sub>6</sub>H<sub>6</sub>, dissolution of the residue in EtOH, and pptn. as 2:4-dinitrophenylhydrazone, m.p. 267–268° (decomp.). Chocolate is shown by a similar method (but using ligroin instead of C<sub>6</sub>H<sub>6</sub>) to contain 1–1·3% of santonin. R. S. C.

Titration of certain alkaloids. M. G. MELLON and J. TIGELAAR (J. Amer. Pharm. Assoc., 1932, 21, 672-675).—Modified Me-red (1.25 g. Me-red and 0.825 g. methylene-blue per litre of H<sub>2</sub>O) is the best indicator for the titration of brucine, strychnine, and atropine. Potentiometric methods using the quinhydrone electrode are more generally applicable, but are not as simple or rapid as the volumetric determinations. E. H. S.

Determination of alkaloids in calabar beans and aconites. F. GSTIRNER (Pharm. Zentr., 1932, 73, 465-467).—Standard volumetric methods are improved. R. S. C.

Exotic and naturalised essences in the southwest and part of Central France. P. BUFFAULT (Bull. Inst. Pin, 1932, 127—132, 145—149).—Afforestation and tree replacements in the Landes, Perigord, Agenais, Limousin, and the Massif Central have been particularly successful with: *Pinus laricio corsica*, Math.; *Picea excelsa*, Link.; *Larix europæa*, D.C.; *Pseudo-touga Douglasii*, Carr.; *Pinus strobus*, Hamilt.; *Picea sitchensia*, Varr., *Abies Nordmannianes*, Spach.; and *A. concolor*, Lindl. Preferably these are mixed: *Pinus sylvestris*, L.; *Picea excelsa*, Link.; and Larix europæa, D.C., succeed well together in the Cantal, and a mixture of *Pinus sylvestris*, L., and *P. laricio corsica*, Math., has also given good results. C. H.

Composition of essence of Pinus longifolia.  $\alpha$ - and  $\beta$ -Carenes. G. DUPONT and [MLLE.] M. JOFFRE (Bull. Inst. Pin, 1932, 142—144).—The non-acid products of ozonisation of carene from essence of *P. longifolia* give the semicarbazone, m.p. 210—211°, of the cyclic ketone corresponding with  $\beta$ -carene (about 4%) in head fractions), and the mono- and di-semicarbazides of the aldehydo-ketone from  $\alpha$ -carene. The Raman spectra of the fractions of the essence show the presence of pinene, nopinene, and  $\alpha$ - and  $\beta$ -carenes, the last in much less amount than  $\alpha$ -carene. C. H.

Pine-needle extracts. C. A. ROJAHN and H. FILSS (Pharm. Ztg., 1932, 77, 829—833).—Sulphite waste liquors have average S and ash contents of  $7 \cdot 2$  and  $20 \cdot 8\%$ , respectively. The corresponding figures for pine-needle extract (A) are 0.65 and 7.3%. These figures may be used for detection of adulteration of A. R. S. C.

Essential oil of Wintera colorata. J. MELVILLE and (in part) A. A. LEVI (J.S.C.I., 1932, 51, 210r; cf. A., 1926, 981).—The oil obtained by steam-distillation of the leaves contains  $\beta$ -pinene,  $\alpha$ -limonene, dipentene, dicyclic sesquiterpenes giving cadalene and azulene with S, (?) geraniol and citronellol, a sesquiterpene alcohol, m.p. 67°, and similar non-cryst. alcohols giving cadalene and azulene on dehydrogenation. The alcohols are present mainly as acetates. The hot principle extracted from the leaves with light petroleum can be conc. in a fraction, b.p. 180°/12 mm, A. A. L.

Sandalwood and its substitutes. J. C. MABY (Analyst, 1932, 57, 515).—Two photomicrographs are given illustrating the tangential longitudinal sections of sandalwood and kalamet wood. T. McL.

Chemical and physical determinations on the gum and volatile oils of asafœtida. J. F. CLEVENGER (J. Amer. Pharm. Assoc., 1932, 21, 668—670).—Characteristics of 15 samples of the gum, typical of 41 lots entering New York over a period of 3 years, are given. The EtOH-sol. extractive, determined by the U.S.P. IX method with a suitable correction for H<sub>2</sub>O (cf. B., 1928, 765), varied from 54.5 to 74.7% and H<sub>2</sub>O 2.8—11.8%. The U.S.P. IX method alone and the U.S.P. X method are both inaccurate. The yields of volatile oil were 7.5—12.0 c.c. per 100 g., having  $d_{20}^{20}$  0.906—0.973,  $\alpha^{20}$  —9.0° to +9.3°,  $n^{20}$  1.493—1.518, and 8 15.3—29.0%. E. H. S.

Analysis of essential oils. C. LAGNEAU (Ann. Falsif., 1932, 25, 80—88).—The difficulties are reviewed and the respective vals. of physical and chemical methods are assessed. A plea is made for the establishment of international standards. H. J. D.

Ca gluconate.—See VII.

See also A., Aug., 827, Colorimetric determination [of cholesterol]. 840, Trypanocides from  $\beta$ -naphthylamine-4 : 6 : 8-trisulphonic acid. 843, Syntheses of pharmacologically important amines. 844, Germicidal aromatic sulphides. 845, Esters of dialkylaminomethyl alcohols. 846, Novocaine analogues. 856, Amyrilenes. 862, Hypnotic hydantoins. 865, Rauwolfine. Alkaloids of Heliotropium lasiocarpum and Cinchona. 866, Strychnos alkaloids. Narceine. 868, Micro-reactions of atropine. 866—7, Vitamins (various). 888, Glucosides of tobacco leaf. Essential oil of Chrysothamnus nauseosus. 889, Asarum oil. Alkaloids of Leontica Eversmanii.

#### PATENTS.

Manufacture of easily soluble salts of acylaminophenolarsinic acids. I. G. FARBENIND. A.-G. (B.P. 373,269, 21.2.31. Addn. to B.P. 341,405; B., 1931, 463).—Other alkali metals are substituted for one or both equivs. of Na in the process of the prior patent. The products of both patents are also made by treating the mono-alkali metal salt with a second equiv. of the same or a different alkali. Na Li, Na K, and K Li salts of 3-acetamido-4-hydroxybenzenearsinic acid, and the Na<sub>2</sub> salt of the 3-benzamido-compound are described. C. H.

Manufacture of hormone-like substances [from coal etc.]. SCHERING-KAHLBAUM A.-G., Assees. of S. ASCHHEIM (B.P. 375,363, 19.1.31. Ger., 17.1.30).— Bituminous coal or the alkaline solution of humic acids derived from brown coal is extracted with an org. solvent immiscible with  $H_2O$ , e.g., CHCl<sub>3</sub>, Et<sub>2</sub>O, or ligroin, and the extract, after removal of the solvent, is again extracted with an org. solvent miscible with  $H_2O$ , e.g., EtOH. Tar oils, petroleum, etc. may be similarly treated except that the order in which the solvents is used is reversed. Substances are obtained which in their action of excitation of rutting are equiv. to the female sexual hormone. A. B. M.

Production of prostate hormone. F. B. ELDRED, Assr. to REED & CARNRICK (U.S.P. 1,835,853, 8.12.31. Appl., 1.11.27).—Fresh prostate glands are comminuted and dried in vac., fats are removed by extraction with light petroleum, and the residue is dried and ground to a coarse powder which is stirred with 5% H<sub>2</sub>SO<sub>4</sub> for 8—10 hr. and then with H<sub>2</sub>O for 8 hr. Both extracts are combined, centrifuged, and nearly neutralised with NaOH. A slight excess of Na<sub>2</sub>CO<sub>3</sub> is then added to ppt. the hormone, which is separated by centrifuging, washed with 0.2% aq. Na<sub>2</sub>CO<sub>3</sub>, and dried.

A. R. P.

Org. compounds containing SH.—See III. Gum solutions.—See XVII.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsion: analysis for nonhalide silver and soluble bromide. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1932, 8, 711-727). --Various methods have been investigated. The method of Weigert and Lühr (double fixation in  $Na_2S_2O_3$ ) for determining non-halide Ag is found to be reliable; Schmidt and Pretschner's method (HNO<sub>3</sub> extraction) is too insensitive. Sol. bromide may be quantitatively extracted with three changes of dil. acid (H<sub>2</sub>SO<sub>4</sub> etc.), Ag-gelatin complex formation being inhibited. Electrometric titration (at  $p_{\rm H} \ge 2$ ) is best for liquid emulsions and is preferable for all determinations. J. L.

Measurement of graininess [of photographic emulsions]. S. D. THREADGOLD (Phot. J., 1932, 72, 348—351).—A new, non-visual method of measurement, giving definite vals., is proposed. The densities of a photographic deposit are measured to parallel light  $(D_{\parallel})$ , and to diffuse light  $(D_{++})$ , at a series of densities, with an adapted B.P.R.A. photo-electric densitometer. From the straight-line graph,  $\log D_{\parallel} : \log D_{++}$  (cf. B., 1916, 385), a "graininess ratio"  $10D_{\parallel}/D_{++}$ , when  $D_{++} = 0.5$ , is obtained. The order of the results for a series of emulsions is in good agreement with visual measurements. J. L.

Increase of the sensitivity of dichromated gelatin. L. LOBEL and DUBOIS (Bull. Soc. Franç. Phot., 1932, 19, 57–62).—The optimum  $p_{\rm H}$  for the sensitisation of dichromated gelatin with CeCl<sub>3</sub> (cf. Tritton, B., 1929, 623) is about 3; at  $p_{\rm H}$  3.5 partial hardening occurs, and at  $p_{\rm H}$  2.5 the gelatin strips very easily but the sensitisation is small. The theory of these results is discussed. J. L.

Standardisation of negative material for photography. R. LUTHER (Z. wiss. Phot., 1932, 31, 83–95). —The proposals of the German Standards Committee for Phototechnique are detailed. The Davis and Gibson filters (A., 1931, 592) are used, but, contrary to American proposals, a step-wedge is preferred for grading exposure. A standard metol-quinol (with  $K_2CO_3$ ) developer is prescribed, and the evaluation of the results is considered in detail. J. L.

Calculation of the correct and most economical concentrations of elon and hydroquinone in a borax developer for motion-picture film. A. M. GUNDELFINGER (J. Franklin Inst., 1932, 214, 223—236). —The development time, temp., and agitation of the developer being const., with an elon-borax-sulphite developer (wt. of borax per gal. = wt. of elon per gal. [E] + 8 g.), a linear relation exists between  $\gamma$  and log E. This holds also for an elon-quinol-borax-sulphite developer; with this developer  $\gamma$  also varies linearly with log Q (Q = quinol concn.). The relation between  $\gamma$ , log E, and log Q is calc. A graphical method for the calculation of the optimum composition of a developer, consistent with good development and cheap cost, is described. J. L.

Ives' polychrome process of colour printing. F. E. Ives (Brit. J. Phot., 1932, 79, Col. Suppl., 29-31). --Full working details of the process are given (cf. B., 1932, 657). J. L.

### Measuring temp. distribution etc.-See I.

See also A., Aug., 821, Photochemical reactions. Photo-reduction of  $In_2O_3$ . Nature of latent images. Examination [of fluorescence] with ultra-violet light. 826, Determination of Ag.

### PATENT.

## Cellulose acetate film.—See V.

# XXII.—EXPLOSIVES; MATCHES.

# PATENTS.

Manufacture of black powder. R. L. HILL, Assr. to ATLAS POWDER Co. (U.S.P. 1,833,573, 24.11.31. Appl., 25.10.28).—A low explosive for blasting comprises a mixture of 40—98% of granules of black powder with 60-2% of granules of  $\mathrm{NH}_4\mathrm{NO}_3$  or  $\mathrm{NH}_4\mathrm{ClO}_4$ , the granules of the two constituents having previously been coated with graphite to reduce the hygroscopicity of the mixture. A. R. P.

Production of a [nitroglycerin] explosive. C. H. CORDIE and W. DE C. CRATER, Assrs. to HERCULES POWDER CO. (U.S.P. 1,833,454, 24.11.31. Appl., 25.9.29). —A 52:48 mixture of  $H_2SO_4$  and  $HNO_3$  is treated at 0—2° with one fifth of its wt. of glycerin added slowly; during the addition of the second third of the glycerin about 7% (based on the wt. of glycerin) of PhMe is added. Diglycerol, glycol, or sugars may wholly or partly replace the glycerin. A. R. P.

## XXIII.—SANITATION; WATER PURIFICATION.

Capacity of drying agents for gas masks. M. BLUMER, C. J. ENGLEDER, and A. SILVERMAN (Ind. Eng. Chem. [Anal.], 1932, 4, 339–340).—The wt. of  $H_2O$  absorbed by a const. vol. of the drying agent decreases in the order  $Al_2O_3$ ,  $Mg(ClO_4)_2$ ,  $CaCl_2$ , BaO,  $CaCl_2$  + soda-lime (1:1), BaO<sub>2</sub>,  $Al_2(SO_4)_3$ , NaOH pellets, Na<sub>2</sub>SO<sub>4</sub>, MgCl<sub>2</sub>, MgSO<sub>4</sub>, CaO. This is not the order of aq. v.p. E. S. H.

Poisoning by carbon monoxide from a gasheater. G. R. LYNCH (Analyst, 1932, 57, 516—518).— A gas-heater burning at  $\frac{1}{2}$  of full gas supply gave only 200 p.p.m. of CO in the products of combustion, but at  $\frac{3}{4}$  supply the exit gases contained 7100 p.p.m. This amount proved lethal in a small, badly ventilated room. T. McL.

Activated sludge process at Salinas, Calif., with particular reference to the causes and control of bulking. T. R. HASELTINE (Sewage Works J., 1932, 4,461-489).—Pronounced short-circuiting in the spiralflow aëration tanks has been reduced by means of diagonal baffles which break up the core of quiescent liquor. By means of sludge re-aëration, the quantity dealt with has been increased by 60% in the same tankcapacity and when using the same amount of air per gal., the vol. necessary to avoid bulking being  $3\cdot 4$ cu. ft. Excessive adsorption of org. matter is the primary cause of bulking, and the best cure is by increased aëration. The amount of sludge used in the plant is small, but it is kept in a highly efficient condition by carrying the process to the nitrification stage. C. J.

Activated-sludge plant at Phœnix, Ariz. D. TRAVAINI (Sewage Works J., 1932, 4, 525—529).—The aëration chambers of this new plant are 330 ft. long, 27 ft. wide, and 14 ft. deep, and have a central longitudinal line of air diffusers raised 4 ft. above the lowest part of the floor. On each side of the diffuser line is a longitudinal paddle wheel operating at 4 r.p.m. against the flow of air, both halves of the floor being of rounded cross-section to suit. The present retention period is  $6\frac{1}{2}$  hr. and an air supply of 0.2—0.3 cu. ft. per gal. is anticipated. The sludge re-aëration tank is of similar design, and surplus sludge will be dealt with

in digesters and on drainage beds. Provision is made for chlorination of the final effluent in the hope of selling it for irrigation purposes. C. J.

Heat and energy relations in the digestion of sewage solids. II. Mathematical formulation of the course of digestion. G. M. FAIR and E. W. MOORE (Sewage Works J., 1932, 4, 428—443).—The rate of gas production is shown to be a satisfactory measure of the progress of the digestion process and formulæ are given connecting the various factors involved. C. J.

Gas yield from sewage sludge. W. RUDDLFS (Sewage Works J., 1932, 4, 444-453).—Under practical conditions in heated tanks the gas yield from domestic sludge will be 0.6-0.75 cu. ft. per cap. per day or 8-11.5 cu. ft. per lb. of total org. matter. Laboratory experiments showed that 75% of the gas yield could be accounted for by the destruction of fats (ether extract), and that nearly all destructible cellulosic material was destroyed and some N lost. C. J.

Sewage filtration and sludge incineration. W. RUDOLFS and W. H. BAUMGARTNER (Sewage Works J., 1932, 4, 490-508).—Crude sewage is passed through an upward-flow filter containing 21 ft. of 11-in. slag at a rate equiv. to  $110 \times 10^6$  gals. per acre per day. In this way, the suspended solids are reduced by 70% and the biochemical O2 demand by 50-60%. A higher degree of purification can be obtained by preliminary coagulation if desired. The slag and accumulated sludge is periodically dried and calcined and the bed remade, the loss of slag being about 2%. Odours produced during incineration can be controlled, and the cost of operation, excluding labour, is estimated to be \$13.5 per 10<sup>6</sup> gals. C. J.

Proposed disposal of sewage grit and coarse and fine screenings by incineration. D. T. TOWNSEND (Sewage Works J., 1932, 4, 509–518).—A complete incinerating plant to deal with 40 tons of centrifuged screenings and grit is to be constructed at Milwaukee at an estimated cost of \$121,000. It is considered that 12-15 gals, of fuel oil will be required per ton of wet material containing 65% of H<sub>2</sub>O, 21% of combustible material, and 14% of ash, and that the total cost, including capital repayment and labour charges, will be \$1.75-2.0 per ton. C. J.

Economic preparation and sale of digested sludge as commercial fertiliser. W. W. TATLOCK (Sewage Works J., 1932, 4, 519—524).—Imhoff tank sludge dried on glass-covered beds  $(10-15\% H_2O)$ , at Dayton, Ohio, can be sold with profit at \$1.0 per 100 lb. An annual production of approx. 300 tons is disposed of, mainly to tobacco growers. C. J.

Lime and zeolite [water-]softening combined. E. I. ROBERTS (Eng. News-Rec., 1932, 108, 913-915).—  $H_2O$  is treated with Ca(OH)<sub>2</sub> and, after settling, carbonation, and filtration, part is softened completely with zeolite and mixed with the remainder of the  $H_2O$ . Operating costs are given. D. K. M.

Brilliant-green bile for coli-aërogenes group determinations. H. E. JORDAN (J. Amer. Water Works' Assoc., 1932, 24, 1027—1053).—An optional procedure for the examination of  $H_2O$  in process of purification in the proposed 7th ed. of "Standard Methods for the Examination of Water and Sewage," using parallel planting in brilliant-green-lactosepeptone-bile is described and discussed. As a result a modified procedure is suggested which it is hoped will save time without loss of efficiency. C. J.

Tests for colour and alkalinity [of water]. C. R. Cox (Water Works' Eng., 1932, 85, 368—370).—The optimum  $p_{\rm H}$  for "colour floc" formation is 4.6—6.2. Each grain (per gal.) of alum requires 6—10 p.p.m. alkalinity for complete reaction. In some cases alum can be partly replaced by  ${\rm H}_2{\rm SO}_4$ . CH. ABS.

Hydrogen-ion concentration in disinfection [of water] by chlorination. W. L. MALLMANN (J. Amer. Water Works' Assoc., 1932, 24, 1054—1061).—The efficiency of Cl<sub>2</sub> disinfection, *e.g.*, of swimming pools, decreases as the  $p_{\rm H}$  increases, between the vals. 6.0 and 9.0. C. J.

Graphical representation of the composition of waters. R. FREY (Ann. Chim. Analyt., 1932, [ii], 14, 49—57).—The mineral constituents of  $H_2O$  are divided into 6 groups, viz., carbonates, sulphates, and chlorides of alkalis and of alkaline-earths, the concns. being expressed in mg.-mols. By plotting these concns. according to a definite convention, using rectangular co-ordinates, 2 triangles may be constructed : (a) for carbonates and sulphates of alkalis and alkaline-earths, (b) for the chlorides, and the areas covered give a graphical representation of the character of the  $H_2O$ . Diagrams for a no. of spa waters are given. H. J. D.

Graphic calculations in water analyses. J. K. SELLERS (Ind. Eng. Chem. [Anal.], 1932, 4, 297—298).— A graphical method of representing the concn. of the various ions is proposed, by means of which it is possible to see at a glance the way in which the ions are combined. E. S. H.

Boiler water.—See I. Bhadravati wood tar.— See II. MeCl in air.—See III. SO<sub>2</sub> in air.—See VII. Corrosion in neutral waters.—See X.

See also A., Aug., 826, Determining Cu and Pb in conductivity  $H_2O$ . 828, Still for very pure  $H_2O$ .

#### PATENTS.

Sewage-disposal apparatus. P. S. WICKERHAM (U.S.P. 1,837,002, 15.12.31. Appl., 22.3.30).—An elongated channel is provided with shelves projecting from each long wall; the shelves do not meet in the centre and are provided with curtain walls at their inner edges and with slots under the curtain walls for discharge of settled sludge into the lower part of the channel. Short scraper conveyors run transversely on the upper surface of the shelves, these conveyors being supported on a carriage which travels the whole length of the channel and as it travels operates scrapers in the bottom of the channel. Skimmers are also provided.

B. M. V.

Filters. Evaporating sea-water.—See I. Gaspurification waste liquors.—See II. Sulphite-pulp liquors. Cuprammonium waste liquors.—See V.

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