

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

APRIL 14 and 21, 1933.\*

### I.—GENERAL; PLANT; MACHINERY.

**Method for interpolating [engineering] data, based on Dühring's rule.** J. H. PERRY and E. R. SMITH (*Ind. Eng. Chem.*, 1933, 25, 195—199).—The use of the method is illustrated for various properties.

J. S. A.

**Loose weight, weight after compacting, and sedimentation volume as characteristics of finely-divided materials.** H. W. GONELL (*Chem. Fabr.*, 1933, 6, 77—81).—The variation of the apparent density of materials such as soot, cement, and fine sand with the particle size, grading, and voids content is discussed. Information regarding these factors may be obtained by determining the bulk density of the loosely packed material, e.g., as poured into a container of standard vol., the density after the container has been shaken by a standard procedure, and the vol. of a known wt. when shaken with a given liquid and allowed to settle. Suitable apparatus is described.

H. F. G.

**Incomplete lubrication.** J. E. SOUTHCOMBE and J. H. WELLS (*Compt. rend. Congr. Graissage*, 1931, 475—486; *Chem. Zentr.*, 1932, ii, 2906).—An apparatus is described. The coeffs. of friction at various velocities from 1 cm. to several m. per min. were determined for mineral oils with or without addition of fatty acid. If the oil film cannot support the wt. placed on it a plastic film is produced, and the coeff. of friction then increases > 100 times. This film behaves in the crit. zone like an extremely viscous liquid.

A. A. E.

**Kinematic viscosity and efflux time.** F. H. GARNER and KELLY (*Compt. rend. Congr. Graissage*, 1931, 213—217; *Chem. Zentr.*, 1932, ii, 2908).—Theoretical and mathematical.

A. A. E.

**Viscosity determinations.** WEICK (*Compt. rend. Congr. Graissage*, 1931, 206—212; *Chem. Zentr.*, 1932, ii, 2908).—Deviations of  $\pm 1\%$  were attained with an Engler viscosimeter.

A. A. E.

**Production of low temperatures, and some results of low-temperature research.** A. VAN IJTERBEEK (*Natuurwetensch. Tijds.*, 1933, 15, 21—27).—A lecture on the theory and practice of liquefying gases, describing briefly discoveries such as that of superconductivity.

H. F. G.

**Filtration. II. Fundamental axiom of constant-pressure filtration.** B. F. RUTH [with G. H. MONTILLON and R. E. MONTONNA] (*Ind. Eng. Chem.*, 1933, 25, 153—161; cf. *B.*, 1933, 207).—Measurements of rate of flow with time of filtrate through a semi-works-scale plate-and-frame press under const. pressure show irregularities at the commencement, due to variable

resistance of the cloth. If when the curve becomes straight a tangent is drawn, it (when produced) gives a val.  $C$ , below the base line of the vol. axis with a corresponding time displacement,  $\theta_0$ , and the course of the filtration is given by  $(V + C)^2 = K(\theta + \theta_0)$ , where  $V$  and  $C$  are measures of the resistance of the cake and cloth, respectively. This equation is shown to hold good for results found by various workers with different materials. Imperfect results are probably due to sedimentation before filtration, but too violent agitation giving reduction in particle size must be avoided. Poiseuille's law (that rate of flow varies directly with pressure) holds good throughout any filtration, and the relation between total resistance and filtrate vol. is such that  $R/(V + C)$  is a const. at all times and for all materials. The implications of these results are discussed.

C. I.

**Theory of industrial filtration.** W. SIEGEL (*Chem. Fabr.*, 1933, 6, 89—92).—In a sand-filter the rate of flow obeys the same laws as in the case of flow through a single capillary (Poiseuille's law, and Darcy's rule:  $V = kP$ , where  $P$  = pressure drop). Turbulent flow may produce discrepancies. A variation in the rate of flow of liquids through either a sand-filter or a coherent medium may be produced by the filtration of a cooling liquid which dissolves the air in the filter pores, or of a liquid the temp. of which is rising and from which gases are liberated. Electrical effects have also been noticed. The relation of the filtration const. to the proportion of solid in suspension is a different curve for different materials. The difficulty of arriving experimentally at a correct val. for the resistance of the filtering medium is discussed. Investigation is required on the effect of the shape of particles. A further complication occurs in the filtration of materials capable of compression (colloids and metal hydroxides). Possible methods of studying the resistance of filter-cakes independently of the resistance of the medium are: (1) the use of the membrane filter (in which case the pores are so fine that no material can enter, and the resistance throughout filtration is the same); (2) the measurement of pressure drop through layers of cake by tube manometers.

C. I.

**Budenheim process of water softening with trisodium phosphate.** P. KÖPPEL (*Papier-Fabr.*, 1933, 32, 109—111).—The feed- $H_2O$  is preheated in a cascade mixed with returned boiler- $H_2O$  and the  $Na_3PO_4$  added. The first step removes dissolved gases and 70% of the  $CaH_2(CO_3)_2$ . The  $Na_2O$  in the boiler- $H_2O$  in the second step removes the remaining  $CaH_2(CO_3)_2$ . The product after addition of  $Na_3PO_4$  is believed to be

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

a  $\text{Ca}_4$  phosphate which forms double salts with sulphates and silicates, the reaction proceeding at  $\lt 70^\circ$ . Oil and Fe are also removed from the  $\text{H}_2\text{O}$  and the treatment removes pre-existent scale. The reaction occupies 1 hr. and the requirement of cryst.  $\text{Na}_3\text{PO}_4$  is 15 g. per cu. m. per degree of hardness. With  $\text{H}_2\text{O}$  containing  $\gt 30^\circ$  of hardness a preliminary  $\text{CaO}$ -soda softening is economical. C. I.

**Boiler feed-water treatment with special reference to corrosion.** J. H. PYBUS (Gas World, 1933, 98, Coking Sect., 15-18).—A lecture. R. N. B.

**Plant life and damage by smoke etc.**—See XVI. **Filters for cane sugar.**—See XVII.

See also A., Mar., 229, **Equilibria in interaction [of salt solutions in boiler-feed  $\text{H}_2\text{O}$ ]**. 233, **Absorption velocity of gases by liquids.**

## PATENTS.

**Liquid heating and vaporising [e.g., for production of phenol].** A. P. BEUTEL, Assr. to DOW CHEM. Co. (U.S.P. 1,856,021, 26.4.32. Appl., 15.3.27).—In the blowing down of liquids heated to a high temp. and under high pressure, e.g.,  $\text{PhCl}$ , aq.  $\text{NaOH}$ , and the resultant  $\text{PhOH}$  at  $350^\circ / \gt 3000$  lb./sq. in., the liquid after reduction of pressure under control of a valve operated by a hydraulic ram is sprayed over a coil carrying the high-pressure liquid, the heat from the latter causing further evaporation of the former. B. M. V.

**Heating of fluids in a still or the like.** J. F. DONNELLY, Assr. to DONNELLY-JENKINS Co. (U.S.P. 1,856,061, 26.4.32. Appl., 25.1.26).—Combustion takes place in a no. of elongated passages alternating with passages conveying an inert fluid, e.g., air and/or steam, which reduces the temp. of the brickwork and the gases to such an extent that a tubular oil heater or other apparatus sensitive to high temp. may be heated both by radiation and by contact with the gases. B. M. V.

**Mulling machine.** H. S. SIMPSON (U.S.P. 1,857,337, 10.5.32. Appl., 2.12.29).—Gearing and protection therefor are described for a pan of the mortar-mill type. B. M. V.

**Mixing apparatus.** W. A. PERVIER, Assr. to C. E. PARKHURST (U.S.P. 1,857,863, 10.5.32. Appl., 3.1.31).—A method of driving a mortar in contact with a fixed pestle is described. B. M. V.

**Mixing and drying machine.** A. B. WEBB, Assr. to RIGHTWAY CORP. (U.S.P. 1,857,798, 10.5.32. Appl., 7.2.30).—A rotating horizontal drum is provided with one or preferably a pair of diametrically opposite buckets merging into the cylindrical wall of the drum and extending the full length, their hooked ends terminating at a radius sufficient to clear a heating means and protecting baffle. Immediately at the trailing side of the buckets are permanent openings for charging (when stationary), for exit of moisture and gases (when rotating), and for discharging (by slow reversal). B. M. V.

**Filtration.** W. P. GEE, Assr. to TEXAS Co. (U.S.P. 1,857,810, 10.5.32. Appl., 24.1.29).—In, e.g., the separation of wax from oil, a continuous disc or drum filter is operated in the usual way during the cake-

forming and washing stages, but the discharge of cake is effected pneumatically with the addition of a spray of the same or different solvent liquid to effect cleansing of the pores of the filter from the inside outwards. B. M. V.

**Filter.** C. W. MOORE, Assr. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,858,900, 17.5.32. Appl., 10.12.28).—In a rotary vac. filter, sedimentation in the prefilter tank is prevented by pumping back some filtrate through jets attached to the stirrer. B. M. V.

**Continuous filter.** J. J. ARMSTRONG (U.S.P. 1,856,452, 3.5.32. Appl., 28.4.30).—In a rotary drum filter the filter medium comprises a spirally wound wire only, but the claims refer mainly to a system of bridges and ribs for supporting the wire. B. M. V.

**Screen organisation for filter outlets.** M. P. and J. W. BROWN (U.S.P. 1,855,904, 26.4.32. Appl., 16.9.31).—A quickly detachable conical strainer is described. B. M. V.

**Centrifugal separator.** M. N. BONSIKOS (U.S.P. 1,858,574, 17.5.32. Appl., 2.9.31).—In a basket-type centrifuge having a raking member for removal of collected solid matter, the sliding member is provided with arcuate segments having apertures coinciding with outlet apertures in the bottom of the basket; each segment is provided with a knife forming in conjunction with the others a continuous annular scraper. The outlet apertures are covered by sliding plates actuated pneumatically, and the scraping is effected by sliding the scraping member complete with its sleeve shaft along the inner shaft of the basket. B. M. V.

**Continuous-operation apparatus for purifying liquids.** A. P. ROBINSON, Assr. to FILTROL Co. OF CALIFORNIA (U.S.P. 1,855,610, 26.4.32. Appl., 7.5.28).—Dirty liquid, e.g., dry-cleaners' solvent, is admitted to the lower part of a vertical vessel together with an absorbent powder or filter aid, is there agitated, and the cleaned liquid allowed to rise through honeycomb baffles to a decantation gutter at the top. B. M. V.

**Still.** R. O. ANDERSON (U.S.P. 1,856,860, 3.5.32. Appl., 1.11.27).—The still is especially suitable for the recovery of volatile matter from oil direct from a well before passing to pipe-line or storage where the volatiles might be lost. The vertical tubes of a calandria convey heating medium, e.g., combustion gases. The calandria is surrounded by a shell, *a*, an inner jacket, *b*, and an outer jacket, *c*. *a* and *c* are connected by short horizontal tubes, *d*, preferably gilled, and an auxiliary heat-transmitting fluid is kept circulating by convection through *adca*. The oil is preheated in external exchangers, by the volatile vapours and outgoing oil in turn, and is then trickled over the gilled tubes in *b*, the products leaving through the heat exchangers, and additional coolers if desired. If the combustion chamber is integral with the apparatus it may be surrounded by an extension of jacket *c*. B. M. V.

**Evaporating apparatus.** H. H. ELLISTON (U.S.P. 1,857,535, 10.5.32. Appl., 16.5.30).—In a tower, the liquid is caused to descend in a sloping zig-zag path over riffles on the lower walls of the passages while it is surrounded on both upper and lower sides by ascending zig-zags of heating medium. Previously, the liquid is

heated in a "water" jacket surrounding the apparatus and in the >-shaped spaces between the passages for heating medium. B. M. V.

**Vapour condensation.** N. S. OLSEN, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,857,394, 10.5.32. Appl., 9.4.27. Renewed 15.1.31).—The vapours pass upwards alternately through annular baffles and around conical deflectors supported on a central mast. Returned oil (or other condensate), after cooling or addition of other cooling medium, is supplied to the cones through the hollow mast. B. M. V.

**Precipitating [crystallising] apparatus and process.** J. SKOGMARK (U.S.P. 1,857,884, 10.5.32. Appl., 3.11.28).—Liquor in a fit state for crystallising is cooled in the inner of horizontal concentric tubes, the outer tubes having countercurrents of H<sub>2</sub>O. The tubes are kept clean by running a continuous wire rope through them, and the deposition of crystals takes place in tanks surrounding both rope pulleys. B. M. V.

**Measuring water contents [of solids or liquids].** E. MITTELSTEINER (U.S.P. 1,858,409, 17.5.32. Appl., 28.12.29. Ger., 19.12.27).—A sample of the material is introduced into a closed vessel in which the atm. has been brought to steady conditions of temp. and pressure, preferably such that any H<sub>2</sub>O introduced would take the form of superheated steam. After introduction of the sample, the temp. is maintained by a thermostat and the increase in pressure measured. The manometer may have a movable scale adjustable to the initial pressure as zero and graduated in wt. of H<sub>2</sub>O. B. M. V.

**Gas and liquid contact apparatus.** W. G. LAIRD, Assr. to HEAT TREATING Co. (U.S.P. 1,858,158, 10.5.32. Appl., 6.10.27).—In a bubbler of the perforated plate type, shafts and caps are used to distribute the upflow of vapour evenly. B. M. V.

**Producing a spray mixture of liquid and gas.** W. D. CARTER (U.S.P. 1,856,316, 3.5.32. Appl., 17.4.28).—In a gas pump of the type comprising a vaned rotor placed eccentrically in a casing and sealed by a centrifugally-formed ring of liquid, provision is made for admitting liquid from the seal into the outlet pipe for gas through a spraying device. Priming is effected and the seal kept replenished by means of cups formed in the end face of the rotor acting in conjunction with grooves in the inside of the end of the casing. B. M. V.

**Apparatus for separating solids from gases.** E. ANDERSON, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,856,685, 3.5.32. Appl., 19.5.28).—The separation is effected by a conical strainer to the upper (wide) end of which the dirty gas is admitted tangentially. A portion of fairly clean gas may be withdrawn from the interior of the apex (or from the dust collector) by a fan and passed to the incoming stream through an injector to aid the flow, the increase in the downward flow of the gas inside the cone thereby aiding the transport of solid matter to a collecting chamber below the apex. B. M. V.

**Gas-scrubbing apparatus.** K. S. VALENTINE, Assr. to TURBO-MIXER CORP. (U.S.P. 1,856,120, 3.5.32. Appl.,

24.10.29).—In a vertical tank turbine, runners are rotated in the apertures of a no. of superposed annular diaphragms; the tank is filled with washing liquid to a level above the topmost impeller and the gas is admitted to the bottom of the tank. B. M. V.

**Filter for gaseous substances.** W. BOKENKROGER (U.S.P. 1,857,348, 10.5.32. Appl., 14.5.28).—A no. of perforated plates with raised edges to act as distance pieces are nested with the perforations staggered and are coated with a sticky liquid. B. M. V.

**Tilting-hearth furnace.**—See X. **Dust pptn.**—See XI. **Treating effluent waters.**—See XXIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Spectroscopy as an aid to coal-seam examination and identification.** J. O. SAMUEL (Chem. and Ind., 1933, 155—156).—Coal is ashed by the wet-combustion method and the ash is examined by a modification of the technique of Lewis (A., 1932, 486). The particular stratum to which a seam belongs may be identified by this means. T. McL.

**Gob-fires.** I. Explosions in sealed-off areas in non-gassy [coal] seams. II. Revival of heatings by inleakage of air. T. N. MASON and F. V. TIDESWELL (Safety in Mines Res. Brd., 1933, Papers 75 and 76, 30 and 19 pp.).—I. A non-explosive atm. is probably produced by freely burning goaf fires in a restricted space, the atm. being vitiated by combustion products. Gases distilled from heated material near a fire (e.g., a shielded fire) in an extensive goaf may form an explosive atm. before the O<sub>2</sub> content is reduced below that corresponding to the lower limits of flame propagation. Evolution of firedamp from neighbouring strata increases the risk of explosion. Danger of formation of an explosive atm. exists during development of active fires in roadside coal and dirt packs and is greatest during and immediately after sealing-off. With fires of small size in comparison with the sealed-off area, the formation of an explosive mixture is delayed and may never occur in the absence of firedamp. With an advanced and concealed fire, the time that elapses before explosion is shorter the more advanced is the fire; and the longer is the delay in sealing-off advanced heatings or fires the more violent are the subsequent local inflammations. The liability of explosion of atms. behind stoppings can be judged from analyses of samples drawn from within the sealed-off area.

II. Increase in heating activity of roadside coal and dirt packs was examined on the basis of analysis of the atm. in the goaf and of temp. distribution in the goaf and pack. Inleakage of air, giving 5—7% increase in atm. O<sub>2</sub> content, produced a marked increase in the heating of the pack. The presence of small-size material in the lower part of the pack caused propagation of a zone of combustion, without increased intensity of heating, in 10—15% O<sub>2</sub>. It is concluded that a fire may be maintained in coal or dirt packs, or may increase in activity, if the atm. of the stopping contains < 5% O<sub>2</sub>. Signs of revival of heating were detected when the O<sub>2</sub> content rose to 3%. The extinction of a safety-lamp flame is no criterion of the capacity of an atm. to

extinguish heating (as distinct from flame); the continued presence of 1–2% O<sub>2</sub> might maintain the heating.

H. E. B.

**Calorific value of pure coal substance.** A. PURDON and S. SAPIRS (Fuel, 1933, 12, 40–46).—The observed apparent decrease in the calorific val. of the pure coal substance (*A*) with increasing ash content for coals from the same mine is due principally to the difference between mineral matter and ash. The effect of this difference is appreciable when the ash is > 5%. The true mineral matter may be determined by Mayer's method (B., 1929, 966), which, however, is rather laborious. For coals from the same mine the ratio mineral matter/ash may be determined periodically and used to correct determinations of calorific val. of *A*. If it is not possible to determine the mineral matter the approx. val. 1.13 for this ratio may be used. A. B. M.

**Development of Dakota lignite. VIII. Oil-steam atmosphere for dehydrating Dakota lignite.** A. M. COOLEY, JUN., and I. LAVINE (Ind. Eng. Chem., 1933, 25, 221–224; cf. B., 1932, 869).—Addition of oil to the steam used for drying lignite improved the strength of the product. Both the character and quantity of the oil had some influence. Best results were obtained by incorporating a suitable quantity of emulsified asphalt with the steam. A Velve lignite dehydrated by steam in the presence of emulsified asphalt gave superior results for standard drop and slacking tests, and also burned more slowly than when dehydrated by steam alone. The tendency for spontaneous ignition was apparently little affected by steam-asphalt drying.

G. M.

**Coalification of lignite by heating under pressure with superheated steam.** H. KLEIN (Berg. Hüttenm. Jahrb. mont. Leoben, 1932, 80, 55–60, 102–115; Chem. Zentr., 1932, ii, 2900).—Long treatment (3000–5000 hr.) leads to an increase in C. At 35 atm. products similar to non-coking bituminous coal were obtained. Gasification of coal substance also took place. The changes are attributed to colloid-chemical reactions.

A. A. E.

**Comparison of methods for determination of water in coal and tar.** W. FRANKE (Braunkohlenarch., 1932, No. 36, 39–42; Chem. Zentr., 1932, ii, 2770).—H<sub>2</sub>O is removed from coal with boiling PhMe and treated with CaC<sub>2</sub>. Vals. are practically the same as those obtained by Dolch's cryo-method, and are > those obtained by the xylene method.

A. A. E.

**Determination of sulphur and halogens in combustible materials.** W. GROTE and H. KREKELER (Angew. Chem., 1933, 46, 106–109).—The combustion is effected in a quartz tube into which two quartz filter-plates are sealed. The gases, after leaving the tube, enter at the lower end a vertical absorption vessel packed with glass balls and having a fine sintered-glass filter-plate inserted at about one third of the height from the bottom. Total S is determined by Grewe's method, in which the sample, if solid, is mixed with Al dust; the combustion is complete within 5–10 min. If the sample contains much N, Raschig's benzidine-phenolphthalein method of titration should be used. Results are much more accurate than when the single filter only

is used, as SO<sub>3</sub> mist readily escapes. Halogen is determined as Ag halide in a similar manner, but with Na<sub>2</sub>SO<sub>3</sub> solution in the absorption vessel. An electrostatic pptn. apparatus for SO<sub>3</sub> is described; it may be used for the separate determination of SO<sub>2</sub> and SO<sub>3</sub> in a mixture with air.

H. F. G.

**Commercial [coal] slacks.** W. FRANCIS (Fuel, 1933, 12, 66–71).—Proximate analyses, determinations of fusain (B., 1931, 5), and of resistant residue on rational analysis (B., 1931, 466) have been carried out on sieved fractions of five Nottinghamshire slacks. In general, the ash content increased and the calorific val. (on the ash-free, dry basis) decreased with decreasing particle size; the relatively low ash content of the finest grade (through a 200-mesh I.M.M. sieve) was exceptional. The fractions of this grade were small in amount (< 9%) and consisted almost entirely of vitrain and fusain. The fusain in the unground slacks was distributed throughout the different sizes and amounted to 2.6–10.6% of the coal. Grinding the slacks caused the durain to concentrate in the larger sizes, the clarain in the intermediate fractions, and the vitrain and fusain in the finest sizes; after grinding, e.g., > 70% of the fusain appeared in the fraction passing 200-mesh.

A. B. M.

**Flocculation of [coal]-washery water that is in continuous use.** J. O. SAMUEL (Proc. S. Wales Inst. Eng., 1933, 48, 393–413).—Flocculation of washing H<sub>2</sub>O in the settling tank of a Baum washery has been greatly accelerated by the use of chemical reagents. Alkaline hydroxides have the greatest effect, and Ca(OH)<sub>2</sub> or aq. NH<sub>3</sub> may be used in practice. Excess of the reagent stabilises the suspension. Flocculation is unaffected in waters containing varying degrees (up to 17) of temporary hardness. The clay constituent was more readily flocculated than the fine-coal suspension, although normally the clay is the more difficult to settle out from the untreated H<sub>2</sub>O. In practice, the amount of Ca(OH)<sub>2</sub> added was 0.06%, but the action is better if 0.1% is used. Once added the Ca(OH)<sub>2</sub> was found to flocculate the H<sub>2</sub>O adequately for a fortnight. The treatment is best carried out when the concn. of suspended matter lies between 3.0 and 3.5%, because coal < ½ in. in size is only seriously affected by the suspended matter above this concn. With larger amounts of suspended matter, or with the addition of too much reagent, colloidal clay is adsorbed on the surface of the coal, and the fine coal becomes dirty, remains wet, and is unsaleable. Practical experience has shown that this method of flocculation greatly improves the quality of the coal < 1 in. in size, and reduces the labour otherwise necessary in cleaning the settling tank.

G. E. F.

**Influence of constituents of the seam on physical and chemical properties of coke and semi-coke.** H. BROCHE and H. NEDELMANN (Glückauf, 1932, 68, 769–779; Chem. Zentr., 1932, ii, 3035).—A high dull : bright coal ratio reduces the strength, porosity, and S content, and increases the reactivity and m.p. of ash, of the coke. In high-temp. coking the strength at first rises. Differences in reactivity between 1500° and 2000° were not observed.

A. A. E.

**Blending of coals for coke making.** W. DAVIDSON (Gas World, 1933, 98, Coking Sect., 8—13).—The volatile matter of Scottish coals at the end of the plastic stage is reduced by blending with finely-ground, low-volatile materials, e.g., low- or high-temp. cokes. This is accompanied by a corresponding increase in size and in resistance to shatter. If the size of the added high-temp. coke is  $> \frac{1}{4}$  in. or the % added is large the cokes are very brittle owing to lack of homogeneity of the blend. The same relationship is found to hold for blends of two coking coals and for a blend of a coal with its own coke. Optimum resistance to shatter is obtained with blends containing 20% of either coal. An apparatus is described for the continuous determination of volatile matter of coals when heated progressively to 950°. Higher vals. at the end of the plastic range are obtained by heating at 2°/min. than at 5°/min.; hence, greater resistance to shatter would probably be obtained by carbonising at a more rapid rate of heating. R. N. B.

**Expansion and contraction of coal caused respectively by the sorption and discharge of gas.** H. BRIGGS and R. P. SINHA (Proc. Roy. Soc. Edin., 1933, 53, 48—53).—The linear expansion of specimens of coal subjected at room temp. to the action of (a) CH<sub>4</sub>, (b) CO<sub>2</sub>, at 300 lb. per sq. in. was determined. On releasing the pressure, the specimens contracted. Coals were found to react less strongly to CH<sub>4</sub> than to CO<sub>2</sub>. W. H. B.

**Carbonisation of screened, mixed, and blended coals.** H. HOLLINGS (Gas J., 1933, 201, 370—376).—The throughput of a retort and the quality of the coke produced may be increased by blending a non-caking coal or coke breeze with certain caking coals, especially if the blend be further briquetted. The coke breeze must be crushed below  $\frac{1}{4}$  in., as otherwise the quality of the large coke is affected. Coal may be screened so that the fines are used in horizontal and the screened coal in continuous vertical retorts, which have then an increased throughput. In the latter it is probable that size of coal has a greater influence than blending, as is shown by the following daily throughputs: unscreened Yorkshire coal 4.8, screened Yorkshire coal ( $> 1$  in.) 8.0, briquetted blend of Durham coal and a non-caking coal 6.5 tons. Change of source of coal supply results in irregular working of the plant; this can be overcome by mixing (as distinct from blending, i.e., crushing below  $\frac{3}{8}$  in. before mixing) the coals before carbonisation. Screening and blending plant is described. By prior carbonisation of non-caking coal at low temp. the ratio of caking to non-caking coal in a blend may be increased. R. N. B.

**Nitrogen in peat and in its carbonisation products.** G. KEPPELER and H. HOFFMANN (Brennstoff-Chem., 1933, 14, 41—47).—On carbonising different types of peat at 450—800°, 50—70% of the N remained in the coke, 10—18% was evolved as NH<sub>3</sub>, and only 3—13% appeared in the tar. 70—80% of the N in the tar was present in the pitch and neutral oil. The bases in the tar were separated and examined; they consisted principally of *tert.* bases and contained derivatives of C<sub>5</sub>H<sub>5</sub>N and quinoline. A compound which was isolated as the picrate (m.p. 160—161°) was shown to be, probably, dihydroquinoline. A. B. M.

**Absorptive power of charcoal preparations.** M. LEINZINGER (Magyar Gyóg. Társas. Ert., 1932, 8, 283—290; Chem. Zentr., 1932, ii, 2681).—Granulating and tableting animal C reduces greatly its adsorptive power towards methylene-blue, but little that towards PhOH. This is due to the use of fats in tableting. Combination of animal C with pharmacologically active substances is therefore liable to lead to mutual inactivation. A. A. E.

**Action of water vapour on methane.** C. MATIGNON and M. SÉON (Compt. rend., 1932, 195, 1345—1348).—The reaction CH<sub>4</sub> + 2H<sub>2</sub>O = 4H<sub>2</sub> + CO<sub>2</sub> has been examined with a view to H<sub>2</sub> manufacture. When a mixture of industrial CH<sub>4</sub> (CH<sub>4</sub> 63.7, N<sub>2</sub> 14.4, H<sub>2</sub> 10.0, CO 7.3, O<sub>2</sub> 3.8%) with large excess of H<sub>2</sub>O (up to 80 times theoretical) is passed over the following catalysts at 1000°, remaining in contact therewith for 7 sec., the % H<sub>2</sub> in the emerging gases noted after each was obtained: ZrO<sub>2</sub> 73.9—74.4, zirconised asbestos 33.6—34.4, asbestos sprinkled with ZrO<sub>2</sub> 58.5—60.6, ThO<sub>2</sub> 63.1—63.9, Fe<sub>2</sub>O<sub>3</sub> 42.4—43.9, Fe<sub>2</sub>O<sub>3</sub> + 10% K<sub>2</sub>CO<sub>3</sub> 32.9—33.6, Al<sub>2</sub>O<sub>3</sub> 40.2—41.4; zircon (mineral) gave a poor yield. At 1050° using ThO<sub>2</sub> practically complete conversion into H<sub>2</sub> and CO<sub>2</sub> occurred (80.9—82% H<sub>2</sub>); at 960° 67.8—69.4% H<sub>2</sub> was obtained, but at 860° the yield varied greatly with the amount of H<sub>2</sub>O, 1000 times theoretical being necessary for complete conversion (cf. A., 1925, ii, 532; 1930, 43; 1933, 254). C. A. S.

**Determination and calculation of the flame temperatures of complex mixtures of combustible gases.** B. LEWIS, H. SEAMAN, and G. W. JONES (J. Franklin Inst., 1933, 215, 149—167).—The flame temp. of mixtures of air with H<sub>2</sub>, CH<sub>4</sub>, and CO containing varying quantities of N<sub>2</sub> and CO<sub>2</sub>, and with coal gas, producer gas, etc., have been determined as a function of the composition. The max. flame temp. of complex mixtures may be calc. on the basis of the mixture law by regarding all the inert gases (i.e., N<sub>2</sub> and CO<sub>2</sub>) as being either associated with only one of the combustible constituents, or distributed among the combustible gases in the same proportions as the latter are present in the mixture. Comparison of the observed and calc. results indicates that the max. flame temp. of industrial gas mixtures may be calc. to within 50°, or, if  $< 10\%$  of N<sub>2</sub> and CO<sub>2</sub> are present, to within 20°. H. F. G.

**Thomas recording gas calorimeter.** R. S. JESSUP (Bur. Stand. J. Res., 1933, 10, 99—121).—The instrument is described. The total heating val. of gases may be measured to within 1%. H. J. E.

**[Filming] oil for gas holders.** E. YOUNG (Gas World, 1933, 98, 10—11).—Filming oil should have viscosity 150—180 sec. (Redwood) at 21°, flash point 177°, and a low surface tension; addition of 1—2% of cresylic acid to the oil acts as a sterilising agent. Filming of the outside of the holder, to prevent corrosion, should be done separately with a thin layer of oil of low surface tension. R. N. B.

**Determination of creosote content of lignite tars.** III. Action of sodium on phenols in presence of water and the gasometric determination of the latter. IV. Curve for conductivity: mol. wt. of creosote, and determination of the latter.

W. FRANKE (Braunkohlenarch., 1932, No. 36, 1—19, 20—38; Chem. Zentr., 1932, ii, 2770).—III.  $H_2O$  in creosote is determined gasometrically with  $CaC_2$ .

IV. Preliminary. A. A. E.

**Relations between viscosity and constitution of hydrocarbons of high mol. wt.** G. HUGEL (Compt. rend. Congr. Graissage, 1931, 146—161; Chem. Zentr., 1932, ii, 2906).—Destructive hydrogenation of coal tar gave a mixture of hydrocarbons of which few possessed side chains; only in the heavy fractions were, e.g., dimethylantracene and tetramethylpicene present. Side chains were introduced into aromatic nuclei and the products were then catalytically hydrogenated and their viscosities determined. Ring-closure always increases viscosity. For the same mol. wt. the viscosity increases with the no. of rings in the mol. The higher is the mol. wt. the flatter is the viscosity curve. By introduction of long side chains the viscosity curve can be appreciably improved. A. A. E.

**Activated carbon for removal of phenols from crude ammoniacal liquor.** F. SIERP (Gas- u. Wasserfach, 1933, 76, 105—109).—The removal of phenols from gas liquor by activated C is efficient, but steam treatment for regeneration is too expensive in view of the high b.p. and presence of tar. The C is therefore washed with  $C_6H_6$ , the phenols are recovered, and residual  $C_6H_6$  is removed with steam. In a plant working in the Ruhr the liquor, freed from tar in a separator packed with Fe turnings, enters one of 2 or 3 adsorbers of Fe with internal protective lining, being heated to 60—65° by the hot  $C_6H_6$ . The working period for an adsorber is 4 hr., the C then containing 7.5% of phenols, and the washing efficiency is 99.5%. The gradual accumulation of tar and resins renders it eventually necessary to regenerate the C. The  $C_6H_6$  used for washing is also brought to 60—65° and is fed downwards, acquiring a mean phenol content of 1.5%, so that 1 pt. of  $C_6H_6$  is required for 8 pts. of liquor. It passes through a  $H_2O$  separator to the still. Costs are discussed, but the process is designed rather to avoid effluent difficulties than as a source of profit. C. I.

**Improved methods of examining mineral oils.** S. KYROPOULOS (J. Inst. Petroleum Tech., 1932, 18, 1010—1012). J. C. VLUGTER, H. I. WATERMAN, and H. A. VAN WESTEN (*Ibid.*, 1013—1014).—A reply to criticisms by Vlугter and co-workers (B., 1932, 536), and a rejoinder. H. S. G.

**New methods of dewaxing and acid-refining mineral oils.** N. O. BACKLUND (J. Inst. Petroleum Tech., 1933, 19, 1—15).—The use of  $C_2HCl_3$  as the solvent in the dewaxing of oils results in a smaller quantity of solvent being necessary than in the older benzene process, a considerably shortened period of cooling, and the possibility of working at a higher temp. Due to using a heavy solvent, the wax is obtained by centrifugal separation as the lighter component, and can be continuously discharged from the central part of a suitable type of centrifugal bowl. A comparison of acid-refining by centrifugal means with the older types of air-blowing and mechanical mixing processes shows the advantages of the max. utilisation of  $H_2SO_4$ , exclusion of air during the whole process, shortest possible

time of contact between the oil and sludge, and a final acid-free oil. H. S. G.

**Rapid analysis of alkaline sludge.** A. B. MALY-ATSKI (Azerbaij. Neft. Choz., 1932, No. 10, 87—92).—2N-NaOH (20—25 c.c.) is added to the sludge (200 g.) and diluted to 500 c.c. (1) 100 c.c. are extracted with  $Et_2O$  and the dried ( $Na_2SO_4$ ) extract is evaporated, the residue (oil) being weighed. (2) 100 c.c. are acidified with 10%  $H_2SO_4$  or HCl (Me-orange) and extracted with  $Et_2O$  (residue on evaporation, crude naphthenic acids). (3) Procedure (2) is repeated on the aq. solution from (1), giving purified naphthenic acids. (4) The acid val. of the naphthenic acids is determined on separate portions of  $Et_2O$  extracts from (2) and (3). CH. ABS.

**Artificial ageing of mineral oils.** F. EVERS and R. SCHMIDT (Petroleum, 1933, 29, No. 4; Mitt. "Internat. Petroleum-Komm.," 1—7).—The theory of the oxidation method is discussed and the manipulation of the apparatus is described (cf. B., 1930, 801; 1931, 749). A quant. study of the variation of fluorescence with age of various oils has shown that by means of the described method the artificial ageing of an oil can be carried further than is possible by other methods.

A. B. M.

**Effect of high temperatures on mineral oils.** A. MAILLARD (Compt. rend. Congr. Graissage, 1931, 308—317; Chem. Zentr., 1932, ii, 2905).—Heating with a Cu arc gave untrustworthy results. Experiments using a heated C filament indicated the effect of dissolved  $O_2$  but gave no definite indication of the origin or purity of the oil. A. A. E.

**Catalytic action of substances present in natural waters on oxidation of petroleum oils.** K. I. IVANOV and N. N. PETIN (J. Gen. Chem. Russ., 1932, 2, 748—754).—The acidity of petroleum distillates, pretreated with aq.  $SO_2$ , is greatly increased after washing with Moscow tap- $H_2O$  (I). This effect is not observed with distilled  $H_2O$ , and occurs only to a small extent with "artificial" tap-water (II) prepared from distilled  $H_2O$  and pure salts. The catalytic properties of (I) are unaffected by ultrafiltration or treatment with  $Cl_2$  or  $O_3$ , but are largely removed by successive treatment with  $H_2SO_4$  and  $Ca(OH)_2$ ; subsequent treatment with  $O_3$  does not restore activity. Distilled  $H_2O$  is not activated by  $O_3$ , which greatly enhances the activity of (II), whilst  $Cl_2$  has the opposite effect. Dniepr river- $H_2O$  and sea- $H_2O$  have an activity equal to that of (I). R. T.

**Isolation of the three xylenes from an Oklahoma petroleum.** J. D. WHITE and F. W. ROSE, JUN. (Bur. Stand. J. Res., 1932, 9, 711—720).—The *o*-, *m*-, and *p*-xylenes occur in the ratio 3 : 3 : 1, to a total extent of 0.3%. The aromatic constituents may readily be extracted by liquid  $SO_2$ . C. W. G.

**Bright stocks from paraffin-base crude oil of Baku.** V. E. PARKHOMENKO (Azerbaij. Neft. Choz., 1932, No. 10, 78—81).—Two processes, producing respectively 4.27 and 11.9% of bright stock, appear feasible. CH. ABS.

**Antidetonating properties of gasolines from American and Baku oil fields.** V. I. ELANSKI

(Azerbaij. Neft. Choz., 1932, No. 10, 81—87).—Straight-run gasolines from Baku have  $C_8H_{18}$  equiv. 37—47 and cracked gasolines (liquid phase) 27—37; (vapour phase) 62—72. CH. ABS.

**Effect of lead tetraethyl on octane number [of gasoline].** L. E. HEBL, T. B. RENDEL, and F. L. GARTON (Ind. Eng. Chem., 1933, 25, 187—191; cf. B., 1932, 790).—The relation between the concn. of  $PbEt_4$  and the  $C_8H_{18}$  no. of a gasoline is examined empirically, and a definition is thereby developed for the "Pb susceptibility" of any gasoline. The  $C_8H_{18}$  no. of an "ethylised" gasoline is determined by 5 distinct factors (described) which, when incorporated in their proper relation in an "Et blending chart," enable the Pb susceptibility of a gasoline to be determined from the  $C_8H_{18}$  nos. of 2 blends containing different concns. of  $PbEt_4$ . Wide differences in the testing conditions do not invalidate these relationships. Pb susceptibilities of gasolines from various sources are given. E. S.

**[Character of] detonation [in knock in internal-combustion engines].** M. SERRUYS (Compt. rend., 1932, 194, 1894—1896; 195, 1228—1230, 1376—1379).—The detonation in knock lasts for  $(1-0.5) \times 10^{-4}$  sec. and occurs towards the end of combustion; pressures of the order of 100 kg. per sq. cm. are produced. When knock begins the temp. at certain points rises suddenly (by  $> 100^\circ$ ). C. A. S.

**Total heats of gas oils at elevated temperatures and pressures.** W. W. GARY, L. C. RUBIN, and J. T. WARD (Ind. Eng. Chem., 1933, 25, 178—183).—The total heats of 4 cracking-plant gas oils in both liquid and vapour states were determined at temp. from  $370^\circ$  to  $482^\circ$  in a flow calorimeter consisting of: (a) a measured-input electric heater, and (b) a  $H_2O$ -flow calorimeter for absorbing the heat imparted to the oil by (a). The results from (a) and (b) are confirmatory. The data obtained are compared with equations based on sp. gr. for predicting the variation of total heat with temp. The total heats of vapours from all stocks at low pressures are represented by Weir and Eaton's equation (B., 1932, 327). For virgin stocks the Bureau of Standards equation (Misc. Publ. 97, 1929) is most reliable:  $C = (1/d)(0.38 + 0.00045t)$ , where  $C =$  sp. heat, B.Th.U./lb./ $^\circ F$ . The authors have developed from their data an equation for liquid gas oil and refractory cracking stocks from  $40^\circ$  to  $15^\circ$  A.P.I. ( $d = 0.825-0.966$ ), viz.,  $H = (15d - 27) + (0.7615 - 0.400d)t + (0.000625 - 0.000440d)t^2$ .

T. H. B.

**Influence of refining on the stability of transformer oil.** FLAMANC (Compt. rend. Congr. Graissage, 1931, 92—116; Chem. Zentr., 1932, ii, 2771).—Results obtained by Weisz and Salomon's method are recorded. A. A. E.

**Ageing of steam-turbine oils.** T. SALOMON (Compt. rend. Congr. Graissage, 1931, 373—392; Chem. Zentr., 1932, ii, 2769).—Weisz and Salomon's method for testing transformer oils is applied to steam-turbine oils. The relation between sediment formation and emulsification is examined. A. A. E.

**Resistance of mineral oils to oxidation.** M. VAN RYSELBERGE (Compt. rend. Congr. Graissage, 1931,

270—285; Chem. Zentr., 1932, ii, 2771).—The artificial ageing of a mineral oil should be conducted in presence of only those metals with which the oil will come in contact in practice. The period of heating should be long. The velocity of absorption of  $O_2$  by the oil during ageing should be measured, and the emulsifying properties of the aged oil should be determined. A. A. E.

**Importance of refraction values of benzines.** P. LOB (Angew. Chem., 1933, 46, 91—92).—Determinations of refractive index ( $n_D$ ) give valuable indications as to the nature of the constituents of hydrocarbon mixtures, since different mixtures may have practically the same  $d$  and even similar b.p. ranges although there is an appreciable difference between their  $n_D$  vals. M. S. B.

**Total heats and specific heats of petroleum waxes.** C. M. GATELEY (Fuel, 1933, 12, 26—29).—The total heats, from the m.p. to  $300^\circ$ , of 4 Persian and 3 Burmese waxes, a white ceresine, and an ozokerite have been determined. The sp. heats can be represented by the equation  $s = a + bt$ , where  $a$  varies from 0.4883 to 0.4948 and  $b = 0.0009$ . The sp. heat of any of the waxes can be represented with sufficient accuracy for engineering requirements by  $s = 0.492 + 0.0009t$ , and the total heat by  $H = 0.492t + 0.00045t^2 - 20.40$ . A. B. M.

**Production, selection, and application of lubricating oils.** F. J. SLEE (Proc. Tech. Sect. Paper Makers' Assoc., 1932, 13, 224—237).—A brief review.

**Physical properties and structure of lubricating films.** V. VIEWEG (Kolloid-Z., 1932, 61, 198—208).—A review of published work, particularly on the relation between lubricant and bearing metal. E. S. H.

**Relation between temperature and viscosity in lubricating oils.** DE CAVEL and ROEGIERS (Compt. rend. Congr. Graissage, 1931, 238—257; Chem. Zentr., 1932, ii, 2769—2770).

**Wt. and vol. of finely-divided materials. Lubrication.**—See I. Creosote as wetting agent.—See VI. Solid  $CO_2$  from flue gas. Absorption of  $H_2S$ . Oxidising  $H_2S$  by air.—See VII. Cements for petroleum bore-holes. Properties of bitumen-mineral matter mixtures.—See IX. Paraffins from carnauba wax.—See XII. Plant life and smoke prevention.—See XVI. [Gas from] fermented fibres.—See XVIII.

See also A., Mar., 216, Physical structure of coal. 231, Gaseous combustion at high pressures. 234, Production of  $H_2$  by the water-gas reaction. 253, Aachen chalk coals.

## PATENTS.

**Retort [for low-temperature distillation of carbonaceous materials].** J. N. VANDEGRIFT, Asst. to INTERNAT. BITUMEN OIL CORP. (U.S.P. 1,857,171, 10.5.32. Appl., 24.3.28).—An inclined rotary cylinder is entirely surrounded by a stationary heating chamber except at the necessary supporting rollers. Combustion takes place in the base of the setting under tiles of, e.g., SiC, which become radiant and heat the lower part of the cylinder; the gases are allowed to come in contact only with the upper part of the cylinder. The heating is

divided into definite zones by transverse walls preferably provided with flues for cooling air. B. M. V.

**Retorts for distillation of coal and other carbonaceous substances.** LOW TEMPERATURE CARBONISATION, LTD., W. A. BRISTOW, and J. P. POSTLETHWAITE (B.P. 386,766, 7.12.31).—The cover-plates of retorts of the type described in B.P. 277,079 (B., 1927, 837) are provided with  $H_2O$ -seals which are maintained by a supply of  $H_2O$  from the  $H_2O$ -jacket of the off-take pipe. A. B. M.

**Production of fuel from coking coal or the like.** INTERNAT. BITUMENOIL CORP., Assees. of J. N. VANDEGRIFT and C. POSTEL (B.P. 386,343, 27.5.31. U.S., 29.5.30).—The dried and preheated coal is distilled by heating it suddenly to  $650$ – $870^\circ$  while it is confined under pressure in a battery of retort tubes (*A*). These are preferably vertical, narrow, e.g., about 5 in. in diam., and slightly coned; they are externally heated. Preheating tubes (*B*) are so arranged that when preheating (which is effected by the flue gases from the combustion chambers surrounding *A*) is completed *B* can be moved over and discharged into *A*; the former are then removed again, a hood is lowered on top of the retort casing, and a no. of pistons are forced down to rest on top of the coal in *A*. Carbonisation is continued for 80–100 min., and the coke is then discharged into a cooling receptacle. The product breaks into dense, wedge-shaped lumps having a higher volatile matter content near the sharp edge than near the outer surface of the wedge (average 6–10%). It forms a suitable domestic fuel. A. B. M.

**Apparatus for producing fuel from coking coal or the like.** INTERNAT. BITUMENOIL CORP., Assees. of J. N. VANDEGRIFT, C. V. LINN, and C. POSTEL (B.P. 386,314, 11.6.31. U.S., 5.3.31. Addn. to B.P. 386,343; cf. preceding abstract).—Improvements in the design of the carbonisation plant of the prior patent are described; e.g., a molten-metal seal is provided for the removable hood which covers the battery of retorts, the rods of the pistons which compress the coal in the retorts are attached to a plate within the hood so that only the means for actuating the plate need pass through the hood, etc. A. B. M.

**[Carbonisation] retort construction.** INTERNAT. BITUMENOIL CORP., Assees. of J. N. VANDEGRIFT (B.P. 386,335, 6.6.31. U.S., 5.3.31).—Retorts are lined with tubes made of rolled sheet metal, e.g., Cr steel. The metal linings are surrounded by protective casings of refractory material, preferably firebrick. Batteries of vertical retorts of this description are suitable for the process of B.P. 386,343 (preceding). A. B. M.

**Water-flow calorimeters.** A. L. NORTON (B.P. 386,934, 23.11.31. Cf. B.P. 385,084; B., 1933, 213).—The  $H_2O$  from the calorimeter (*A*) flows into an inlet tube which communicates with a water-level indicator. The  $H_2O$  leaves the latter through an opening of restricted area so that the  $H_2O$ -level indicates the rate of flow of  $H_2O$  from *A*. A. B. M.

**Manufacture of producer and like gas.** F. COOKE and A. L. HOLTON (B.P. 386,832, 2.4.32).—The hot gas from the producer (*A*) is passed through a series of towers (*B*) wherein it is sprayed with tar. Crude tar is supplied

to the last tower, is collected at the bottom thereof, and supplied to the next preceding tower, and so on. The tar purifies the gas from dust and is distilled in the process. Some of the gas which has passed through *B* may be returned to *A* so that the tar vapours are cracked therein (or in the carburettor and superheater) and the gas is thereby enriched. A. B. M.

**Treatment of hydrocarbon oil produced in gas manufacture.** A. R. ALBRIGHT, Assr. to KOPPERS CO. (U.S.P. 1,859,015, 17.5.32. Appl., 10.10.28).—Crude light oil obtained by stripping wash oil is treated with  $H_2SO_4$  and then with aq. NaOH containing tar acids, e.g., PhOH, which have been extracted from the transfer liquid which has removed the tar acids from still waste. D. K. M.

**Treatment of sludge acid [from purification of coke-oven light oil etc.].** E. B. CONKLIN, Assr. to SEMET-SOLVAY CO. (U.S.P. 1,855,878, 26.4.32. Appl., 20.8.28).—Acid sludge is allowed to separate as completely as possible and, after decantation of the free acid, the remaining sludge is heated and fed through a distillation zone where it is continuously mechanically agitated in the presence of a current of steam, thereby vaporising light hydrocarbons, which are condensed. The residue from the distillation zone is continuously discharged. H. S. G.

**Cracking of [hydrocarbon] oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,855,879, 26.4.32. Appl., 13.11.25. Renewed 1.9.28).—A no. of separate oils of varying character are passed under pressure through independent cracking coils to separate conversion zones from which the vapours are led to a common vapour zone maintained at a lower pressure, a dephlegmator, and a condenser. The reflux condensate is separated from the vapours and further cracked without being returned to any of the original coils. H. S. G.

**Treatment or manufacture of hydrocarbons.** H. DREYFUS (B.P. 386,669, 9.7.31).—Hydrocarbons are cracked by passing them in the liquid or vapour phase through a bath of molten inorg. compounds, e.g., alkali or alkaline-earth halides, containing suitable catalysts, e.g., Si, Mg, or Al silicates,  $MoO_3$ , CaO, Ni, etc. The bath is maintained preferably at  $600$ – $800^\circ$ , and cracking may be effected under increased or diminished pressure, according to the character of the required product. The process may be used to produce motor spirit from petroleum, or gaseous hydrocarbons from light oils etc. A. B. M.

**Apparatus for distilling oils.** H. H. MORETON, Assr. to O. B. ENGLISCH and C. F. CRAIG (U.S.P. 1,855,685, 26.4.32. Appl., 8.10.28).—An oil-purifying apparatus consists of a no. of vapour chambers each provided with a vent pipe discharging into a main flue. Consecutive chambers are connected alternately at the top and bottom by pipes having a loop extending around the flues. H. S. G.

**Refining of oils and the like by steam treatment.** G. ZOTOS (B.P. 386,993, 23.6.31).—Steam at  $500^\circ/200$  atm. is passed into oil and the whole transferred to a dephlegmator. The vapour leaving the top of this vessel is condensed, the oil in the reflux is returned to



the reaction chamber, and the  $H_2O$  still under pressure is either treated with  $CaO$  and returned to the boiler or may be used to preheat the boiler feed. D. K. M.

(A, B) **Treatment [purification] of petroleum hydrocarbons (B) in the vapour phase.** R. CROSS, Assr. to CROSS DEVELOPMENT CORP. (U.S.P. 1,859,027—8, [A, B] 17.5.32. Appl., [A] 4.11.27, [B] 8.11.27).—(A) Liquid Na is injected by means of high-pressure gas from the pressure chamber or from an external source into the vapours leaving the dephlegmator in a cracking process. After separation of the sludge, from which Na may be recovered, the vapours are condensed. (B) The vapours are bubbled through layers of molten alkali metal, e.g., Ca, Na, on trays in a tower. Polymerised products are withdrawn from the heating tower and returned to the cracking plant, and the vapours free from S are dephlegmated in the upper part of the tower and condensed. D. K. M.

**Treatment [purification] of hydrocarbon oils.** S. H. DIGGS, J. M. MCGEE, and T. S. COOKE, Assrs. to STANDARD OIL CO. (U.S.P. 1,858,394, 17.5.32. Appl., 5.3.28).—The distillate obtained by the pressure distillation of crude oils is mixed with 98%  $H_2SO_4$  (by passing through an orifice plate) and passed through a vessel at  $< 38^\circ$ . Sludge is separated and the oil steam-distilled. D. K. M.

**Treatment of hydrocarbon oils.** J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,855,486, 26.4.32. Appl., 29.1.31).—Cracked hydrocarbon distillates are subjected in heated vaporous form to treatment with  $SO_2$  and  $O_2$ -containing gas in the presence of  $Fe_2(SO_4)_3$ . H. S. G.

**Treatment [oxidation] of hydrocarbon materials.** P. K. FROLICH, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,858,822, 17.5.32. Appl., 14.10.27).—Hydrocarbons, e.g.,  $CH_4$ ,  $C_4H_{10}$ , preheated by flowing through coils inside the reaction chamber are oxidised by air at  $350-600^\circ / > 1000$  (preferably 1500—3000) lb. per sq. in. The product is dephlegmated under conditions that keep the unchanged hydrocarbon liquid; the fixed gas leaving the top of the dephlegmator (A) is scrubbed by a suitable solvent, e.g.,  $H_2O$ , ethylene glycol. The liquid leaving the bottom of A is washed counter-currently with the solvent, giving a solution of the oxidation products (acids, ketones, aldehydes, alcohols) and the unchanged hydrocarbon vapour; the last-named, after scrubbing with aq. NaOH, is recycled. D. K. M.

**Sweetening of hydrocarbon oils.** A. R. MARTIN, P. J. KELLY, and K. H. REPATH, Assrs. to PAN AMER. PETROLEUM & TRANSPORT CO. (U.S.P. 1,858,635, 17.5.32. Appl., 25.4.27).—Oil and "doctor" solution are passed through a baffled mixing chamber and then a settler in which doctor solution separates and is re-used. S is added to the oil containing PbS and after passing through a baffled chamber the PbS is separated in a settler. The PbS is treated with 98%  $H_2SO_4$  and air and the  $PbSO_4$  is washed and reconverted into doctor solution by the addition of aq. NaOH. D. K. M.

**Method and reagent for treating wet [petroleum] oils.** J. J. CANFIELD, Assr. to EMPIRE OIL & REFINING CO. (U.S.P. 1,858,186, 10.5.32. Appl., 27.1.28).—The oil- $H_2O$  mixture is treated with a hydrocarbon-

substituted derivative of a polycyclic sulpho-aromatic compound (e.g., crude isopropyl-naphthalenesulphonic acid; prep. described), a low-f.p. liquid halogen derivative of an aromatic hydrocarbon (e.g.,  $PhCl$ ),  $H_2O$ , and cresylic acid or a phenol. H. S. G.

**Removal of wax from oil.** (A) S. B. FUNSTEN and I. M. PERKINS, (B) J. E. SCHOTT, Assrs. to ATLANTIC REFINING CO. (U.S.P. 1,857,370 and 1,857,404, 10.5.32. Appl., [A] 24.6.29, [B] 5.7.29).—In (A)  $< 2$  wt.-% of a high-boiling wax-agglomerating compound contained in coal tar, and in (B) a petroleum distillation residue of low A.P.I. gravity, is added to the oil prior to treatment by one of the usual wax-removing processes. H. S. G.

**Lubricating oil.** J. ROBINSON, Assr. to STANDARD OIL CO. (U.S.P. 1,855,203, 26.4.32. Appl., 30.6.27).—Turbine oils are produced by treating a lubricating oil with a crit. amount of conc.  $H_2SO_4$ , as predetermined on an actual sample of the oil. H. S. G.

**Oil-cracking apparatus.** H. THOMAS, Assr. to SUN OIL CO. (U.S.P. 1,863,173, 14.6.32. Appl., 8.6.27).

**Heating and cracking of oil.** J. C. BLACK and E. W. GARD (U.S.P. 1,859,691, 24.5.32. Appl., 16.11.26).

**Cracking of hydrocarbons.** E. C. HERTHEL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,859,966—7, 24.5.32. Appl., [A] 16.4.27, [B] 16.12.29).

**Cracking of hydrocarbon oils.** E. C. HERTHEL and T. DE C. TIFFT, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,859,510, 24.5.32. Appl., 7.6.28).

**Oil conversion.** W. G. LEAMON, Assr. to LEAMON PROCESS CO. (U.S.P. 1,861,399, 31.5.32. Appl., 2.11.23).

**Distillation of oil.** E. G. RAGATZ, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,862,063, 7.6.32. Appl., 31.12.28).

**Petroleum distillation system.** H. W. HOW, Assr. to STRUTHERS WELLS-TITUSVILLE CORP. (U.S.P. 1,862,363, 7.6.32. Appl., 11.7.29).

**Separation of crude petroleum and natural gas.** W. MCGRAW and R. C. ADAMS, Assrs. to TRUMBLE GAS TRAP CO. (U.S.P. 1,860,896, 31.5.32. Appl., 11.4.28).

**Treatment of hydrocarbons.** T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,862,507, 7.6.32. Appl., 26.6.26).

**Refining of hydrocarbons.** W. V. ISCHIE, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,859,968, 24.5.32. Appl., 1.5.29).

**Preparation of lubricants.** E. H. LESLIE, Assr. to E. B. BADGER & SONS CO. (U.S.P. 1,860,838, 31.5.32. Appl., 7.3.28).

**Heating of fluids. Mixing and drying machine. Filtration [of wax from oil]. Still. Vapour condensation.**—See I.  $NH_3$  from  $NH_3$  liquor.—See VII. Composition for roads.—See IX. Lacquers.—See XIII. Fertilisers from gas liquors.—See XVI. Treating effluent waters.—See XXIII.

### III.—ORGANIC INTERMEDIATES.

**Action of steam on  $CH_4$ . Phenols from ammoniacal liquor. Isolating xylenes from petroleum.**—See II. [Products from] hemicelluloses. Cymene

in sulphite liquor.—See V. Paraffins from carnauba wax.—See XII. COME<sub>2</sub>-BuOH by fermentation.—See XVIII.

See also A., Mar., 254, Colorimetric determination of CHCl<sub>3</sub>. 258, Synthesis of tartaric acid, and its determination. 266, Prep. and storage of nitrosomethylcarbamide. 267, Prep. of PhOH. 269, Reduction of aromatic NO<sub>2</sub>-compounds to amines. *m*-Nitroaniline. 274, Oxidation of PhMe to PhCHO and BzOH.

## PATENTS.

Manufacture of organic compounds [hydrohalides of vinylacetylene]. E. I. DU PONT DE NEMOURS & Co. (B.P. 387,325, 24.7.31. U.S., 22.10.30).—Vinylacetylene is combined with aq. H halide preferably in presence of Cu<sub>2</sub>Cl<sub>2</sub>, AuCl<sub>3</sub>, CaCl<sub>2</sub>, MgCl<sub>2</sub>, Hg<sub>2</sub>Cl<sub>2</sub>, or HgCl<sub>2</sub>, or the corresponding bromides. Products described are: β-chloro-αγ-butadiene, b.p. 60°, αγ-dichloro-β-butylene, b.p. 128°; chloromethylallene, b.p. 86–88°; β-bromo-αγ-butadiene, b.p. 42–43°/165 mm.; αγ-dibromo-β-butylene, b.p. 56–59°/6 mm.

C. H.

Manufacture of alcoholic oxidation products of olefines. H. DREYFUS (B.P. 387,372, 4.8.31).—Olefines are treated with O<sub>2</sub> in presence of H<sub>2</sub>O, preferably an aq. solution of Mn or Fe salts, I, or per-salts. C<sub>2</sub>H<sub>4</sub> gives glycol.

C. H.

Production of methyl alcohol and other carbon compounds and catalytic agents therefor. A. T. LARSON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,844,129, 9.2.32. Appl., 14.4.27).—A catalyst for the reduction of CO to MeOH is made by fusing and reducing a mixture of CuO and an oxide of Mn, W, Zn, Cd, or Mo.

C. H.

Production of liquid preparations of tribromoethyl alcohol. BYK-GULDENWERKE CHEM. FABR. A.-G. (B.P. 387,432, 28.10.31. Ger., 4.11.30).—The alcohol is mixed with H<sub>2</sub>O-sol. esters, one of which is a urethane, e.g., urethane with EtOAc, MeOAc, or Et lactate.

C. H.

Purification of alcohols. R. M. ISHAM, Assr. to DOHERTY RES. Co. (U.S.P. 1,845,665, 16.2.32. Appl., 30.7.27).—*tert*-Alcohols are removed by preferential dehydration at 250° in presence of a catalyst, e.g., ThO<sub>2</sub> on pumice.

C. H.

Manufacture of aliphatic anhydrides and their recovery. H. DREYFUS (B.P. 387,365, 27.7.31).—The H<sub>2</sub>O in vapours from thermal anhydriation of AcOH etc. is removed by reaction with a hydrocarbon, e.g., CH<sub>4</sub>, in presence of CaWO<sub>4</sub> or NaPO<sub>3</sub> at 600–650°, the products being, if desired, passed over Ni at 700–750°.

C. H.

Esterification of organic acids with separation of the esters in highly concentrated form. INVENTION GES. F. VERWALTUNG U. VERWERTUNG CHEM.-TECH. PATENTE G.M.B.H. (B.P. 387,573, 13.6.32. Ger., 13.6.31).—The esterification is effected in 2 stages: (a) with excess alcohol, (b) with excess acid; the vapours from (a) are passed to (b), and the acid from (b) is passed, at a temp. > the b.p. of the ester, to (a). Part of the alcohol is preferably removed by washing the vapours

from (a) with H<sub>2</sub>O or acid at a temp. > the b.p. of the ternary mixture. The prep. of MeOAc and *n*-BuOAc is described.

C. H.

Manufacture of esters from aldehydes, and of catalysts suitable for use therein. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 387,621, 19.9.32. Ger., 25.4.32).—Aldehydes (MeCHO) are treated with a compound of ZnCl<sub>2</sub> with a chloroaluminium alkoxide, prepared, e.g., by refluxing Al, AlCl<sub>3</sub>, EtOH, and EtOAc, and adding ZnCl<sub>2</sub>.

C. H.

Controlling molecular aggregation in the polymerisation of vinyl esters. CANADIAN ELECTRO PRODUCTS Co., LTD., Asses. of K. G. BLAIKIE, G. O. MORRISON, and T. P. G. SHAW (B.P. 387,353, 30.4.31. U.S., 30.4.30).—Polymerisation is effected in presence of sufficient diluent (PhMe) to give, at the temp. employed, a product of the desired softening point and viscosity, the proportion being maintained, e.g., by returning to the mixture under reflux a portion only of the condensate together with fresh mixture richer in vinyl ester. The temp. is preferably kept const.

C. H.

Polymerisation of vinyl compounds and catalyst therefor. CANADIAN ELECTRO PRODUCTS Co., LTD., Asses. of K. G. BLAIKIE (B.P. 387,323 and 387,335, 27.4.31. U.S., 25.4.30).—(A) The peroxide catalyst (Ac<sub>2</sub>O<sub>2</sub>) is made in a solvent (PhMe) which does not retain in solution the non-catalytic by-products; a saturated aliphatic aldehyde (0.05% MeCHO) may be present. Vinyl acetate with Ac<sub>2</sub>O<sub>2</sub> in PhMe at 70° gives a white, horn-like mass, softening at 134°. (B) For the prep. of the catalyst an org. acid anhydride (Ac<sub>2</sub>O) is oxidised with a per-salt (NaBO<sub>3</sub>) in a suitable solvent (PhMe).

C. H.

Manufacture of enolic acetone derivatives from a mixture of acetone and its enolic form. B. N. LOUGOVOY, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,844,430—1, 9.2.32. Appl., [A] 26.5.26, [B] 27.5.26).—COME<sub>2</sub> is treated with 0.01–0.1% of KOH or other alkali at 15° to give a mixture containing 12–15% of diacetone alcohol, which is neutralised with tartaric acid and distilled. The recovered COME<sub>2</sub> is again enolised, a longer time being necessary, and condensed to diacetone alcohol mixture, the process being repeated. (B) The enolisation of the recovered COME<sub>2</sub> is accelerated by addition of CH<sub>2</sub>O or a *tert*. alcohol.

C. H.

Stabilisation of lead tetra-alkyl. W. S. CALCOTT and A. E. PARMELEE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,843,942, 9.2.32. Appl., 10.1.31).—Hydroxyalkylamines, e.g., tri-(β-hydroxyethyl)amine, are added to PbEt<sub>4</sub> or PbMe<sub>4</sub>, preferably in solution in C<sub>2</sub>H<sub>4</sub>Br<sub>2</sub> and C<sub>10</sub>H<sub>7</sub>Cl.

C. H.

Reduction of unsaturated [maleic and fumaric] acid compounds. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,844,394, 9.2.32. Appl., 20.4.29).—In the reduction of Na maleate or Me maleate or fumarate to succinates, Ni, Fe, Co, Cu, Zn, or Cd is used as catalyst in the form of chromates, borates, aluminates, titanates, zirconates, or base-exchange compounds or other salts not reduced to metal by H<sub>2</sub>; e.g., a Na Al silicate is leached with HCl and treated successively with Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and Ni(NO<sub>3</sub>)<sub>2</sub> solutions.

C. H.

**Dehydrogenation of hydrogenated heterocyclic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 387,507, 4.2.32).—The vapour is led over a catalyst comprising a metal of group VI (or a compound thereof), with or without carrier, at 250–550°. Suitable catalysts are molybdic acid, tungstic acid,  $WS_2$ , or a mixture of  $ZnO$ ,  $MgO$ , and molybdic acid or of  $CuO$ ,  $Cr_2O_3$ , and  $BaO$ . Examples are piperidine, tetrahydrodiphenylene oxide, tetrahydrocarbazole, quinolines from *ar*-tetrahydro- $\alpha$ - and  $\beta$ -naphthylamines, 1:2:3:4-tetra- and deca-hydroquinolines. C. H.

**Manufacture of piperidine pentamethylenedithio-carbamate.** [Vulcanisation accelerator.] ROBINSON BROS., LTD., and F. C. RAWSTRON (B.P. 387,496, 20.1.32).—Piperidine (2 mols.) and  $CS_2$  are separately dissolved in 3–7 vols. of a solvent in which the product is insol. (e.g., petroleum, b.p. 135–150°), and the solutions are mixed, with cooling. [Stat. ref.] C. H.

**Manufacture of phenol from chlorobenzene.** DR. F. RASCHIG GES.M.B.H. (B.P. 387,832, 26.1.32. Ger., 26.1.31).—In the manufacture of  $PhOH$  from  $PhCl$  and steam over a catalyst,  $PhOH$  and  $HCl$  are removed without condensing the excess  $PhCl$  and steam, e.g., by washing with alkaline  $H_2O$ ; the  $HCl$  may first be removed, e.g., with a limited quantity of  $H_2O$ . C. H.

**Condensation products from [triaryl]phosphine oxides and phenolic compounds.** W. LOMMEL and H. MÜNZEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,844,015, 9.2.32. Appl., 24.10.29. Ger., 31.10.28).—A phenol is combined with a triarylphosphine oxide or its hydrate to give compounds,  $PAR_3(OH)(OAr)$ . Compounds (m.p. in parentheses) are obtained from  $PPh_3O$  and  $PhOH$  (106°), *o*-, *m*-, and *p*-cresols (89–90°, 67°, 44–45°), *o*- and *p*- $C_6H_4Cl \cdot OH$  (105–106°, 80–81°), thymol (67–68°), chlorothymol (65–66°), 2:4- and 2:6-di- and 2:4:6-tri-chlorophenols (54–55°, 97–98°, 70–71°), *m*-5-xylene (83–84°), 6-chloro-*o*- and *m*- and 3-chloro-*p*-cresols (91–92°, 45°, 64–65°), *o*- and *p*-hydroxydiphenylmethanes (88–89°, 117°), *Et p*-hydroxybenzoate (104–105°), *p*-nitrophenol (107–108°), pyrocatechol mono-*Et* ether (93–94°), resorcinol (119–120°), *p*- $C_6H_4(OH)CHO$  (66–67°),  $\alpha$ - and  $\beta$ - $C_{10}H_7 \cdot OH$  (106–107°, 89–90°), diethyl-*m*-aminophenol (81–82°), *ar*-tetrahydro- $\beta$ -naphthol (120°), *isobutyl*-phenol (73–74°), 4-chloro-2-*isobutyl*-phenol (100–101°), 4:1- and 1:2-benzyl-naphthols (117–118°, 104–105°), dibromo- $\beta$ -naphthol (77–78°); and from tri-*o*-tolylphosphine oxide and  $PhOH$  (149–150°). C. H.

**Reduction of nitro-compounds.** S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,845,759, 16.2.32. Appl., 22.5.24).—In reductions of aromatic  $NO_2$ -compounds with  $Fe$ ,  $H_2O$ , and a little acid a vessel rotating about a more or less horizontal axis and provided with baffles is used to avoid stirring with stirrers. C. H.

**Manufacture of amine salts.** L. E. MILLS, Assr. to Dow CHEM. Co. (U.S.P. 1,843,693, 2.2.32. Appl., 14.9.29).— $NH_2Ph \cdot HCl$ , m.p. 198°, and *o*- $NH_2 \cdot C_6H_4Me \cdot HCl$ , m.p. 202°, are obtained colourless by interaction of liquid arylamine with a countercurrent of  $HCl$  gas at such temp. that the salt is liquid. C. H.

**Manufacture of *p*-isopropyl- $\alpha$ -methylhydrocin-namaldehyde [ $\beta$ -*p*-isopropylphenylisopropaldehyde].** A. KNORR and A. WEISSENBORN, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,844,013, 9.2.32. Appl., 18.10.29).— $\beta$ -*p*-isopropylphenylisopropaldehyde, b.p. 133–137°/9 mm., which has a cyclamen odour, is obtained by hydrogenation of the corresponding cin-namaldehyde, preferably in presence of  $Ni$ . C. H.

**Separation of phenylphenols [*o*- and *p*-hydroxydiphenyls].** E. C. BRITTON and F. BRYNER, Assrs. to Dow CHEM. Co. (U.S.P. 1,843,706, 2.2.32. Appl., 5.12.29).—The mixture is dissolved at 85° in aq.  $NaOH$  equiv. to the *o*- and 25% of the *p*-compound. Pure *p*-compound separates on cooling. A m.-p. curve for mixed *o*- and *p*-hydroxydiphenyls is given. C. H.

**Purification of keto [aroylbenzoic] acids.** A. O. JAEGER and H. J. WEST, Assrs. to SELDEN Co. (U.S.P. 1,845,751, 16.2.32. Appl., 26.10.29).—The acid (*o*-benzoyl- or *o*- $\alpha$ -naphthoyl-benzoic) is converted into alkali salt and impurities are oxidised with  $NaOCl$ ,  $Cl_2$  and  $NaOH$ , or alkaline  $KMnO_4$ . C. H.

**Production of styrene.** NAUGATUCK CHEM. Co., Asses. of O. H. SMITH and G. W. JARGSTORFF (B.P. 387,397, 12.8.31. U.S., 22.8.30).—Halogenated  $PhEt$  is refluxed at 155–175° with the salt of an org. base, e.g.,  $C_5H_5N$  hydrochloride, sulphate, or phosphate,  $NHEt_2 \cdot HCl$ ,  $NH(CH_2Ph)_2$  sulphate or hydrochloride. C. H.

**Manufacture of betaine hydrochloride.** K. BROMIG, Assr. to DEUTS. GOLD- u. SILBER-SCHNEIDANST. VORM. ROESSLER (U.S.P. 1,844,929, 16.2.32. Appl., 7.11.27. Ger., 15.11.26. Cf. B.P. 320,589; B., 1930, 77).—Molasses schlempe is refluxed with 25%  $HCl$  for 3–8 hr., alkali salts are filtered off, and after concn. to  $\frac{2}{3}$  vol. glutamic acid is pptd. by saturation with  $HCl$ . The filtrate, again conc. to  $\frac{2}{3}$  vol. in vac., gives cryst. betaine hydrochloride. C. H.

**Manufacture of wetting, cleaning, and emulsifying agents.** CHEM. FABR. VORM. SANDOZ (B.P. 387,398, 12.8.31. Switz., 5.12.30).—Esters of glycols or glycerols,  $C_2H_4(OH)(OY)$ ,  $C_3H_5(OH)(OX)(OY)$ , or  $C_3H_5Cl(OH)(OY)$ , where  $X = H$ ,  $Ar$ , or aralkyl, and  $Y = Ar$  or aralkyl, with fatty acids above  $C_9$  are sulphonated. Thus, the oleic ester of glycerol monoxylyl ether is treated with 100%  $H_2SO_4$  at  $-5^\circ$  to  $8^\circ$ . C. H.

**Production of dibenzanthrone.** L. C. DANIELS, Assr. to SELDEN Co. (U.S.P. 1,844,381, 9.2.32. Appl., 26.1.31).—Benzanthrone is heated with alkali in presence of fructose or crude formose. C. H.

**Production of  $PhOH$ .**—See I. Oxidation of hydrocarbons.—See II. Azo intermediates.—See IV. Products from butadiene.—See XIV. Treating effluent waters.—See XXIII.

#### IV.—DYESTUFFS.

Casein pigments.—See XIII.

See also A., Mar., 220, Solubility of dyes. 225, Dye sols. 258, Light-sensitive carotene dye. 285, Cyanine dyes. Chlorophyll [and azo dye from 2:4-dipropionyl-3:5-diethylpyrrole].

## PATENTS.

**Manufacture of [acid] dyes of the triarylmethane series.** I. G. FARBENIND. A.-G. (B.P. 387,956, 12.9.32. Ger., 12.9.31).—Dyes containing the group  $>N\cdot CH_2\cdot CH_2\cdot SO_3H$  are prepared. Examples are oxidised products from: *p*-diethylaminobenzaldehyde and *N*-*o*-tolyltaurine (red-violet);  $CH_2O$  and *N*-phenyl-*N*-ethyltaurine (blue-violet); Michler's hydrol and *N*-*m*-tolyltaurine (blue-violet); 3'-amino-4:4'-bis(diethylamino)triphenylmethane and  $CH_2Cl\cdot CH_2\cdot SO_3H$  (blue-green). C. H.

**Manufacture of [pigments and wool] dyes [from benzoquinones].** I. G. FARBENIND. A.-G. (B.P. 387,565, 26.5.32. Ger., 26.5.31. Addn. to B.P. 313,094; B., 1931, 337).—The dyes of the prior patent are also obtained by heating at 150–300° benzoquinone, which may carry halogen, alkyl, or aryl in positions 2 and/or 5, and/or halogen in positions 3 and/or 6, with arylamines,  $NH_2\cdot Ar\cdot NHAr'$  or  $NH_2\cdot Ar\cdot NArR$ , in which Ar = arylene, Ar' = aryl, and R = alkyl, or Ar·N·Ar' is a carbazole residue. A metal chloride condensing agent and an oxidant (di- or tri-nitrophenol) may be present, and the products may be sulphonated. Examples include: chloranil with 2-amino-5-ethylcarbazole; 2-phenyl-*p*-benzoquinone with *p*-aminodiphenylamine; 2:3:5-trichloro-*p*-benzoquinone with 2-amino-5-ethylcarbazole. C. H.

**Manufacture of azo dyes and intermediate products.** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 387,360, 6.7.31).—2:3-Hydroxynaphthoic arylamides  $C_{10}H_6(OH)\cdot CO\cdot NH\cdot C_6H_4RR'\cdot NH_2$ , in which the  $NH_2$  is in position 4, and R (position 3) and R' (position 6) each represents alkyl or alkoxy, are made from the corresponding arylamines,  $NH_2\cdot C_6H_4RR'\cdot NO_2$  or  $NH_2\cdot C_6H_4RR'\cdot NHAc$ . The products are treated in substance or on the fibre with  $HNO_2$  to give fast blacks. An example is 2:3-hydroxynaphthoic acid condensed with 5-acetamido-4-methoxy-*o*-toluidine in PhMe with  $PCl_3$ , hydrolysed with alcoholic  $H_2SO_4$ , padded with  $NaNO_2$  on cotton, and treated with dil. HCl. C. H.

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

**Fine structure and mechanical properties of fibres.** H. MARK (Trans. Faraday Soc., 1933, 29, 6–10).—The stress-strain relation, calc. on the assumptions that the strength of a cellulose fibre is proportional to the areas of contact of adjacent micelles and that stretching orients the micelles in the direction of stretching, is similar to the experimental relation. A. G.

**Relations between the refractive indices and the behaviour of cellulose fibres.** J. M. PRESTON (Trans. Faraday Soc., 1933, 29, 65–71).—The degree of orientation of the micelles, and hence the extensibility of cellulose fibres, can be calc. from the val. of  $n$ , and this also distinguishes mercerised from unmercerised fibres. A. G.

**Emulsification of mixed liquids of high mol. wt. [in wool-scouring processes].** J. B. SPEAKMAN and N. H. CHAMBERLAIN (Trans. Faraday Soc., 1933, 29, 358–365).—The amount of mineral oil left in wool after a standard scour increases with increasing b.p.

(chain length) of the oil, owing to increasing adhesion. Removal is facilitated by the addition of 6% of oleyl alcohol to the oil; larger amounts are ineffective owing to increasing adhesion, due to its polar character. Large amounts of olive oil, oleic acid, and oleonitrile also assist the removal of mineral oil. A. G.

**Physical significance of crimp or waviness in the wool fibre.** S. G. BARKER [with E. HILL] (Trans. Faraday Soc., 1933, 29, 239–257).—Crimp in wool fibres may be due to dehydration after emergence from the follicle, to physico-chemical forces acting within the follicle, to surface-tension effects on the newly-extruded hair, to the rotation of the bulb of the follicle, or to a combination of these causes. A. G.

**Electrical resistance of wool fibres.** M. C. MARSH and K. EARP (Trans. Faraday Soc., 1933, 29, 173–192).—The resistance of wool fibres is independent of voltage, polarity, and time. Between 40 and 82% a 10% decrease in R.H. causes an 8–9-fold increase in resistance, and when fibres are taken through a cyclic change of humidity hysteresis is observed. Wools of different types are similarly affected by humidity changes. Resistance ( $R$ ) and  $H_2O$  content ( $M$ ) are related thus:  $R = CM^{-15.0}$ . For falling humidity  $R$  is lower than for rising humidity.  $R$  is increased 8-fold by washing with distilled  $H_2O$ . Steaming loose fibres does not affect the resistance, but steaming under tension increases it by 60% owing to the regain being diminished. Steaming and setting at 80% extension greatly increases  $R$ . Conduction of electricity occurs through  $H_2O$  channels between the micelles and the rapid rise of  $R$  with diminishing  $M$  is due to the breaking down of  $H_2O$  paths. A. G.

**Modification of natural cotton cellulose by swelling and by degradation.** S. M. NEALE (Trans. Faraday Soc., 1933, 29, 228–234).—The reactions of cotton cellulose can be explained without assuming the existence of micelles if the structure is regarded as continuous but slightly imperfect. A. G.

**Degree of ripening of cotton.** O. ROEHRICH (Trans. Faraday Soc., 1933, 29, 218–227).—The degree of ripening is measured by a coeff. of maturation obtained by combining the hair diam. and the hair wt. per cm. A. G.

**Chemical degradation of linen cellulose.** C. R. NODDER (Trans. Faraday Soc., 1933, 29, 317–324).—For linen degraded by neutral hypochlorite and by acids, respectively, the solubility no. ( $S$ ) and the viscosity in 2% solution ( $\eta_2$ ) are related by the equations  $6/\sqrt{S-1} = \log \eta_2 + 1.85$ , and  $4.4/\sqrt{S-1} = \log \eta_2 + 1.85$ . For substances of the same viscosity the Cu no. is higher for attack by neutral hypochlorite than by acids, and a mechanism is suggested to account for these differences and for the strength changes which occur during modification and during subsequent alkali-boiling. A. G.

**"Regain" of silk of different origins.** W. S. DENHAM and A. L. ALLEN (Trans. Faraday Soc., 1933, 29, 316–317).—The  $H_2O$  regains of Italian, Chinese, and Japanese mulberry silks are the same and < the regain of tussah silk. A. G.

**Tensile properties of silk filaments.** W. S. DENHAM and T. LONSDALE (Trans. Faraday Soc., 1933, 29, 305—316).—The load-extension diagram for silk filaments is linear for small loads, but at higher loads the extension increases progressively more rapidly; at R.H. > 85% there is no linear portion. At R.H. 65% Young's modulus for the linear part of the curve is about 0.8, and the tensile strength about  $6 \times 10^9$  dynes per sq. cm. With increasing R.H. the breaking load falls and the extension at break rises; the breaking load also increases with increasing rate of loading. The strain may be divided into elastic, epibolic, and plastic parts, the proportion of the last-named increasing with the amount and duration of the loading. A. G.

**Technical significance of hemicelluloses.** R. RUNKEL (Papier-Fabr., 1933, 31, 75—77, 86—89, 97—100).—Spruce wood, red beech wood, and cereal straw contain, respectively, 24, 33, and 28% of hemicelluloses, a large proportion of which are pentosans in the last two. These are the source of AcOH and furfuraldehyde during dry distillation. If pentosans are removed from beech wood by mild acid hydrolysis the residue yields little furfuraldehyde, but the amount of phenols is increased, probably due to less phenol-aldehyde condensation. In the saccharification of wood utilisation of hemicelluloses may lead to an increased yield of fermentable sugars. Xylan gives a cryst. xylose and a fermentable xylose solution by acid hydrolysis, and the latter yields EtOH, AcOH, COMe<sub>2</sub>, and lactic acid. Hemicelluloses may be important in rayon as they can be xanthated and acetylated. In bast fibres pectins of differing properties cement together the crystallites, the hairs, and the hair bundles, and in making textiles and paper the removal of this adhesive must be carried to the correct stage. The strength of paper depends on the cementing substances, and if wood is so treated that the pulp contains hemicelluloses the yield is larger, less milling is required, and the paper is stronger. Hemicelluloses and lignin in spent liquors from soda-cellulose may be converted into AcOH, COMe<sub>2</sub>, etc. A. G.

**Influence of mild hydrolysis on chemical composition of wood.** J. WIERTELAK (Przemysł Chem., 1933, 17, 1—5).—The yield of reducing substances from white-pine sawdust hydrolysed with H<sub>2</sub>O under 8.17 atm. steam pressure attains a max. val. of 12.5% after 180 min.; using 0.5—2% acids in place of H<sub>2</sub>O the corresponding figure is 21%. The production of sugars takes place chiefly from  $\gamma$ -cellulose, the content of which falls from 16 to 9.4% after hydrolysis with H<sub>2</sub>O, to 1.52% with 0.5% H<sub>2</sub>SO<sub>4</sub>, and to 0.3% with 2% H<sub>2</sub>SO<sub>4</sub>; in addition, the Cross and Bevan cellulose content falls from 53.45 to 41.6—45%, and the cellulose pentosan content from 4.3 to 0.4—2.7%. At the same time, the  $\beta$ -cellulose content rises from 0 to 0.84% after hydrolysis with H<sub>2</sub>O, and to 7.6—8.8% with 0.5—2% H<sub>2</sub>SO<sub>4</sub>. The contents of other constituents are practically unaffected by mild hydrolysis. R. T.

**Wood pulp and its raw materials. I. General. II. Acetylation of cellulose pulp. III. Chlorination of mulberry.** T. NAKASHIMA and M. NEGISHI (J. Cellulose Inst., Tokyo, 1932, 8, 255—259, 259—263, 263—266). I. The difference between rayon and paper

pulp was investigated. The  $\alpha$ -cellulose content of rayon pulp is always higher than that of paper pulp, whilst the  $\beta$ -cellulose content of the latter is almost double that of the former. The difference between the two types of raw materials is, however, not great, so that paper pulp should function satisfactorily in rayon manufacture.

II. Unbleached cellulose pulp was purified with 90% NaOH and acetylated by Kita's method. COMe<sub>2</sub>-sol. acetates from varying celluloses were thus prepared and compared. Bleaching the acetates with KMnO<sub>4</sub> caused increase in viscosity, but solubility and Ac content remained unaltered.

III. Examination has shown that mulberry is actually superior to the other bast fibres as a raw material. Alkali purification was more effective than chlorination and gave products with 89.51—93.13%  $\alpha$ -cellulose.

V. E. Y.

**Refining of wood pulp.** K. NAKAMURA (J. Cellulose Inst., Tokyo, 1932, 8, 266—269).—The  $\alpha$ -cellulose content of bleached pulp is raised by 5% by treatment with CaO and Na<sub>2</sub>SO<sub>3</sub>. A small addition of NaOH is advantageous, but excess must be avoided. The bleaching should preferably be carried out at low temp.

V. E. Y.

**[Sulphite-pulp] digester lining developments.** A. F. RICHTER (Paper Trade J., 1933, 96; T.A.P.P.I. Sect., 67).—The developments and trends in the design and construction of such linings are reviewed. H. A. H.

**Penetration in sulphite[-pulp] cooking.** R. DE MONTIGNY (Pulp and Paper, Canada, 1933, 34, Conv. issue, 109—111).—Preliminary experiments showed that with initial evacuation of air-dry spruce chips and consequently more rapid penetration by the bisulphite cooking liquor, the cooking time could be shortened by almost eliminating the penetration period, without producing burnt chips. The denser jack pine did not respond to evacuation, and appreciable burning still took place. The natural resins in jack pine also inhibit aq. penetration. The results (unpublished) of work bearing on this subject, done by O. MAASS and co-workers, are summarised.

H. A. H.

**Action of sulphurous acid on cellulose.** L. P. MOORE and H. HIBBERT (Pulp and Paper, Canada, 1933, 34, Conv. issue, 103—107).—The effect of an excess of H<sub>2</sub>SO<sub>3</sub> on two forms of purified cellulose at different temp. and for different periods (from 2 to 16 hr.) has been studied. At 30°, increasing concn. of acid (from 0.1 to 4.0% SO<sub>2</sub>) markedly decreases the cuprammonium viscosity, but has no effect on the  $\alpha$ -cellulose content or Cu no. At 100° increasing acid concn. decreases  $\alpha$ -cellulose content and increases Cu no., but has no effect on viscosity. These effects are tentatively ascribed to the decreasing H<sup>+</sup> activity of SO<sub>2</sub> solutions at higher temp.

H. A. H.

**Origin of cymene in sulphite[-cellulose] liquor.** J. O. MURTO (Suomen Kem., 1932, 5, [A], 121—126).—The cymene results probably from dehydrogenation of the terpenes present by the action of S.

H. F. G.

**Changes in the cellulose during the maceration process in paper manufacture.** E. L. SELLEGER (Chem. Weekblad, 1933, 30, 44—46).—A general account,

with a comparison between papermaking and the production of cellulose ester films. H. F. G.

**Solubility and swelling of ethylcelluloses in organic solvents.** S. N. YSHAKOV and R. S. ALEXANDROV (Plast. Massi, 1932, Nos. 2—4, 1—6).—The action on two ethylcelluloses (containing different proportions of OEt) of org. liquids in which they swell or disperse has been examined. The one with the lower OEt no. disperses the less readily. The actions of different org. liquids depend on their nature and on the chain-length and the distribution of the polar groups. The best solvents are those which react most energetically with the groups of the cellulose derivatives. Characteristic of many solutions of ethylcelluloses is their gelatinisation when cooled. T. H. P.

**Theory of cellulose nitration.** M. SENDO (J. Cellulose Inst., Tokyo, 1932, 8, 290—304).—Cellulose was nitrated at 25° with mixed acids of varying composition and the products were studied. The N content was found to increase until it was in equilibrium with the nitrating acid. The esterifying capacity of the bath has been estimated in terms of the free energy of the nitrating acids. Farmer's view of the nitration mechanism is discussed. In addition to having a dehydrating action, the H<sub>2</sub>SO<sub>4</sub> present in the nitration bath also causes swelling of the cellulose. V. E. Y.

**Fractionation of nitrocotton.** (Preliminary note.) **Dissolution of nitrocotton in acetone-water.** S. YAMAMOTO (J. Cellulose Inst., Tokyo, 1933, 9, 4—9).—On treating nitrocelluloses of various N contents with mixtures of COMe<sub>2</sub> and H<sub>2</sub>O in different proportions, the solubility for any given N content increases with increasing COMe<sub>2</sub> content of the solvent mixture, at first slowly and then very rapidly. In the region of slow increase fractionation is possible by diffusion methods, whilst in that of rapid increase it may be done by repeating the dissolution and pptn. The % COMe<sub>2</sub> necessary for complete dissolution of the material is directly proportional to its N content. B. P. R.

**Determination of the degree of ripening of viscose.** Z. KAWATA (J. Cellulose Inst., Tokyo, 1933, 9, 10—13).—Results given by the NH<sub>4</sub>Cl method (A) vary with small variations in the composition of the viscose, but the method gives an accurate val. Those obtained by the NaCl method (B) vary similarly, but to a less extent than with method A. Method B is suitable for factory use when an approx. val. is required. For viscose containing 8% of cellulose and 7% of alkali an NH<sub>4</sub>Cl no. of 10—9.5 corresponds to a NaCl no. of 4—3.5. Method B is suitable for comparing the degrees of ripening of viscoses of different composition, but if method A is used for this purpose the samples taken must contain equal wts. of cellulose. B. P. R.

**Colloid chemistry of the viscose process.** H. L. BREDÉE (Chem. Weekblad, 1933, 30, 51—54).—A general description is given of the spinning process, and of such factors as the influence of the Na<sub>2</sub>SO<sub>4</sub> content of the bath on the strength of the thread and of the tension on the orientation of the micelles. H. F. G.

**Technique of viscose, [cellulose] acetate, and gelatin films and similar structures.** M. HALAMA

(Kolloid-Z., 1932, 61, 240—246).—The production of wrapping papers of cellulose and other products is described. The properties and applications of these films are discussed. X-Ray examination indicates that the axis of the thread-like micelles is perpendicular to the plane of the film. E. S. H.

**Spinning process in the preparation of artificial silk.** Properties of technical spinning solutions. O. FAUST (Kolloid-Z., 1932, 61, 257—264).—A lecture, in which the relations between spinning properties, the viscosity of the solution, and the orientation of thread-like micelles are discussed. E. S. H.

**Cohesion of artificial silk fibres in relation to their structure.** L. A. VAN BERGEN (Chem. Weekblad, 1933, 30, 55—58).—The influence of the differences of structure between the outer layers and the core, resulting from the non-uniform distribution of velocity across the jet, on the mechanical properties of viscose threads is discussed. H. F. G.

**White papers from southern pines [of U.S.A.]. III. Pulping longleaf pine for strong, easy-bleaching pulp.** M. W. BRAY and C. E. CURRAN (Paper Trade J., 1933, 96; T.A.P.P.I. Sect., 62—66).—Semi-large-scale investigation shows that longleaf pine (*Pinus palustris*), cooked by the sulphate process and bleached by the two-stage chlorination-hypochlorite method, gives an easy-bleaching sulphate pulp very similar in strength and colour to that obtained from loblolly pine. The yield (41%) and the bleach requirement are not affected by the rate of growth or the density of the wood (calc. on a wt. basis). The re-use of black liquor (40% of the total vol.) in the cooking process gives a slightly higher yield, but increases the bleach requirement a little; pulp strength is unaffected. H. A. H.

**Estimation of dirt and shives in pulp and paper.** J. D'A. CLARK, R. S. VON HAZMBURG, and R. J. KNOLL (Paper Trade J., 1933, 96; T.A.P.P.I. Sect., 54—56).—A new visual method is described. It is claimed to be many times more accurate than a numerical count. The quantity of dirt is determined in terms of its "equiv. black area" (estimated area of a contrasting spot in terms of the area of that size of a perfectly black spot on a pure white background which would make the same visual impression), and reported in p.p.m. Shives are reported in terms of their actual area, also in p.p.m. The method is sufficiently rapid for control purposes. H. A. H.

**Dendritic growths in paper.** J. STRACHAN (Paper Maker, 1933, 85, 49—52 ts).—The conditions governing the growth of dendrites in paper are discussed. Such growths are considered to take place in two stages. The metallic Cu particles in the paper are first converted into CuSO<sub>4</sub> by the Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> used in sizing, the CuSO<sub>4</sub> then being reduced to CuS by the H<sub>2</sub> liberated from the reaction between metal and acid. This stage is reached comparatively quickly. The CuSO<sub>4</sub> is later slowly oxidised to basic sulphate during several years. The black and the greenish-brown products can be differentiated. The tree-like growths are due to diffusion and adsorption of CuS along the cellulose fibre surfaces, the greatest growth usually taking place,

therefore, in the machine direction of the paper. Fe particles, unlike Cu, form Liesegang rings by diffusion, there being no cryst. growth. Photomicrographs are given.

H. A. H.

**Case for rosin-wax [paper] sizes.** R. B. LADOO (Chem. Met. Eng., 1933, 40, 89—90).—Rosin-wax sizes are more efficient than ordinary rosin-soap sizes, largely because of their greater covering power, which is due to smaller particle size and lower m.p. It is claimed that many papermaking troubles such as foaming of the stock, brittleness, lack of permanence, and curling of the finished paper are diminished by their use. Paper so sized is less pervious to aq. alkali, EtOH, and printing inks, and possesses a higher gloss. The process is economical.

H. A. H.

**Filler uptake of unsized paper.** R. LORENZ and R. STOPP (Papier-Fabr., 1933, 31, 25—33, 41—46, 53—57).—When paper is filled with china clay the wt. of filler in the paper is proportional to that added to the pulp. It increases with increasing thickness of the paper, with increased milling, with increasing coarseness of the clay, and, within the practical range, with increasing rate of filtration. The incorporation of the filler is due to filtration only, and, if the paper is < a certain thickness, all the clay passes through. The gauze side of the paper contains a larger proportion of coarse clay particles than the upper side; the two sides are thus different, especially as the finer particles appear whiter.

A. G.

**Testing cigarette papers for elasticity.** K. MANDL (Papier-Fabr., 1933, 31, 113—114).—Apparatus and procedure are described.

**School materials [paper].**—See XIII. **Determining swelling [of artificial silk].** Latex thread. Use of rubber [as adhesive].—See XIV. **Fermentation of fibrous materials.**—See XVIII.

See also A., Mar., 216, **Physical structure of cellulose fibre, cellulose nitrates, etc.** 248, **Automatic titrating devices [for the paper industry].**

## PATENTS.

**Preparing resinous wood for digestion by the bisulphite process.** G. ROSEN, Assr. to CELLULOSE ROSEN SOC. ANON. (U.S.P. 1,857,695, 10.5.32. Appl., 16.5.31. Fr., 7.8.30).—The chips are pretreated, preferably in a closed circulating system, with dil. aq.  $\text{Ca}(\text{OH})_2$  (1—2% CaO on the wt. of dry wood) for about 3 hr. at  $\geq 110^\circ$ . This treatment may be repeated once or twice with fresh liquor. The chips are finally washed, preferably with aq. 1% NaCl, to facilitate subsequent penetration of  $\text{SO}_2$ .

D. J. N.

**Manufacture of [cellulose] products from corn-stalks, sugar cane, and similar vegetable material.** G. TAUBE, Assr. to I. B. BRODIE, S. MUSHER, and B. M. RUBIN (U.S.P. 1,857,985, 10.5.32. Appl., 20.8.30).—The material, after crushing and screening to remove pith, is placed in a digester which is then evacuated and filled with  $\text{H}_2\text{O}$ . After 8—12 hr. the  $\text{H}_2\text{O}$  is run away and the digester again evacuated and filled with digestion liquor (2% aq. NaOH). The material is then cooked for 2—3 hr. at 2—3 atm., washed, and, if desired, bleached.

D. J. N.

**Treating [cooking] fibrous material.** T. W. BACCHUS, Assr. to HERCULES POWDER CO. (U.S.P. 1,856,453, 3.5.32. Appl., 23.9.30).—Cellulosic material and digestion liquor are continuously pumped through a long pipe the outlet from which is controlled by a pressure valve or stand-pipe to give the required digestion pressure. The digester pipe is heated near the inlet and cooled near the outlet; the intermediate section (about 3000 ft. long), in which digestion takes place, is insulated.

D. J. N.

**Preparation of fibrous cellulose acetates.** C. F. BOEHRINGER & SOEHNÉ G.M.B.H. (B.P. 387,533, 24.3.32. Ger., 24.3.31).—After pretreatment with AcOH, cellulose is acetylated at  $\geq 40^\circ$  with  $\text{Ac}_2\text{O}$ ,  $\text{SO}_2$ , and a catalyst ( $\text{HClO}_4$ , its salts with weak bases, or its salts with strong bases together with a free mineral acid), in the presence of substances preventing the acetylation product from going into solution, e.g., PhMe.

F. R. E.

**Manufacture of delustrated cellulose acetate yarn.** VISCOSE CO. (B.P. 386,216, 4.6.32. U.S., 16.9.31).—5—20% of chlorinated  $\text{C}_{10}\text{H}_8$  ("Halowax") on the wt. of cellulose acetate is added to the spinning solution.

D. J. N.

**Preparing cellulose xanthate.** R. DOSNE, Assr. to CANADIAN INTERNAT. PAPER CO. (U.S.P. 1,857,948, 10.5.32. Appl., 26.8.29. Can., 12.4.29).—Standard ageing conditions (72 hr. at  $20^\circ$ ) can be adopted for alkali-celluloses prepared from different pulps if there is incorporated with the alkali-cellulose the requisite quantity (determined by trials) of a sulphite or bisulphite which retards ageing in direct proportion to the amount present. A formula is given for calculating the approx. quantity of  $\text{Na}_2\text{SO}_3$  required for a particular pulp.

D. J. N.

**Production of low-viscosity cellulose esters.** C. J. MALM and A. ANDERSEN, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,857,562, 10.5.32. Appl., 29.6.29).—The dry ester is exposed to a current of ozonised air at, e.g., room temp. until the viscosity is reduced to the required degree.

D. J. N.

**Manufacture of artificial filaments.** E. PRINCE, Assr. to DU PONT RAYON CO. (U.S.P. 1,856,401, 3.5.32. Appl., 14.6.24. Fr., 21.12.23).—A high spinning speed can be satisfactorily maintained when spinning fine filaments if the temp. is reduced in the vicinity of the spinning jets by, e.g., cooling the spinning solution and/or introducing a cold (room temp.) evaporative medium at both ends of the cell simultaneously and drawing off the vapour-laden medium at some intermediate point, e.g., at or near the middle. Heating devices are placed in the lower half of the cell.

D. J. N.

**Manufacture of artificial threads [from resins].** I. G. FARBENIND, A.-G. (B.P. 387,976, 19.10.32. Ger., 19.10.31).—A solution in an org. solvent, e.g., cyclohexanone, of an artificial resin, e.g., a polymerised vinyl compound, a mixed polymerisation product of a vinyl compound with  $\text{CH}_2\text{=CCl}_2$  or maleic anhydride, or of esters of aliphatic or aromatic dicarboxylic acids with polyhydric alcohols, together with a softening agent if desired, is spun into a pptg. bath containing AcOH,  $\text{NaClO}_4$ , or NaCNS, and the swollen threads are stretched in several increasing steps and then shrunk before collecting on the receiving device.

F. R. E.

**Production [drying] of artificial filaments with uniform shrinkage.** VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 387,918, 4.7.32. U.S., 2.7.31).—The freshly-spun filaments, while still on perforated or permeable bobbins, are covered with a watertight and air-impermeable jacket, *e.g.*, a cloth of sufficient thickness, and dried by withdrawing  $H_2O$  solely from the interior. After re-spooling, with simultaneous twisting if desired, they are moistened and again dried from the interior towards the exterior. F. R. E.

**Manufacture of regenerated silk yarn.** S. YAMAMOTO (B.P. 387,890, 19.5.32).—Natural silk or fibroin dissolved in ammoniacal Cu or like solution is spun into a coagulant consisting of a solution of one or more alkali salts [ $Na_2SO_4$  or  $(NH_4)_2SO_4$ ], mixed with insufficient free acid to convert them into acid salts, together with other sol. coagulant salts ( $MgSO_4$  or alum). F. R. E.

**Absorbent paper.** F. H. McCORMICK and G. L. SCHWARTZ, ASSRS. to CELASTIC CORP. (U.S.P. 1,857,100, 3.5.32. Appl., 25.8.31).—Cellulose fibres of papermaking length, *e.g.*, kraft pulp, jute, are dispersed in a solution of a swelling agent, *e.g.*, 8–35% aq. NaOH at 25°, and the reaction is stopped (by dilution) when the increase in freeness of the pulp (due to curling of the fibres) is  $> 14\%$ , and before any substantial proportion of the non- $\alpha$ -cellulose constituents of the pulp is dissolved ( $> 30$  min.). Products made from such pulp, optionally in admixture with untreated pulp, show high porosity coupled with good strength, and are used as a base for impregnating compositions. D. J. N.

**Manufacture of waterproof and insulating paper.** J. KNAGGS, and PORTALS, LTD. (B.P. 387,248, 2.8.32).—Waterleaf paper is impregnated with an emulsion containing a protein (casein or gelatin) and rubber latex, balata, or the like, and then passed through a bath containing a hardening agent for the protein ( $CH_2O$ ), colloidal S, and a vulcanisation accelerator. After being set aside for 12–24 hr. in a wet condition, the paper is air-dried and calendered. Part of the protein in the impregnating emulsion may be replaced by a natural or synthetic resin. D. J. N.

**Treatment [strengthening and waterproofing] pulp articles and material therefor.** A. L. CLAPP (U.S.P. 1,857,498, 10.5.32. Appl., 27.9.28).—Moulded articles made from cellulose pulp (preferably unsized) are impregnated with aq.  $Na_2SiO_3$  ( $d$  1.07–1.12) containing kieselguhr (1%) and optionally an oxide pigment (2%) in suspension and are then dried at 93–149°. D. J. N.

**Surface-finishing of paper or fibre board.** A. L. CLAPP (U.S.P. 1,857,497, 10.5.32. Appl., 14.2.27).—Unsize board is treated on one or both sides, before or during calendering, with a solution of a glaze-producing agent, *e.g.*, aq.  $Na_2SiO_3$  ( $d$  1.16), and is then heavily calendered between hot rolls before penetration of the size is deep enough to induce crushing. A waterproof effect may be obtained by incorporating with the board during manufacture substances, *e.g.*, asbestos (10–50%) or CaO (5–20%), which react with  $Na_2SiO_3$  to form waterproofing agents. D. J. N.

**Dressing or sizing of paper.** CHEM. U. SEIFENFABR. R. BAUMHEIER A.-G. (B.P. 386,018, 6.8.31. Ger., 6.8.30.

Addn. to B.P. 385,344; B., 1933, 200).—The resin emulsion described in the prior patent is used.

D. J. N.

**Paper-article manufacture.** A. L. CLAPP (U.S.P. 1,857,496, 10.5.32. Appl., 16.7.26. Renewed 9.11.31).—Asbestos-cellulose paper (*e.g.*, 40:60), preferably containing 10–15% of CaO, is impregnated with aq.  $Na_2SiO_3$  ( $d$  1.26), dried to a  $H_2O$  content of 20–30%, and moulded under heat (71–82°) and pressure (500 lb./sq. in.). If an article resembling crockery is required, a finely-divided inorg. filler (75% clay and 25% siliceous) is dusted on the surface before moulding. D. J. N.

**Manufacture of [rubbered] sheet material.** DUNLOP RUBBER CO., LTD., L. BROWN, and F. W. WARREN (B.P. 387,451, 25.11.31).—A suitable textile backing, *e.g.*, canvas, hessian, or paper, or equiv. felted material, *e.g.*, cotton or wool fibre bonded, *e.g.*, with rubber, is covered on one side with a layer of disintegrated fibrous material, *e.g.*, comminuted paper, cotton, or wool, which is retained by an adhesive coating, and on the other side with a layer of sponge rubber. Alternatively, the textile backing may be omitted and the sponge rubber, produced as a froth of aq. dispersion, may, in sheet form, be provided on one side with an adhesive layer to which a coating of suitable disintegrated material is firmly attached. D. F. T.

**Manufacture or treatment [reduction of electrification] of artificial sheets, films, foils, etc.** H. DREYFUS (B.P. 387,710, 12.8.31).—The de-electrifying substance, consisting of alkylolamines and/or their derivatives, the  $\omega$ -aminoalkylamides of fatty acids, the di- or poly-hydric alcohol derivatives in which some only of the OH groups are esterified by higher fatty acids, or mixtures thereof, in solution or suspension in  $H_2O$  or an org. liquid (paraffin oil), is added to the solution of the material composing the sheets etc. (org. derivatives of cellulose), to the pptg. bath used in their manufacture, or is applied to the sheets etc. continuously with or subsequent to their production. F. R. E.

**Manufacture of fibrous compositions containing filler and binder substances.** RICHARDSON CO. (B.P. 386,991, 16.5.31. U.S., 6.4.31).—In the processes of B.P. 341,542 and 383,636 (B., 1931, 389; 1933, 102), the filler, instead of being added in the beater, is mixed with the thermoplastic agent before addition of the fibrous material. D. J. N.

**Prep. of bleach liquors.**—See VII. **Preserving straw etc.**—See IX. **Artificial leather.**—See XV.

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

**Theory of the dyeing process. Influence of acid dyes on animal fibres.** E. ELÖD (Trans. Faraday Soc., 1933, 29, 327–345).—Wool absorbs acids from  $H_2O$  solutions in amounts depending on the  $p_H$ , and an equilibrium is attained which can be reached from either side. The first process in acid dyeing is the formation of a protein salt with the mineral acid. This brings a Donnan equilibrium into play, and the colour anions distribute themselves in the same ratio as those of the colourless acid. The amount of dye absorbed and the rate of absorption pass through maxima at  $p_H$  1.3. That chemical combination takes place between the



wool and the dye is shown by a diminished capacity of dyed wool to combine with HCl. In agreement with the Donnan equilibrium equations the addition of a neutral salt ( $\text{Na}_2\text{SO}_4$ ) decreases the rate of dyeing, hence accounting for the levelling action of salts. The full basicity of wool is developed only at a low  $p_{\text{H}}$  owing, e.g., to internal salt formation. A. G.

**Printing of sulphur dyes on cotton.** B. J. CHESHIRE (Sealed Note No. 2010, 2.7.10. Bull. Soc. Ind. Mulhouse, 1932, 98, 555—557). Report by G. TAGLIANI (*Ibid.*, 557—558).—Attack of the Cu printing rollers by the printing paste is avoided by using (instead of  $\text{Na}_2\text{S}$ ) a paste obtained by cooling to  $5^\circ$  a mixture of 2 kg. of 40%  $\text{CH}_2\text{O}$  and 1 kg. of  $\text{NaHSO}_3$  ( $d$  1.33) and adding gradually 1 kg. of  $\text{Na}_2\text{S}$  solution (65%  $\text{Na}_2\text{S}$ ); this paste is stable in the printing paste but becomes reactive during the 5 mins. steaming which follows printing. TAGLIANI reports favourably on tests using Thionol Red Brown 5R, Brilliant Green 2G, and Yellow G. Replacement of conc. aq.  $\text{Na}_2\text{S}$  by cryst.  $\text{Na}_2\text{S}$  yields an unsatisfactory and less stable product. A. J. H.

**Printing of black and grey reserves under sulphur colours by means of nitrosodimethylaniline.** L. GOUBYRIN (Sealed Note No. 1995, 25.4.10. Bull. Soc. Ind. Mulhouse, 1932, 98, 559—561). Report by A. LAUTER (*Ibid.*, 561).—Fabric is printed with a paste containing  $\text{NMe}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{NO}$  and  $m\text{-C}_6\text{H}_4(\text{OH})_2$ , and (for grey effects)  $\text{ZnCl}_2$ , then dried and steamed for 5—6 min., and afterwards over-printed with a S-dye liquor, washed, soured with 5%  $\text{H}_2\text{SO}_4$ , washed, and soaped;  $\text{ZnCl}_2$  may be partly substituted by  $\text{Al}(\text{OH})_3$ . LAUTER reports that the resulting effects are fast to washing. A. J. H.

**Some difficulties in cotton yarn mercerisation.** P. KRAIS (Trans. Faraday Soc., 1933, 29, 355—357).—The dyeing properties of mercerised cotton are affected by the conditions of drying, by traces of acid, and by the action of light and air. A. G.

**Creosote from wood tar as wetting agent in dry mercerisation.** W. POHL (Chem.-Ztg., 1933, 57, 142—143).—Light creosote ( $1\frac{1}{2}$  g.) added to the alkaline mercerising bath (1 litre) so facilitates wetting by the bath and consequent mercerising effect that the cotton may be introduced dry without any preliminary steeping. Certain wood-tar fractions of high b.p. have a similar effect. D. F. T.

**Prep. of bleach liquors.**—See VII. **Textile-aid industry.**—See XII. **Colouring rubber.** **Rubber-impregnated fabrics.**—See XIV.

#### PATENTS.

**Dyeing materials made of or containing cellulose acetate in black shades.** SILVER SPRINGS BLEACHING & DYEING Co., LTD., F. E. MASON, and T. G. ALLEN (B.P. 386,117, 5.1.22).—The method of dyeing aniline-black described in B.P. 337,746 (B., 1931, 198) is modified, whereby absorption of  $\text{NH}_2\text{Ph}$  by acetate silk previous to oxidation is effected by exposing the silk to the vapour of  $\text{NH}_2\text{Ph}$ . A. J. H.

**Production of ornamental effects on materials made of or containing cellulose esters or ethers.**

BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 387,343, 28.7.31).—Broken-pattern effects are produced by printing a pattern with a dye having no affinity for the cellulose ester or ether, and over-printing with a swelling agent (Et lactate); the pattern is coloured only where the swelling agent has been applied. Examples of suitable dyes are Formyl-violet 5BN, Chromazurine E, Cupranil-brown B, Alkali-blues, Metanil-yellow, Tartrazine, Chloramine-black E2B, Modern-black N, etc. C. H.

**Textile treating [desizing] process and composition.** H. C. GORE, S. JÓZSA, and C. N. FREY, ASSRS. to STANDARD BRANDS, INC. (U.S.P. 1,859,084, 17.5.32. Appl., 11.1.30).—Desized fabric having improved wettability is obtained by adding to the usual desizing bath, which contains a starch-liquefying enzyme, an emulsion of oleic acid prepared with substances (e.g., naphthalenesulphonic acids, pine oil, Hexanol, Tetralin, and  $\text{C}_2\text{HCl}_3$ ) which do not adversely affect the enzyme. A. J. H.

**Undressing [desizing] of textiles.** RÖHM & HAAS A.-G. (B.P. 386,912, 22.8.32. Ger., 31.8.31).—Starch sizes are removed from textile materials by treatment at  $35\text{--}40^\circ$  with a starch-liquefying enzyme the action of which is accelerated by the addition of an aq. extract of yeast. A disinfectant (PhMe) may be added to the bath. A. J. H.

**Production of silk improved in weight, durability, and appearance.** F. W. WEBER, ASSR. to MAYWOOD CHEM. WORKS (U.S.P. 1,858,474 and 1,859,188, 17.5.32. Appl., [A] 2.2.27, [B] 25.1.28).—(A) Silk is weighted with  $\text{SnCl}_4$  and  $\text{Na}_2\text{HPO}_4$  in the usual manner and then further weighted with acetates of the rare-earth metals (free from Th) and  $\text{Na}_2\text{HPO}_4$ ; the preliminary Sn weighting protects the silk from deterioration by the rare-earth metal phosphates. (B) Silk is impregnated with a mixture of  $\text{SnCl}_4$  and a Zn salt (e.g.,  $\text{ZnCl}_2$  or  $\text{ZnSO}_4$ ), then squeezed, and treated with dil. aq.  $\text{Na}_2\text{HPO}_4$ ; citric acid is preferably added to the Sn-Zn salt solution in order to keep any Ca, Mg, and Fe impurities in solution. A. J. H.

**Art of coating [pile fabric with rubber compositions].** A. W. DROBILE, ASSR. to COLLINS & AIKMAN CORP. (U.S.P. 1,855,387, 26.4.32. Appl., 20.9.29).—The fabric is impregnated from the back without penetration through to the face, for the purpose of "anchoring" the pile threads, with a latex compound reinforced by dispersion therein of coagulated rubber which has previously been masticated with a protective colloid, e.g., casein, rosin soap, and saponin. A. J. H.

**Impregnation of fabrics with soaps [for waterproofing].** CHEM. U. SEIFENFABR. R. BAUMHEIER A.-G. (B.P. 386,700, 6.8.31. Ger., 6.8.30).—An impregnating liquor is used consisting of an emulsion of a fatty acid or resin soap of a multivalent metal (e.g., Al, Zn, Pb, Mg) together with a waterproofing substance (e.g., paraffin wax, bitumen) and an emulsifying agent (e.g., glue, starch); the liquor is stabilised by passage through a homogenising machine. A. J. H.

**Lubrication [for reducing electrification] of textile fibres.** G. LECOMTE, ASSR. to DU PONT RAYON Co. (U.S.P., 1,856,713, 3.5.32. Appl., 27.4.28. Fr.,

24.12.27).—Sizes containing emulsified, oxidised, semi-drying vegetable oils of the cottonseed or rapeseed type are applied, particularly to acetate silk yarns.

A. J. H.

**Purifying liquids.**—See I. **Wetting etc. agents.**  
See III. **Impregnating [fibrous material].**—See XIII. **Treating effluent waters.**—See XXIII.

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

**Installation and manufacturing costs of sulphuric acid.** R. MORITZ (Chim. et Ind., 1933, 29, 278—283).—The Petersen tower process and a process comprising the use of tall chambers with bottom communication are compared. With the latter, employing hot burners, and concentrating before the Glover tower, and even without intensive working, the cost is much lower. Acid of  $d$  1.843, obtained by this process, is not appreciably dearer than acid of  $d$  1.712 by the Petersen process. The tall chambers can utilise gases of low  $\text{SO}_2$  content. The process is also cheaper than the contact process, although the latter retains its utility for the production of oleum. W. J. W.

**Detergency of alkaline salt solutions. III. Deflocculating and emulsifying power.** F. D. SNELL (Ind. Eng. Chem., 1933, 25, 162—165; cf. B., 1932, 1118).—Emulsifying and deflocculating power were measured by sedimentation of burnt umber impregnated with oil. Materials with colloidal properties— $\text{Na}$  oleate,  $\text{Na}_2\text{SiO}_3$ , and  $\text{Na}_2\text{PO}_4$ —are better deflocculating agents than non-colloidal substances, irrespective of  $p_{\text{H}}$ . The emulsifying power of builder and soap are additive, and independent of  $p_{\text{H}}$ . Below  $p_{\text{H}}$  10.2,  $\text{Na}_2\text{HPO}_4$  has emulsifying powers, but is not advantageous in presence of soap on account of the low  $[\text{OH}^-]$ . J. S. A.

**Preparation of ammonium sulphate from gypsum by the action of ammonium carbonate solution.** I. S. TELETOV and A. D. VELESCHINETZ [with S. M. GUREVITSCH, G. I. DMITRENKO, and I. A. ROGATSCHEVA] (Ukrain. Chem. J., 1932, 7, [Sci.], 141—168).—Suspensions of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (I) in aq.  $(\text{NH}_4)_2\text{CO}_3$  (II) at  $40^\circ$  give 94% yields of  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{CaCO}_3$ . (I) should be ground to pass through a sieve of 1600—4900 meshes per sq. cm.; no advantage is gained by finer subdivision. (I) and (II) should be present in theoretical proportions; the most convenient concn. of (II) is that corresponding to 13—14%  $\text{NH}_3$ . The addition of  $(\text{NH}_4)_2\text{SO}_4$  to the reaction mixture increases the yield by 2—3%. The yields obtained from (I) dehydrated at  $200^\circ$  are about 5% < from the dihydrate. R. T.

**Decantation and filtration, with subsequent washing of sludge, in the preparation of ammonium sulphate from Artemov gypsum.** P. Z. KOVAL [with I. Z. JASNOGORODSKI and Z. L. ALEXANDROVITSCH] (Ukrain. Chem. J., 1932, 7, [Sci.], 169—177).—Suspensions of  $\text{CaCO}_3$  in aq.  $(\text{NH}_4)_2\text{SO}_4$  (cf. preceding abstract) are more conveniently separated by filtration than by decantation. R. T.

**Preliminary technical-economic estimates for production of ammonium sulphate from gypsum.** G. I. GORSCHTEIN and N. A. VISCHNIEVSKI (Ukrain.

Chem. J., 1932, 7, [Tech.], 156—182).—Teletov and Veleschinetz's method (cf. *supra*) involves a capital outlay 25% < that for production from  $\text{NH}_3$  and  $\text{H}_2\text{SO}_4$ , and the cost of production is 12—20% lower.

R. T.

**Purification of brine by ammoniation.** W. C. HSIEH, E. O. WILSON, and T. P. HOU (Ind. Eng. Chem., 1933, 25, 165—167).— $\text{Ca}$ ,  $\text{Mg}$ , and oxides of  $\text{Fe}$  and  $\text{Al}$  are removed from sea-brine by ammoniation. Some loss of  $\text{NaCl}$  and  $\text{Na}_2\text{CO}_3$  occurs, the ppt. containing  $\text{MgCO}_3$ ,  $\text{Na}_2\text{CO}_3$ , and  $\text{NaCl}$  in equimol. ratio, possibly as triple salt. The optimum concn. of  $\text{NH}_3$  is 20—40 g./litre; settling of the ppt. occurs best at  $> 55^\circ$ . J. S. A.

**B.p., viscosity, density, and vapour pressure of brines obtained by a mixed process of salt separation of carnallites.** A. P. PALKIN, M. A. OPUHTINA, N. P. IUNSKAYA, E. N. ORLOVA, and B. N. FEDCHENKO (Kali, Russia, 1932, No. 5—6, 38—41).—Properties of solutions containing  $\text{MgCl}_2$ ,  $\text{KCl}$ , and  $\text{NaCl}$ , obtained in Giprom's method of separation, are recorded.

CH. ABS.

**Dissolving carnallite through a well.** Y. M. KHEIFETZ (Kali, Russia, 1932, No. 7, 33—35).

**Dissolving carnallite deposits through a well boring by Preobrazhenski's method.** E. I. AHUMOV and B. B. VASILIEV (Kali, Russia, 1932, No. 5—6, 27—37).

**Dehydration of carnallite and magnesium chloride.** I. G. SCHTSCHERBAKOV (Kali, Russia, 1932, No. 7, 36—37).—Dehydration to 2.5%  $\text{H}_2\text{O}$  and 3%  $\text{MgO}$  is described.  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  may be dried at  $400^\circ$  and chlorinated in presence of  $\text{C}$ ; the product contains 0.5—1.0%  $\text{MgO}$  and 0.5—2.0%  $\text{C}$ . CH. ABS.

**Graphic calculations in carnallite technology.** Y. E. VILNYANSKI and Z. S. BANNUIH (Kali, Russia, 1932, No. 5—6, 42—49).—Equilibria in the system  $\text{MgCl}_2$ — $\text{KCl}$ — $\text{NaCl}$ — $\text{H}_2\text{O}$  are expressed graphically.

CH. ABS.

**Preparation of bleach liquors from liquid chlorine and [quick]lime.** O. WURZ (Papier-Fabr., 1933, 31, 49—53, 62—64).—Technical details are given. The quicklime should contain  $\geq 0.2\%$   $\text{Fe}_2\text{O}_3$ ; if  $\text{Mn}$  is present the liquor is red, but this is harmless. During chlorination a rise of temp. to  $42^\circ$  does not increase the loss of  $\text{Cl}$  as  $\text{Ca}(\text{ClO}_3)_2$  or  $\text{CaCl}_2$  if the liquor is alkaline (as is always necessary). For an escape of  $\text{Cl}_2$  the best spray is aq.  $\text{Na}_2\text{CO}_3$ . A. G.

**Tricalcium phosphate as a caking inhibitor in salt and sugar.** H. V. MOSS, T. W. SCHILB, and W. G. WARNING (Ind. Eng. Chem., 1933, 25, 142—147).—The efficiency of  $\text{Ca}_3(\text{PO}_4)_2$  (I) as a caking inhibitor was found to depend on the ratio  $\text{CaO} : \text{P}_2\text{O}_5$ , the content of  $\text{Fe}$  and  $\text{Al}$  phosphates, the degree of hydration, and particle size.  $\text{CaHPO}_4$  is poor as a caking inhibitor, the best  $\text{CaO} : \text{P}_2\text{O}_5$  ratio being approx. 3.4 : 1. Addition of 3—11%  $\text{AlPO}_4$  improved the efficiency, as did reduction of particle size. The most efficient samples showed an ignition loss of 6—12%. (I) was the most effective filler tested, basic  $\text{MgCO}_3$  ranking next. 0.5% of (I) completely inhibited caking in sugar and conferred free-flowing properties. Sugars containing (I) are more suited for icing than starch-filled sugars. J. S. A.

**Preliminary technical-economic estimates for production of superphosphate and phosphorite meal from various Ukrainian phosphorus-containing minerals.** G. I. GORSCHTEIN, L. M. CHAIT, N. A. VISCHNIEVSKI, and A. I. DROFAN (Ukrain. Chem. J., 1932, 7, [Tech.], 130—155).—The production of superphosphate from low-grade phosphorites cannot compete with that from apatite concentrates. R. T.

**Preparation of superphosphate from Schtschigrov phosphorite and from its mixtures with Podolian phosphorite or with apatite concentrates.** S. P. FARASCHJAN and R. M. TSEKINOVSKA (Ukrain. Chem. J., 1932, 7, [Sci.], 135—140).—Superphosphate containing 15%  $H_2O$  and 10.3—11% assimilable  $P_2O_5$  (decomp. coeff. 90—95%) can be prepared from Schtschigrov phosphorite (I), containing 17.75%  $P_2O_5$ . The viscosity of the mixture of (I) with  $H_2SO_4$  is, however, too high for factory practice. This difficulty can be overcome by using mixtures with Podolian phosphorite or with apatite concentrate (50—25%). R. T.

**Manufacture of superphosphate from mixtures of high-grade Podolian phosphorites or Chibin apatite concentrates and Ukrainian low-grade phosphorites.** I. S. TELETOV, S. P. FARASCHJAN, and (in part) F. V. NAJMAN (Ukrain. Chem. J., 1932, 7, [Sci.], 113—134).—Kroleviets and Izjum phosphorites cannot be used alone for the manufacture of superphosphate, owing to the low assimilable P content of the product; that obtained from mixtures of the above with Podolian phosphorite or with Chibin apatite concentrate is, however, quite satisfactory. R. T.

**Manufacture of superphosphate from Kroleviets phosphorites and from their mixtures with Podolian phosphorites.** L. M. CHAIT (Ukrain. Chem. J., 1932, 7, [Tech.], 115—129).—The findings of Teletov and co-workers (cf. preceding abstract) are confirmed for production on a factory scale. R. T.

**Preparation of compounds of carbon and nitrogen with metallic elements.** A. PERRET (Bull. Soc. Ind. Mulhouse, 1933, 99, 10—20).—Cyanamides may be produced by the reaction  $XCO_3 + 2NH_3 \rightleftharpoons XCN_2 + 3H_2O$  if the velocity of the  $NH_3$  stream is sufficiently high to remove the  $H_2O$  ( $X = Ca, Sr, \text{ or } Ba$ ). Small yields are obtained below  $750^\circ$  and the reaction is not possible with  $MgCO_3$ . The following reactions have also been investigated: (1)  $Ca \text{ carbamate} \rightarrow CaCN_2 + CO_2 + 2H_2O$ , (2)  $Ca(NCO)_2 \rightarrow CaCN_2 + CO_2$ , (3)  $Ca \text{ cyanurate} \rightarrow 3CaCN_2 + 3CO_2$ , (4)  $CaO + 2NH_3 + CO \rightarrow CaCN_2 + 2H_2O + H_2$ . It is improbable that any of these methods can replace the usual process. E. S. H.

**Copper sulphate from Ural copper ores and from copper waste.** II. Decomposition of copper sands with sulphuric acid. III. Treatment of waste from dust-settling chambers. I. G. SCHTSCHERBAKOV and A. K. RASPOPINA (Tzvet. Met., 1930, 1342—1347, 1486—1490; cf. B., 1931, 1047).—II. 6%  $H_2SO_4$  extracted 60% of (0.2—4.5%) Cu in 10 min.; after 1 hr. the residue contained  $> 0.2\%$  of the original Cu.

III. The waste contains 0.5—6.0% Cu (46—73% sol. in  $H_2O$ ; 14—31% sol. in dil.  $H_2SO_4$ ). The dust is treated with  $H_2SO_4$ , or with  $SO_2$  at  $> 600^\circ$ . CH. ABS.

**Specific gravity of fuller's earth.** K. YAMAMOTO and H. IWASAKI (Rep. Soc. Appl. Chem. Waseda, 1932, 17, 1—8).—The earth when dried at  $120^\circ$  has  $d$  2.4; heating at  $200^\circ$  gives a max. val., structural change apparently taking place at  $600$ — $800^\circ$ . The  $d$  is higher after alkali- than after acid-treatment; colloidal  $SiO_2 \cdot xH_2O$  is present. A. A. E.

**Influence of the conditions of preparation on the activity of a nickel-manganese-aluminium catalyst.** F. FISCHER and K. MEYER (Brennstoff-Chem., 1933, 14, 47—50).—The effect of varying the washing conditions, the pptg. agent, the supporting material, and the method of pptn. has been studied. A catalyst of optimum activity was obtained with the method of prep. previously described (B., 1931, 748). An equally active catalyst was obtained from commercial Ni, Al, pyrolusite, and  $K_2CO_3$  as from the pure materials. A. B. M.

**Determination of carbon dioxide in air.** Y. KAUKO (Suomen Kem., 1932, 5, [B], 54).—For const. concn. of basic ions, the total concn. of  $CO_2$ ,  $H_2CO_3$ ,  $HCO_3'$ , and  $CO_3''$  in the system base- $CO_2$ - $H_2O$  is, over a considerable range, almost independent of the  $p_H$ . When the  $CO_2$  partial pressure is altered equilibrium is attained very rapidly, and as a consequence the  $CO_2$  content of air may be determined potentiometrically or colorimetrically ( $\pm 0.0003\%$ ) within about 2 min. H. F. G.

**Solid carbon dioxide from flue gas by application of the Joule-Thomson effect.** F. E. E. GERMANN (Ind. Eng. Chem., 1933, 25, 150—152).—In the usual  $CO_2$  absorption process for prep. of solid  $CO_2$  the escaping gas still contains 8—11%  $CO_2$ , an original gas of at least 17%  $CO_2$  being required. A new method is designed to use flue gases containing 10%  $CO_2$ . It depends on the retardation of condensation by divergence from Henry's law at high pressures, whereby solid  $CO_2$  may be obtained from a highly compressed gas mixture by simultaneous reduction of temp. and pressure. A semi-commercial plant has been erected, using a pressure of 1700—3000 lb. per sq. in. and a temp. of  $-50^\circ$ , the original compression eliminating practically all  $H_2O$  vapour. The gas mixture is expanded to 10 atm. with cooling to  $-104^\circ$ , by which means 90% of the  $CO_2$  is condensed as solid. Using natural gas as fuel, the production cost is estimated at \$25 per ton. C. I.

**Absorptive capacity of luxmasse, bog-iron ore, mixtures thereof, and other iron oxides, for hydrogen sulphide.** MAINZ and MÜHLENDYCK (Brennstoff-Chem., 1933, 14, 50—54).—Coke-oven gas containing 8—10 g./cu. m. of  $H_2S$  was passed through the absorptive material contained in 3 towers (8 cm. diam.; 45 cm. high) until the  $H_2S$  content of the issuing gas rose to 0.05 wt.-%. Luxmasse absorbed 10—15 times as much  $H_2S$  as did bog-Fe ore. A 1:1 mixture of the two containing 25% of  $H_2O$  gave an absorption of 61% (calc. on the dry material) at  $20$ — $34^\circ$  and with a gas velocity of 3—5 mm./sec.; with higher or lower  $H_2O$  contents, at higher temp., or with higher gas velocities, the absorption fell off. Caking of the mass was dependent on its  $H_2O$  content and temp. The absorptive capacity of flue dust for  $H_2S$ , even after preheating in air, was too low, and its resistance to the passage of the gas

too high (on account of its low porosity), for it to be suitable for this purpose. A. B. M.

**Oxidation of hydrogen sulphide solutions by air.** M. KAPP (Bull. Soc. d'Encour., 1932, 131, 649—654).—In a continuously-operating industrial form of apparatus for use in connexion with the removal of  $H_2S$  from gas,  $NiSO_4$  solution trickles into a scrubber through which pass a current of air and the  $H_2S$  solution from the washer. The S and  $NiS$  produced are filtered off, and the liquor passes through a layer of  $BaCO_3$ , to ppt.  $H_2SO_4$ , and a second filter; it then enters a settling tank, where fine solids deposit and tar etc. rise to the surface, and is finally used for washing further gas. Cost data are given. For the rapid determination of  $H_2S$  in industrial liquors, 500 c.c. of the liquor are added to an excess of 20%  $CdSO_4$  solution (to prevent oxidation), and the whole is boiled for 10 min. after addition of  $H_2SO_4$ . The evolved gas is passed into standard I solution, which is titrated with aq.  $Na_2S_2O_3$ . H. F. G.

**Determining S and halogens in fuels etc. [ $H_2$  by] action of water vapour on  $CH_4$ .**—See II. Accumulator acid. Electrolysing thiosulphate solutions.—See XI. Fertiliser.—See XVI. Potash from molasses.—See XVII. Liquid  $O_2$  explosives.—See XXII. Detecting CO traces in air.—See XXIII.

See also A., Mar., 224, Permeability of Pd to  $H_2$ . Prussian-blue hydrosols. Prep. of colloids. Conc. hydroxide sols. 225, Measuring concn. and dispersion of suspensions [in salt solutions]. 230, Electrolysis of aq. NaI. 233, Kinetics of formation of  $H_2SO_4$ . Absorption of  $CO_2$  by aq.  $Na_2CO_3$ . 234, Oxidation of sulphites. Production of  $H_2$  by the water-gas reaction. Activation of  $H_2$ . 235,  $NH_3$  oxidation by Pt catalysts. Electrolysis of dry liquid  $NH_3$ . 238, Pure radon. 239, Prep. of metallic cyanamides. Drying agent from  $Al_2(SO_4)_3$ . 240, Separation of Ce from other Ce earths. "Blue acid." Properties of  $O_3$ . Hydrolysis and synthesis of nitrosylsulphuric acid. 242, Conductimetric analysis of acids. Detection and determination of halogens. 243, Determining HCN in air etc. 244, Spectral analysis of Ru in carnallite, and of salt mixtures. 248, Determining  $SO_2$  in air. 312, Prep. of Bi salts of fatty acids.

#### PATENTS.

**Construction of lead chambers for manufacture of sulphuric acid.** R. MORITZ (B.P. 387,737, 3.9.31. Fr., 3.9.30).—Chambers of  $\sphericalangle$  15—25 m. in height,  $\sphericalangle$  5—25 m. in width, and  $\sphericalangle$  2.5—5 m. in depth, and having area of walls in sq. m. : vol. in cu. m.  $\sphericalangle$  1.2 : 1, communicate with each other at the bottom through narrow passages which extend the whole width of the chambers and through which the acid and gases flow in opposite directions. Ducts constructed, e.g., of bricks are provided in the passages to divide up the gas stream, and in the chambers to cause the gases to ascend at the centre and to descend near the walls. L. A. C.

**Regeneration of waste sulphuric acid [from oil refining].** H. FRISCHER (B.P. 387,569, 8.6.32. Ger., 25.2.32).—The acid is conc. in the presence of  $HNO_3$  or

higher oxides of N<sub>2</sub> and the NO evolved is converted back into  $HNO_3$  etc. for re-use. L. A. C.

**Production of pure hydrogen fluoride.** I. G. FARBENIND. A.-G. (B.P. 387,614, 31.8.32. Ger., 2.9.31).—Crude HF, obtained, e.g., by the action of  $H_2SO_4$  on fluorspar, is passed through cold  $H_2SO_4$  or oleum and pure HF is expelled by heating the acid at 60—100°. L. A. C.

**Production of pure liquid ammonia from ammonia liquor.** R. ZANIBONI (B.P. 387,596, 22.7.32. It., 28.7.31).—The  $NH_3$  liquor is distilled in a CaO column and the distillate passed through a dephlegmator into an  $NH_3$  absorbent, e.g.,  $H_2O$ ,  $NH_4CNS$ ,  $NH_4CN$ ,  $NH_4NO_3$ ,  $NH_4I$ , I, which is cooled during the absorption and then heated in a closed condenser system so that the  $NH_3$  gas liquefies under the pressure generated. Absorption and liquefaction may be repeated. L. A. C.

**Treatment of basic phosphate [Thomas] slags.** HOESCH-KÖLN NEUESSEN A.-G. F. BERGBAU U. HÜTTENBETRIEB (B.P. 387,439, 12.11.31. Ger., 12.11.30).—The slag is treated with sufficient acid or acid salt, preferably  $HNO_3$ , to dissolve so much CaO that  $H_2SiO_3$  just begins to pass into solution and other constituents remain undissolved. The  $Ca(NO_3)_2$  solution obtained is, e.g., treated with  $NH_3$  and  $CO_2$ , with  $NH_4HCO_3$ , or with  $(NH_4)_2SO_4$ . L. A. C.

**Treatment of bauxite, clay, and intermediate aluminium-bearing ores.** L. G. JENNESS, Assr. to INTERMETAL CORP. (U.S.P. 1,858,272, 17.5.32. Appl., 9.8.29).—Bauxite or clay is calcined at 900° to remove  $H_2O$ , then treated with  $S_2Cl_2$  at 400—450° to volatilise  $FeCl_3$  and  $TiCl_4$ . The Al is then sublimed as  $AlCl_3$  by treatment with  $S_2Cl_2$  at 600—700°. A. R. P.

**Caustic silicate [from kaolin, for gas masks etc.].** P. O. ROCKWELL (U.S.P. 1,857,402, 10.5.32. Appl., 5.4.30).—Granular kaolin or pumice is heated for 10—20 min. in 20—90% aq. NaOH at 100—160°, removed from the liquor, drained, and heated  $> 300^\circ$  without previous washing, to obtain a granular product having a high adsorptive power for  $H_2O$  and acid vapours. A. R. P.

**Economic recovery of sulphur from smelter smoke.** A. M. THOMSEN (U.S.P. 1,857,414, 10.5.32. Appl., 18.4.27).— $SO_2$  gases from roasting furnaces are passed with air and steam over hot NaCl to form  $Na_2SO_4$  with the evolution of HCl which is dissolved in  $H_2O$ . The  $Na_2SO_4$  is converted into  $Na_2CO_3$  and CaS by the Leblanc process, and the CaS treated with the HCl liquor to produce  $H_2S$ , which is then burned with a limited supply of air to obtain S and  $H_2O$ . A. R. P.

$TiO_2$ .—See XIII. Fertilisers.—See XVI.

#### VIII.—GLASS; CERAMICS.

**Using urtite in glass-making.** V. V. VARGIN and N. B. ZHUK (Keram. i Steklo, 1932, 8, No. 8, 6—9).—Urtite (nephelite 85, apatite 5, dark-coloured minerals 10%; alkali 17—21%;  $Al_2O_3$ :alkali 1.43) from Murman gives glasses which melt more rapidly, devitrify less readily, are less viscous, and more easily refined than nephelite glasses. CH. ABS.

**Density of some soda-lime-silica glasses as a function of composition.** F. W. GLAZE, J. C. YOUNG,

and A. N. FINN (Bur. Stand. J. Res., 1932, 9, 799—805).—With the aid of the diagram given it is possible to predict the density of a glass from its composition, and the compositions of glasses of equal density.

C. W. G.

**Influence of the characteristics and treatment of the raw material on the properties of fused-silica products, and effect of treatment of the fused products on their properties.** B. MOORE (Trans. Ceram. Soc., 1933, 32, 45—63).—The influence is described of the composition, physical structure, and treatment of the raw materials on the properties of fused-SiO<sub>2</sub> products (formation of pinholes and spots, softening and devitrification tendencies, fluorescence, transparency, dielectric strength, transmission capacity for light radiation, and production of strain). The effect on fused-SiO<sub>2</sub> products of rate of cooling, change of temp., glazing, and prolonged contact with common materials of chemical industry is also described.

A. L. R.

**Clays of south-east England.** H. G. DINES (Trans. Ceram. Soc., 1933, 32, 64—90).—Their geological formations are described, with diagrams; the clays are classified and described, and their relative importance as raw materials for bricks is indicated.

A. L. R.

**Determination of plasticity of clays, kaolins, and other plastic materials.** P. SCHABLIKIN and K. GALAHITSKAJA (Sprechsaal Keram., 1932, 65, 675—676; Chem. Zentr., 1932, ii, 2707).—A method employing a few drops of a suspension and glass plates separated by a measurable distance is employed.

A. A. E.

**Refractory plugs and spouts for ingot moulds in iron metallurgy.** P. P. BUDNIKOV and S. P. LOGINOV (Domez, 1932, No. 6, 30—35).—The following mixtures were used: (1) 25% refractory plastic clay (38—40% Al<sub>2</sub>O<sub>3</sub>), 25% chamotte (38—39% Al<sub>2</sub>O<sub>3</sub>), and 50% Ceylon graphite (all < 1 mm.); (2) 35% refractory plastic clay (38—39.5% Al<sub>2</sub>O<sub>3</sub>, < 1 mm.) and 65% chamotte (< 0.5 mm.). Tests are recorded.

CH. ABS.

**Fireclay from tripolites.**—See IX.

See also A., Mar., 229, Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> refractories. 235 and 240, Katô kaolin. 251, Kaolin. Shales and bricks of Georgia. 252, North Carolina slags and shales.

## PATENTS.

**Heat-absorbing glasses.** JENAER GLASWERK SCHOTT & GEN. (B.P. 387,778, 9.11.31. Ger., 13.11.30).—The glasses comprise < 0.2% FeO, < 25% P<sub>2</sub>O<sub>5</sub> and/or B<sub>2</sub>O<sub>3</sub>, and < 10% Al<sub>2</sub>O<sub>3</sub>, and contain less SiO<sub>2</sub> than P<sub>2</sub>O<sub>5</sub>. P compounds with reducing properties, e.g., Fe<sub>3</sub>P, are added to the batch or to the molten glass.

L. A. C.

**Optical glasses.** JENAER GLASWERK SCHOTT & GEN. (B.P. 388,118, 29.10.31. Ger., 5.11.30).—Glass having  $n_D > 1.60$  comprises > 20% of TiO<sub>2</sub> and < 35% of oxides of bi- and ter-valent elements (BaO, SrO, CaO, MgO, ZnO, B<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>), including < 10% of alkaline-earth oxides.

L. A. C.

**WC refractory.**—See X.

## IX.—BUILDING MATERIALS.

**Ukrainian tripolites in the cement and fireclay industries.** P. P. BUDNIKOV and V. V. BELOVODSKI (Ukrain. Chem. J., 1932, 7, [Tech.], 103—112).—Kutejnikovsk tripolite (75—85% SiO<sub>2</sub>, 4—12% Al<sub>2</sub>O<sub>3</sub>, 2—6% Fe<sub>2</sub>O<sub>3</sub>, 0—0.13% MgO) can be used for the production of cement and fireclay.

R. T.

**Cements and their physico-chemical behaviour as luting materials in petroleum bore-holes.** A. TINNES (Petroleum, 1933, 29, No. 1, 1—11).—The prep., constitution, and properties of the various hydraulic cements are critically discussed. Special attention is paid to waterproofing and to the destructive action of acids, alkalis, salt solutions, sea-H<sub>2</sub>O, oils, etc. A mixture of "Siccifax" cement (blast-furnace slag cement + bituminous material) and micro-asbestos is strongly recommended in preference to Portland cement.

J. A. S.

**Cements and well cementing: effect of chlorides on the setting and hardening of cement.** A. REID and J. T. EVANS (J. Inst. Petroleum Tech., 1932, 18, 992—1006).—The presence of NaCl either in the mixing-H<sub>2</sub>O or in the medium under which the cement sets slightly increases the rate of hardening during the initial stages, but this is succeeded by a marked retrogression in the tensile strength of the cement, which is more evident if chlorides are present from the commencement, i.e., in the mixing-H<sub>2</sub>O. The rate of hardening at temp. up to 60°, even with 40% gauge grout, is not sufficiently rapid to cause trouble in cementing wells.

H. S. G.

**Concrete pavement failures due to high-lime cement.** I. PAUL (Eng. News-Rec., 1933, 110, 212—213).—Failures in New York have been traced to high-CaO cements in which the CaO has been increased progressively to meet the requirements of high strength. The problem of increasing the life of concrete roads can be solved by reducing the content of CaO so that the cement contains < 40% of 3CaO.SiO<sub>2</sub>.

C. A. K.

**Physical properties of mixtures of bitumen and finely-divided mineral matter.** A. EVANS (J. Inst. Petroleum Tech., 1932, 18, 957—991).—It is shown that the m.p., hardness, and tensile strength are raised and the ductility is decreased by increase in the following factors, two or more of which may operate simultaneously: proportion of filler, hardness of the bitumen, fineness of the filler, and bitumen absorption of the filler. Chemical composition does not appear to be a factor, but the structure of the filler is important. The ductility of a bitumen is no guide to the ductility of an asphaltic cement made therefrom.

H. S. G.

**Wt. and vol. of finely-divided materials.**—See I. Clays [for brick-making].—See VIII. Determining swelling [of wood].—See XIV. Ply-wood industry. See XV.

See also A., Mar., 239, Hydrated Ca aluminate.

## PATENTS.

**Improving the properties, more especially the strength and imperviousness to water, of cement mortar, plaster, and concrete.** K. WINKLER & Co.

G.M.B.H. (B.P. 387,429, 24.10.31. Ger., 24.10.30).— $H_2O$ -sol. salts of Al, Fe, or Cr are added to a cement mix. C. A. K.

**Composition for bituminous roads.** J. RADCLIFFE (B.P. 387,324, 26.5.31).—The whole or part of a bituminous binder (4–12%) is incorporated in a porous friable absorbent, *e.g.*, coke, which is then mixed with the aggregate to facilitate manipulation when cold. The binder is released by crushing pressure on the coke. C. A. K.

**Preservation of wood, straw, and the like.** R. FALCK (B.P. 387,819, 13.1.32).—A non-leachable preservative consists of an aq. colloidal solution of  $As_2S_3$  prepared by passing  $H_2S$  into an aq. solution containing 1–2%  $As_2O_3$ , 1–5% sugar, and 0.3%  $NH_3$  solution (25%  $NH_3$ ); for certain purposes the amount of  $H_2S$  passed is controlled so as to leave excess of  $As_2O_3$  in the solution. F. R. E.

**Putty masses. Impregnating [cellular material].**—See XIII.

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

**Graphical method of calculating blast-furnace charge.** N. A. KOSTULEV (Domez, 1932, No. 7, 1–18).—The Mathesius method is criticised. The charge is preferably determined by a method commencing with the calculation of the slag of required composition. CH. ABS.

**Refined pig irons: their influence on the production of high-duty iron castings.** J. E. HURST (Metallurgia, 1933, 7, 115–116).—The use of refined and alloy pig irons for such castings is reviewed. The properties of the refined irons and of mixtures with roll scrap are compared with vals. for cold-blast Fe from various sources. E. H. B.

**Corrosion of weldings of soft steel.** F. MEUNIER (Compt. rend., 1933, 196, 271–273).—The resilience of the joint between plates of Thomas steel united autogenously with plates of open-hearth steel is compared with the potential in 0.1N-KCl relative to the HgCl electrode, and also with the content in O and N of the joining metal. Resilience decreases markedly with increased content of O; increase of N seems to have a similar but less marked effect (*cf.* B., 1928, 674). There is little relation with the potential, though to some extent a high potential accompanies low resilience. C. A. S.

**Production of ferro-alloys.** S. S. STEINBERG and P. S. KUSAKIN (Tzvet. Met., 1930, 1471–1480).—Molybdenite (87.5%  $MoS_2$ ) was smelted with Fe filings, coke, and CaO, alloys containing Mo 80, C 5–6, and > 0.10% S being obtained. Fe–Cr alloys containing  $\nabla$  0.5% C were obtained from crude Fe–Cr and magnetite or chromite. Fe–V alloys were obtained from Fe–V ore ( $V_2O_5$  48,  $Fe_2O_3$  26%) and  $(NH_4)_3VO_4$ , using C and Fe–Si as reducing agents; the alloys contained V 36.93–44, Si 1.18–1.23, C 0.48–1.80%. CH. ABS.

**Production of chromium-tungsten steel in the basic open-hearth furnace.** A. SHEVCHENKO (Domez, 1932, No. 6, 47–49). CH. ABS.

**Dressing Minussinsk copper ore.** B. A. DAVIDOVICH (Tzvet. Met., 1931, 461–476).—Glafrin and Yulia ores contain, respectively, Cu 4.15, 2.8; S 5.19, 3.60;  $SiO_2$  26.10, 29.96; CaO 8.86, 29.44;  $Fe_2O_3$  12.48, 7.60;  $Al_2O_3$  11.61, 7.44;  $CO_2$  1.52, 11.92%; Ag 20 g. per ton, trace; traces in each case of Zn, Pb, As, Sb, and Au. Flotation concentrates contained 21–25.97% Cu (90.36–97.99% of original Cu content), and 1.62–2 g. Au and 89–149.8 g. Ag per ton. CH. ABS.

**Flotation of copper pyrites from the Kalatinsk mine.** S. I. MITROFANOV (Tzvet. Met., 1930, 1362–1368).—Flotation of the ore (Cu 1.9, Fe 44, S 47%) gives a concentrate containing 18% Cu, equiv. to 85% extraction. In smelting, only 75% of the Cu is extracted. CH. ABS.

**Concentration of Djezkazgan copper oxide ores.** V. A. NAUMOV (Tzvet. Met., 1930, 992–1010).—The ore contains Cu 14.65,  $SiO_2$  66.21,  $Fe_2O_3$  2.62,  $Al_2O_3$  1.73, MgO 0.80, CaO 0.37,  $CO_2$  13.62%; it should be finely ground (85% < 200-mesh) in a medium of liquid: solid = 1.5 : 2 in presence of basic resin and Na silicate. Flotation is carried out in several stages. A 95% extraction, with 36–37% Cu in the concentrate and 1–1.4% in the tailings, can thus be effected. CH. ABS.

**Flotation of copper oxide ores.** B. I. ROZOV and S. D. SUKHOVLSKAYA (Tzvet. Met., 1930, 208–213).—Malachite is treated with  $Na_2S$  solution before flotation. CH. ABS.

**Flotation of oxidised copper ores from Koktas-Djartas and Koktas-Djal.** K. A. SIMONOV (Tzvet. Met., 1931, 331–336).—The ores contain, respectively, Cu 2.74, 4.07; Fe 1.96, 1.68; S 0.0015, 0.029;  $SiO_2$  59.44, 70.22; CaO 0.6, 0.82%; Ag 33 g., 19 g.; Au < 0.5 g., < 2 g. per ton. Max. concn. of Cu (84.5%) was obtained by flotation with ore ground to 100-mesh. CH. ABS.

**Flotation of Kounrad oxidised copper ore.** S. N. ZUBAREV (Tzvet. Met., 1931, 322–331).—The ore (Cu 1.91,  $SiO_2$  74.15, S 0.14, CaO 0.34, MgO 0.64, MnO 0.55,  $Fe_2O_3$  1.62,  $Al_2O_3$  13.95,  $CO_2$  2.95%, Ag 11 g. per ton, As and Au traces) was successfully conc. by flotation. CH. ABS.

**Khalilov oxidised copper ore.** A. S. SLADKOV (Tzvet. Met., 1931, 1202–1205).—On flotation, about 50% of the Cu in the ore (1% Cu) remained in the tailings. CH. ABS.

**Flotation of Sibaeusk copper-iron ore.** L. M. ALEXEEV and B. I. ROZOV (Tzvet. Met., 1930, 1672–1690).—The deposits contain (average) Cu 2.09, Fe 22.4%, Ag 21 g., Au 1 g. per ton of ore. Flotation is recommended. CH. ABS.

**Separation of copper from slags by flotation.** S. I. MITROFANOV (Tzvet. Met., 1930, 714–718).—58–80% of the Cu, present as Cu,  $CuFeS_2$ , and  $Cu_2S$ , in the slag ( $SiO_2$  20–22, Fe 50,  $Al_2O_3$  2, Zn 2, S 2.5, Cu 2%) was recovered at  $pH$  2–8. CH. ABS.

**Determination of frothing properties in flotation reagents.** S. A. KUZIN (Tzvet. Met., 1930, 1481–1485).—A method and apparatus are described. CH. ABS.

**Hydrometallurgical treatment of Perm copper sandstones.** N. P. ASEEV, K. F. BELOGLAZOV, and N. S. GREIVER (Tzvet. Met., 1930, 167—208).—The solubility of chrysocolla in dil.  $H_2SO_4$  is practically equal to that of malachite. The petrographic nature, structure of separate grains and composition of the ore were studied. By the action of 10—12%  $H_2SO_4$  (7 pts. : 1 pt. Cu) on the Cu mineral ground to < 6 mm., 90% of the Cu is extracted. The smaller is the grain, the greater is the acid consumption, which varies also with the progress of lixiviation. CH. ABS.

**Calculation of minimum copper content in matte required in a plant of a given capacity.** S. N. BARABOSHKIN (Tzvet. Met., 1931, 1065—1071).

**Influence of oxygen on properties of copper.** W. BRONIEWSKI and S. JASLAN (Compt. rend., 1933, 196, 174—177).—Up to 8% of  $Cu_2O$  was melted with electrolytic Cu in vac. and annealed at 450° for 1 hr. Curves relating %  $Cu_2O$  to various properties show no influence on e.m.f. of dissolution or thermoelectric power, but the temp. coeff. of the latter factor and the elastic limit respectively decrease and increase to 0.8%  $Cu_2O$ , and then remain const. Electric conductivity decreases slightly, whilst its temp. coeff. rises to a low max. for the  $Cu_2O$ -Cu eutectic, and then declines slowly; hardness and tensile strength show similar but more marked maxima, whilst striction and elongation on rupture decrease very rapidly. These facts indicate a max. solubility of  $Cu_2O$  of 0.8%, and a eutectic containing 4—5% (cf. A., 1906, ii, 356; 1930, 161; B., 1923, 980A). C. A. S.

**Treatment of thick copper boiler-plate and red merchant copper.** V. G. SERDYUKOV (Tzvet. Met., 1930, 516—530).—To avoid cracks caused by excess of oxide, a sample from the furnace is first subjected to a bending test. Brittleness is also caused (> by oxides) by absorption of reducing gases; hence before rolling, ingots should be heated in a slightly oxidising atm. CH. ABS.

**Testing the tinning of rubber-insulated copper wire.** A. R. MATTHIS (Kautschuk, 1933, 9, 4—8).—A piece of wire of 4 sq. dm. surface is coiled and the exposed Cu ends are waxed; it is then rotated with 50 c.c. of aq.  $NH_3$  (d 0.91), in a horizontal, wide-necked bottle closed with a cork covered with Sn foil, for 5 min. The dissolved Cu is then determined. Judged by a comparison of the details and results with those of other methods, the new test has distinct advantages. D. F. T.

**Notes on the copper-rich alloys.** H. C. ANSTAY (Metallurgia, 1933, 7, 117—118).—Recent developments in Cu alloys are reviewed, consideration being given to the effects of small additions of Sn, Ag, Al, Be, etc. on their mechanical and electrical properties. E. H. B.

**Concentration of Turlansk lead ore.** V. I. TRUSHLEVICH and K. A. SIMONOV (Tzvet. Met., 1931, 173—203).—The ore contains Pb 16.94 (chiefly cerussite), Fe 32.46,  $Al_2O_3$  3.63,  $SiO_2$  8.45, Ca 5.43, Mg 0.69, Zn 3.58, S 0.49%, Au 0.6 g. and Ag 36 g. per ton. Conc. by one-step flotation is preferred; concentrates containing 56.0% Pb (93.1% of original Pb content)

were obtained. Attempted concn. of Zn in the tailings was unsuccessful. CH. ABS.

**Concentration of Turlansk oxidised lead ore on a Wilfley table.** V. A. VANYUKOV and V. Y. KOROLYUK (Tzvet. Met., 1930, 1690—1695).—The ore contains (average) 18% Pb. The concentrates contained 35.8—38.2% Pb (55.0—75.8% of Pb content). CH. ABS.

**Influence of impurities on properties of lead.** I. Introduction. J. N. GREENWOOD. II. (A) Purification of lead by electrolysis. (B) Comparison of the crystallisation of commercial and electrolytic leads. (C) Photomicrography of lead. R. S. RUSSELL (Proc. Austral. Inst. Min. Met., 1932, No. 87, 135—143, 145—152, 152—161, 162—166).—I. The intercryst. corrosion of Pb is dependent on the grain size and the presence of internal stress below the elastic limit. Grain size depends on the impurities present and on the previous mechanical and thermal history of the specimen. The tendency of a Pb to undergo intercryst. corrosion may be ascertained by immersing the specimen for some days in a solution containing 400 g. of  $Pb(OAc)_2$  and 100 c.c. of  $HNO_3$  (d 1.16) per litre and then bending the metal through 10°.

II (A). Commercial Port Pirie Pb containing Cu 0.0002, Bi 0.0023, Cd 0.0002, Sb 0.0040, Fe 0.0007, Zn 0.0005, S 0.0003, and Ag 0.0003% was further refined by electrolysis in 5% aq.  $Pb(ClO_4)_2$  containing 5%  $HClO_4$  and 0.05% peptone; the cathode Pb contained Cu 0.0001, Bi 0.0004, Sb 0.00011, Fe 0.00036, Zn 0.00023, and Ni 0.00002%. (B) Samples of these two leads (A and B, respectively) and of a specially fire-refined Pb (C) containing Cu 0.0002, Bi 0.0009, Cd 0.0001, Sb 0.0004, Fe 0.0002, Zn 0.00023, and Ag 0.00055% were rolled to varying degrees of reduction, annealed at 18°, 50°, 75° and 175° for varying periods, and examined for grain growth. (B) recrystallised completely in 1½ hr. at 18° after 5% reduction, whereas (A) and (C) were only partly recrystallised in 24 hr. after 20% reduction. These differences are ascribed to the small differences in the composition noted above. (C) The crystal structure of Pb is best developed by etching with a 7.5% solution of  $(NH_4)_2MoO_4$  in 3N- $HNO_3$  for 10 sec. A. R. P.

**Testing the purity of high-grade lead.** R. S. RUSSELL (Proc. Austral. Inst. Min. Met., 1932, No. 87, 167—174).—Spectrographic analysis of electrolytic Pb with 0.002% of impurities revealed the presence only of Fe, whereas chemical analysis showed traces (< 0.0005%) of Cu, Bi, Sb, Ni, and Zn. In fire-refined Pb containing 0.008% of impurities the spectrograph revealed the presence of all found chemically. Sb can be determined satisfactorily by the turbidimetric method as  $Sb_2S_3$ , and Bi colorimetrically with KI. No good rapid methods for traces of Zn or Fe appear to exist. A. R. P.

**Corrosion of chemical lead.** P. F. THOMPSON (Proc. Austral. Inst. Min. Met., 1932, No. 87, 193—206).—Determinations of the potential of Pb sheet in 63%  $H_2SO_4$  indicated that the high negative potential frequently observed either is due to contamination with Fe with which the Pb comes in contact during casting

and working, or is derived from segregation of the minute quantities of Fe in the metal along the grain boundaries. It is suggested that this Fe is the cause of failures of chemical Pb by corrosion, and not the Sb content.

A. R. P.

**Removal of copper from Ridder crude lead.** M. P. VERKHOVITZEV (Tzvet. Met., 1930, 90—101).—By heating the crude Pb with S or metallic sulphides, preferably PbS, the Cu was reduced from 3.11—6.52 to 0.054—0.070%, whilst 92% of the Au and 90% of the Ag were retained in the Pb.

CH. ABS.

**Rust preventers.** S. SALM (Allgem. Oel- u. Fett-Ztg., 1933, 30, 87—88).—A brief review.

E. L.

**Quantitative emission-spectral analysis. IV. Determination of zinc in pure aluminium and aluminium alloys.** J. CLERMONT (Z. anal. Chem., 1932, 90, 321—330; cf. B., 1932, 1083).—A table of homologous pairs of lines for the analysis of Al containing Zn is given. The limit of detectability, using an arc spectrum, is 0.02% Zn. Spectrographic determinations of Si and Fe in Al (about 0.1% each) were mostly within  $\pm 10\%$  of the analytical figures.

F. L. U.

**Tests for aluminium foil from the viewpoint of its suitability as a wrapping material for cut bread.** K. SEIDEL (Z. Unters. Lebensm., 1933, 65, 104—106).—Solubilities in 100 c.c. of 0.25N- and 0.5N-AcOH, 0.25N- and 0.5N-lactic acid, 0.25N-H<sub>3</sub>PO<sub>4</sub>, and an aq. extract of pumpernickel are recorded after 3 days at 20°. A satisfactory specimen (100 mg.) lost 0, 10, 2.8, 4.5, 135.3, and 0 mg./100 sq. cm., respectively.

J. G.

**Fluidity and pourability of very light alloys.** L. LOSANA (Alluminio, 1932, 1, 237—243; Chem. Zentr., 1932, ii, 2867).—Measurement of the consts. is described; determinations were made with Mg-rich alloys. Zn affects the pourability of Mg more than does Al.

A. A. E.

**CuSO<sub>4</sub> from Cu ores.**—See VII. **Refractory plugs etc. for ingot moulds.**—See VIII. **Electrolysing Ag-bearing solutions.**—See XI. **Rust-preventing paints.**—See XIII. **Tinned Pb tubes for tooth-pastes.**—See XX.

See also A., Mar., 217, **Magnetic susceptibility of Fe-Si.** 218, **Permeability of Pd to H<sub>2</sub>.** Na<sub>4</sub>Pb. Pb alloys, Au-Mn. 219, **Pt-Cu, Pt-Ni, Al<sub>2</sub>Zn<sub>3</sub>, Mg-Zn.** 220, **Cu-Sn, Ag-Zn, Cr-C, Fe-Si-P.** **Structure of ternary hyper- and hypo-eutectics.** 230, **Cd-Ag.** 233, **Austenite-pearlite transformation.** 235, **Electrodeposition of Zn.** **Electrochemical behaviour of Pt.** 239, **Sublimation of Mg.** 242, **Analysis by electrolysis and spectrography.** 244, **Spectral determination of Au in ores.** 248, **Diffusion of light and corrosion of metals.**

## PATENTS.

**Elongated tilting hearth furnace.** H. MASCHMEYER (U.S.P. 1,856,716, 3.5.32. Appl., 24.10.30. Ger., 7.11.29).—The furnace has the shape of a horizontal truncated cone lined with refractory to give an elliptical cross-section to the melting chamber. Melting is effected by means of a blast burner introduced at the plane of the major axis of the ellipse.

A. R. P.

**Silicothermic smelting process and charge therefor.** W. C. READ, Assr. to ELECTRO METALLURG. Co. (U.S.P. 1,856,592, 3.5.32. Appl., 2.9.30).—For the prep. of ferro-alloys containing Mo and/or W a mixture of Si with MnO<sub>2</sub> as primer, Fe<sub>2</sub>O<sub>3</sub>, and MoO<sub>3</sub>, or WO<sub>3</sub>, or salts thereof, is heated until a self-propagating reaction occurs with the production of a ferro-alloy low in Mn and Si. Ferrosilicon may replace Si and Fe<sub>2</sub>O<sub>3</sub>.

A. R. P.

**Production of sintered alloys.** R. R. WALTER (U.S.P. 1,856,607, 3.5.32. Appl., 4.12.30. Ger., 4.4.30).—The powdered metals are mixed with 1—2% H<sub>2</sub>O and a small quantity of a vegetable drying oil, e.g., soyabean oil, tung oil, pressed into shape, allowed to remain exposed to the atm. until the oil hardens, and then sintered in the usual way.

A. R. P.

**Reduction of [iron] oxide ores.** L., F. C., and F. E. KERN (U.S.P. 1,858,274, 17.5.32. Appl., 22.4.29).—A plastic mixture of Fe ore, bituminous coal, and dil. HCl or aq. chlorides is formed into briquettes, which are dried and heated gently to expel volatile matter and then more strongly to reduce the Fe<sub>2</sub>O<sub>3</sub> to metal. The product is crushed and subjected to magnetic separation to obtain Fe sponge free from S and P.

A. R. P.

**Preparation of metal [iron] sheets for annealing.** L. C. DREFAHL, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,856,751, 3.5.32. Appl., 17.3.30).—Sticking together of Fe sheets during pack-annealing is prevented by immersing the sheets, after pickling in dil. acid, in dil. aq. Na<sub>2</sub>SO<sub>4</sub>, NaCl, Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>HPO<sub>4</sub>, or Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, and annealing without washing or drying.

A. R. P.

**Manufacture of malleable iron.** E. L. IVES, Assr. to H. A. BRASSERT & Co. (U.S.P. 1,858,548, 17.5.32. Appl., 24.1.29).—Pig Fe is smelted with Fe ore in a cupola up which are passed the hot products of combustion from burning oil or fine coal in a series of combustion chambers disposed around the cupola above the tuyères.

A. R. P.

**Metal [cast-iron] roll.** W. H. SEAMAN (U.S.P. 1,857,825, 10.5.32. Appl., 27.6.27).—The metal contains C 3—3.75, Mn 0.35—0.5, S 0.05—0.08, Ni 0.2—1, Si 1—1.5, and Cr 0.75—2% and has a structure comprising a central core of grey or mottled cast Fe surrounded by a zone of secondary white Fe and a thin outer skin of close-grained white Fe.

A. R. P.

**Manufacture of corrosion-resistant metal [iron or steel] bodies such as plates, rods, or tubes.** PLYKROME CORP. (B.P. 387,525, 12.3.32. U.S., 3.4.31).—Mild-steel sheet is covered with a thin sheet of soft Fe low in C and Si, then with a protective sheet of Cr-Fe or Cr-Ni-Fe alloy, and the whole is welded, hammered, or forged into a uniform composite sheet of the desired thickness.

A. R. P.

**Coating surfaces of iron and steel.** W. M. PHILLIPS and G. M. COLE, Assrs. to GEN. MOTORS CORP. (U.S.P. 1,856,261, 3.5.32. Appl., 26.1.29).—The article is made the cathode in a boiling solution containing Zn(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> and 0.05—0.13% of free H<sub>3</sub>PO<sub>4</sub>, with or without NaH<sub>2</sub>PO<sub>4</sub>, until a dense black coating is produced.

A. R. P.



**Production of phosphate coatings on metals [iron].** METAL FINISHING RESEARCH CORP. (B.P. 386,739, 2.11.31. U.S., 7.7.31).—The coating bath comprises a solution containing  $Zn(H_2PO_4)_2$ ,  $Fe(H_2PO_4)_2$ ,  $CuSO_4$ , and a small quantity of a mild oxidising agent, e.g.,  $NaNO_3$ , which has no action on the  $Fe^{II}$  salt. A. R. P.

**Magnetic [iron-nickel] alloys.** W. S. SMITH, H. J. GARNETT, W. F. RANDALL, TELEGRAPH CONSTRUCTION & MAINTENANCE CO., LTD., and DEUTS.-ATLANTISCHE TELEGRAPHENGES. (B.P. 386,682, 22.7.31. Addn. to B.P. 357,947; B., 1932, 67).—Small quantities of one or more of the elements Cr, Mo, Mn, W, Cu, Al, Si, V, Co are added to the alloys previously claimed. The following alloys are specifically mentioned: (a) Ni 76.9, Fe 17.8, Mn 0.7, Mo 3, and Ag 1.5%; (b) Ni 77.2, Fe 17.8, Ag 0.9, Mn 0.8, and Mo 3%; and (c) Ni 78.5, Fe 18, Mo 3, Mn 1, Si 2, and Be 1.2%. The heat-treatment comprises annealing for 1 hr. at  $1000^\circ$ , slow cooling (4 hr.) to  $600^\circ$ , quenching, and annealing at  $375^\circ$  for 5 hr. A. R. P.

**Production of [iron-nickel alloy] magnetic materials.** (A) C. P. BEATH and (A, B) H. M. E. HEINCKE, ASSRS. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,859,067 and 1,859,087, 17.5.32. Appl., [A, B] 29.10.27. Renewed [B] 20.11.31).—Oxidised Fe-Ni alloys with < 25 (20)% Ni are rolled at  $1325^\circ$  to obtain a fine-grained structure and when the temp. falls to just above the brittle range the alloy is (A) allowed to cool normally, or (B) quenched, to obtain a product which can readily be comminuted to yield a finely-cryst. powder suitable for the manufacture of dust cores. A. R. P.

**Zirconium high-speed steel.** Y. KAMISHIMA (B.P. 387,386, 5.8.31).—Addition of 1–10% Zr to high-speed steel containing W and Cr as the chief alloying constituents is claimed to prevent loss of hardness when the alloy is used at high temp. A. R. P.

**Preparation and purification of [iron-silicon] alloys.** P. H. BRACE, ASSR. to WESTINGHOUSE ELECTRIC & MANUFG. CO. (U.S.P. 1,858,386, 17.5.32. Appl., 7.11.29).—Steel is melted in a high-frequency induction furnace under a layer of  $SiO_2$  while a current of  $H_2$  is passed over the surface of the metal to remove C, S, and P, and to reduce the  $SiO_2$  to Si which alloys with the Fe. A. R. P.

**Hack-saw blade [of chromium-tungsten steel].** H. M. GERMAN, ASSR. to UNIVERSAL STEEL CO. (U.S.P. 1,858,705, 17.5.32. Appl., 11.7.29).—The steel contains 3–12 (7–8)% Cr, 2–12 (3–8)% W, and  $\geq 3$  (0.2–0.5)% V, and the sheets from which the blades are cut are heat-treated to produce an even distribution of hard nodular grains of cementite in the structure. A. R. P.

**Manufacture of metallic [iron-tungsten-cobalt] alloys.** C. P. MILLER (B.P. 387,322, 21.4.31).—The alloys contain Fe with 20–35 (30)% Co, 15–30% Mo and/or W (23% W), 0.5–3 (1.5)% V, 0.2–0.6 (0.3)% Mn, and < 0.3% C, Si, and other impurities together. [Stat. ref.] A. R. P.

**Hard metal [tungsten carbide] compositions.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of R. PALMER (B.P. 387,427, 8.10.31. U.S., 8.10.30).—A mixture of WC and thick ammoniacal starch paste is pressed into shape and heated to obtain a porous

sintered mass which is then immersed in molten Cu or Ag until the pores are filled. The process may be used for making welding electrodes, burnishing implements, bearings, or electrical contacts. [Stat. ref.] A. R. P.

**Manufacture of shapes from homogeneous [tungsten carbide] alloys of great hardness.** E. SCHNEBEL, ASSR. to F. KRUPP A.-G. (U.S.P. 1,855,994, 26.4.32. Appl., 19.7.29. Ger., 17.7.25).—W powder is melted with 10% of TiC in a graphite crucible in  $H_2$  and cast into  $H_2O$ -cooled Cu moulds. The brittle ingot is crushed to a fine powder which is pressed into shape, sintered at  $1750^\circ$  in  $H_2$ , and cooled rapidly to  $1500^\circ$  and then very slowly to room temp. A. R. P.

**[Tungsten carbide] refractory compositions.** C. A. LAISE, ASSR. to CALLITE PRODUCTS CO., INC. (U.S.P. 1,858,244 and 1,858,300, 17.5.32. Appl., [A] 14.5.30, [B] 13.5.30).—Claim is made for sintered products comprising an intimate mixture of (A) 60–85% WC, 2.5–15% SiC, 0.1–1% BN, and 1–30% Co, and (B) 60–90% WC, 2–25%  $Al_2O_3$ , 0.1–1% BN, and 5–30% Co, Ni, or Fe. A. R. P.

**Alloy containing zirconium and tungsten for the principal constituent.** Y. KAMISHIMA (B.P. 387,699, 7.8.31).—The alloy consists of W with 20–90% Zr. Up to 20% of one or more of the metals Fe, Al, Mn may be added as binder, and in addition 0.5–10% Ta may be included to increase the hardness. A. R. P.

**Manufacture of alloy for tools and working appliances.** BAYERISCHE METALLWERKE A.-G. (B.P. 387,684, 7.5.31. Ger., 7.5.30).—Claim is made for a sintered alloy comprising an intimate mixture of 20–79.5% of at least two carbides of the group Si, Zr, Ti, Ce, Th, at least one metal of the Cr, Mo, W group, and/or at least one metal of the group Fe, Co, Ni, Mn. The carbides are preferably pressed into the desired shape, which is then impregnated with the metal or metals of the other groups. A. R. P.

**Reducing and converting metals [e.g., chromium] and making alloys.** P. A. E. ARMSTRONG (U.S.P. 1,855,176, 26.4.32. Appl., 29.7.27).—In the manufacture of Ni-Cr alloys or Cr steels the Ni or Fe is melted under a slag and the Cr introduced by adding a mixture of  $CrF_3$ , CaO, and a reducing agent, e.g., Al or Si. The process is also applicable to the prep. of alloys of V, U, Ta, Ti, or Nb. A. R. P.

**Recovery of [heavy metals from] oxidised ores [by flotation].** S. A. FALCONER and L. J. CHRISTMANN, ASSRS. to AMER. CYANAMID CO. (U.S.P. 1,858,007, 10.5.32. Appl., 1.6.31).—A mixture of Na mercaptobenzthiazole and  $Na_2S$  or NaHS is used as collector. A. R. P.

**Treatment of gold ores containing cyanicides [e.g., telluride ores].** W. P. WILLIAMS. From LEEWILLS CO., LTD. (B.P. 387,548, 22.4.32).—Telluride Au ores are treated by the flotation process to obtain a telluride concentrate, which is agitated with 10–100 lb. of CaO per ton and aerated until the cyanicide is destroyed. Both concentrate and tailing are then cyanided in the usual way. A. R. P.

**Recovery of copper [from scrap].** H. M. BURKEY and L. E. COLE, ASSR. to AMER. METAL CO., LTD. (U.S.P.

1,858,236, 17.5.32. Appl., 19.8.30).—The material is blown with fluxes in a converter to obtain blister Cu and the rich Cu slag produced is run through a bed of incandescent coke to reduce its Cu content, both slag and reduced Cu being returned to the converter with fresh quantities of scrap. A. R. P.

**Improving the electrical conductivity of copper.** METALLGES. A.-G. (B.P. 387,516, 25.2.32. Ger., 27.2.31).—The molten metal is deoxidised with 0.005–0.1% Li, the amount used being sufficient to leave 0.002–0.005% Li in the cast metal. A. R. P.

**Manufacture of copper powder.** C. F. SHERWOOD, Assr. to MORAINÉ PRODUCTS Co. (U.S.P. 1,856,661, 3.5.32. Appl., 2.6.27).—Cu is pptd. by means of Fe from aq.  $\text{CuSO}_4$ , the ppt. is washed and dried in a non-oxidising atm., and the powder rendered denser by grinding in a ball mill, preferably with Sn powder, graphite, and salicylic acid, to obtain a mixture from which porous bearings may be pressed up. A. R. P.

**[Manganese-nickel-copper] alloy.** N. B. PILLING, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,858,415, 17.5.32. Appl., 23.9.24).—Claim is made for an alloy containing Cu 15–55, Ni 47–22, and Mn > 20%, having a negative temp. coeff. of resistance >  $1 \times 10^{-4}$  per °C. The alloy with Cu 24, Ni 36, and Mn 40% has a coeff. of  $-0.0032$ . A. R. P.

**Hydrometallurgical treatment of [lead-zinc] ores.** U. C. TANTON (U.S.P. 1,858,944, 17.5.32. Appl., 20.8.24).—Oxidised or roasted Pb or Zn ores are leached with brine and the solution electrolysed with insol. anodes to yield Pb or Zn and  $\text{Cl}_2$ ; the gas is converted into  $\text{CaCl}_2$  for use in subsequent leaching operations to prevent accumulation of  $\text{SO}_4^{--}$  in the leach liquor. A. R. P.

**[Tin-zinc] alloy.** H. S. REAM, JUN., and F. J. HEIN, Assrs. to SHENANGO-PENN MOLD Co. (U.S.P. 1,859,169, 17.5.32. Appl., 5.5.31).—An alloy of Ni 26, Cu 13, Fe 1, Zn 30, and Sn 30% is claimed; it is produced by adding the Zn and Sn to the necessary amount of molten Monel metal. Addition of 1% of the alloy to the ordinary Zn galvanising bath reduces the amount of Zn taken up by the Fe. A. R. P.

**[Cadmium-zinc alloy] coating material for metal articles.** R. AUERBACH, Assr. to W. STEINHORST (U.S.P. 1,855,377, 26.4.32. Appl., 5.4.29. Ger., 7.4.28).—An alloy of Cd 30% and Zn 70% is used for the hot-galvanising of Fe and steel articles. A. R. P.

**Apparatus [kettle] for use in refining lead bullion and similar operations.** G. K. WILLIAMS (U.S.P. 1,856,678, 3.5.32. Appl., 9.9.30. Austral., 7.11.29).—The kettle consists of a slightly tapering steel vessel (A) with hemispherical bottom surmounted by a truncated conical section (B) between the flange of which and that of A is a ring of channel cross-section having its flanges directed towards the inside of A and welded at their inner ends to the flanges of A and B, respectively. At the upper end of B is an inflow launder (C) provided with a downwardly projecting baffle by means of which a submerged inlet opening is formed. An outflow pipe (D) extends from the bottom of A to a point in B on the opposite side of, but at the same level as, C, where it

terminates in a horizontal portion. The temp. of B is maintained well above the m.p. of Zn, while that at the lowest point of A is kept at just above the temp. of the Pb-Zn eutectic. Pb and Zn are fed continuously into C and pass through a molten layer of Zn-Ag alloy in B whilst desilverised Pb flows continuously from D.

A. R. P.

**Recovering certain metals [indium and gallium] of the third periodic group.** F. G. McCUTCHEON, Assr. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,855,455, 26.4.32. Appl., 25.3.26. Renewed 10.10.31).—The Pb residues obtained from the redistillation of leady Zn are melted with In so that the resulting alloy contains < 5% In. The alloy is granulated and heated with conc.  $\text{H}_2\text{SO}_4$  until the Pb falls to a powder without being converted into  $\text{PbSO}_4$  while the remaining metals are converted into sulphates, which are extracted with  $\text{H}_2\text{O}$ . The solution is treated with  $\text{H}_2\text{S}$  to ppt. Ge and heavy metals and the In and Zn are recovered by electrolysis, Zn being deposited almost completely before the In. The electrolyte is then treated with an excess of NaOH, filtered, and electrolysed for Ga. A. R. P.

**Refining and purification of metals [nickel-silver].** G. F. DORAN (U.S.P. 1,857,947, 10.5.32. Appl., 14.5.31).—The molten metal or alloy is deoxidised with a mixture of  $\text{KMnO}_4$  and excess of C. A. R. P.

**Heat-treatment of strong aluminium alloys.** N. V. HYBINETTE, Assr. to NICALUMIN Co. (U.S.P. 1,858,092, 10.5.32. Appl., 10.12.29).—The strength of alloys with > 95% Al and their resistance to intercryst. corrosion are considerably improved by carrying out the ageing treatment in several stages at gradually rising temp. from 20° to 150°. A. R. P.

**Production of alloys of beryllium and aluminium.** E. BÄGGLI and E. BURGER (B.P. 387,849, 4.10.32).—Beryl is heated with  $\text{Na}_2\text{SiF}_6$  at 750° and the product leached with 10% HCl. The filtered solution is treated with aq.  $\text{NH}_3$  and the ppt. of  $\text{Be}(\text{OH})_2$  and  $\text{Al}(\text{OH})_3$  is collected, washed, and dissolved in HF. The solution is evaporated to obtain a mixture of fluorides and oxyfluorides of Al and Be which are electrolysed in fused NaF-BaF<sub>2</sub> electrolyte at 1200° to give a Be-Al alloy containing about 25% Al. A. R. P.

**Corrosion-proof aluminium alloys.** A. STRASSER and W. GERMANN (B.P. 387,562, 17.5.32).—Claim is made for an Al alloy containing 0.5–5.8 (2)% Ti, 0.5% Mn, and 1–6 (3)% of an intermediate alloy containing 98–98.5% Pb and 2–1.5% Na. The Pb is stated to keep the Ti in solid solution. A. R. P.

**Corrosion-resistant age-hardenable aluminium composite metals.** DÜRENER METALLWERKE A.-G., and K. L. MEISSNER (B.P. 387,615, 31.8.32. Ger., 12.9.31).—Claim is made for a composite metal comprising a high-Cu-Al alloy coated on both sides with a similar alloy low in Cu, both alloys age-hardening under similar thermal treatment. *E.g.*, the base alloy (A) may contain 4–4.5% Cu, 0.5–0.7% Mg, and 0.3–0.6% Mn, and the coating alloy 0.5–3% Cu with the same Mn and Mg as A. A. R. P.

**Surface treatment [colouring] of aluminium or aluminium alloys.** R. S. DUNHAM, Assee. of M.

TOSTERUD (B.P. 387,806, 17.12.31. U.S., 17.12.30).—Oxide coatings on Al produced by chemical or electrolytic means are impregnated with a mordant, *e.g.*, K Sb tartrate,  $\text{Na}_2\text{SiO}_3$ , tannic or phosphotungstic acid, and then immersed in a solution of a basic dye until the desired depth of colour is obtained. A. R. P.

**Decreasing shrinkage in aluminium-bronze castings.** A. PACZ (U.S.P. 1,838,632, 29.12.31. Appl., 3.11.28. Ger., 3.12.27).—Addition of 1.5–2.5% Si, 1–3% Fe, and < 1% Mn to Al bronze (3–9% Al) reduces the shrinkage on casting. The Fe is added after the Al and is previously mixed with a flux containing F and an alkali metal, *e.g.*,  $\text{Na}_2\text{SiF}_6$ . A. R. P.

**Aluminium-magnesium alloys.** R. S. ARCHER and L. W. KEMPF (U.S.P. 1,856,615, 3.5.32. Appl., 21.5.30).—An alloy of Al with 10–16 (10%) Mg and 0.2–1.5% Mn, having tensile strength < 60,000 lb. per sq. in. and Brinell hardness 90, is claimed. A. R. P.

(A) Electrolytic production of iron. (B, C) Treatment of iron ores, scrap iron, or other ferruginous substances. VEREIN. STAHLWERKE A.-G. (B.P. 386,790 and Addn. B.P. 387,265 and 387,629, [A] 12.1.32, [B] 29.8.32, [C] 29.9.32. Ger., [A] 19.1.31, [B] 16.1.32, [C] 15.1.32).—The ore or scrap is treated with  $\text{Cl}_2$  at 300° to produce liquid  $\text{FeCl}_3$ , which is (A) electrolysed, or (C) treated, with  $\text{H}_2$  to obtain metallic Fe. (B) In the electrolytic process the anode is made of a compressed mixture of finely-divided Fe ore and graphite which continuously supplies  $\text{FeCl}_3$  to the electrolyte. A. R. P.

**Electrolytic recovery of copper from alloys.** T. LEWIN (B.P. 386,896, 22.7.32).—In the electrolytic refining of scrap Cu alloys to obtain pure Cu, the anode slime is roasted and used for the purification of the electrolyte from suspended and dissolved oxides of Fe, Sn, Sb, etc., steam being passed into the mixture during the operation. A. R. P.

[Maintenance of] chromium-plating [baths]. C. H. HUMPHRIES, Assr. to METALS PROTECTION CORP. (U.S.P. 1,857,547, 10.5.32. Appl., 25.11.27).—The bath is regenerated by addition of commercial  $\text{CrO}_3$  and the  $\text{SO}_4^{2-}$  content is maintained within the desired limits by addition of  $\text{BaCrO}_4$  from time to time. A. R. P.

**Manufacture of thin solid insulating films [on metals], having a high dielectric strength.** A. A. SAMUEL (B.P. 387,437, 9.11.31. Fr., 8.11.30).—Films are deposited electrolytically on anodes of Al, Mg, Ta, and their alloys from electrolyte comprising a very weak acid and a very weak base or org. substance containing OH groups which are not easily dissociated. Thus, *e.g.*, a film is deposited on Al from electrolyte containing  $\text{H}_2\text{O}$ ,  $\text{H}_3\text{BO}_3$ , and triethanolamine (1:1:1). Alternatively, rosin (3 pts.) and triethanolamine (1 pt.) may be used. J. S. G. T.

**Pb chambers for  $\text{H}_2\text{SO}_4$ . S from smelter smoke.**—See VII.

## XI.—ELECTROTECHNICS.

**Electrolysis of silver-bearing thiosulphate solutions.** K. HICKMAN, W. WEYERTS, and O. E. GOEHLER (Ind. Eng. Chem., 1933, 25, 202–212).—Such electrolysis results in production of  $\text{Ag}_2\text{S}$  except under con-

trolled conditions. Agitation and acidity are necessary and the permissible c.d. varies directly with the Ag content. Although a little gelatin (derived, *e.g.*, from the photographic film) is beneficial, much interferes with plating. The use of promoters (cf. B.P. 364,711; B., 1932, 431) for plating in presence of gelatin is described, such compounds yielding a colloid Ag complex which is reduced to Ag by melting. In certain cases a promoter is formed in the bath by interaction of  $\text{Na}_2\text{S}_4\text{O}_6$  and gelatin. The gelatin in these solutions migrates to the cathode and is deposited in particles of a size to interfere with the building of the Ag crystal lattice. The promoter maintains electric continuity through the colloid layer.  $\text{Ag}_2\text{S}$  is always formed in traces.  $\text{Na}_2\text{SO}_3$  is essential to the process. It prevents the acidic decomp. of  $\text{Na}_2\text{S}_2\text{O}_3$ , destroys  $\text{Na}_2\text{S}_2\text{O}_4$ , which is injurious, and redissolves  $\text{Ag}_2\text{S}$ . Suitable cells are constructed of ebonite-lined metal with pipe-lines of rubber. Agitation is either by air-stirring, rotating electrodes, or by paddles between the electrodes. The electrical efficiency varies inversely with the c.d., and the deposit contains 97–98% Ag. The products of electrolysis in solution are  $\text{Na}_2\text{SO}_4$  and thionates. C. I.

**Cadmium electrode for storage-battery testing.** W. J. HOLMES and R. ELLIOTT (Trans. Electrochem. Soc., 1932, 62, 159–174).—Auxiliary Cd electrodes of various sizes and shapes were placed in various positions in the electrolyte of a Pb accumulator and the p.d. between such electrodes and the positive and negative plates of the accumulator measured by means of a voltmeter of 241 ohms internal resistance. Errors due to concn. polarisation at the Cd are much greater when connected to the positive plate, but in any case are minimised by using a Cd electrode of large surface. The readings of p.d. are somewhat affected by the position of the Cd electrode owing to inclusion of some part of the internal-resistance drop between the plates. A hollow conical electrode cast from Cd of ordinary purity and immersed for a few days in accumulator acid before use is recommended. Cd of higher purity leads to greater polarisation errors. For investigating causes of loss of capacity of an accumulator, readings of p.d. between Cd and each of the plates should be taken at intervals during continuous discharge. Under the conditions recommended the method is accurate enough for all ordinary testing purposes, and the readings are not appreciably affected by the presence of reasonable amounts of Fe or Cu in the electrolyte. H. J. T. E.

**Determination of organic matter in accumulator sulphuric acid solutions.** R. G. MYERS (Philippine J. Sci., 1932, 49, 581–585; cf. B., 1931, 391).—The acid (7 g.) is treated with  $\text{Pb}(\text{OAc})_2$  solution and EtOH, with gentle boiling followed by vigorous agitation. After decantation, the ppt. is washed twice on a centrifuge. The solution is then freed from Pb with  $\text{H}_2\text{S}$  and evaporated to dryness. The residue is treated with 3 c.c. of boiling  $\text{H}_2\text{O}$  and sufficient  $\text{Ba}(\text{OH})_2$  to remove Fe, evaporated to dryness, heated to const. wt. at 105°, and ignited. Results varied from 0.075% to 0.088% and were independent of the age of the acid. C. I.

**Electrical conductivity of commercial dielectrics and its variation with temperature.** J. D. CLARK

and J. W. WILLIAMS (J. Physical Chem., 1933, 37, 119—131).—The conductivities of 6 synthetic materials and 4 kinds of wood have been determined over a range of temp. The sp. vol. conductivity,  $\mu$ , varies with abs. temp.,  $T$ , according to the equation  $\mu = ke^{-E/RT}$ , where  $E$  is the energy of liberation of the carrier of the current in g.-cal., and  $k$  is a const. having the same dimensions as  $\mu$ .  
E. S. H.

Resistance of wool fibres.—See V. Purifying Pb. Metallic films.—See X. Insulating varnishes. Accumulator jars.—See XIII. Electrodepositing rubber.—See XIV. Determining P in soil extracts. Field electrometer.—See XVI. Determining ash in beet sugar.—See XVII. Electrodialysis of amylase solutions.—See XVIII.

See also A., Mar., 211, Photo-electric polarimetry. 217, Magnetic susceptibility of Fe-Si. 225, Measuring suspensions [in salt solutions]. 230, Electrolysis of aq. NaI. 235, Electrolysis of dry liquid  $\text{NH}_3$ . Electrodeposition of Zn. Electrochemical behaviour of Pt. 242, Analysis by electrolysis and spectrography. 249, Electric precipitators for air analysis. 261, Prep. of aldonic acids.

## PATENTS

[Self-baking] electrodes for electric furnaces. NORSKE A./S. FOR ELEKTROKEM. IND. (B.P. 387,585, 2.7.32. Norw., 7.7.31).—Supporting members ( $A$ ) project into the electrode casing ( $B$ ) so that they are baked into the electrode mass, and electrode suspension gear is attached to  $A$  outside  $B$ .  
J. S. G. T.

Insulating [dielectric] materials. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS-GES. (B.P. 387,744, 14.9.31. Ger., 15.9.30).—A mixture of chloronaphthalenes ( $I$ ) and a small proportion of org. substances of high mol. wt. and sol. or colloidal dispersible in ( $I$ ), e.g., rubber, balata, polymerised fatty oils, cellulose derivatives, or resins, is claimed.  
J. S. G. T.

Insulation of magnetic bodies [e.g., permalloy dust]. H. LATHROP, ASSR. TO BELL TELEPHONE LABS., INC. (U.S.P. 1,857,201, 10.5.32. Appl., 2.7.31).—The dust is mixed with one-third of the desired amount of insulating material, e.g., talc and/or kaolin, and with a solution of  $\text{Cr}(\text{OAc})_3$  and  $\text{Na}_2\text{SiO}_3$  to form a paste, which is dried; a second one-third of the talc and/or kaolin is then added and the same process repeated, and finally the last third is added and the drying repeated.  
A. R. P.

Electrostatic dust precipitation. STURTEVANT ENG. CO., LTD., and H. W. WAGNER (B.P. 387,452, 25.11.31).—Passage of pptd. dust into the stream of gas under treatment is prevented by arranging the gas streams to form jets above or at the upper part of the dust-collecting chamber and below or at the lower end of the separating tubes.  
J. S. G. T.

Depolarising [porous positive] electrodes for electric batteries. SOC. ANON. LE CARBONE (B.P. 387,809, 22.12.31. Fr., 28.2.31).

Insulating paper. De-electrified sheets etc.—See V. Coating Fe or steel. Magnetic alloys. Hard

metal compositions. Improved Cu. Pb-Zn ores. In and Ga. Be-Al alloys. Fe and its ores. Cu from alloys. Cr-plating. Insulating films on metals.—See X.

## XII.—FATS; OILS; WAXES.

Detection of foreign [hardened] fats in cacao butter. B. PASCHKE (Z. Unters. Lebensm., 1932, 64, 561—564).—The sample is refluxed with  $\text{EtOH}$  and  $\text{H}_2\text{SO}_4$  for 6 hr., the solution diluted, and the Et esters are extracted in a mixture of  $\text{Et}_2\text{O}$  and light petroleum, the extract being washed until acid-free and dried over  $\text{Na}_2\text{SO}_4$ . The  $\text{Et}_2\text{O}$  is then removed by evaporation, the residue heated at  $95^\circ$  for 2 hr. and distilled in a vac. of 10 mm. until half (weighed, to within 0.3 g.) remains as residue ( $R$ ). The corresponding distillate is redistilled in a similar way, giving two fractions ( $A$  and  $B$ ). The sap. val. then is found in each case, the limits of ( $B - R$ ) and ( $B - A$ ) for pure expressed cacao butter being 7.1—8.1 and 3.9—5.1 respectively [refractivity at  $40^\circ$  ( $r$ ) 47.0—47.4]. Since the vals. are, respectively, 19.1 and 12.8 for hardened arachis oil ( $r$  51.0), and 39.2 and 24.2 for hardened train oil ( $r$  46.3), 10% of either in the sample is detectable (cf. B., 1931, 449).  
J. G.

Rapid determination of the acetyl value of fats. R. DELABY and V. BREUGNOT (Bull. Sci. pharmacol., 1932, 39, 354—361; Chem. Zentr., 1932, ii, 2759).—The oil (1—3 g.) is boiled with 10 c.c. of a mixture of  $\text{Ac}_2\text{O}$  (1 pt.) and  $\text{C}_5\text{H}_5\text{N}$  (2 pts.);  $\text{AcCl}$  is not essential.  
A. A. E.

Detection of resin in viscous fats. SOC. PAIX ET CRE. (Compt. rend. Congr. Graissage, 1931, 80—82; Chem. Zentr., 1932, ii, 2908).—The Storch-Morawski reaction is described and its val. criticised.  
A. A. E.

Boiling of a soap base for toilet [milled] soap [manufacture]. R. KRINGS (Allgem. Oel- u. Fett-Ztg., 1933, 30, 84—87).—Introductory. Six processes available for the prep. of fully-saponified, stable soap bases, including soaps settled on a lye, are outlined; recent applications of the "emulsion" (cold- or semi-boiling) processes are included.  
E. L.

Constitution of special soaps derived from chloro-hydroxy-fatty acids. A. BEYER (Tiba, 1932, 10, 831—837, 915—921).—The characteristic properties of Imbert's soap (cf. B., 1907, 25) are due to the presence of free OH groups, to the structure of the mol., and to its electro-colloidal nature. It consists of a hydrate of  $\sphericalangle 2$  mols. of free poly-acid (with  $\sphericalangle 1$  free  $\text{CO}_2\text{H}$  group) and  $\sphericalangle 2$  mols. of Na salt. It has no lactonic or other cyclic grouping.  
CH. ABS.

Significance of free alkali in soap. G. KNIGGE (Fettchem. Umschau, 1933, 40, 30; cf. B., 1933, 74, 197).—A very reliable method for the determination of free alkali in soap is the Davidsohn method for soft soaps (B., 1927, 883).  
E. L.

Significance of the fatty-acid carboxyl group in the textile-aid industry. H. T. BÖHME A.-G. (Fettchem. Umschau, 1933, 40, 30—31). H. STADLINGER (Ibid., 31—32).—Polemical against Lindner (cf. B., 1933, 28, 197). Priority in the prep. of sulphonated fatty alcohols is claimed for H. T. Böhme A.-G.  
E. L.

**Cooking temperatures of [China] wood oil.** E. BASTIAN (Farbe u. Lack, 1933, 106; cf. B., 1933, 275).—The oil (35–40 kg.) should be heated uniformly and rapidly in a 200-litre kettle by gas or oil burners to the temp. at which spontaneous heat evolution begins (predetermined with a small sample), the kettle then withdrawn, and sufficient raw oil added to cool the whole to 180°. Reference is made to variations in the consistency of the product when large and small quantities of oil are heated. S. M.

**Effect of cooling of [China] wood oil.** F. FRITZ (Farbe u. Lack, 1933, 105–106).—Fresh samples which have been carefully stored solidify at approx. 0°. The crude methods of extraction and collection still employed in China are responsible for brown, dark, or partly oxidised samples which remain liquid.

F. KÖHLER adds that the formation of a buttery mass at 0° is very slow with oils which have been transported and stored above 10°. Hongkong oils show less tendency to solidify than those from Hankow. S. M.

**Refining of castor oil.** R. HEUBLUM (Allgem. Oel- u. Fett-Ztg., 1933, 30, 77–78).—Details are given of successful commercial refining of expressed castor oil by treatment with NaOH without prefiltration; the presence of albuminous matter favours the separation of a coarse-grained soap stock which readily settles out. E. L.

**Preparation of paraffins from carnauba wax by means of catalytic hydrogenation.** H. I. WATERMAN, P. DE KOK, and A. J. TULLENERS (J. Inst. Petroleum Tech., 1933, 18, 1007–1009).—Hydrogenation carried out in a Bergius autoclave, using catalysts of Mo-C (cf. B.P. 331,199; B., 1930, 940) and Ni-kieselguhr (A., 1931, 1246), resulted in substantial yields (up to 70% on the wt. of wax) of a hard white waxy material possessing the properties of a pure paraffin. H. S. G.

**Lubrication.**—See I. **Pasty adulterants.**—See XIX. **Analysis of skin creams.**—See XX.

See also A., Mar., 223 and 224, **Soap solutions.** 248, **Determining m.p. of fats etc.** 256, **Prep. of glyceryl stearates.** 258, **Catalytic hydrogenation of fatty oils.** 312, **Prep. of Bi salts of fatty acids.**

#### PATENTS.

**Refining of vegetable and animal oils, fats, and waxes.** L. ROSENSTEIN and W. J. HUND (B.P. 387,962, 20.9.32. U.S., 22.9.31).—The free fatty acid content of fats or waxes is reduced by, e.g., distillation to a suitable limit (e.g., 3% for coconut oil) before extracting the residual impurities etc. with liquid NH<sub>3</sub> or alkylolamines. E. L.

**Wetting etc. agents.**—See III. **Leather.**—See XV.

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

**“Chalking” [of paint films].** VII. R. KEMPF (Farben-Ztg., 1933, 38, 559–562).—A further detailed report (cf. B., 1932, 895) is given on the degree of chalking and its relation to general weathering properties of TiO<sub>2</sub>, Ti white, lithopone, and white-lead paints. The seasonal influence on chalking is very notable with southern and

less so with northern exposure. The Ti paints chalk most and white-lead least; lithopone is intermediate. S. S. W.

**Rust-preventive lead paints.** M. MEIER (Oesterr. Chem.-Ztg., 1933, 36, 28–29).—A discussion of the rustproofing properties (for Fe) of paints containing compounds of Pb, directing attention to the reactions between these compounds and linseed oil, especially to the formation of Pb<sub>2</sub>O. E. S. H.

**Electrolytic pigment corrosion.** H. WAGNER and G. HEINTZ (Korrosion u. Metallschutz, 1932, 8, 225–226; Chem. Zentr., 1932, ii, 3150).—With Mars-red and chrome-orange, painted on galvanised Fe with water-glass, reduction by Zn is recorded. A. A. E.

**Apparent densities of lithopones.** K. LINS (Farbe u. Lack, 1933, 101–102).—Light lithopones ( $d$  1.11) differ from normal lithopone ( $d$  1.43) in the greater vol. of air retained between the particles in consequence of less extensive calcination. Their decreased wettability necessitates more oil and more grinding to produce uniform paints and enamels. The latter also possess lower hiding power, are therefore relatively more costly, dry more slowly, and subsequently become yellow in the dark more readily than those made from normal lithopone. S. M.

**[Poisonous metals in] school materials.** E. MERRES and R. TURNAU (Z. Unters. Lebensm., 1933, 65, 182–185).—No such metals were found in 19 samples of moulding compositions and 36 of coloured chalks; of 72 paints, 6 (green and sienna) contained 0.014–0.30% As, 5.2–7.2% Pb, and 2.5–22.0% Cr; and of 14 samples of coloured paper, one contained 0.45 mg. As/100 sq. cm. J. G.

**Aqueous casein pigments.** N. S. SOLOVIEV, V. VOITZEKHOVSKI, M. PESKIN, and A. KOSTENKO (Za Ovlad. Tekh. Kozh. Proizvod., 1931, No. 2, 17–18).—Russian and German dyes are compared. The effect of treatment with gum, shellac, alginate, etc. is described. CH. ABS.

**Newer chemistry of coatings.** C. ELLIS (Ind. Eng. Chem., 1933, 25, 125–132).—A résumé is given of the various types of synthetic resins available on commercial and laboratory scales. Their chemistry and mol. structure are outlined, special attention being paid to methods of modification in order to prevent polydimensional growth to a size inducing insolubility and infusibility. The development of cheap solvents, e.g., hydrogenated naphthas, acetic esters, is also discussed. S. S. W.

**Service requirements of insulating varnishes.** R. H. ARNOLD and L. E. FROST (Ind. Eng. Chem., 1933, 25, 133–135).—A general account is given of the use and testing of insulating varnishes. It is considered that present laboratory tests in this field do not yield reliable forecasts of behaviour on the production scale. S. S. W.

**Varnishes and lacquers for rubber footwear.** D. D. WRIGHT (Ind. Eng. Chem., 1933, 25, 140–141).—The functions, nature, and methods of application of varnishes etc. to rubber footwear are outlined. Retention of freedom from tack after packing, light-resistance,

extreme elasticity, and durability are the main requirements of such finishes. The difficulties encountered, *e.g.*, pitting, tackiness, "silveriness," cracking, poor adhesion, discoloration, etc., are briefly discussed.

S. S. W.

**Some effects of baking shellac varnish films.** M. RANGASWAMI and R. W. ALDIS (Indian Lac Res. Inst., Res. Note No. 4, Jan., 1933, 2 pp.).—Films which were baked so that their temp. was raised from 60° to 140° during 1½—2 hr. did not "blush," were not attacked by immersion in H<sub>2</sub>O and subsequent drying, and showed decreased H<sub>2</sub>O absorption and increased scratch hardness, abrasion-resistance, adhesion, and flexibility.

S. M.

**Shellac-drying oil combinations.** I. R. W. ALDIS (Indian Lac Res. Inst., Bull. No. 12, 1933, 4 pp.).—Powdered shellac is "dissolved" by linseed oil at 380—390°/1 atm., but there is danger of gelling. If the linseed oil is preheated with  $\frac{1}{36}$  of its wt. of PbO, incorporation of the shellac takes place at 290°; with increasing proportions of PbO to  $\frac{1}{6}$ , the temp. necessary is lowered to 220°. Oxides and carbonates of Na, K, Ca, Ba, and Mg, glycerin, rosin, ester gum, Albertol III L, stearic acid, Ph<sub>3</sub>PO<sub>4</sub>, tolyl phosphate, and triacetin can also be used as incorporating agents. Oil-sol. modified shellacs are made by heating rosin and shellac with either CaO or glycerin. Shellac "dissolves" also in tung oil if an incorporating agent is present. Long-oil varnishes are prepared by heating any of these "solutions" with sufficient blown or thickened oil at 270°; thinning is effected with the usual diluents. S. M.

**Explanation of the polymerisation of resols from refractometric measurements.** A. SCHMID and G. DE SENARCLENS (Helv. Chim. Acta, 1933, 16, 10—19).—The  $n_{\text{He}}$  yellow of a neutral resol (from PhCHO and CH<sub>2</sub>O; details of prep. given) maintained at 85° increases rapidly at first and then more slowly to a final val.; the same final val. is reached at 72° and 78°, but in these cases the initial rise is not so great as at 85°. When the resol contains 1.01—2.66% NaOH the  $n$ -time curves are similar to the above, but higher vals. of  $n$  are found with increase in concn. and the curves are eventually parallel to one another. The same final val. is reached at 72° or 85° when the resol contains the same concn. of NaOH. It is concluded that the NaOH accelerates condensation (as does raised temp.) but does not influence the true polymerisation; the position of chemical equilibrium is probably altered by the alkali.

H. B.

**Production of accumulator jars from plastic masses in the Karbolit works.** V. I. LISEV (Plast. Massi, 1932, Nos. 2—4, 24—29).—The process is described, with illustrations of mixers, presses, etc.

T. H. P.

**Ethylcelluloses.**—See V. Determining plasticity. —See VIII. Colouring rubber. Printing industry and rubber.—See XIV.

See also A., Mar., 224, Prussian-blue hydrosols. Prep. of colloids.

## PATENTS.

**Pigmented oil composition and process of inhibiting oxidation thereof.** J. C. EMHARDT, Assr.

to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,857,274, 10.5.32. Appl., 21.3.24).—Nitrocellulose compositions containing lithopone or TiO<sub>2</sub> pigments and a fatty oil having a tendency to rancidify, *e.g.*, castor oil, together with a derivative of citric or malic acid as rancidity retarder (1—5 wt.-% of the oil) are claimed. S. S. W.

**Manufacture of titanium oxide.** TITANIUM PIGMENT Co., Inc., W. F. WASHBURN, and F. L. KINGSBURY (B.P. 387,720, 13.8.31. U.S., 14.8.30).—Pptn. of TiO<sub>2</sub> hydrates is accelerated and pigments of improved colour and hiding power are obtained by hydrolysing a solution of a Ti salt, *e.g.*, Ti(SO<sub>4</sub>)<sub>2</sub>, in the presence of suspended particles of both CaSO<sub>4</sub> and hydrated TiO<sub>2</sub> (cf. B.P. 364,562; B., 1932, 315). The latter may be pptd. *in situ* or separately pptd. by addition of a neutralising agent to a solution of a Ti salt in a mineral acid and then introduced. S. M.

**Manufacture of writing inks.** NAMIKI MANUFG. Co., LTD. From KABUSHIKI KAISHA NAMIKI SEISAKUSHO (B.P. 387,844, 25.2.32).—The formation of ppts. and the corrosion of nibs in Fe-tannic acid inks are reduced by addition of substances which render positive the charges on the colloidal particles present, *e.g.*, P, As, Sb, Bi, or their compounds. S. M.

**Lacquer and lacquer enamel containing certain petroleum products.** B. N. LOUGOVOY, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,855,681, 26.4.32. Appl., 16.11.25).—Substantial proportions of certain petroleum distillates containing unsaturated compounds, *e.g.*, cracked petroleum having a boiling range of 35° between 100° and 200°, are used in nitrocellulose lacquers. As clarifier, 4—5% of alcoholic material, *e.g.*, BuOH, diacetone alcohol, is added. S. S. W.

**Cellulose ether compositions.** H. H. HOPKINS and J. B. SEGUR, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,855,744, 26.4.32. Appl., 24.5.28).—An alkyl cellulose ether, *e.g.*, ethylcellulose, is dispersed in a mixture of 1 pt. of a monohydric alcohol, *e.g.*, BuOH, and 0.33—6.0 pts. of an aliphatic hydrocarbon boiling between 30° and 250°, *e.g.*, petrol. S. S. W.

**["Flat"] nitrocellulose lacquer.** G. H. MUTERSBAUGH, M. ZUCKER, H. D. HEISER, and W. A. SHOPE, Assrs. to GLIDDEN Co. (U.S.P. 1,856,653, 3.5.32. Appl., 8.1.31).—Neutral metallic tungates, *e.g.*, Zn tungate, are added as flattening agents to clear nitrocellulose lacquers. S. S. W.

**Manufacture of resinous compositions from phenols and aldehydes.** BRIT. THOMSON-HOUSTON Co., LTD., Asses. of E. F. FIEDLER (B.P. 387,932, 26.7.32. U.S., 27.7.31).—A phenol is partly condensed with an aldehyde in the presence of an alkaline catalyst, *e.g.*, Ca(OH)<sub>2</sub>. The resinification is then completed in the presence of an acid catalyst, *e.g.*, H<sub>2</sub>SO<sub>4</sub>, a small amount of montan wax, CaO, hexamethylenetetramine, and fillers being also incorporated, and the mass is moulded under heat and pressure. Good moulding properties, finish, H<sub>2</sub>O-resistance, and mechanical strength are claimed. S. S. W.

**Shellac substitutes.** I. S. MELLANOFF, Assr. to KEMIKAL, INC. (U.S.P. 1,857,691, 10.5.32. Appl., 20.5.29).—100 pts. of casein are dissolved in 400—800

pts. of  $H_2O$  containing 16 pts. of  $Na_2B_4O_7$  and 10 pts. of 26%  $NH_3$  solution. 4 pts. of this solution are added slowly to 5 pts. of a  $PhOH-CH_2O$  reaction product (condensed in the presence of catalyst to a stage where it is substantially transparent and remains liquid on cooling and stirring) together with 1 pt. of sulphonated castor oil and 1 pt. of 10%  $NH_3$  solution, if required, to prevent gelation. S. S. W.

**Preparation of potentially reactive phenolic condensation products.** O. A. CHERRY and F. KURATH, Assrs. to ECONOMY FUSE & MANUFG. CO. (U.S.P. 1,857,357, 10.5.32. Appl., 9.4.28).—Furfuraldehyde and  $PhOH$  are refluxed for 3 hr. in the presence of an alkaline catalyst, e.g.,  $NaOH$ , and after neutralisation, e.g., with  $H_2C_2O_4$ , and cooling, the condensation product is heated for about 50 min. with a methylene-containing substance, e.g., commercial  $CH_2O$ , in the presence of  $NH_3$ . After cooling, furfuralamide is stirred in until the odour of  $CH_2O$  disappears, and the resinous layer is then separated. S. S. W.

**Manufacture of synthetic resins.** BRIT. THOMSON-HOUSTON CO., LTD., Asses. of J. G. E. WRIGHT (B.P. 387,928, 21.7.32. U.S., 21.7.31).—A flexible "alkyd" resin is compounded with polymerised vinyl chloride and filler at approx.  $95^\circ$  for  $\frac{1}{2}$  hr. and after moulding under heat and pressure the product is "cured" outside the mould for about 22 hr. at  $80-100^\circ$ . The product is acid-, alkali-,  $H_2O$ -, and flame-proof. S. S. W.

**Production of vitreous polymerised styrene from mixtures of styrene with materials.** I. OSTROMISLENSKY and W. A. GIBBONS, Assrs. to NAUGATUCK CHEM. CO. (U.S.P. 1,855,413, 26.4.32. Appl., 16.1.31).—Mixtures of styrene ( $\leq 40\%$ ) and high-boiling aromatic hydrocarbons, e.g.,  $PhEt$ , are heated with access of air at  $80-200^\circ$  for 8–150 hr., and the polymerised styrene is separated and purified. (Cf. U.S.P. 1,703,950; B., 1929, 467). S. S. W.

**Moulding, coating, filling, impregnating, and binding.** I. S. MELLANOFF, Assr. to KEMIKAL, INC. (U.S.P. 1,857,690, 10.5.32. Appl., 25.9.28).—Fibrous or cellular material is treated successively with 5–10% and 15–30% solutions of the syrupy condensation product of an alkaline solution of peat with aldehydic and phenolic substances (cf. U.S.P. 1,681,155; B., 1928, 866), and the impregnated material is subjected to heat and pressure. S. S. W.

**Production of plastic masses, varnishes, putty masses, and the like.** DEUTS. HYDRIERWERKE A.-G. (B.P. 387,534, 24.3.32. Ger., 28.3.31).—Plasticisers suitable for cellulose varnishes are made by condensing a dicarboxylic acid, or its ester or anhydride, e.g., phthalic anhydride, with equiv. quantities of an alcohol above  $C_7$ , e.g., lauryl alcohol. S. M.

**Polymerised vinyl esters.**—See III. **Dye pigments.**—See IV. **Resin threads.**—See V. **Plastics from butadiene.**—See XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**[Rubber] latex round thread. Its manufacture and properties.** E. A. MURPHY (Trans. Inst. Rubber Ind., 1932, 8, 328–344).—Manufacturing details are

given. Advantages of the latex process, relative to the older method using dry rubber, are greater length and smoothness of the threads, wider range of counts, and higher tensile strength. D. F. T.

**Impregnating fabrics with rubber [latex].** E. A. HAUSER and (Miss) M. HUENEMOERDER (Trans. Inst. Rubber Ind., 1932, 8, 316–327).—For waterproofing, complete impregnation of the fabric is not essential, but perfect adhesion of the rubber to the fabric must be obtained; the greater is the penetration of the rubber into the interstices of the fibre, however, the better the anchorage. To-day by treatment of cord with  $C_6H_6$  or similar solvent capable of destroying the  $H_2O$ -repellent properties of the fibre, or by the addition of special wetting agents to the latex, it is possible with latex to effect as good impregnation as can be obtained with rubber solutions. D. F. T.

**Technical red-colouring of rubber goods.** R. KRECH and B. WURZSCHMITT (Chem.-Ztg., 1933, 57, 141–142).—The possible advantages to be derived from replacement of the older red pigments for rubber, e.g.,  $Sb_2S_3$  or  $HgS$ , by org. colours are discussed from the legal and technical aspects. D. F. T.

**Electrodeposition of rubber from Revertex and Revultex.** N. BUDILOFF (Kautschuk, 1933, 9, 1–4, 20–25).—Diluted Revertex is free from aggregated particles and is of similar consistency to  $NH_3$ -preserved latex. In order to obtain homogeneous non-porous deposits on a Zn electrode, however, the addition of  $NH_3$  is necessary. The wt. of deposited rubber under comparable conditions is approx. proportional to the amount of current passed, the best yield being obtained with a c.d. of 0.1 amp./sq. dm. In the formation of the deposit the coagulant effect of Zn ions dissolved from the anode plays an important part, and deposition will continue for some time after the current has been stopped. Zn and Cd are the most suitable metals for the anode. Compounded Revertex can be electrodeposited successfully but, presumably on account of diminished adhesiveness between its globules, diluted Revultex (i.e., Revertex containing vulcanised rubber globules) when compounded with fillers fails to give satisfactory anode deposits although, in the uncompounded condition and after the addition of  $NH_3$ , good deposits are obtainable which dry without cracking. D. F. T.

**Use of rubber in the printing industry.** G. L. RIDDELL (Trans. Inst. Rubber Ind., 1932, 8, 294–315).—Possible applications of rubber to printing plates, rollers, blankets, etc. are described. Rubber is also used in some photogravure inks, and latex is finding use as an adhesive in the bookbinding trade. D. F. T.

**Preservatives in a [rubber] tyre-tread stock.** T. R. DAWSON (J. Res. Assoc. Brit. Rubber Manuf., 1933, 2, 1–9).—The perishing of tyre rubbers under the influence of heat and light assumes serious importance in the tropics. Samples of tyre-tread stock, containing, respectively, tolueneazotolueneazo- $\beta$ -naphthol (I) and quinol (II) and vulcanised over a range of periods, when aged at  $70^\circ$  revealed a definite beneficial effect of (II) and a feeble influence by (I). The latter also retarded vulcanisation and bloomed during the ageing. Natural

outdoor ageing over 13 months confirmed the efficacy of (II) and demonstrated that (I) is more effective in sunlight than in the dark.

D. F. T.

**Interfacial relationships between rubber and fillers. I. Tear-resistance of vulcanised rubber.** G. LEFCADITIS and F. H. COTTON (Trans. Inst. Rubber Ind., 1932, 8, 364—389).—Known methods for measuring tear-resistance of rubber are critically examined. In view of the need for more trustworthy and consistent results, a method is evolved in which tearability is recorded in terms of the mean of the initial and final loads required to tear a 4-mm. length of a butt-ended test-piece punched with a standard dumb-bell die from the inner disc of a 6-mm. ring test-piece. The results of this test are compared with those of older methods. In many cases it is impossible to effect tearing across the grain; measurement on non-grained test-pieces is therefore adopted. Incorporation of fine china clay reduces the tear-resistance of vulcanised rubber; low-temp. accelerators, however, give a vulcanised product of exceptional resistance. The effect of a mixture of china clay and C black is below the algebraic sum of the effects of the 2 components separately.

D. F. T.

**Determining the swelling of vulcanised rubber in organic liquids by means of a voluminometer.** D. J. VAN WIJK (Kautschuk, 1933, 9, 18—20).—Advantages in accuracy and time are gained by measuring the vol. instead of the wt. or linear dimensions of the swollen test-piece. The voluminometer described may be used equally well for other materials such as wood, gelatin, or artificial silk.

D. F. T.

**Tinning of rubber-insulated Cu wire.**—See X. Varnishes etc. for rubber.—See XIII.

## PATENTS.

**Manufacture of rubber.** SOC. ITAL. PIRELLI (B.P. 387,267, 2.9.32. Ital., 17.10.31).—Vulcanisable rubber mixtures containing powerful accelerators are softened by the addition of a small proportion (*e.g.*, 2—10%) of the partly depolymerised product obtained by heating rubber to near its m.p., *e.g.*, at 250°. The need for mastication is thereby reduced and premature vulcanisation is avoided.

D. F. T.

**Manufacture of synthetic rubber.** E. I. DU PONT DE NEMOURS & Co. (B.P. 387,340, 24.7.31. U.S., 14.5.31).—An aq. emulsion of  $\beta$ -chloro- $\alpha\gamma$ -butadiene readily polymerises to a latex. A suitable emulsifying agent is Na oleate. A protective colloid (protein, pectin, or resin), anti-oxidants,  $\text{NH}_3$ , etc. may be added.

**Preparation of useful [rubber-like and other] substances from halogen-substituted butadienes.** E. I. DU PONT DE NEMOURS & Co. (B.P. 387,363, 24.7.31. U.S., 28.2.31).—A  $\beta$ -halogeno- $\alpha\gamma$ -butadiene is polymerised, *e.g.*, in presence of air,  $\text{O}_2$ , heat, light, catalysts, or under pressure. As catalysts peroxides of Bz, Na, Pb, or H, or oxidised turpentine may be used, and the rate of polymerisation controlled by addition of solvents or anticatalysts (phenols, quinones, amines, aromatic  $\text{NO}_2$ -compounds, mercaptans, thio-ethers, especially quinol or pyrogallol). A rubber-like plastic is obtained by stopping the polymerisation before products insol. in

$\text{C}_6\text{H}_6$  are formed; this may be cured by heat alone, *e.g.*, at 50—180°. 74 examples are given.

C. H.

**Manufacture of vulcanisation products resembling rubber.** A. CARPMAEL, and I. G. FARBENIND. A.-G. (B.P. 387,381, 4.8.31. Addn. to B.P. 339,255 and 342,314; B., 1931, 264, 407).—When vulcanised in the presence of finely-divided C, mixed polymerisates derived from hydrocarbons of the butadiene type and from polymerisable non-hydrocarbon substances, such as Et sorbate or cinnamate,  $\gamma$ -chloro- $\beta$ -methylbutadiene, and 3-chlorostyrene, yield rubber-like products of high elasticity and strength.

D. F. T.

**Manufacture of goods of or containing rubber or similar material.** DUNLOP RUBBER Co., LTD., ANODE RUBBER Co., LTD., D. F. TWISS, E. A. MURPHY, and A. NIVEN (B.P. 387,671, 7.8.31. Addn. to B.P. 350,450; B., 1931, 770).—Aq. dispersions, *e.g.*, of rubber, are treated so as to form granular ppts. by addition of coagulating agents, such as AcOH,  $\text{Na}_2\text{SiF}_6$ ,  $\text{ZnSO}_4$ , other than compounding ingredients formed by pptn. *in situ*. By coating rubber articles with such a granular ppt., a matt surface-finish is imparted.

D. F. T.

**Moulding of articles of, or containing, rubber, guttapercha, or similar organic substances.** DUNLOP RUBBER Co., LTD., ANODE RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 387,670, 7.8.31).—Articles are produced with a standard surface finish, ranging from a smooth matt to a coarse granular texture, by operations such as dipping, moulding, or spraying, using formers or moulds which have previously been coated with the particular type of flocculent or granular ppt. required (cf. preceding abstract). Instead of using the master-former or mould thus obtained, replicas thereof may be used produced by known methods.

D. F. T.

**Vulcanisation [of rubber].** G. P. DAVIS (B.P. 387,934, 2.8.32).—Chlorophyll activates, accelerates, and permanently fixes the vulcanisation of rubber or rubber latex in the presence of vulcanising agents, preferably including  $< 1\frac{1}{2}\%$  of Zn or Pb compounds or other activator or accelerator. The vulcanisation process is effected by ordinary factory methods or by solar heat and light at room temp. and atm. pressure.

D. F. T.

**Accelerator for vulcanisation of rubber.** A. CAMBRON, ASSR. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,858,577, 17.5.32. Appl., 28.3.28. Ger., 19.1.27).—Condensation under essentially anhyd. conditions is effected between a straight-chain aliphatic aldehyde ( $\text{C} > 1$ ), *e.g.*, MeCHO or crotonaldehyde, and a primary arylamine, such as  $\text{NH}_2\text{Ph}$ , in the presence of an acidic catalyst, *e.g.*,  $\text{ZnCl}_2$  and HCl, successively, and without a solvent. The product can be finely powdered and will not agglomerate after grinding.

D. F. T.

**Treatment of rubber with aldehyde derivatives.** S. M. CADWELL, ASSR. to NAUGATUCK CHEM. Co. (U.S.P. 1,855,336, 26.4.32. Appl., 14.5.26).—Deterioration of rubber is retarded by adding, before vulcanisation, a substantially non-accelerating condensation product, prepared from an aliphatic aldehyde (1—1 $\frac{1}{2}$  mols.) and an arylamine (1 mol.) in strongly acid solution, *e.g.*, MeCHO- $\text{NH}_2\text{Ph}$ , m.p.  $< 150^\circ$ , containing bimol. ethyldeneaniline.

D. F. T.



**Vulcanisation of rubber.** G. H. STEVENS (U.S.P. 1,856,596, 3.5.32. Appl., 30.9.25).—Vulcanisation is accelerated by a member of the class constituted by carboditolyimide and its homologues and their polymeric modifications. D. F. T.

**Vulcanisation of rubber.** C. O. NORTH and W. SCOTT, ASSRS. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,856,819, 3.5.32. Appl., 21.11.29).—Vulcanisation is accelerated by the heptaldehyde derivative of a reaction product obtained from a primary arylamine (2 mols.) and an aliphatic aldehyde, e.g., of butylideneaniline. D. F. T.

**Vulcanisation of rubber or the like.** NAUGATUCK CHEM. CO., ASSCES. of W. F. TULEY (B.P. 387,454, 27.11.31. U.S., 19.12.30).—Rubber containing S, a metallic oxide, and an org. accelerator is vulcanised in the presence of an acidic or substantially neutral compound containing the group  $\text{NHR}'\cdot\text{CO}\cdot\text{NR}''\text{R}'''$  (R', R'', and R''' representing H or hydrocarbon radicals). These compounds, which may also be used in the form of their salts or of mol. compounds with metallic salts, do not affect vulcanisation at mixing or milling temp., but at normal vulcanising temp. they liberate  $\text{NH}_3$  or amines which promote the vulcanisation reaction. D. F. T.

**Rubber composition and method of preserving rubber.** C. W. BEDFORD, ASSR. to B. F. GOODRICH CO. (U.S.P. 1,855,788, 26.4.32. Appl., 11.9.28).—Rubber is treated with the condensation product of an aliphatic aldehyde (< 2 mols.), e.g., aldol, and a naphthylamine (1 mol.), e.g.,  $\alpha\text{-C}_{10}\text{H}_7\text{-NH}_2$ , prepared in the presence of a small proportion of an acid. D. F. T.

**Antioxidant [for rubber].** L. B. SEBRELL, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,857,981, 10.5.32. Appl., 4.5.31).—Naphthanthraquinone and its  $\text{NH}_2$ -derivatives are comparatively sol. in rubber and, when incorporated, exhibit marked anti-ageing influence. D. F. T.

**Vulcanisation accelerator.**—See III. **Waterproof paper.** **Rubbered sheet material.**—See V. **Rubber-coated fabric.**—See VI. **Artificial leather.**—See XV.

## XV.—LEATHER; GLUE.

**Terminalia pallida** as a tanning material. K. S. CHOUDARY (J. Soc. Leather Trades' Chem., 1933, 17, 68—70).—The analysis of *Tella karaka* (*Terminalia pallida*) pods and bark showed, respectively (%): tans 20.8, 14.6; sol. non-tans 22.75, 4.7. The tannin content of the liquors when exposed to the atm. was rapidly diminished by fermentation, but less acid was formed than with *T. chebula* liquors. D. W.

**Effect of tannin, acidity, and temperature on the cinchonine test for detection of sulphite-cellulose waste liquor [in vegetable tanning extracts].** O. GERNGROSS and H. HERFELD (Collegium, 1933, 66—75; cf. B., 1933, 159).—The error due to sulphiting is diminished and ultimately eliminated as the acidity and temp. of the solution are increased, but the sensitivity of the test is diminished. The sulphiting error is eliminated by carrying out the test at 70°, but the limit of sulphite-cellulose waste detectable in an extract is increased from 3% to 6—7%. D. W.

**Determination of the  $p_{\text{H}}$  values of tanning extracts.** Report No. 3 of the British Section Committee [of the Society of Leather Trades' Chemists]. D. BURTON (J. Soc. Leather Trades' Chem., 1933, 17, 57—59; cf. B., 1933, 33).—Good agreement was obtained with determinations by means of the glass electrode in different types of apparatus; it was shown to be superior to the bubbling  $\text{H}_2$  electrode. Precautions must be taken to prevent the escape of  $\text{SO}_2$  from bleaching extracts during the determination. D. W.

**Testing of tannins with animalised cotton cloth.** A. GANSSER (Boll. Uff. Staz. sperim. Ind. Pelli, 1933, 11, 1—4).—When a revolving bath is used in this test, part of the gelatin leaves the tissue and ppts. some of the tannin, and avoidance of this by pretreating the cloth with  $\text{CH}_2\text{O}$  is said to alter the colour of the tannin. With a stationary bath, however, good results are obtained, even when  $\text{CH}_2\text{O}$  is used, although the tanning time is increased from 12 to 24 hr. The tanning of fishing nets is referred to. T. H. P.

**Effect of acids on vegetable[-tanned] leather.** I. V. KUBELKA and E. WEINBERGER (Collegium, 1933, 89—105).—15.7% of hide substance was sol. in  $\text{H}_2\text{O}$  in a chestnut-tanned leather containing 5% HCl which had been allowed to remain 1 month, and a similar leather containing 1% HCl was completely destroyed after 6 months. The deteriorating effect of  $\text{H}_2\text{SO}_4$  was not measurable until the leather had been allowed to remain 1 month, when it was > the effect of HCl, and the proportion of hide substance sol. in  $\text{Na}_2\text{CO}_3$  compared with that sol. in  $\text{H}_2\text{O}$  and  $\text{Na}_2\text{CO}_3$  was < the proportion obtained for leathers containing HCl. The initial deterioration of leather containing  $\text{H}_2\text{C}_2\text{O}_4$  was nearly the same as that in leather containing  $\text{H}_2\text{SO}_4$ , but after allowing the leather to remain for long periods the damage to the leather was not as great as that in leather containing  $\text{H}_2\text{SO}_4$ . The tensile strength of leather was increased and the  $\text{H}_2\text{O}$ -sol. matter diminished by treating it with 1—10% AcOH. Acid-free vegetable-tanned leathers contain a max. of 0.3—0.5% hide substance sol. in  $\text{H}_2\text{O}$  and  $\text{Na}_2\text{CO}_3$ ; a higher val. (> 0.8—1.0%) indicates acid hydrolysis of the vegetable-tanned leather. D. W.

**Influence of neutralisation, mordant, and fixative on the colour and uniformity of dyed chrome-tanned leather.** I. N. IZAKSON (Za Ovlad. Tekh. Kozh. Proizvod., 1931, No. 2, 17).—The best results were obtained with leather having a liquor of  $p_{\text{H}} < 6$ . A slightly acid medium should first be used owing to rapid absorption of the dye. Mordants and fixatives (best, K Ti oxalate) are discussed. CH. ABS.

**Liming of fellmongered sheepskins.** R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1933, 17, 60—68).—Different types of fellmongered sheepskins have been examined microscopically and photomicrographs are reproduced. The grain layer in sheepskins fellmongered with  $\text{Ca}(\text{OH})_2$  was better than, and the corium inferior to, those in skins fellmongered with  $\text{Na}_2\text{S}$ . The corium in poor  $\text{Ca}(\text{OH})_2$ -fellmongered skins was further deteriorated by liming, but good skins were slightly improved. The corium in  $\text{Na}_2\text{S}$ -fellmongered

skins was improved by further liming, and the quality of different skins was rendered more even thereby. The slightly better condition of  $\text{Na}_2\text{S}$ -felled skins was not maintained after tanning. D. W.

**Investigating lime liquor [from leather treatment] with a glass electrode.** S. I. SOKOLOV, A. G. PASUNSKI, and N. P. PESKOV (Za Ovlad. Tekh. Kozh. Proizvod., 1931, No. 2, 14—15).—A glass electrode containing  $\text{Li}_2\text{O}$  10,  $\text{CaO}$  10, and  $\text{SiO}_2$  80% was used. Solutions containing  $< 0.25\%$   $\text{Na}_2\text{S}$  have an almost const.  $p_{\text{H}}$  of 12.2—12.3, whilst in a saturated solution  $p_{\text{H}} = 12.0$ . Saturated  $\text{Ca}(\text{OH})_2$  has  $p_{\text{H}}$  12.6. The solubility of  $\text{Ca}(\text{OH})_2$  decreases with addition of  $\text{Na}_2\text{S}$ . Addition of  $> 10\%$   $\text{NaCl}$  to  $\text{Ca}(\text{OH})_2 + \text{Na}_2\text{S}$  slightly lowers the  $p_{\text{H}}$ .  $\text{KCNS}$  scarcely affects  $p_{\text{H}}$ ;  $\text{As}_2\text{S}_3$  only slightly decreases  $[\text{Ca}^{++}]$ . Addition of  $(\text{NH}_4)_2\text{SO}_4$  to saturated  $\text{Ca}(\text{OH})_2 + 0.25\%$   $\text{Na}_2\text{S}$  progressively decreases the  $p_{\text{H}}$ . Considerable lowering of  $p_{\text{H}}$  can be effected only by addition of weak bases ( $\text{NH}_4$  salts). In fresh lime liquors  $p_{\text{H}}$  is practically const. at 12.2—12.4. CH. ABS.

**Salting of skins with soda-salt.** II. M. BERGMANN and L. SELIGSBERGER (Ledertech. Rundsch., 1932, 24, 73—76; Chem. Zentr., 1932, ii, 2912—2913).—The absorption of  $\text{Na}_2\text{CO}_3$  and  $\text{NaCl}$  over various periods was measured; absorption is greater for medium than for fine grain-size. Mechanical separation during transport occurs with effloresced or calcined  $\text{Na}_2\text{CO}_3$ , but not with soda crystals. A. A. E.

**Effect of various additions to marine salt for use as a cure for hides and skins.** A. GUTHRIE and M. S. SASTRY (J. Soc. Leather Trades' Chem., 1933, 17, 50—56).—"Red heat" was diminished, though not entirely eliminated, in raw hides and skins by additions to the  $\text{NaCl}$  of 3% of the following salts in diminishing order of effect:  $\text{Na}_2\text{SiF}_6$ ,  $\text{ZnCl}_2$ ,  $\text{H}_2\text{C}_2\text{O}_4$ , "Atlas preservative," but was not diminished by additions of  $\text{NaHSO}_4$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaHSO}_3$ , or "Perchloron." D. W.

**Rapid detection of follicular scabs in unhaired hide.** M. BERGMANN, W. HAUSAM, and E. LIEBSCHER (Ledertech. Rundsch., 1932, 24, 77; Chem. Zentr., 1932, ii, 2912).—A microscopical procedure is described. A. A. E.

**Testing "Gratan," used in the preservation of fish skins.** Y. N. KAPLUNOV (Za Ovlad. Tekh. Kozh. Proizvod., 1931, No. 2, 14).—A 0.5% solution of "Gratan," containing cyclic hydrocarbons condensed with phenol, gave the best results. CH. ABS.

**Quality of fish glues: their physical examination.** D. JENNINGS (Chem. Trade J., 1933, 92, 105).—Fish glues are liquid, should be slightly acid to phenolphthalein but neutral to litmus, and should remain unaltered after 1 month's storage at 37—38°. The colour is affected by the fish stock from which it has been manufactured and by the method of clarification used. Skin-stock glues are clear; fish glues are opaque. The latter should be tested for white pigments (e.g.,  $\text{ZnO}$ ) and alum. Partial decomp. is indicated by a putrid odour which remains after the removal of any essential oils by steaming. Glues with a high gel point do not spread uniformly nor give uniform strength in joints. Best fish glues gelatinise at 10—7.5°. The viscosity is

increased by increasing the concn. and by adding  $\text{H}_3\text{BO}_3$  or  $\text{NaF}$ , and diminished by  $\text{PhOH}$ , cresol, and metal chlorides. The drying qualities of liquid glues are compared by noting the times required for thin films of the glues on glass to dry at 20° and R.H. 20%; the dry films should not become liquid or tacky at 25° and R.H. 75—80%. Glues containing  $> 0.2\%$   $\text{NaCl}$ ,  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ , or  $\text{ZnCl}_2$  are unsatisfactory. D. W.

**Adhesion in the ply-wood industry.** A. HECQUET (Chim. et Ind., 1933, 29, 262—267).—The manufacture and methods of application of glues for multi-ply woods are described. A satisfactory adhesive must (a) enter the panel without thorough penetration, (b) be sufficiently stable to allow easy prep. and application, (c) gel rapidly after application, (d) possess max. tensile strength ( $T$ ) and  $\text{H}_2\text{O}$ -resistance ( $R$ ). The  $T$  of various adhesives made from casein, gelatin, blood-albumin, and synthetic resins are tabulated. One made from 22% of casein, 73% of  $\text{H}_2\text{O}$ , and 5% of  $\text{Ca}(\text{OH})_2$  displayed good  $T$  and  $R$  vals., but gelled too rapidly; substitution of the  $\text{Ca}(\text{OH})_2$  by  $\text{Na}_2\text{CO}_3$  increased the stability but decreased  $R$ , and the surface of the panel also tended to be faulty. Curves are plotted to show the effect of  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{SiO}_3$ , and  $\text{NaF}$  on the stability of casein glues. The max. contents of fatty matter, acid val. (as  $\text{H}_2\text{SO}_4$ ), and ash in caseins of good quality are 0.4, 0.5, and 0.2% respectively; adhesives having higher vals. are of uncertain behaviour, especially after drying. Ordinary gelatin is low in  $T$  and  $R$ . Blood-albumin evolves a bad odour and undergoes rapid putrefaction when heated; synthetic resins ( $\text{CH}_2\text{O}$  type) are satisfactory. S. M.

**Gelatin films.**—See V. **Casein pigments.**—See XIII. **Determining swelling [of gelatin].**—See XIV.

#### PATENTS.

**Tanning of hides and extraction of tanning liquors from myrobalans, bark, and the like.** J. FORSTER (B.P. 387,449, 24.11.31).—A relatively long tanning drum ( $A$ ) is divided into three co-axial compartments (by partitions extending perpendicular to the axis of  $A$ ) each of which is subdivided by radial partitions extending parallel with the axis of  $A$  into a no. of smaller compartments. The last-named are provided with openings for feeding and emptying, and are also interconnected for the transference of liquors. Tanning materials are leached on the counter-stream principle in some of these, and hides are suspended in others and tanned with liquors of increasing concn. obtained from the leaching compartments. D. W.

**Manufacture of leather.** CHEM. FABR. SIEGFRIED KROCH A.-G., and J. H. EPSTEIN A.-G. (B.P. 387,542, 11.4.32. Ger., 13.5.31).—Leather is fat-liquored with an emulsion of a vegetable oil containing 85—95% of esters of unsaturated fatty acids of the oleic acid series which solidify entirely or partly at  $< -6^\circ$ , e.g., apricot, peach-kernel, almond, or bleached tea-seed oils. The oils may be sulphonated or emulsified with other sulphonated oils. D. W.

**Manufacture of artificial leather.** E. GARTNER and M. RUCH (B.P. 387,414, 9.9.31. Ger., 16.9.30).—A fibre fleece of artificially spun fibres lying mainly in one direction is impregnated with rubber latex and guided through drying rolls or cylinders. D. W.

## XVI.—AGRICULTURE.

**Effect of acidification on the productivity of ordinary chernozems.** P. SAJEV (Z. Pflanz. Düng., 1933, 27A, 407—418).—Among chernozems of similar nutrient and humus contents, higher crop yields occur on the more acid types. Applications of CaO to these soils reduces the mobility of humus, N, and (notably) of P. Artificial acidification produced the reverse effects. Addition of different acids, to produce the same  $p_H$ , had varying effects on crop yields. The action of  $\text{NaHSO}_4$  was similar to that of superphosphate. Acids applied in quantities too small to affect the current season's crop increased the yield of oats in the second season. A. G. P.

**Exchangeable bases of some East Anglian soils derived from the Jurassic and cretaceous sediments, with special reference to their marine origin.** M. L. M. SALGADO (J. Agric. Sci., 1933, 23, 18—30).—Contrary to the views of Taylor and Woodman (B., 1931, 688), the difficulty of working certain Kimmeridge and Oxford clays and gault soils results merely from their high content of silt and clay and not from a preponderance of Na-clay. The exchangeable Na content of the surface soils was small, but increased somewhat with depth. A. G. P.

**Determination of adsorptively bound calcium in soils containing calcium carbonate.** S. S. JARUSSOV (Z. Pflanz. Düng., 1933, 27A, 386—402).—The treatment with neutral salt solutions of carbonate-containing soils exhibiting hydrolytic acidity results not only in the release of adsorbed Ca, and the dissolution of some  $\text{CaCO}_3$  as  $\text{Ca}(\text{HCO}_3)_2$  by the direct action of the neutral salt, but also in the decomp. of  $\text{CaCO}_3$  by the acid soil. Sol. Ca salts are produced and the  $\text{CO}_2$  liberated brings further amounts of  $\text{CaCO}_3$  into solution. As a result of these secondary reactions the methods of Hissink, Tiurin, Schmuck, and of Gehring for determining exchangeable Ca yield unduly high vals. in soils of this type. The following method overcomes this source of error. The sample (5—10 g.) is treated with 500 c.c. of  $N\text{-KCl}$  and 1 g. of  $\text{CaCO}_3$ . After keeping for 12—16 hr. the mixture is shaken for 30 min. and filtered. In the filtrate  $\text{Ca}^{++}$  and total alkalinity (Me-orange) are determined. The adsorbed Ca is calc. as total  $\text{Ca}^{++} - 2(\text{HCO}_3^-) + L$ , where  $L$  is the solubility of  $\text{CaCO}_3$  in  $N\text{-KCl}$ , determined separately under conditions similar to the above. For old limed podsols the above method and those of Tiurin and of Schmuck proved most suitable; others gave high vals. Tiurin's method should be used for soils containing natural carbonates but no  $\text{MgCO}_3$ . In this case other methods, including the author's, tend to yield low results. A. G. P.

**Rate of decay in relation to soil types and vegetative covering in Glamorgan.** W. E. ISAAC (J. Agric. Sci., 1933, 23, 31—72).—Apparatus is described for determining the rate of  $\text{CO}_2$  production during the decomp. of org. matter in soils under standardised conditions. Differences in the rate of decomp. of ground linseed meal in the soils examined were small, but, in general, were paralleled by differences in fertility. Seasonal variations in the decomposing power of a

particular soil exceed differences exhibited by soils of different origin. A. G. P.

**Determination of unsaturation of soils by ammonia absorption.** J. DI GLÉRIA and L. KOTZMANN (Mezőg.-Kutat., 1932, 5, 270—278; Chem. Zentr., 1932, ii, 3292).—The method is more characteristic, and less dependent on the original concn. of the absorbing material, than Hissink's, Gedroiz', or the conductometric method. A. A. E.

**Determination of sulphate in soil extracts.** J. S. HOSKING (Chem. Eng. Min. Rev., 1933, 25, 139—140).—The HCl extract of soil (25—100 c.c.) is evaporated with a few c.c. of 0.1*N*- $\text{KMnO}_4$  to destroy org. matter. The dry residue is treated with dil. (1 : 10) HCl, filtered, and, if org. matter is still present, boiled with a further small quantity of  $\text{KMnO}_4$  solution. The liquid is diluted to 125—150 c.c., filter-paper pulp is added, and after cooling to 70°, treated with 1 : 1 aq.  $\text{NH}_3$  until a ppt. forms, when a further addition of 15 c.c. of 1 : 1 aq.  $\text{NH}_3$  is made and the whole filtered. The filtrate is evaporated to about 50 c.c., neutralised with  $N\text{-HCl}$ , and a further 2.5 c.c. of the acid are added. After boiling for a few min., the liquid is again filtered, diluted to 250—300 c.c., heated to boiling, and treated with excess of  $M\text{-BaCl}_2$  (for every 12 mg.  $\text{SO}_3$  add 1.5 c.c. of  $\text{BaCl}_2$ , with 5 c.c. in excess), all the reagent being added at once. The liquid is boiled for 5 min. and set aside overnight on a low-heat plate. The  $\text{BaSO}_4$  is then collected on a filter, washed, and weighed as usual. A. G. P.

**Colorimetric determination of phosphorus in citric acid [soil] extracts.** R. R. WARD (Soil Sci., 1933, 25, 85—97).—To 120 c.c. of the customary citric acid extract of soil are added 50 c.c. of conc.  $\text{HNO}_3$ , 15 c.c. of conc. HCl, and 10 c.c. of 20%  $\text{H}_2\text{SO}_4$ . The mixture is evaporated to fuming ( $\text{SO}_3$ ), hot  $\text{H}_2\text{O}$  is added, and, after boiling, the liquid is filtered into an electrolytic cell provided with a Hg cathode and a spiral Pt anode. Electrolysis is continued for 16 hr. with a cathode c.d. of 0.03—0.05 amp. per sq. cm. (6—8 volts). The liquid is then removed, treated with a few drops of  $\text{H}_2\text{O}_2$  to destroy permanganate formed during electrolysis, and conc. to about  $\frac{1}{2}$  vol. Phosphate is then determined by Zinzadze's method (A., 1930, 725). A. G. P.

**Conductivity method in agrochemistry.** A. A. KHALIZEV (Khim. Sotzial. Zemled., 1932, 1, No. 2, 31—46).—The conductivity of soil extracts is given by  $u = aC^n$ , where  $C$  is the ratio soil :  $\text{H}_2\text{O}$  and  $a$  and  $n$  are consts. Suspended soil particles usually increase the conductivity of the extracts. The conductivity of the particles is characteristic for each soil. CH. ABS.

**New field apparatus for electrometric  $p_H$  measurements.** A. UHL (Z. Pflanz. Düng., 1933, 27A, 418—424).—A special form of electrode is described. A. G. P.

**Carbon dioxide treatment [of plants] in field soils.** A. BÖMER and P. RINTELEN (Z. Pflanz. Düng., 1933, 12B, 49—85).—The effect is examined of climatic conditions on the concn. of  $\text{CO}_2$  maintained in the atm. adjacent to the soil surface by a system of pipe-line distribution. Treatment with  $\text{CO}_2$  increased the yields

of a no. of crops, the strength of straw, the hectolitre wt., and wt. per 1000 grains in rye and oats and induced firmer heading in cabbage. A. G. P.

**Making phosphate fertilisers at Trail [B.C.].** W. C. WEBER (Chem. Met. Eng., 1933, 40, 72—75).—In this works  $H_3PO_4$  is produced at 30—32%  $P_2O_5$  concn. directly. "Triple superphosphate" is made by further concn. to 39%  $P_2O_5$  in single-effect vac. evaporators and mixing with ground phosphate rock. Alternative products are  $(NH_4)H_2PO_4$  and mixtures thereof with  $(NH_4)_2SO_4$ . These require acid containing 34—37%  $P_2O_5$ . For the evaporators Pb, hard Pb, and Cu (Pb-covered) are the constructional materials. The products are nodulised by repeated recirculation; the overall conversion efficiency is 91—93%  $P_2O_5$  and 99%  $N_2$ . C. I.

**Importance of sulphates as fertilisers.** G. BERTRAND and L. SILBERSTEIN (Compt. rend., 1932, 195, 1349—1352, and Chim. et Ind., 1933, 29, 255—257).—Comparative yields of rape grown in (a) a natural soil poor in S (0.114 g. per kg. of which 0.013 was rendered insol. by the Ba present); (b) as (a) but with addition of enough Ba to render all S insol.; and (c) as (a) but with addition of 0.250 g. per kg. S as  $Na_2SO_4$ , the same amounts of P, K, and  $NO_3$  being added in all cases, were as (a) 100 : (b) 59.7 : (c) 183.3, thus demonstrating clearly the need for the presence of  $SO_4$ " (cf. B., 1927, 951; A., 1930, 262). C. A. S.

**Hygroscopicity of fertiliser salts: reciprocal salt pairs.** A. R. MERZ, W. H. FRY, J. O. HARDESTY, and J. R. ADAMS (Ind. Eng. Chem., 1933, 25, 136—138; cf. B., 1929, 448).—When relatively little  $H_2O$  is added to the stable salt pair at a given temp. that salt of the reciprocal pair which is less sol. in the saturated solution of the stable pair forms the third solid phase, and the solution formed is that of highest v.p. Addition of  $H_2O$  to the unstable salt pair brings about double decomp., the third salt remaining depending on the proportions of the original mixture. Where solid solutions are formed there may be one or no triple-salt point; the solution formed is that of max. solubility (i.e., max. hygroscopicity). Where double salts exist there may be 3 or more triple-salt points. Stable salt pairs and v.p. are given for saturated solutions of various fertiliser salt pairs at 30°. The hygroscopicity no. is expressed by  $100 - R.H.$  of air in equilibrium with the saturated solution. The hygroscopicity of mixtures is usually  $>$  that of any salt in the solid phase. Where one of the stable salt pair is a non-fertiliser salt, the highest v.p. alone is of importance; in the converse case the lowest v.p. may determine the hygroscopicity of the system. J. S. A.

**Fertiliser value of dried leaves of fruit trees.** A. V. DAHLBERG (Fertiliser Green Bk., 1932, 13, No. 10, 22—23).—Analyses of dried prune, peach, and apple leaves are recorded. CH. ABS.

**Organic manure from sewage, town refuse, and waste vegetation.** J. J. RAO and V. SUBRAHMANYAN (J. Indian Inst. Sci., 1932, 15A, 89—106).—Mineral starters, e.g.,  $NaNO_3$ ,  $(NH_4)_2SO_4$ , and  $CaCN_2$ , have no beneficial effect on the rate of decomp. or manurial val. of composts of town refuse and sewage. The best results are obtained with activated sludge, there being

evidence of the fixation of atm.  $N_2$  in this case, but in all others a definite loss. The rotting process requires about 70 days and offensive odours and insects can be controlled by mixing the compost and spraying with Bordeaux mixture or bleaching powder. The microflora present are largely fungi, but the use of the Cu mixture suppresses these in favour of *Actinomyces* without affecting the rate of decomp. or the manurial val. of the final product. Pot and field trials indicate that this material is superior in manurial val. to either farmyard manure or combined chemical fertilisers. Large quantities at a reasonable cost are readily obtainable in India, especially as waste vegetation, e.g., leaves, can be incorporated with advantage. When nightsoil is used for this purpose it is advisable to permit some liquefying action to take place and to dil. with sewage before use. A mixture of activated sludge and refuse rapidly dewateres the former and decomposes the latter and produces an inoffensive humus. C. J.

**Soil-absorbing complex, plant fertilisers and soil improvement.** K. K. GEDROIZ (Khim. Sotzial. Zemled., 1932, 1, No. 1, 40—49).—A discussion of the relation between the soil and the plant, the absorbing complex being regarded as the seat of fertility. There is a definite relation between Ca and Mg with respect to plant growth. CH. ABS.

**Effect of physiologically acid fertilisers on podsol soils and their availability to plants.** A. T. KIRSANOV (Khim. Sotzial. Zemled., 1932, 1, No. 2, 17—24).—By proper manipulation, acid fertilisers, e.g.,  $(NH_4)_2SO_4$ , on podsol soils may mobilise other plant foods and improve crop yields. CH. ABS.

**Yield law of plants.** A. RIPPEL and R. MEYER (Z. Pflanz. Düng., 1932, 27A, 257—302).—The nature of the yield curves of plants varies with the species, the medium in which the plant grows, and the relative proportions of the nutrients. The effect of these factors on the position of the point of max. yield is examined and the importance of the period of harvesting in comparative tests is emphasised. The slope of the descending (toxic) portion of the growth curve as affected by increasing salt concns. in the nutrient differs with the plant species. The "Anstiegstangente" of the growth curve (i.e., the limiting val. of the tangent to the growth curve as the nutrient factor approaches zero) is different for various species, but is the same for different varieties of the same plant; also for the same species of plants in a similar stage of maturity it is largely independent of external conditions. The val. of the tangent decreases as the period of max. yield is shortened. A. G. P.

**Effect of ammonium sulphite on plant growth.** A. H. LEWIS and F. B. MARMOY (J. Agric. Sci., 1933, 23, 1—5).—In pot cultures, additions of  $(NH_4)_2SO_3$  [up to 1% of the  $(NH_4)_2SO_4$  used] did not reduce the germinative capacity of seeds or the growth and dry matter yields of mustard or rye. A. G. P.

**Manuring of meadow-hay land. Effect of manures and lime on botanical composition and yield of hay.** W. A. JACQUES (J. Agric. Sci., 1933, 23, 146—160).—Better crops were obtained by manuring with dung than with artificial fertilisers.  $NaNO_3$  was more effective than  $(NH_4)_2SO_4$ . Crop increases due to liming

were greater on plots receiving artificial fertilisers than on those dressed with dung. Continuous use of  $(\text{NH}_4)_2\text{SO}_4$  with or without CaO did not improve the botanical composition of the herbage. Continuous use of  $\text{NaNO}_3$  reduced bent and sweet vernal grasses and increased the proportion of cocksfoot. Dung produced a better hay than complete artificial fertilisers and when used repeatedly reduced the proportion of undesirable plants in the herbage, in comparison with other treatments.

A. G. P.

**Establishment of pasture on virgin peat.** D. CLOUSTON (Scot. J. Agric., 1932, 15, 280—286).—The scirpus peat described may be reclaimed by thorough preliminary drainage and the application of complete fertilisers together with CaO, without which fertilisers are ineffective.

A. G. P.

**Investigations on irrigated pastures. I. Yield and botanical composition of an irrigated permanent pasture under various systems of pasture management.** A. E. V. RICHARDSON. **II. Chemical composition of irrigated pastures at Wood's Point, S. Australia.** H. P. C. GALLUS (Counc. Sci. Ind. Res., Australia Bull., 1932, No. 71, 45 pp.).—I. Mowing at 28-day intervals throughout the year produced high dry-matter yields the composition of which approached that of a protein-rich concentrate. Yields were still greater under a 28-day rotational grazing system. By prolonging the growth period to 56 days the yield of pasture was increased and its nutrient ratio was more suitable for fattening sheep. Applications of superphosphate increased yields throughout the year.  $(\text{NH}_4)_2\text{SO}_4$  increased more particularly the bi-monthly cut immediately following its application.

II. The average N and P contents of irrigated pasture were high throughout the year, winter vals. being rather higher than those of summer. Differences in yield and botanical composition between mown and grazed pasture were reflected in their chemical composition. Grass mown at 56-day intervals had higher N and P contents than that mown every 28 days.

A. G. P.

**Relation between carbon dioxide and elemental nitrogen assimilation in leguminous plants.** P. W. WILSON, E. B. FRED, and M. R. SALMON (Soil Sci., 1933, 35, 145—165).—Clover plants grown in an atm. containing artificially increased amounts of  $\text{CO}_2$  (up to 0.1%) produced larger leaves, longer tops, more stocky growth, more numerous nodules, and had higher carbohydrate and fixed N contents. Amounts  $> 0.1\%$   $\text{CO}_2$  did not cause correspondingly greater development. In plants grown in agar with 0.1%  $\text{CO}_2$ , the increased N fixed was proportionally  $<$  the increased dry matter produced. This disproportionality was not apparent in sand cultures until the  $\text{CO}_2$  content was raised to 0.4%. Nodules were larger on tap roots and tended also to invade the laterals with the higher concns. of  $\text{CO}_2$ .

A. G. P.

**Statistical study of nitrogen fixation by clover plants.** P. W. WILSON, P. WENCK, and W. H. PETERSON (Soil Sci., 1933, 35, 123—143).—Interrelationships between the amounts of N fixed, the no. and distribution of nodules, and the growth characteristics of clover plants in sand and agar cultures are examined. A. G. P.

**Influence of rye and oat straws on growth and yield of certain vegetables.** B. E. GILBERT and F. R. PEMBER (Soil Sci., 1933, 35, 115—122).—The incorporation with soil of the resistant org. matter of straws retarded the growth of a no. of crops, the effect being most marked where the N content of the straw was low. Applications of N fertilisers to remedy this defect would need to be far above the economic limit.

A. G. P.

**Relation of caliche to desert plants.** F. SHREVE and T. D. MALLERY (Soil Sci., 1933, 35, 99—113).—The formation of caliche (a calcareous hardpan common in south-western United States) is attributable to interrupted penetration of rain  $\text{H}_2\text{O}$  under arid conditions. Even very thin layers of caliche retard the movement of  $\text{H}_2\text{O}$  in soils. The growth of *Larrea (corvilleda) tridentata* in mixtures of sand or clay with caliche was inversely related to the amount of caliche present. The latter did not appreciably affect the Mg or Ca contents of the plants. The effect of caliche on plant growth is related more closely to the physical than to the chemical properties of the soils. A. G. P.

**Effect of sulphur compounds in breaking the dormancy of potato tubers and in inducing changes in enzyme activities of treated tubers.** L. P. MILLER (Contr. Boyce Thompson Inst., 1933, 5, 29—81).—A no. of S compounds examined broke the dormancy period of freshly harvested potatoes. In addition to this property  $\text{Me-CS-NH}_2$  (I) differed from all other compounds in retarding the sprouting of non-dormant tubers. Potatoes absorbed considerable amounts of (I), but actual sprouting did not occur before (I) had disappeared from the tissues.  $\text{KCNS}$  and  $\text{CS}(\text{NH}_2)_2$  were also absorbed, but did not delay sprouting. Treated tubers showed increases in catalase and peroxidase activity to extents varying in the order thiosemicarbazide  $>$   $\text{H}_2\text{S}$  and  $\text{EtSH}$   $>$   $\text{NH}_2\text{-CS}_2\text{-NH}_2$  (II) and (I). The same order of effect occurred in treated non-dormant tubers, but the actual increases in activity were smaller. These enzymic changes occurred within 24—48 hr. after treatment. The breaking of dormancy and stimulation of the activity of the above enzymes were not parallel phenomena. The S compounds did not increase enzyme activity in expressed potato juice. Juices from treated tubers show a slightly raised  $p_{\text{H}}$  val. and in the whole tubers there was an increased sugar content and respiratory activity. Tubers treated with (II) contained considerable amounts of CNS'.  $\text{Me}_2\text{S}_2$  and  $\text{Et}_2\text{S}_2$  are readily reduced to the corresponding mercaptans by the tissues. All S compounds examined reduced the tyrosinase activity of potato juice.

A. G. P.

**Influence of artificial reduction of leaves on the yield of sugar beets.** N. I. ORLOVSKI (Nauk. Zapiski Tzuk. Prom., 1932, No. 21—22, 227—237).—Removal of leaves reduces the sugar yield.

CH. ABS.

**Climatic and soil characteristics in relation to sugar cane agriculture in Central America.** F. SALDAÑA-DÁVILA (Internat. Sugar J., 1933, 35, 27—29).—Factors which contribute to the remarkable conditions of cane cultivation in parts of Central America often at altitudes of 2000 ft. are stated. In some parts

juice densities of 21—22° Brix and purities of 95° are common, and upwards of 50 tons of cane per acre can often be obtained from plant cane which has received moderate attention. J. P. O.

**Damage [to plant life] by effluent gas and smoke, and its prevention.** H. WISLICENUS (*Angew. Chem.*, 1933, 46, 104—105).—The problem, in its relation to plant life, is reviewed, and means of removing SO<sub>2</sub> etc. are discussed. Damage will probably be minimised in the future by the centralisation of the offending factories in large electric power and gas-producing stations, and the gradual elimination of coal furnaces in the smaller factories. H. F. G.

**Factors affecting the efficiency of contact insecticides. III. Further chemical and toxicological studies of pyrethrum.** F. WILCOXON and A. HARTZELL (*Contr. Boyce Thompson Inst.*, 1933, 5, 115—127; cf. B., 1932, 441).—Methods of purification of pyrethrins and for the partial separation of pyrethrins I and II are described. Pyrethrin I is more toxic than II to *Aphis rumicis*. The toxicity of pyrethrum flowers is slightly > that of purified extracts. External application of pyrethrins, under conditions in which no tracheal penetration is possible, results in the intoxication and death of certain insects. Actual penetration of the tissues is demonstrated by the use of stains. A. G. P.

**California red scale and its control in the Lower Rio Grande valley of Texas.** S. W. CLARKE and W. H. FRIEND (*Texas Agric. Exp. Sta. Bull.*, 1932, No. 455, 35 pp.).—Fumigation with HCN effectively kills the insects, but reinfestation was fairly rapid. Best results were obtained with oil emulsions, quick-breaking types made with slow-drying oils being more effective than those with rapidly-drying oils. A. G. P.

**Vegetable weevil (*Listroderes obliquus*).** O. H. LOVELL (*Calif. Agric. Exp. Sta. Bull.*, 1932, No. 546, 19 pp.).—Best control in carrot, turnip, or spinach is obtained by dusting with Na<sub>2</sub>SiF<sub>6</sub> or BaSiF<sub>6</sub> (30—40 lb. per acre). A. G. P.

**Superphosphate.**—See VII.

See also A., Mar., 244, **Determining K [in soil extracts]**. 247, **Soil microscope**. 253, **North-western peat and muck soils of U.S.A.** **Acid-alkali coeff. of peat.** **American loess soils.** **Climate, clay composition, and soil type.** **Illinois soils.** 254, **Soil-profile examination.** **Geology and soils of Perak.** **Profiles of drift soil in north-east Scotland.** 328, **Mineral contents of Egyptian cereals.**

#### PATENTS.

**Manufacture of mixed fertilisers containing ammonium nitrate which are stable when stored.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 382,368, 5.9.31).—The fertilisers, especially (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>-NH<sub>4</sub>NO<sub>3</sub>, in granular form are sprayed with fused (preferably eutectic) mixtures of Mg(NO<sub>3</sub>)<sub>2</sub>, alkali nitrates, and, if desired, alkaline-earth nitrates and/or urea; e.g., a mixture, m.p. 138°, of 54 pts. of Mg(NO<sub>3</sub>)<sub>2</sub>, 44 pts. of NaNO<sub>3</sub>, and 2 pts. of Ca(NO<sub>3</sub>)<sub>2</sub> may be added. L. A. C.

**Production of fertilisers [from ammoniacal gas liquors].** C. J. HANSEN, Assr. to KOPPERS Co. (U.S.P. 1,855,856, 26.4.32. Appl., 7.2.30. Ger., 1.7.29).—The liquors containing (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, NH<sub>4</sub>CNS, and free NH<sub>3</sub> are heated with H<sub>3</sub>PO<sub>4</sub> at 180° in an autoclave to yield (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> with the separation of liquid S. A. R. P.

**Manufacture of phosphate-silicate fertiliser.** L. KERN (U.S.P. 1,855,190, 26.4.32. Appl., 31.10.25. Renewed 15.7.30).—Coarsely ground phosphate rock is mixed with an equal wt. of crushed CaO and 3—6 times its wt. of kieselguhr containing 50—80% H<sub>2</sub>O, which slowly slakes the CaO, whereby the rock is converted into Ca phosphate-silicate which serves as a source of readily assimilable P<sub>2</sub>O<sub>5</sub> and as a suitable fertiliser for acid soils. A. R. P.

**Manure.** R. A. G. YOUNG (B.P. 387,694, 4.8.31).—Poultry droppings mixed with a drying agent, e.g., gypsum, are dried in a current of hot air and ground. L. A. C.

**Thomas slag.**—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Storing sugar beets for a long period.** A. M. PSHENICHNUI and G. S. ZISMAN (*Nauk. Zapiski Tzuk. Prom.*, 1932, No. 21—22, 45—50).—Beets were preferably piled and covered with earth alone; daily losses of sugar were less in winter than in autumn, and increased in early spring. Treatment with SO<sub>2</sub> prevents deterioration of sugar in beets during storage. CH. ABS.

**Colloids of sugar beets during prolonged storage and manufacture.** M. Z. KHELEMSKI and E. P. KOSHEVEROVA (*Nauk. Zapiski Tzuk. Prom.*, 1932, No. 21—22, 57—63).—No const. relation was found between the amount of colloids and [H<sup>+</sup>], viscosity, surface tension, reducing substances, or purity of juices. From December to April there is a continuous increase (69%) of colloids in diffusion juice. CH. ABS.

**Production of sugar from dried beet.** ANON. (*Internat. Sugar J.*, 1933, 35, 36).—A syrup of 50° Brix extracted directly from dried beet does not filter easily without previous liming. After liming, however, it is found that a syrup of 35° Brix will filter without undue rise of pressure in the filter-press, and that if the filtrate be conc. up to 50° Brix by using it to extract further dried beet this conc. syrup can be limed and filtered without trouble. Unlimed, filtered syrup at 50° Brix or near was neutralised with NaOH, and the brilliant liquor boiled to a massecurite, the high-green syrup obtained from it being defecated with lime, filtered, and re-boiled. On machining these two massecurites, an off-colour white sugar was obtained, which was of a sufficiently good quality for sale to the confectionery and bakery industries. J. P. O.

**Amount of sugar which can be extracted from the cut surfaces of sugar beets.** F. KRYŽ (*Z. Zuckerind. Czechoslov.*, 1933, 57, 165—167).—From roots containing 18.5% of sugar, the sugar extractable from a cut surface is 0.004 g. per sq. cm. Using this figure as a basis and estimating the average exposed surface of topped roots arriving at the factory, which

have undergone the average amount of injury due to broken-off tails, to breakages, and to other damage, it is found that on the average about 0.034 pt. of sugar per 100 pts. of intact topped roots can be washed out during transport in the flumes. J. P. O.

**Flocculation of beet juice.** J. BARBAUDY (Bull. Assoc. Chim. Sucri., 1933, 50, 9—12).—Juices expressed from pulped beets and diluted 8-fold showed a point of optimum flocculation about  $p_H$  3.2, measured 24 hr. after the addition of acid. For juice diluted only 2-fold the optimum was  $p_H$  4.35, agreeing well with vals. obtained by other workers with fresh undiluted juices. Fresh juice diluted 4.5-fold, but not treated with acid, showed  $p_H$  6.65, which changed to  $p_H$  5.67 in 24 hr. and reached the optimum flocculation point,  $p_H$  3.38, in 7 days. J. H. L.

**Rapid refractometric method for approximate determination of sugar in carbonation scums.** V. STANĚK and K. ŠANDERA (Z. Zuckerind. Czechoslov., 1933, 57, 145—148).—The method consists in determining refractometrically the amount of sugar in carbonation press-cake. It is accurate to about  $\pm 0.3\%$  of sugar. J. P. O.

**The Oliver-Campbell filter compared with the plate-and-frame press in the cane-sugar factory.** W. A. POWE (Internat. Sugar J., 1933, 35, 33—35).—Advantages claimed for the Oliver-Campbell as compared with the plate-and-frame filter for handling cane muds are: lower first cost for equipment to deal with the same amount of material; lower building cost; less filtering area; lower annual cost of filter medium; saving in labour; lower sugar losses; less wash- $H_2O$ ; and lower power consumption. In addition the former filter offers a clean station, and easy operation and control. Whole-juice filtration can be accomplished in a pressure leaf filter using "Hyflo-Supercel" as filter aid (0.2 kg. per metric ton of cane) and allowing for 0.1 sq. m. of filter area per metric ton of cane. Evaporator syrup can best be filtered in leaf-type pressure filters, such as the Sweetland, using 0.3—1 kg. of "Hyflo" and 0.15—0.2 sq. m. of filter area per metric ton of cane. J. P. O.

**Use of Lafeuille rotary crystallisers at Carlota [cane-sugar factory, Philippines].** W. PATERSON (Bull. Assoc. Chim. Sucri., 1932, 49, 427—435; from Sugar News).—Two 500-hectolitre Lafeuille crystallisers (B., 1926, 928, 992; 1929, 499) were used for low-grade massecuites in the 1931—2 campaign, concurrently with 28 ordinary ones of U-type. The grain for a Lafeuille charge was formed with factory syrup in a vac. pan, fed with run-offs of 48—49% purity, and then transferred at 93° Brix to the Lafeuille already under vac. and fed with run-offs of 70—75° Brix and 49% purity, during vigorous mixing and heating by steam at 4—8 lb./sq. in. until the charge amounted to 380 hectolitres. After concn. to 98° Brix heating was discontinued for 15—20 min. and then cold  $H_2O$  was circulated, slowly at first, and the charge cooled to 42° in 4 hr.,  $H_2O$  being added as required to give a final massecuite of 97° Brix. The cooled charge was at once reheated to 66—68° in 2½ hr. to facilitate centrifuging. The whole series of operations occupied 15—18 hr., compared with 5—6

days in ordinary crystalliser work. Centrifugal capacity was increased 2—3-fold owing to the reheating to 66—68° and practically no sugar was redissolved thereby. The purity of the final molasses was slightly lower than that attained in ordinary working. Other advantages were the complete control of temp. and Brix rendered possible by the Lafeuille plant. J. H. L.

**Incrustations in evaporators [in beet-sugar factories].** J. VONDRÁK and T. NEMES (Z. Zuckerind. Czechoslov., 1933, 57, 153—159).—Examination of the analyses of 79 samples of evaporator incrustations from Czechoslovakian beet-sugar factories showed the predominating constituent to be carbonates in 15 cases, magnesia in 5,  $SO_2$  in 2,  $SO_3$  in 2, and  $SiO_2$  in 27. It is concluded that, in general, scale formation in these evaporations is not due to any defective manipulation, but is unavoidable. J. P. O.

**Practical calculation of the amount of molasses in the raw [beet] factory.** A. DOLINEK (Z. Zuckerind. Czechoslov., 1932, 57, 135—136).—Examples are given of formulæ utilising the conductivity ash for the calculation of the expected molasses (per 100 roots), one being as follows, in which the sugar content of the molasses is assumed to be 50% and the yield  $0:10G_p A_d/P_d$ , in which  $G_p$  = total production of polarisation sugar per 100 roots,  $A_d$  the ash content of the thick-juice, and  $P_d$  the polarisation of the thick-juice. J. P. O.

**Determination of sucrose in final molasses.** F. M. FERNANDEZ (Sugar News, 1932, 13, 477—482).—Using dry basic Pb acetate for clarification, the following two modifications gave almost identical results with factory final molasses: (1) both direct and invert readings in the presence of Pb; (2) direct reading after removal of the Pb, and invert reading in the presence of Pb. These results did not differ much from the modification in which both direct and invert readings are taken after removal of the Pb. J. P. O.

**Potash recovery from molasses.** H. S. HARKNESS (Internat. Sugar J., 1933, 35, 42—43).—A furnace which was used in a Hawaiian factory for the incineration of molasses is described. A recovery of 2.5—3.0% of ash (on the molasses) was obtained, the ash containing  $K_2SO_4$  32, KCl 21.1, and  $K_2CO_3 + KOH$  3.1%, in all about 35%  $K_2O$ . J. P. O.

**Utilisation of final molasses and filter-press mud.** M. S. FILOSOFOV (Nauk. Zapiski Tzuk. Prom., 1932, No. 21—22, 121—123).

**Analysis of mineral content of Steffen's waste [from sugar beet].** E. BARTOW and W. M. BENNINGHOFF (Ind. Eng. Chem., 1933, 25, 175—177).—Samples from various parts of the United States were saturated with  $CO_2$  to remove Ca and evaporated, ashed, and the ash was analysed. The yield of concentrate from the dil. waste is 1—5% and the ash contains on an average about 34% K. The K and Na contents do not vary with the geographical origin of the waste, though the  $SO_4$  and Cl<sup>-</sup> contents do. C. I.

**Rapid approximate determination of invert sugar in raw [beet] sugar.** A. DOLINEK (Z. Zuckerind. Czechoslov., 1933, 57, 190—192).—Modifying the method

of Staněk and Pavlas (B., 1931, 1154), the author employs a reagent containing 10.98 g. of  $K_3Fe(CN)_6$ , 37.6 g. of  $K_2CO_3$ , and 0.1 g. of methylene-blue per litre; it should be < 14 days old and shaken before use as the indicator is not completely dissolved. A solution of 16 g. of sugar sample in 50 c.c. of  $H_2O$  is heated in a small Erlenmeyer flask, and 15 c.c. of the reagent are heated separately. As soon as the sugar solution begins to boil, the hot reagent is added, followed by a 10 c.c. rinsing of hot  $H_2O$ . Boiling is continued, and the time required for decolorisation is measured from the addition of the reagent (not the rinsing). The following data, selected from the author's table, show % invert sugar in sugar samples, corresponding to different times required for decolorisation: for 29, 34, 40, 58, 68, 85, 135, 220, and 365 sec., 0.22, 0.17, 0.14, 0.10, 0.09, 0.08, 0.07, 0.06, and 0.05%, respectively. J. H. L.

**Influence of solution concentration on accuracy of the conductometric determination of the ash content of raw [beet] sugars.** B. LAZAR (Z. Zuckerind. Czechoslov., 1932, 57, 129—135).—Conductivity determinations were carried out with solutions containing 26, 13, 6.5, and 5 g. of raw beet sugar per 100 c.c., but the differences in the results for the conductometric ash between the high and low contents were practically negligible. In the presence of  $H_2C_2O_4$  considerable irregularities were observed in the results for the conductometric vals., compared with those obtained in the presence of HCl. J. P. O.

**Use of pure glucose for standardising Fehling's solution.** H. MAIN (Internat. Sugar J., 1933, 35, 43).—In using an invert sugar solution for the standardisation of Fehling's solution, there is no means of checking its accuracy other than by titration against a Cu solution. A solution of glucose, the purity of which can readily be controlled by polarisation, is therefore recommended. J. P. O.

**Refining of sugar directly from beet sugar without recrystallisation.** I. F. ZELIKMAN, A. I. SICHARENKO, and A. V. VELICHKEVSKI (Nauk. Zapiski Tzuk. Prom., 1932, 21—22, 65—95).—Granulated sugar is obtained when ripe, healthy, non-stored beet is worked; the yield from massecuite of colour  $\gt$  10 Stammer units is 40—45%. Important factors in manufacturing refined sugar directly from beet juice without recrystallisation are: acceleration of the process (especially where high temp. is maintained), replacement of the second carbonation by sulphitation and filtration, application of activated carbons and three-strikes massecuite, and boiling with careful separation of run-offs. CH. ABS.

**Dampening of white sugar in the store.** J. VONDRÁK (Z. Zuckerind. Czechoslov., 1933, 57, 159—160, 167—168).—Such dampening may be prevented by heating or ventilating the building so as always to maintain the temp. of the sugar above dewpoint. Any considerable fall of temp. is to be avoided. Hygroscopicity measurements must constantly be observed inside and outside the store, and the temp. of the sugar in the interior of the bags must periodically be read. J. P. O.

**Spontaneous ignition of sugar.** A. BRÜNING (Centr. Zuckerind., 1932, 40, 612—614).—A case of

spontaneous ignition of powdered sugar in a chocolate factory is reported. It occurred in a large sheet-Fe box under the sugar-mill, the temp. of the sugar in which did not exceed 47°. Attempts were made to induce spontaneous ignition of sugar at this temp., but without effect, excepting, however, when it was mixed with 1% of floor sweepings. J. P. O.

**Determination of inulin and lævulosin applied to the analysis of coffee substitutes and to the question of caramelisation.** C. I. KRUISHEER (Chem. Weekblad, 1933, 30, 154—166).—Inulin is completely broken down into its constituents by warming with 3% HCl at 68—70° for 10 min.; the fructose content, however, is not exactly equal to the inulin because the mol. wt. of the latter varies according to its origin. Pure inulin always contains a notable proportion of combined glucose. From a study of the results of progressive hydrolysis supplemented by fermentation, it has been found that commercial samples of inulin and samples prepared from fresh chicory root, dried root, *Helianthus tuberosus*, dahlia, and ox-eye (*Buphthalmum*) contain about 10% of combined glucose, which can be reduced only to 2.5% by repeated repptn. from EtOH and/or baryta. During the roasting process to produce "peekoffie" and chicory the inulin is partly broken down to form lævulosin and the time for complete inversion is increased to 3 hr., although the roasting takes place under virtually anhyd. conditions in absence of acids, and small amounts of fructose may also be formed. The ratio of fructose to glucose after hydrolysis falls from 10:1 to 4:1, but is still sufficiently high to serve for the detection of roasted inulin products in coffee. Hydroxymethylfurfuraldehyde (I) is formed to 2% with traces of furfuraldehyde. The latter may be determined by steam-distilling a neutral  $Et_2O$  extract and pptn. of furfuraldehyde in the distillate and (I) in the residue as phloroglucides (cf. B., 1930, 80). Roasted coffee and pure coffee extracts contain no ketoses or lævulosin, aldoses are present as polymerides, and the  $Et_2O$  extract contains only traces of (I). Hence chicory can be detected, after hydrolysis, by excess of fructose over glucose; burnt sugar, by lævulosin and an excess of glucose over fructose. A study of the caramelisation process shows that when sucrose is heated several reactions take place simultaneously. In the primary stages not only glucose and difructose anhydrides, but also fructose and diglucose anhydrides and internal anhydrides, are formed. Lævulosan (A., 1921, i, 544) is completely hydrolysed by 3% HCl in 10 min. at 70°, whilst lævulosin (B., 1930, 1043) requires 3 hr. Wohl's lævulosin is a mixture with lævulosan; so also is *iso*-saccharosan (A., 1924, i, 1045, 1046) containing lævulosin and glucosin, glucosan (A., 1920, i, 594), caramelan, and caramelen. In the caramelisation of sucrose the glucosan residues are fairly stable to heat; the degradation of the fructose residues is responsible for the formation of caramel. Hence glucose heated alone does not caramelize, but this can be brought about in technical practice at a lower temp. by heating with alkali, when more profound decomp. occurs of the type when alkaline solutions of monosaccharides are heated, lævulosin and glucosin being absent. S. C.



**Preparation of rye starch.** A. SCHULERUD (Z. ges. Getreide-Mühlenw., 1932, 19, 140—141; Chem. Zentr., 1932, ii, 2750).—The purest starch is obtained by treatment of the meal for 1—2 hr. with 40% EtOH and purification of the centrifuged starch with H<sub>2</sub>O, 70% EtOH, and 96% EtOH. The resulting starch contains 0.12% ash and 0.73% protein. A. A. E.

**Hemicelluloses [and saccharification]. [Sugars by] hydrolysis of wood.**—See V. **Caking of sugar.**—See VII. **Leaf removal and sugar-beet yield. Sugar cane agriculture.**—See XVI. **Determining sucrose in beer.** COMe<sub>2</sub>-BuOH fermentation.—See XVIII. **Determining sugars in condensed milk. Honey and diastase.**—See XIX. **Beet-factory waste-water.**—See XXIII.

See also A., Mar., 211, **Polarimetry of sugars.** 260, **Analysis of individual carbohydrates. Products of oxidation of sugars. Determination of fructose and sorbose. Synthesis of sucrose.** 261, **Electrolytic prep. of aldonic acids. Action of aq. alkali on starches etc. Acetolysis of starch. Prep. of sol. starch.**

#### PATENTS.

**Betaine hydrochloride.**—See III. **Products from sugar cane.**—See V.

### XVIII.—FERMENTATION INDUSTRIES.

**Electrodialysis of amylase solutions.** H. LÜERS and R. LECHNER (Woch. Brau., 1933, 50, 49—51).—Malt extract, papain malt extract, and an aged malt extract showed after electrodialysis a 6—12-fold increase in saccharifying power, whilst the liquefying action decreased and, in the case of EtOH-pptd. green malt extract, disappeared almost completely; the saccharifying action of barley amylase was not increased. The N content remained unchanged, and the ash content almost disappeared. The acidity of the enzyme solution increased during electrodialysis, and this was shown to favour the destruction of the liquefying enzyme. The collected results of the electrodialysis, normal dialysis, and fractional pptn. of amylase solutions indicate that the saccharifying and liquefying powers are separate functions of a  $\beta$ -saccharogen and possibly an  $\alpha$ -dextrinogen amylase, respectively. R. H. H.

**Process for producing absolute alcohol by azeotropic distillation, using trichloroethylene.** R. FRITZWEILER and K. R. DIETRICH (Internat. Sugar J., 1933, 35, 29—32, 71—74).—In the above process C<sub>2</sub>HCl<sub>3</sub> is used as the dehydrating agent, the dehydration efficiency and decantation efficiency of which are superior to those of C<sub>6</sub>H<sub>6</sub>, petrol, or mixtures of these. It follows that the layer of high H<sub>2</sub>O content contains far less dehydrating agent and occupies less vol. than the materials at present used without the dehydration being substantially inferior. Plant is described for the dehydration of high-proof EtOH. Starting with 94° spirit, the steam consumption is stated to be 98 kg. per hectolitre of EtOH at 99.8—100% produced. Some particulars are also given of the production of abs. EtOH by this process directly from mash. J. P. O.

**Acetone-butyl alcohol fermentation.** V. N. SHAPOSHNIKOV, A. Y. MANTEIFEL, and F. M. CHISTYAKOVA (Bull. Nauch.-Issledov. Khim.-Farm. Inst., 1931, 75—77).—The relatively highest yield of COMe<sub>2</sub> (7.3%) was obtained, for maize, at 8.5% maize. The consumption of starch parallels its hydrolysis, the fermenting liquid always containing sugar. Fermentation with a high concn. of flour always shows the same final concn. of sugar (0.18—0.28%), in spite of the fact that starch is entirely consumed, making the increase in yield of COMe<sub>2</sub> appear disproportionate to the amount of starch consumed. CH. ABS.

**Mechanical equipment for continuous fermentation of fibrous materials.** A. M. BUSWELL and C. S. BORUFF (Ind. Eng. Chem., 1933, 25, 147—149).—Fermentation of maize stalks, sewage screenings, etc. is checked if the material is allowed to collect at the surface of the tank as a mat suspended by gas bubbles. Good results are obtained by the use of a drum, with feed and discharge at opposite ends, which is rotated twice a day. With this apparatus up to 2.1 kg. of the stalks per cu. m. of tank capacity were fed per day, giving an average gas production of 42% of the tank capacity per day. The gas contained 52% of CH<sub>4</sub>. Over-feeding results in the accumulation of org. acids. The NH<sub>3</sub>-N content of the liquor should be <100 p.p.m. and the temp. 25—30°. C. I.

**Relation between sugar content and attenuation.**

**II. Sugar content of attenuated beers.** W. PIRATZKY (Woch. Brau., 1933, 50, 51—54; cf. B., 1932, 443).—The method of sugar determination described earlier (B., 1932, 443) is applied to attenuated beers. Two general equations applicable to the fermentation of all pure-malt worts are derived, which indicate that (1) residual sugar content (as maltose) expressed as % residual extract is a const. (21.7), and (2) extract fermented = sugar fermented. The extract of attenuated beer always exhibits the same reducing power to Fehling's solution, viz., 25—26 (maltose = 100), and is composed of stable dextrin (75%) and maltodextrin (25%). A brewery bottom yeast ferments worts only until the existing maltose is consumed. R. H. H.

**Kolthoff-Kruisheer method for determination of sucrose in beer.** W. WŁOSTOWSKA (Przemysł Chem., 1933, 17, 18—22).—The beer is clarified and decolorised by Pb(OH)OAc and NaHCO<sub>3</sub>, the filtrate heated with HCl in order to invert sucrose, and fructose determined by Kruisheer's method (B., 1930, 388). The experimental error is >1.5%. R. T.

**Foam on beer.** G. KRAUSS (Woch. Brau., 1932, 49, 409—414, 417—421).—Factors favourable to the production and stability of foam on beer are: less acid  $p_H$  of the beer or wort; use of normal rather than overgrown, and warm rather than cold-stored malts; soft H<sub>2</sub>O containing small amounts of salts, e.g., Fe, Al, and Cu sulphates; low yeast dosage; blending of the beers; small casks for storage and higher bunging pressures. Stability increases with the amount of hop constituents in the beer, decreases on prolonged boiling of the hops, and varies with the age and strain. Humulone and total resin (without humulone) stabilise and increase the amount of foam. Maize as a malt substitute and

oxidation of the wort in the cooler are not unfavourable. Boiling of 50% of the total mash gives a better stability than 75%. Filtration of a cloudy beer improves the foaming ability, but decreases that of a bright beer. Clarification of the beer in the cask with chips, which have no effect on the foaming qualities, is therefore recommended. The use of dark beers and malts for colouring, conduction of the beer through long hoses, and exposure to light are also injurious. Use of bottles contaminated with grease, mineral oil, and soda injures the beer in every respect. Pasteurisation only slightly reduces the stability, but the injury increases with time and temp. of pasteurisation. R. H. H.

**Measurement of beer foam.** F. M. WIENINGER (Woch. Brau., 1932, 49, 273—278).—The amount and stability of the foam are determined by passing air at 0.3 atm. upwards through a porous glass filter-plate into a column of 200 c.c. of the beer at 10° in the apparatus described. Air pressure of 0.8 atm. is necessary for decarbonated beers. Compared with other processes the present method gives the greatest amount of foam with a very slow rate of subsidence. C. R.

**Biological cleanliness of brewery hosing.** I. JANENSCH (Woch. Brau., 1933, 50, 69—71).—Methods of sterilising brewery hoses and pipes, e.g., by hot-H<sub>2</sub>O brushing, steam, soda, formalin, are discussed and shown to be inefficient in many cases. The biological condition of the hose is determined by rinsing with sterile H<sub>2</sub>O which is incubated in hopped wort—gelatin, wort—tartaric acid, Stockhausen yeast extract, and pasteurised beer media. Hosing "disinfected" by brewery methods contained wild elliptical yeasts, torula, sarcinae, diplococci, moulds, bacilli, and bacteria. R. H. H.

**Enzymic treatment of sweet musts with the "Bayer" filtration enzyme.** A. MEHLITZ (Konservern-Ind., 1932, 19, 539; Chem. Zentr., 1932, ii, 2889).

**Determination of benzoic acid in Argentine wines.** J. TESTA (Requind, 1932, 1, 21).—The official German method has been modified. CH. ABS.

**Determination of citric acid in wine.** W. BARTELS (Z. Unters. Lebensm., 1933, 65, 1—37).—Methods depending on conversion into and determination of COMe<sub>2</sub> are compared with pptn. as citrates. A modification of Kogan's method, which may be used for red wines and in the presence of glycerin, is described, Ba citrate being pptd. by BaCl<sub>2</sub> in the presence of alcoholic NH<sub>3</sub> and distilled with AcOH and a min. excess of KMnO<sub>4</sub>, and the distillate titrated to starch with 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> in the presence of an excess of 0.1N-I. The 19 wines of various origins examined contained 0—29 mg. of citric acid per 50 c.c., and 92—102% of the 8—19 mg. per 50 c.c. added was determined by the method. Small quantities of citric acid produced during fermentation or derived from the grapes may be present as a natural constituent of wines. J. G.

**Occurrence of sodium chloride in wine.** J. L. MERZ (Wein u. Rebe, 1932, 14, 144—145; Chem. Zentr., 1932, ii, 2751).—Washing of vats with sea-water is mentioned. A. A. E.

**Baden white wines of the 1930—1932 season.** E. REMY (Z. Unters. Lebensm., 1932, 64, 548—553).—

The EtOH content of wine may be calc. from the abs. surface tension of the wine (*W*) from the formula  $(73 - W)/n$ , where *n* = (ratio of the abs. surface tensions of H<sub>2</sub>O and EtOH) — 0.2; the error is  $\pm 0.13$  g. EtOH/100 c.c. There is little difference between the abs. and relative surface tensions for the wine and its distillate. Full physical data and chemical analyses for 10 wines (71—94% EtOH) are tabulated. The mean As content was 0.5, and Cu 1.0 mg./litre; Ca/K was 9—17, and Ca/PO<sub>4</sub> 2.4—6.8. The low reduction val. by Tillmans' test (A., 1932, 310, 658) indicates negligible vitamin-C contents. J. G.

**Physical and chemical changes occurring during the storage of wine distillates on the large scale.** H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1933, 65, 208—220).—Storage of 40 and 60% Charente wine distillates in completely and in half-filled wooden casks for 3½ years in (a) a cool, well-ventilated, (b) a moist, and (c) a warm, dry cellar at 2—25° and 30—89%. R.H. produced a 6-monthly loss in cask-wt. of 0.4—1.0 kg., the losses being greatest for (b) and about equal for (a) and (c). The changes in EtOH content (+0.5 to +0.8 vol.-%) were similar in all 3 cases, but if calc. in litres/100 litres of pure EtOH showed losses of 0.4—6 kg. in 6 months, the greatest losses occurring in the half-filled casks and, except under conditions (b) where there was little seasonal variation, in summer. There were considerable decreases in aldehydes, increases in acids, slight irregular decreases in higher alcohols, and periodic rises and falls in esters. J. G.

**Detection and determination of aldehydes in spirits.** S. HÄHNEL (Svensk Kem. Tidskr., 1933, 45, 27—42).—The sample (previously distilled if sugar or similar material is present) is made up to 100 c.c. with H<sub>2</sub>O or MeCHO-free EtOH, so that the resulting solution contains 30% of EtOH. To 10 c.c. of this solution 3 c.c. of fuchsin bisulphite solution are added, and after keeping the whole at 15° for 45 min. the tint produced is matched in a Pulfrich photometer; the MeCHO content is given by the val. of the extinction coeff. of a 10-mm. layer of liquid. The accuracy is about 2% over a range of 10—100 mg. MeCHO per litre of the 30% EtOH solution. If smaller amounts of MeCHO are in question, the liquid should previously be conc. by distillation. H. F. H.

**Flavour of "Kasutorishōchū."** K. SHOJI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1933, 20, 87—102).—This Japanese whisky is obtained by steam-distillation of the press cake from fermented polished rice. Among the substances found to be present by polarographic and chemical studies were MeCHO, furfuraldehyde, Ac<sub>2</sub>MeOH, Pr<sup>o</sup>OH, tert.-amyl and higher alcohols, and various esters of valeric acid. E. B. H.

**Milk diastase. Origin of diastase.**—See XIX. See also A., Mar., 261, Prep. of sol. starch. 313, New oxidation enzyme. Determining starch-liquefying power of amylase. 315, Choline-esterase.

#### PATENTS.

**Rectification of alcohol.** USINES DE MELLE, and H. M. GUINOT (B.P. 387,589, 6.7.32. Fr., 6.7.31).—

Gasoline used as entrainer prevents the rise of fusel oil during rectification. A ternary azeotropic mixture of gasoline,  $H_2O$ , and  $EtOH$  free from fusel oil is withdrawn laterally from the top of the column. Volatile impurities are removed in the usual way. The  $EtOH$  is conc. by the azeotropic method and the entrainer removed in one operation, using a small amount of  $MeOAc$  as "uniter." R. H. H.

**Manufacture of sugar-free beer.** E. W. HARVEY (B.P. 387,639, 22.11.32).—Wort is fermented until free from fermentable sugar, a portion is conc. under vac. at  $49-54^\circ$ , and added to 12 vols. of the unconc. fermented wort. Saccharin or xylose is added, with or without flavouring or preservative, and pure  $EtOH$  is used to bring to the desired beer strength. R. H. H.

**Desizing of textiles.**—See VI.

### XIX.—FOODS.

**Chemistry in the flour industry.** A. J. HAMMER (Chem. and Ind., 1933, 52, 152—155).—A *résumé* is given of the position of cereal chemistry. Tests are required for wheat and flour to eliminate the baking test. T. McL.

**Rôle of the measurement of viscosity in mill chemistry.** T. RUEMELE (Z. Unters. Lebensm., 1933, 65, 199—207).—Graphs relating  $\eta$  and time (0—6 hr.) for solutions of 200 mg. of gluten in 75 and 100 c.c. of 0.02N-lactic acid at  $27^\circ$  show that for the best glutes (*e.g.*, garnet quality) the relation is linear, whilst for others  $\eta$  increases more rapidly in a given time, and a curve is obtained. The % difference between  $\eta$  for these 2 solvents decreases from 53 to 51 for garnet gluten, and increases from 25 to 52 for poorer glutes during 5 hr., and is therefore an indication of quality. Similar tests with 12 different flours showed that the ratio of these two  $\eta$  vals. gives a relative baking no. in close agreement with that derived from the increase in vol. and pore size (after Mohs and Neumann). A suitable viscosimeter is described. (Cf. B., 1931, 943.) J. G.

**Determination of maize in wheat flour.** W. S. SMIRNOW (Z. ges. Getreide-Mühlenw., 1932, 19, 141; Chem. Zentr., 1932, ii, 3029).—Flour (15 g.) is treated with 95—97%  $EtOH$  (50 c.c.) and kept for 1 hr. at  $75^\circ$ ; of the filtrate 10 c.c. are treated with  $N-NaOH$  (2 c.c.) and 5%  $CuSO_4$  (3—5 drops) and kept at  $75^\circ$  for 15 min. In presence of maize a deep reddish-violet colour, stable for 2—3 days, is produced. The method can be used quantitatively. The presence of rye or barley does not interfere. A. A. E.

**New pastry adulterants and their detection.** H. DILLER (Z. Unters. Lebensm., 1932, 64, 532—540).—Soya-bean flour (*cf.* following abstract) has been found (25—30%) in macaroni and may be detected from the high fat and N contents; rice, rye, and potato flours are detected microscopically. A common egg substitute is "heliocithin," or a yellow glutinous liquid (0.6% fat, 16% N, 80%  $H_2O$ , and 0.02% lecithin- $P_2O_5$ ). Soya-lecithin, which is also used for this purpose (B., 1931, 87), is a brown turbid oil (3%  $H_2O$ , 89% fat, and 0.9% lecithin- $P_2O_5$ ). The I val. of the light petroleum

extract is 100—110, and this provides a means (preferable to *n*) for detecting it in, *e.g.*, macaroni, the normal I val. of which is 77—81. J. G.

**Two vegetable flours.** D. KALTSCHewa (Z. Unters. Lebensm., 1932, 64, 540—545).—The prep., properties, and analyses in the raw and cooked state are detailed for Bulgarian soya-bean flour (*cf.* preceding abstract), and for pea flour (*Cicer arietinum*, L.):  $H_2O$  4.5, 2; N 39.4, 24.0; fat 24.5, 7.6; fibre 2.7, 1.7; ash 4.6, 2.3; lecithin 2.0, 1.6; I val. of oil 118, 114; sap. val. 184, 233, respectively. J. G.

**Loss of substance in the baking of bread.** K. EBLE and R. BRETSCHNEIDER (Z. Unters. Lebensm., 1932, 64, 554—555).—The  $P_2O_5$  content of the dry bread is a better indication of the ash and degree of milling of the flour used, and of the loss of substance on baking, than the  $NaCl$  determination suggested by Gerum (B., 1932, 365). J. G.

**Detection of permanently pasteurised milk.** R. STROHECKER and J. SCHNERB (Z. Unters. Lebensm., 1933, 65, 85—95).—The decolorisation times (*T*) in the usual form of the Schardinger (methylene-blue) test at  $60^\circ$  are: for raw milk 1—2.75, for milk pasteurised at  $63-65^\circ$  3—30, and at  $59-61^\circ$  1.75—4 min. *T* is unaffected by acidity, lowered by abnormally high fat contents, and, in the case of milks heated in the laboratory, increased on storage for 5—48 hr., although for milks heated in the dairy *T* first increases and then decreases after 48 hr. Additions of > 25% of pasteurised to raw milk are detectable, but after 24 hr. *T* gradually returns to the val. for raw milk. Sol.  $O_2$  in raw or heated milk raises *T*, but removal of it in a stream of  $CO_2$  or  $N_2$  brings *T* to a val. slightly lower than that obtained in air (*supra*). J. G.

**Detection of milk pasteurised by the holding method.** W. LAUE (Milch. Zentr., 1933, 62, 13—16, 29—33, 41—47).—200 determinations were carried out according to Hock's method (B., 1929, 995). Sound milk gave good results; diseased and sour milks showed abnormalities. E. B. H.

**Mercurimetric determination of chlorides in milk.** E. GEYER and A. ROTSCHE (Z. Unters. Lebensm., 1933, 65, 66—71).—Fats and proteins are pptd. with 20%  $Al_2(SO_4)_3$  and 2N- $NaOH$ , or with dil.  $AcOH$ , and 50 c.c. of the resulting serum are titrated with  $N/35.5-Hg(NO_3)_2$  in the presence of dil.  $HNO_3$  and  $Na$  nitroprusside until a bluish opalescence appears, the reagent being prepared from  $HgO$  in dil.  $HNO_3$  and standardised against  $N/35.5-NaCl$ . The results are higher than those obtained by Volhard's method, but confirm Mohr's method. J. G.

**The f.-p. depression constant of milk during a lactation period.** R. BAUER (Z. Unters. Lebensm., 1933, 65, 42—49).—Monthly determinations of *d*, fat, solids, *n*,  $\Lambda$ , f.-p. depression, and yields of milk are tabulated for the lactation periods of 15 cows. The f.-p. depression const. varies irregularly between 52.6 and 57.0, the variation for an individual cow being about 3 units. The val. for colostrum is 47.7 (fat 9.5—12.2, solids 19.7, *n* 39.0);  $\Lambda$  is lower than for ordinary milk (*cf.* B., 1928, 910; 1930, 738). J. G.

**Milk diastase.** H. KLUGE (Z. Unters. Lebensm., 1933, 65, 71—84).—A modification of Rothenfusser's method (B., 1931, 315, 1025) for the determination of diastase val. is described in which varying quantities of milk serum prepared by pptn. in succession with  $\text{Pb}(\text{OAc})_2$  and  $\text{NaCl}$  and buffered to  $p_{\text{H}}$  6.9 are digested with starch for 3 hr. at  $39^\circ$  and tested with I. Raw milk has a mean diastase val. of 1.312, which is lowered temporarily to  $< 0.292$  if permanently pasteurised and to  $< 0.146$  if heated momentarily at  $75^\circ$ . It is unchanged on storage (raw milk), destroyed by boiling, and decreased by preservatives. The Orla-Jensen creamometric method (B., 1932, 748), but not the above method, will distinguish between milk heated for 30 min. at  $60$ – $63^\circ$  and at  $55^\circ$ . J. G.

**Formation of an aroma resembling that of amyl alcohol in milk.** A. CUNNINGHAM (Proc. World's Dairy Cong., 1931, ii, 1—5).—The aroma was caused by white and orange micrococci from the bedding. CH. ABS.

**Copper contents of milk and milk preparations.** E. REMY (Z. Unters. Lebensm., 1932, 64, 545—548).—Full analyses of 10 varied samples are given. For cow's, human, and goat's milk, respectively, mean Cu contents were 0.13, 0.14, and 0.09 mg.-%; Fe 0.5, 0.14, 0.20; Ca/K 1:1.3, 1.3, and 2.1. A small reduction val. (4—20 c.c./100 g.) was obtained by Tillmans' 2:6-dichlorophenol-indophenol method (A., 1932, 310, 658), but it was not established that this was due to vitamin-C. J. G.

**Determination of sucrose and lactose in sweetened condensed milk.** H. LORENZ (Z. Unters. Lebensm., 1932, 64, 564—569).—The most reliable polarimetric and Cu-reduction methods are compared, and correction curves are given. The Monier-Williams method is considered the most exact, although White's method (in which lactose is determined by Cu reduction, and sucrose after inversion, on aliquot portions of the alkaline  $\text{CuSO}_4$  serum) is simplest, quickest, and requires the least equipment. Deviations of the latter from the A.O.A.C. method (cf. B., 1926, 992) amount to  $\pm 0.3\%$  for sucrose and  $0$ – $0.47\%$  for lactose. J. G.

**Body of cream.** G. T. PYNE and J. LYONS (Proc. World's Dairy Cong., 1931, iii, 37—42).—In milk separated immediately after the temp. was raised max. viscosity was produced at  $32$ – $44^\circ$ . Previous chilling of milk raises the separating temp. for max. viscosity. The viscosity of cream kept at  $40^\circ$  for 30 min. diminished. Agglutinin appears to play no part. CH. ABS.

**Control of the off-flavour appearing in strawberry ice cream.** C. D. DAHLE and E. C. FOLKERS (Ice Cream Trade J., 1932, 28, No. 12, 37—39).—The off-flavour is due to acidity of the berries and Cu content of the mix; it is prevented by keeping the Cu content down to approx. 0.8 p.p.m. CH. ABS.

**Determination of gases in cheese.** H. HOSTETTLER (Proc. World's Dairy Cong., 1931, ii, 127—128).—An apparatus is described. Determination of  $\text{H}_2$  is important. Holes contain  $\text{CO}_2$  ( $> 96\%$ ),  $\text{N}_2$ , and only traces of  $\text{H}_2$ ; gases obtained by evacuation of the curd contain up to 99.85%  $\text{CO}_2$ . CH. ABS.

**[Detection of degree of ageing and preservation of] eggs.** K. ELBE, H. PFEIFFER, and R. BRETSCHNEIDER (Z. Unters. Lebensm., 1933, 65, 100—104).—Eggs preserved in  $\text{CaO}$  or  $\text{Na}_2\text{SiO}_3$  give a red to yellow colour and fresh eggs a violet colour if 1 c.c. of albumin is shaken with 2 c.c. of amyl alcohol and 0.5 c.c. of alizarin solution. A yellow to green stain is obtained if the tip of a raw or old unpreserved egg is dipped in thymol-blue ( $\text{EtOH}$  solution), whilst preserved eggs turn blue. Bell and Doisy's  $\text{P}_2\text{O}_5$  reaction is negative when applied to the albumin of eggs  $< 2$  weeks old, but older eggs (preserved or otherwise) produce a blue-green colour. The  $p_{\text{H}}$  of a  $\text{MeOH}$ -serum is not a reliable age test. Erasion of marks of origin may be detected by the deep blue stain visible in ultra-violet light. J. G.

**Iodine content of iodine eggs.** J. STRAUB (Z. Unters. Lebensm., 1933, 65, 97—100).—The egg is evaporated with  $\text{K}_2\text{CO}_3$  and ignited, the residue being extracted several times with  $\text{H}_2\text{O}$  and finally with  $\text{EtOH}$ , and the extract re-ignited each time. I is then determined colorimetrically or (for  $> 40 \times 10^{-6}$  g.) by titration with  $0.005N$ - $\text{Na}_2\text{S}_2\text{O}_3$  to starch after neutralisation with  $\text{H}_2\text{SO}_4$  to Me-orange, addition of  $\text{Cl}_2$ - $\text{H}_2\text{O}$ , and evaporation to 5 c.c. The I content of ordinary eggs was  $4.5$ – $6.8 \times 10^{-6}$  g., and rose to 195 after 2 days, and to 2500 after feeding the hen for 10 days on a diet containing I. Inorg. I was 670 and 522, org. I 40 and  $422 \times 10^{-6}$  g., and total I 0.02 and 0.09% in the white and yolk, respectively. J. G.

**Analysis of mayonnaise and the variability of its egg constituents.** J. L. PERLMAN (J. Assoc. Off. Agric. Chem., 1932, 15, 466—482).—Phospholipin decomp. in mayonnaise may occur either naturally under certain conditions or through chance inoculation of the egg ingredients with certain extra-cellular enzyme-producing bacteria before the dressing is compounded. Four such strains have been isolated from decomposing egg material. Free choline is an end-product. The utility of total fat and lipin- $\text{P}_2\text{O}_5$  determinations is questionable in the case of dressings stored for  $> 1$  or 2 months without refrigeration. Mitchell's  $\text{EtOH}$ - $\text{CHCl}_3$  extraction method, slightly modified, yields the highest fat and lipin- $\text{P}_2\text{O}_5$  vals. for fresh samples, and the tentative  $\text{EtOH}$  extraction method (Methods of Analysis, A.O.A.C., 1930, 356) the highest lipin- $\text{P}_2\text{O}_5$  vals. for samples aged for  $< 2$  months. The approx. age of a dressing may be found by a comparison of the vals. so obtained. A probable gradual increase in the fat and lipin contents of mayonnaise on storage is indicated. W. J. B.

**Distinction between egg and plant lecithin by biological means, using the complement-fixation method.** O. MEZGER, H. JESSER, and M. VOLKMAN (Z. Unters. Lebensm., 1933, 65, 49—54).—It is not possible to obtain a satisfactory distinction between lecithins from soya beans, wheat, and eggs by the precipitin or (Wassermann) blood-hæmolysis reactions. J. G.

**Lecithin preparations of animal and vegetable origin.** F. E. NOTTBOHM and F. MAYER (Chem.-Ztg., 1932, 56, 881—882).—The lecithin content calc. from the choline as determined by titration, with  $\text{Na}_2\text{S}_2\text{O}_3$ , of

the ppt. produced by the action of I solution on the fatty acids is < that calc. from the N or P contents. Egg and soya lecithins are distinguishable by the ratios N : P and residual N (*i.e.*, after deduction of that due to choline) : residual P, which are approx. 0.8 and 1.2, and 0.5 and 1.4, respectively. Methods of separation of lecithin by pptn. with  $\text{COMe}_2$  are described, and show plant lecithin to be associated with 20–35% of a fatty oil (I val. 115, f.p. 13°), whilst egg lecithin yields only 6–14%. J. G.

**Do cacao beans contain lecithin? [Determination of choline.]** F. E. NOTTBOHM and F. MAYER (Z. Unters. Lebensm., 1933, 65, 55–66).—Choline is determined by heating the fat-free beans with  $\text{MgO}$ , the filtrate being acidified with  $\text{HCl}$  and autoclaved. An extract in  $\text{CHCl}_3$  of the resulting solution is decolorised with C and the residue after evaporation is extracted with abs.  $\text{EtOH}$ , which is also evaporated and an aq. extract of the new residue decolorised with C and pptd. with I. The ppt. is then decomposed with  $\text{Ag}_2\text{O}$ , reprecipitated with I, and titrated with 0.1N- $\text{Na}_2\text{S}_2\text{O}_3$ . The choline content of 11 beans and 4 commercial preps. ranged from 0.102 to 0.149%, half of which was sol. and half insol. in hot  $\text{EtOH}$ ; only traces were found in the  $\text{Et}_2\text{O}$  extract, and the lecithin content of the  $\text{EtOH}$ -sol. portion as calc. from the P content is double that of the choline content. The results indicate that free lecithin is absent from cacao beans and that phosphatides other than choline (not necessarily lecithin) may occur in the  $\text{EtOH}$  extract; these may be produced by the action of the hot  $\text{EtOH}$  or by the roasting process (*cf.* preceding abstract). J. G.

**Detection of coffee substitutes.** A. HEIDUSCHKA and H. THOMAS (Z. Unters. Lebensm., 1933, 65, 95–97).—Normal malt coffees were found by Merl's method (B., 1931, 316) to contain 5.2–5.7 mg. of maltol/10 g., normally roasted barley 5.5, wheat 5.6 (3.5 if steeped, roasted, and glazed), rye 6.1, dried, roasted, and glazed malt 1.3 (5.7 if first steeped), and rye (treated similarly) 3.9–5.2. J. G.

**Gravenstrin spot scald.** J. F. HOCKEY (Sci. Agric., 1932, 13, 225–227).—Spot scald occurred when apples containing no starch in the pulp cells and little or no sucrose were exposed to bright light. Temp. and ventilation in storage had little or no effect on the development of the condition. A. G. P.

**Detection of dry plums in plum-pulp mixtures.** G. KAPPELLER and W. REIDEMEISTER (Z. Unters. Lebensm., 1932, 64, 558–560).—The red colour produced with  $\text{NaOH}$  and  $\text{SO}_2$ -magenta (according to Rudolph and Barsch, B., 1932, 239) is unreliable as a test for dry plums since it is produced in the presence of Fe, with an intensity depending on the concn. of the latter. No satisfactory results are obtainable by the I-fixation or the  $\text{NaOH}$  tests (*loc. cit.*). J. G.

**Sugar-feeding honey and the origin of diastase.** K. BRAUNSDORF (Z. Unters. Lebensm., 1932, 64, 555–558).—A sugar-feeding honey having a diastatic val. of 17.9 (sucrose 6.5%, acid val. 1.42, ash 0.12%, negative Fiehe test, and isolated pollen grains visible under the microscope) was examined; this indicates that a

diastatic val. < 10 (*cf.* lit.) is not necessarily characteristic of such honeys. It is considered that the diastase is derived from the bees' bodies, and that the diastase val. is therefore dependent on the racial characteristics. J. G.

**Relation of edible portion and offal, and composition, of fresh-water fish.** METZNER and RÖHLER (Z. Volksernähr. Diät., 1932, 7, 236–238, 255–257; Chem. Zentr., 1932, ii, 3028).—Edible portions are: eel 62.7, bream 55.3, trout 65.9, pike 62.5, carp 60.1, red-eye 66.6%. A. A. E.

**Vitamin content of commercial vitamin-A and -D preparations.** W. HEUBNER and K. SCHÜBEL (Klin. Woch., 1932, 11, 1142–1143; Chem. Zentr., 1932, ii, 2987).—Vigantol gave const. -D vals.; Vigantollebertran and Lofotenlebertran were also rich in -A. Vitatran, Eukraton, and Robugen were low in -A and -D. Medicinal cod-liver oil was rich in -A. Vitophos, Jemalt, and Nestlé's Infants' Food were not appreciable sources of -A, and the first contained also no -D. A. A. E.

**Adsorption of organic acids in mixtures of alcohol and water by activated charcoal, with special reference to acids which occur in foodstuffs.** G. REIF and R. VON DER HEIDE, JUN. (Z. Unters. Lebensm., 1932, 64, 514–532).—Experiments at 20° were carried out under various conditions of concn. of active C (1.2% ash), org. acids, and  $\text{EtOH}$ , and for single and various combinations of 2 or 3 mono-, di-, and tri-basic acids, including isomerides. In general, the lower-C acids show at first a rapid decrease in adsorptive powers with increase in  $\text{EtOH}$  content of the solution, followed by a progressively slower decrease until a point is reached when the adsorption remains const. (for 40–50%  $\text{EtOH}$ ). With rise in the homologous series these curves approach straight lines, and there is some evidence of the inversion of the rule for the higher members.  $\text{AcOH}$  produces a greater reduction in adsorbing power than  $\text{HCO}_2\text{H}$  when added to another (dicarboxylic) fatty acid, and in mixtures of 3 acids (2–10%) the individual adsorbing powers are all lowered. Attempts are made to relate the solubility,  $p_H$  val., and adsorbability, and to explain the results in terms of the structure of the org. acid mol. and its electrical properties in different solvents. J. G.

**[Icing] sugar.**—See VII. **Al foil for wrapping cut bread.**—See X. **Cacao butter.**—See XII. **Spontaneous ignition of sugar.** **Determining inulin and lævulosin.**—See XVII. **"Kasutorishōchū"** [whisky from rice].—See XVIII.

See also A., Mar., 300, Catalase of milk. 323–6, **Vitamins (various).**

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**[Pharmacopœial methods.]** F. VIEBÖCK (Pharm. Presse, 1932, 37, 17–19, 47; Chem. Zentr., 1932, ii, 2694–2695).—The D.A.B. VI methods for the detection of  $\text{AcOH}$  in  $\text{HCO}_2\text{H}$  and  $\text{C}_6\text{H}_4\text{Cl}\cdot\text{CO}_2\text{H}$  in  $\text{BzOH}$ , and the determination of Hg in mercurisalicic acid are criticised and modified. Boie's method for the acidimetric determination of theobromine is also modified. A. A. E.

**Evaluation of potassium iodide ointment.** E. RUPP and G. HAMANN (Apoth.-Ztg., 1932, 46, 1642—1643; Chem. Zentr., 1932, ii, 3279).—The ointment (2 g.) is melted with H<sub>2</sub>O (10 c.c.), cooled, filtered and again melted with H<sub>2</sub>O (5 c.c.), and the fat rinsed with H<sub>2</sub>O. The aq. filtrate is treated with 5 c.c. of FeCl<sub>3</sub> solution (D. A. B.); the I is dissolved, after 2 hr., in CHCl<sub>3</sub> (10 c.c.) and titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. A simplification of the procedure and a colorimetric method for small KI contents are described. A. A. E.

**Analysis of skin creams.** H. PINCASS (Pharm. Zentr., 1933, 74, 132—134).—H<sub>2</sub>O is determined by drying at 80—100° for 24 hr., glycerin either by oxidation with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> after hydrolysis with H<sub>2</sub>SO<sub>4</sub>, the CO<sub>2</sub> evolving being measured, or titrimetrically with KMnO<sub>4</sub>. High acid vals. indicate rancidity or presence of borax or beeswax. Paraffin wax is determined by sulphonating the unsaponifiable matter and extracting with ligroin. The saponifiable constituents are separated into soaps insol. and sol. in 10% EtOH containing 2% KCl, the former representing waxes and the latter vegetable or animal oils, which are roughly identified by the I vals. of the fatty acids. S. C.

**[Assay of] calcium gluconate.** H. J. FISHER (J. Assoc. Off. Agric. Chem., 1932, 15, 456—460).—A tentative volumetric method is described which depends on the determination of the Ca in the ash. W. J. B.

**Polarimetric method for determination of calcium gluconate.** H. J. FISHER and E. M. BAILEY (J. Assoc. Off. Agric. Chem., 1932, 15, 461—466).—When dil. solutions of Ca gluconate are saturated with uranyl acetate the rotations are a linear function of the concn. When solutions containing 1 g. of sample per 25 c.c. are examined in the polarimeter (white light, 200-mm. tubes) the % Ca(C<sub>6</sub>H<sub>11</sub>O<sub>7</sub>)<sub>2</sub>·H<sub>2</sub>O = 4·3(B - A), where A is the rotation in degrees Ventzke of the solution without uranyl acetate, and B is the rotation of a similar solution saturated with uranyl acetate. W. J. B.

**[Determination of] guaiacol.** N. L. KNIGHT (J. Assoc. Off. Agric. Chem. 1932, 15, 429—434).—None of the methods tested, including a gravimetric bromination, a colorimetric, and a NaBrO<sub>3</sub> titration method, has been found satisfactory. W. J. B.

**Determination of potassium guaiacolsulphonate in medicaments.** J. FIALKOV and M. STSCHIGOL (Pharm. Zentr., 1933, 74, 103—105).—1—2 c.c. of HNO<sub>3</sub> (d 1·4) and 0·2—0·3 g. of the substance are evaporated and the residue is ignited. After cooling, 1 c.c. of HNO<sub>3</sub> is added and the evaporation and ignition are repeated. The residue is extracted with H<sub>2</sub>O and the extract treated with 20—25 c.c. of 0·1N-BaCl<sub>2</sub>, and after boiling for 5 min. 25—30 c.c. of 0·1N-(NaOH + Na<sub>2</sub>CO<sub>3</sub>) are added and the mixture is filtered. The filtrate is titrated with 0·1NHCl, using Me-orange. The results of the examination of 20 samples of varying composition are given. E. H. S.

**Identification of atoxyl.** M. WAGENAAR (Pharm. Weckblad, 1933, 70, 233—239).—Characteristic microcryst. pptts. (illustrations are given) are obtained when

atoxyl is crystallised from COMe<sub>2</sub> or when a solution is treated with AgNO<sub>3</sub>, CdSO<sub>4</sub>, or MgSO<sub>4</sub>. Rose-red crystals are obtained when a drop of atoxyl solution is evaporated with HCl and then treated with an oleic acid solution of furfuraldehyde, whilst vanillin or piperonal give a deep yellow colour. A solution in HCl gives an intense orange colour with a wood shaving, and As can be shown by exposing a slide, on which a drop of HgCl<sub>2</sub> solution has been allowed to evaporate, to the gases evolved in a reduction apparatus, when the crystals slowly become deep yellow. S. C.

**Composition of some commercial compound antineuralgics.** O. RIESSER (Arch. exp. Path. Pharm., 1933, 169, 164—179).—An analytical method and the data obtained by its use are given. Phenacetin, caffeine, antipyrine, and pyramidone are the most common constituents. F. O. H.

**Turbidity of morphine solutions.** C. STICH (Pharm. Zentr., 1933, 74, 114—115).—An account of micro-organisms found in morphine solutions is given. S. C.

**Determination of morphine. III.** L. DÁVID (Pharm. Ztg., 1933, 78, 163; cf. B., 1931, 861).—Slight modifications of the original method and its application to morphine determinations in various opium powders, extracts, and tinctures are described. E. H. S.

**Detection and determination of procaine.** J. A. SÁNCHEZ (Semana Med., 1932, II, 807—808).—Conc. aq. furfuraldehyde acidified with AcOH (5 drops per 10 c.c.) gives with procaine, but not stovaine or cocaine, a red colour. Procaine hydrochloride is titrated with NaOH (phenolphthalein), and the liberated base is titrated with H<sub>2</sub>SO<sub>4</sub> (rosolic acid); the vals. should be equal. CH. ABS.

**Determination of theobromine.** N. S. GORYAINOVA (Khim. Farm. Prom., 1932, 6, 227—228).—Powdered cocoa (8 g.), extracted with light petroleum, is treated with dil. H<sub>2</sub>SO<sub>4</sub> and warmed for 20 min., 5—10 g. of MgO in H<sub>2</sub>O then being added. The mixture is heated to form a paste and treated with 100 c.c. of 10% EtOH-CHCl<sub>3</sub>; 75 c.c. of the filtrate are evaporated until the EtOH is removed. The residue is washed by decantation with light petroleum and EtOH and weighed. The purity of theobromine is determined by dissolving 0·2 g. in 5% aq. NH<sub>3</sub> (20 c.c.) and concentrating it to 5 c.c. with 25 c.c. of 0·1N-AgNO<sub>3</sub>. The concentrate is acidified with 30% AcOH, filtered, washed, and titrated with the filter by Folgard's method (1 c.c. 0·1N-AgNO<sub>3</sub> = 0·0180 g.) CH. ABS.

**Tobacco smoke. II.** E. WASER and M. STÄHLI (Z. Unters. Lebensm., 1932, 64, 569—573).—Tests on substances to be inserted in the tips of cigarettes to adsorb nicotine have failed to substantiate fully the manufacturers' claims. SiO<sub>2</sub> gel (cf. A., 1933, 91), which is less effective as an adsorbent for nicotine in smoke than in solution, is more effective than porous clay or glass granules of a similar size. It is less effective than active C, but its efficiency increases as the smoking period increases (e.g., about 30% of the nicotine in smoke is removed from 5 g. of tobacco, smoked in 6·5 min., by 1·3 g. of SiO<sub>2</sub> gel). J. G.

**Determination of camphene in pine oil.** I. M. EZRIELEV and S. S. MAGIDOV (Plast. Massi, 1932, Nos. 2—4, 11—13).—Bertram and Walbaum's method (A., 1894, i, 201) is long and inaccurate. Satisfactory results are obtainable by treating the oil with a mixture of glacial AcOH and 50% H<sub>2</sub>SO<sub>4</sub> of known titre and subsequently determining the acid which does not react by titration. T. H. P.

**Effect of time of harvesting on content and quality of essential oils from valerian rhizome and root.** W. J. STRAŻEWICZ (Pharm. Zentr., 1933, 74, 134—136).—Chemical examination of *Valeriana officinalis*, L., var. *latifolia* Vahl., shows that oil content and quality vary with the time of harvesting, there being a definite fall in both yield and quality from plants harvested before May or later than June. S. C.

**Essential oil of *Salvia sclarea*, L.** B. N. RUTOVSKI and K. A. GUSSEVA (Riechstoffind., 1932, 7, 65—66, 78—79; Chem. Zentr., 1932, ii, 3315).—Oil from the inflorescence (Central Asiatic Russia) (0.06—0.33%) had  $d_{20}^{20}$  0.891—0.931,  $\alpha_D$  —4.5° to —39°,  $n_D^{20}$  1.4572—1.4826, acid val. 0.1—12.7, ester val. 101.8—215.6 (after acetylation 105.7—230.8). Vals. for oil from inflorescence and leaves were: 0.06%, 0.8974, —57.92°, 1.4797, 0.77, 100.48 (133.82), and for that from flowering plants 0.07—0.1%, 0.896—0.927, —19° to —50°, 1.4625—1.4905, 1.1—1.2, 94.0—149.2 (—). Vals. are also given for a Crimean sample. AcOH, *l*-linalool, *l*-nerolidol, and a sesquiterpene resembling  $\beta$ -santalene were present. A. A. E.

**Utilising waste from anise oil.** B. I. RUTOVSKI and K. P. DOLMATOV (Bull. Nauch.-Issledov. Khim.-Farm. Inst., 1931, 13—15).—A by-product (10—15%) contained anethole and estrazole; the latter was isomerised to the former with KOH. CH. ABS.

**Vitamin preps.**—See XIX.

See also A., Mar., 249, Quant. removal of FeCl<sub>3</sub> from aq. solution. 256, Prep. of glyceryl stearates. 279, Mol. compounds of cineole. 281, Novocaine analogues. 288, *Cinchona* alkaloids. 289, Alkaloids (various). 290, New derivatives of *p*-arsanilic acid. Substituted arylarsinic acids. 292, Detection of papaverine. 310, Local anæsthetics of urethane type. Substituted barbituric acids. 311, Solanocapsine. Cardiotoxic glucosides. 312, Prep. of Bi salts of fatty acids. 318, Concn. and purification of diphtheria toxoid. 319, Bactericidal properties of AgCl. Prep. of vagotonin. Isolation of thymocrescin. Prep. of intermedin. Parathyroid hormone. 320, Hormone of the adrenal cortex. Determining adrenaline in gland extracts. 321, Prep. of insulin. 323—6, Vitamins (various). 329, Alkaloids (various). Indian medicinal plants. *Cascara sagrada*. Determining essential oils in plants. Resin of tobacco smoke.

#### PATENTS.

**Making stable solutions of morphine for injection purposes.** F. MERZ (MERZ & Co.) (B.P. 387,850, 7.3.32. Ger., 28.3.31).—Solutions, physically and therapeutically superior to those of morphine alone, are

obtained by the addition of phenyldimethylpyrazolone and adjustment of the  $p_H$  to about 7. E. H. S.

**Liquid preps. of C<sub>2</sub>H<sub>2</sub>Br<sub>3</sub>·OH.  $\beta$ -*p*-isoPropylphenylisopropaldehyde.**—See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Influence of alkali iodides on liability of photographic emulsions to solarisation.** A. CHARRIQUO (Compt. rend., 1933, 196, 172—174; cf. B., 1930, 929).—Addition of increasing amounts of KI (0.5, 1, and 2 g. per 100 g. of AgNO<sub>3</sub> originally used) to an emulsion (of 500 H. & D.) causes an increasing fall in sensitiveness and in density of the negative, and increasing liability to solarisation, accompanied by increase in grain size of AgBr. With the largest addition of KI a completely solarised negative was obtained with "instantaneous" exposure. Addition of KCl has no effect on liability to solarisation or grain size of AgBr. C. A. S.

**Criterion of the speed of negative emulsions for use in routine work.** F. LAPEYRE (Bull. Soc. Franç. Phot., 1933, 20, 49—54).—The speed is chosen as a no. inversely proportional to the min. illumination required for detail in the darkest parts of a grey subject (contrast 1.5) which will give, with  $\frac{1}{6}$  sec. exposure and suitable development time, a print with a complete range of contrast on a paper of density range 1.2. The method for determining this speed is described. J. L.

**Electrolysing thiosulphate solution.**—See XI.

See also A., Mar., 238, Evolution of the latent image. Effects of  $\alpha$ -rays. 258, Light-sensitive carotene dye.

#### PATENTS.

**Filter layers for photographic purposes.** I. G. FARBENIND. A.-G. (B.P. 387,519, 9.3.32. Ger., 9.3.31).—A H<sub>2</sub>O-insol. org. dye lake (e.g., Congo-red pptd. with BaCl<sub>2</sub>) is mixed with an aq. solution (10%) of gelatin, and filter layers are made by pouring the mixture in the usual manner. Such layers are transparent and show no diffusion of the dye, even in the wet state. The dye is easily decolorised by, e.g., acidified aq. Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>. J. L.

**Colour photography.** KODAK, LTD., Assees. of J. G. CAPSTAFF and M. W. SEYMOUR (B.P. 388,062, 17.8.31. U.S., 21.8.30).

## XXII.—EXPLOSIVES; MATCHES.

**Stabilisers and gelatinisers for smokeless powders.** M. GIUA and G. GUASTALLA (Chim. et. Ind., 1933, 29, 268—277).—In the nitration of NH<sub>2</sub>Ph, centralite (I), NHPH<sub>2</sub>, phthalide,  $\alpha$ -naphthylurethane, Et and K oxanilates, 1.1, 4.2, 5.6, 0.8, 1.9, 1.9, and 2.0 NO<sub>2</sub> groups per mol., respectively, were fixed. It was impossible to nitrate a condensation product of Et<sub>2</sub>C<sub>2</sub>O<sub>4</sub> and *m*-xylylidine without decomp. Monoacetyl-*m*-xylylidine gave a NO<sub>2</sub>-compound, but this decomposed on further nitration. Partial hydrolysis occurred when the Ac<sub>2</sub> compound was nitrated. The relative efficiencies of stabilisers are given by their "nitrometric

index," *i.e.*, the amount by which, owing to their absorption of N oxides, they reduce the N val. of nitroglycerin in the nitrometer. Figures obtained for the above compounds were: 0.894, 1.19, 2.51, 0.45, 0.77, 0.77, and 0.77, respectively. In view of the association in smokeless powders of certain NO<sub>2</sub>-compounds with (I), the eutectic points of the following binary systems were determined and are tabulated: (I) with mono-, di-, and tri-nitrotoluenes, picric acid, 1 : 2 : 4-C<sub>6</sub>H<sub>3</sub>Br(NO<sub>2</sub>)<sub>2</sub>, and NHPh<sub>2</sub>. Abs. viscosities were determined of solutions of nitrocellulose, mixtures of nitrocellulose with (I), mixtures of nitro- and acetyl-cellulose in CPhMe and in CH<sub>2</sub>Ac·CO<sub>2</sub>Et. No conclusion could be formed regarding the formation of mol. compounds from nitric esters and stabilisers or gelatinisers of the types in question. W. J. W.

**Liquid oxygen explosives.** T. COULTER and A. E. LANCE (J. Chem. Met. Soc. S. Africa, 1932, 33, 142—160).—In the Weberlox cartridge, cellulose material is used as the absorbent instead of C, so that practically no CO is evolved on explosion. Metallic powders, *e.g.*, Al, are added to raise the explosion temp. For Weberlox-B.E. and -C.G., fired by fuse, the temp. are 4172° and 2150°, and the velocities of detonation 4253 and 3000 m./sec., respectively. When the explosive is initiated by a Loxlite primer, the velocity is much higher. In the Pb-block test a 12 in. × 1½ in. cartridge was equiv. to 0.642 lb. of 60% gelignite. Increased safety in blasting is claimed for Weberlox explosives as compared with liquid O<sub>2</sub> explosives containing C, or NO<sub>2</sub>-explosives. W. J. W.

**Nitration of cellulose. Fractionation of nitro-cotton.**—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Detection of small amounts of carbon monoxide in air.** W. ACKERMANN (Chem.-Ztg., 1933, 57, 154—155).—In 1 litre of air, 0.13 vol.-% of CO can be detected by absorption in dil. blood solution, adding (NH<sub>4</sub>)<sub>2</sub>S, and examining the absorption spectrum, and 0.015 vol.-% can be detected by reaction with 0.005N-PdCl<sub>2</sub>.

E. S. H.

**Absorption of lead by tooth-pastes from tinned-lead tubes.** V. FROBOESE (Z. Unters. Lebensm., 1933, 65, 176—181; cf. B., 1932, 50).—The quantities found in the paste ranged from 0.5 to 37 mg. of Sn/100 g. and 0—1.8 of Pb at the time of purchase, and rose to 139 and 3.1, respectively, after up to 12 weeks' storage. Storage for periods up to 4 years produced increases (up to 357 and 2.8, respectively), decreases, or no change in the Sn and Pb contents, and the amounts found appear to bear no definite relation to the reaction or  $\Lambda$  of the paste. A suitable Pb content for the tubes is > 1%, and the Sn coating usually corresponds to 0.12 g./100 sq. cm. J. G.

**Determination of free carbon dioxide in water containing humus.** Y. KAUKO (Suomen Kem., 1932, 5, [B], 54).—As a result of the phase relationships existing in solutions of the type in which humus usually occurs, the free CO<sub>2</sub> content may be determined by blowing air through the solution and measuring the quantity of CO<sub>2</sub> evolved. H. F. G.

**Detection and determination of iron in drinking water.** L. W. WINKLER (Pharm. Zentr., 1933, 74, 129—132).—The colour reaction with KCNS and Fe<sup>+++</sup> ions is more sensitive than that with K<sub>3</sub>Fe(CN)<sub>6</sub>. A qual. test is carried out by boiling a sample of H<sub>2</sub>O with a little KClO<sub>3</sub> and HCl, adding KCNS, and matching the colour against a similar sample treated subsequently with Na<sub>2</sub>SO<sub>3</sub>. For the determination 100 c.c. are evaporated with 0.1 g. of KClO<sub>3</sub> and 2—3 c.c. of HCl, adding distilled H<sub>2</sub>O to 100 c.c. and then KCNS solution, and matching the colour against a standard prepared from Fe alum solution (1 c.c. = 0.1 mg. Fe). The comparison is best carried out in artificial light, and for quantities > 0.2 mg. Fe per litre the results are accurate to 5% if tests are carried out in triplicate. S. C.

**Colorimetric and gravimetric methods for determination of arsenic, especially in water and filter-mud.** H. SCHRÖDER and W. LÖHR (Z. Unters. Lebensm., 1933, 65, 168—176).—A modified Gutzeit method is described in which the AsH<sub>3</sub> passes through a vertical tube containing in succession Pb(OAc)<sub>2</sub>-paper and -wool and a strip of filter-paper (15 × 2 cm.) impregnated with 5% HgBr<sub>2</sub>. The result is obtained from the length of the stain, which ranges from 0.5 cm. for 2 × 10<sup>-6</sup> g., to 12 cm. for 200 × 10<sup>-6</sup> g., of As. For gravimetric work org. matter is destroyed by acids and H<sub>2</sub>O<sub>2</sub>, HNO<sub>3</sub> is removed, and the As is separated as AsCl<sub>3</sub> by distillation in a stream of CO<sub>2</sub> in the presence of FeSO<sub>4</sub>, N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>SO<sub>4</sub>, borax, and HCl; 100 c.c. of distillate are collected and As<sub>2</sub>S<sub>3</sub> is removed by pptn., redissolved in aq. NH<sub>3</sub>, oxidised with H<sub>2</sub>O<sub>2</sub>, and finally pptd. as Mg<sub>2</sub>As<sub>2</sub>O<sub>7</sub>. J. G.

**Elimination of hydrogen sulphide from [beet-factory] waste waters.** E. BANIK (Centr. Zuckerind., 1932, 40, 748—749).—When the waste waters being returned to the diffusion process are found to contain an objectionable amount of H<sub>2</sub>S they are treated with the necessary amount of NaOCl. J. P. O.

**Removing phenols from ammoniacal liquor.**—See II. **School materials.**—See XIII. **Manure from sewage etc.**—See XVI. **Fermentation of fibrous materials.**—See XVIII.

See also A., Mar., 243, **Determining HCN and (248) SO<sub>2</sub> in air.** 249, **Electric precipitators for air analysis.**

#### PATENTS.

**Extraction of dissolved or suspended liquids, and sparingly-soluble organic liquids in particular, contained, in small quantities, in effluent water.** CARBO-NORIT-UNION VERWALTUNGS-GES. M.B.H. (B.P. 387,541, 8.4.32. Ger., 2.5.31).—The polluted water may be passed through a bed of fine-grained activated C or intimately mixed with a small quantity thereof, in a finely-divided state, and later separated by settlement and decantation. The adsorbed org. substances are expelled from the C by hot gases or steam-distillation and are recovered; the activated C can be used repeatedly and the effluents, in many cases, are suitable for, *e.g.*, steam-raising, dyeing, or extraction purposes. C. J.

**Kaolin for gas masks etc.**—See VII.