

# BRITISH CHEMICAL AND PHYSIOLOGICAL ABSTRACTS

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# BRITISH CHEMICAL AND PHYSIOLOGICAL ABSTRACTS

## A., I.—General, Physical, and Inorganic Chemistry

FEBRUARY, 1943.

### I.—SUB-ATOMICS.

**Absorption phenomena in a condensed spark source.** G. O. Langstroth and W. W. Brown (*Canad. J. Res.*, 1942, 20, A, 173—183).—In the emission spectra of Mg, Hg, and Sn the intensity ratios of certain line pairs are dependent on the amount of the element introduced into the source. As air currents of 1500 cm. per sec. reduce the absorption but currents of 80 cm. per sec. have practically no effect, this is attributed to partial absorption which must occur mainly within the limits of the discharge zone. The radiation involving a transition to a level in the ground term shows greater self-absorption than that associated with a higher final energy level. No appreciable errors are introduced into analytical determinations by variations in the absorption due to varying air currents of normal velocity. J. W. S.

**Lifetime of the zinc line  $\lambda$  3076 Å. ( $4^1S_0-4^3P_1$ ).** H. Bruck (*Compt. rend.*, 1942, 214, 307—309).—Measurements by photographic photometry and comparison with the theoretical curve give a mean val.  $3.0 \times 10^{-8} \pm 5\%$  sec. N. M. B.

**Effect of photo-electrons released in the film and the size of the photometer slit on the breadth of X-ray lines.** A. Kochendorfer (*Physikal. Z.*, 1942, 43, 313—329).—The measured broadening of lines in X-ray spectrograms is determined not only by particle-size and lattice-distortion effects and the width of the X-ray slit, but also by the width of the microphotometer slit and effects due to photo-electrons excited in the film. A detailed theory of these factors is given. Test measurements with 0.325-Å. X-rays show good agreement with theory; with both slits 0.002 mm. in width the true breadth of a  $\sim 0.003$ -mm. line can be measured to  $\pm 5\%$ , an accuracy sufficient to permit exact study of mosaic structure in metals. A. J. E. W.

**Selenium photo-elements. V. Effect of  $\alpha$ -rays.** A. Becker (*Z. Physik*, 1942, 118, 695—705).—Irradiation of Se photo-elements with  $\alpha$ -rays produces no measurable electromotive effect. The photo-effect is decreased owing to the effect of the  $\alpha$ -rays on the photo-electrically effective surface of the element. The effect is not due to alteration of the optical properties of the surface.  $\alpha$ -Rays cause a decrease in the dark resistance of the cell. Electrical changes in the body of the semi-conductor are not responsible because intensive  $\gamma$ -rays produce no effect. A. J. M.

**Variation of thermionic and photo-electric emission constants of silver oxide-cæsium cathodes during activation.** R. Suhrmann and F. W. Dehmelt (*Z. Physik*, 1942, 118, 677—694).—A superficially oxidised  $Ag_2O$  cathode was covered with Cs by vaporisation, and activated by warming to various temp. up to  $180^\circ$ . It was then covered with Cs again for 14 days, and activated at  $171.5^\circ$ . During the two activation processes the thermionic and photo-electric emission were investigated and the consts. determined at the various activation temp. The different behaviour of the emission consts. during the two activation processes is due to differences in the nature of the cathode surface. A. J. M.

**Origin of E layer of ionosphere.** R. Jouaust (*Compt. rend.*, 1942, 214, 441—442).—Photo-electric ionisation at altitudes 100—120 km. must involve metastable excited atoms, e.g., the O ( $^1S$ ) atoms identified in this region by the line 5577 Å., O ( $^1S$ )  $\rightarrow$  O ( $^1D$ ), in the night-sky radiation. Ionisation of this atom requires 9.32 e.v., corresponding to  $\lambda$  1323 Å., which has a sufficiently high intensity at 100 km. O ( $^1S$ ) is produced by three-body recombination, in the presence of an O atom, of ( $^3P$ ) O atoms arising from absorption of solar  $\lambda$  1751 Å. by O<sub>2</sub>. L. J. J.

**Wandering of electrons in alkali halide crystals.** N. Karabascheff (*Z. Physik*, 1942, 118, 718—726).—The current due to the wandering of electrons in KBr crystals increases linearly with time. The rate of increase  $\propto$  square of the applied potential. These observations can be explained by consideration of electron diffusion in an electric field, and quant. equations are given. A. J. M.

**Field-electron microscopical observations on tungsten points.** R. Haefer (*Z. Krist.*, 1942, 104, 1—10; cf. A., 1941, I, 32).—Müller's method of obtaining the cathodic emission pattern of a W point (cf. A., 1937, I, 503; 1938, I, 301) is modified to give a pattern for the whole emitting surface. The cathode is placed at the centre of a

spherical glass bulb carrying a sputtered Pt layer as anode and the fluorescent material. The emitting areas increase in size as the grain-size and the radius of curvature and "smoothness" of the point (shown by electron photomicrograms) are increased by heating; the potential required for excitation also increases. The emission pattern represents a  $5 \times 10^4$  to  $2 \times 10^6$ -fold magnification of the emitting surface (resolving power 2500—10 Å.). The (100), (110), and (211) planes show reduced emission. A. J. E. W.

**Short-lived barium and lanthanum isotopes obtained in uranium fission.** O. Hahn and F. Strassmann (*Naturwiss.*, 1942, 30, 324—328).—The Ba of half-life 14 min., regarded as a primary U fission product, is also present in the active ppt. from emanating U; it is also obtained from Xe. It is, however, not a pure isotope, but a mixture of two, with half-lives 6 and 18 min. The 6-min. Ba yields La with half-life  $74 \pm 5$  min., whereas the 18-min. Ba gives rise to La of half-life  $3.5 \pm 0.35$  hr. A. J. M.

**Problem of isomerism in nuclear physics.** S. Flügge (*Physikal. Z.*, 1941, 42, 221—254).—A review dealing with theories attempting to explain nuclear isomerism. The rotation impulse and  $\gamma$ -activity theories are reviewed, a large no. of experimental results on nuclear isomerism are collected, and an extensive bibliography is given. A. J. M.

### II.—MOLECULAR STRUCTURE.

**Infra-red spectrum of furan.** (Miss) L. W. Pickett (*J. Chem. Physics*, 1942, 10, 660—663).—Infra-red spectrum data for furan vapour, obtained over the range 500—35,000  $\text{cm}^{-1}$ , show a considerable no. of combination and overtone frequencies not previously observed, particularly a strong band at 1580  $\text{cm}^{-1}$ . Two bands at 584 and 624  $\text{cm}^{-1}$  and two at 725 and 763  $\text{cm}^{-1}$  observed at low pressures are probably resolutions of the single bands at 601 and 740  $\text{cm}^{-1}$  respectively obtained by Manzoni-Ansidei at high pressures. Assignments of frequencies are made corresponding with those made by Lord and Miller for pyrrole (cf. A., 1942, I, 313). C. R. H.

**Infra-red spectrum of polyatomic molecules. XVI. Trideutero-methyl chloride and bromide.** H. D. Noether (*J. Chem. Physics*, 1942, 10, 664—668).— $\text{CD}_3\text{Cl}$  and  $\text{CD}_3\text{Br}$  were prepared by reducing  $\text{CD}_3\text{NO}_2$  to  $\text{CD}_3\text{NH}_2$ , HCl by means of HCl and Fe filings in absence of air at  $70^\circ$ , and converting the amine into the Bz derivative in the usual way. After purification the latter was heated with  $\text{PCl}_5$  or  $\text{PBr}_5$ . Infra-red spectrum data over the range 2.8—18  $\mu$ . are recorded for both halides and a complete assignment of the fundamental frequencies is given. C. R. H.

**Near infra-red absorption spectrum of methyl iodide.** R. T. Lagemann and H. H. Nielsen (*J. Chem. Physics*, 1942, 10, 668—671).—New infra-red absorption data for MeI at 3.3, 6.9, 8, and 11.3  $\mu$ . reveal additional detail in each region, and give information on the structure and convergence of the Q branches of some of the bands. C. R. H.

**[Ultra-violet] absorption spectra of organometallic compounds of tin and lead.** L. Riccoboni (*Gazzetta*, 1941, 71, 696—713).—The ultra-violet absorption spectra of  $\text{SnEt}_4$ ,  $\text{PbEt}_4$  (I),  $\text{SnEt}_3\text{Cl}$ ,  $\text{SnEt}_2\text{Cl}_2$ , and  $\text{PbEt}_3\text{Cl}$  (II) are determined in  $\text{C}_6\text{H}_{14}$  and in MeOH [(II) in MeOH and in  $\text{H}_2\text{O}$ ]. The continuous absorption observed is attributed to dissociation. With (I) and (II) there is decomp. and separation of Pb. In the region studied (to  $\nu$  45000  $\text{cm}^{-1}$ ), the logarithm of the extinction coeff. increases fairly uniformly as  $\nu$  increases, in general rapidly, and relatively more with Sn than Pb compounds, and more in  $\text{C}_6\text{H}_{14}$  than in MeOH. The results are discussed. E. W. W.

**Long-wave-length spectra of saturated carboxylic acids, esters, and salts.** H. L. McMurry (*J. Chem. Physics*, 1942, 10, 655—660).—The electron structure and spectra of the  $\text{CO}_2'$  ion and the  $\text{CO}_2\text{R}$  radical (R = alkyl group or H) in saturated mols. are discussed. In the  $\text{CO}_2\text{R}$  radical the longest- $\lambda$  absorption, which occurs at 2100 Å. and is weak, is ascribed to a transition similar to that causing the weak absorption at 2900 Å. in the  $\text{:CO}$  spectrum in aldehydes and ketones. The analogous weak absorption of the  $\text{CO}_2'$  ion at 1900—2000 Å. is masked by a stronger absorption as a result of a transition in which a non-bonding O electron is excited to an antibonding mol. orbital of the unsaturation type. C. R. H.

**Absorption spectra and structures of pyrethrins I and II.** A. E. Gillam and T. F. West (*J.C.S.*, 1942, 671—676).—Details of prep. of pyrethrin I and II concentrates and derived semicarbazones and pyrethrolones are given. Absorption spectra data are recorded, and the following conclusions are reached: (a) in the pyrethrolone fragment of the pyrethrin mols., two separate chromophoric systems are present, each containing > one double linking; thus the postulated trienone chromophoric system is not present; (b) an  $\alpha\beta$ -unsaturated ketone grouping is present in a 5-atom ring; and (c) unsaturation in the side-chain is present as a conjugated diene; the presence of a cumulated diene system is precluded by absorption spectra data.

A. T. P.

**Absorption of piperitenone and related ketones in the ultra-violet.**—See A., 1942, II, 416.

**Absorption spectra of terpenoid compounds.**—See A., 1942, II, 415.

**Raman frequencies of HDO.** Y. P. Rao (*Indian J. Physics*, 1942, 16, 205—209).—Raman spectra of  $H_2O$ - $D_2O$  mixtures (1:9 and 9:1) are reported and discussed. The frequencies of HDO are 2523 and 3363  $cm^{-1}$ .

W. R. A.

**Raman effect in molybdomalic complexes.** (Mlle.) M. Théodoresco (*Compt. rend.*, 1942, 214, 312—315).—For neutral  $NH_4$  molybdomalate solution, 19 Raman frequencies and a broad band at 1570—1688  $cm^{-1}$ , and for the acid salt, 14 frequencies and a band at 1570—1716  $cm^{-1}$ , are reported. Results are discussed in relation to mol. structure.

N. M. B.

**Proper frequencies of halogen derivatives of normal saturated aliphatic hydrocarbons.** M. Parodi (*Compt. rend.*, 1942, 214, 542—544).—Mathematical. A method of evaluating the frequencies for two types of chain compound is deduced. Vals. calc. for 11 compounds are tabulated, and satisfactory agreement with experiment is shown.

N. M. B.

**Raman spectra of thiazole and its derivatives. I. Thiazole. II. Mono- and di-substituted derivatives of thiazole.** R. Manzoni-Ansidei and G. Travagli (*Gazzetta*, 1941, 71, 677—680, 680—685).—I. The Raman spectrum of thiazole (improved prep. from diazotized 2-aminothiazole and  $H_3PO_2$ ) is very similar to that of thiophen, i.e., is of aromatic character, without evidence of an ethylenic linking. Some slight deviations are attributed to the presence of the two hetero-atoms.

II. Raman spectra of 2-chloro- (I) (Gattermann prep.), 2-methyl- (II), 2:5-dimethyl- (III), 2-amino- (IV), and 2-anilino-thiazole (V) are determined. Lines ascribed to aromatic C-H are observed with (I), (II), and (IV), but not with (III) (of which the spectrum was incompletely observed, owing to fluorescence) or (V). With (IV), three lines ascribed to aliphatic C-H are observed, suggesting the tautomerism:  $CMe:CH:N:CMe \rightleftharpoons CMe:CH:NH:C:CH_2 \rightleftharpoons CH_2:C:CH_2:NH:C:CH_2$ .

E. W. W.

**Raman effect. CXXI. Butadiene, pure and in solution.** K. Bradacs and L. Kahovec (*Z. physikal. Chem.*, 1940, B, 48, 63—69).—The Raman spectrum of  $(CH_2:CH)_2$  in isolation and in solution in a no. of org. solvents has been examined with normal and high dispersion, and polarisation data have been determined. The results are in agreement with a rigid *trans*-configuration.

L. J. J.

**Raman effect. CXXII. Association of formamide.** L. Kahovec and H. Wassmuth (*Z. physikal. Chem.*, 1940, B, 48, 70—81).—For liquid and solid  $HCO-NH_2$  (I), and for (I) combined with cryst. salts, Raman frequencies are allocated to chain-, CH-, and  $NH_2$ -linkings. The association of liquid (I) is considerably more modified by crystallisation either alone or with salts than by addition of salts to its aq. solution.

L. J. J.

**Molecular volume and structure. III, IV.** T. W. Gibling (*J.C.S.*, 1942, 661—665, 665—666; cf. A., 1941, I, 324).—III. Corrections to be applied in calculating parachor vals. due to ring formation and interaction between unbonded substituent groups containing C, H, and O are assessed. Polarity in alkylbenzenes and aromatic ethers causes a decrease in the parachor. Parachor vals. agree with a skew configuration for benzil and with a structure for  $Ph_2CO_3$  similar to that of the Me and Et esters.

IV. Parachors of *n*-fatty acids increase with mol. wt. in the same way as those of the alkyl esters up to  $C_5$ . From  $C_5$  to  $C_{18}$  the parachor increment per  $CH_2$  is 40.85 (normal val. 39.8) owing to their being mainly in dimeric forms. Extrapolation of parachor vals. for AcOH at varying temp. to  $0^\circ K$ . gives 260.4 for the parachor of dimeric AcOH.

L. J. J.

**Physico-chemical constants of oxygen as gas-standard.** E. Moles (*Compt. rend.*, 1942, 214, 424—425).—The most accurate available vals. for the mol. vol. of  $O_2$  and the gas const.  $R$  are  $22.4137 \pm 0.0001$  l. and  $0.082056 \pm 0.00001$  l.-atm. The compressibility per cm. is  $1.75 \pm 0.05 \times 10^{-5}$ .

L. J. J.

**Dispersion of liquids and solutions. II. Dispersion of water in the visible and ultra-violet.** A. Kruis and W. Geffcken (*Z. physikal. Chem.*, 1940, B, 45, 438—450; cf. A., 1943, I, 43).— $n^{25}$  of  $H_2O$

has been measured for 74  $\lambda\lambda$  between 6680 and 2120  $\text{Å}$ , with accuracies, depending on  $\lambda$ , of  $1 \times 10^{-6}$  or  $10^{-5}$ . The determination of  $\lambda$  by this method is suggested.

W. R. A.

**Thermodynamics of a relativistic Fermi-Dirac gas.** D. S. Kothari and B. N. Singh (*Proc. Roy. Soc.*, 1942, A, 180, 414—423).—Expressions for the energy, pressure, free energies, and entropy of a Fermi-Dirac ideal gas are derived taking account of the effect of relativistic mechanics. The degenerate and non-degenerate cases are considered.

G. D. P.

**Thermal differences of *o*- and *p*-hydrogen.** K. Schäfer (*Z. physikal. Chem.*, 1940, B, 45, 451—464).—Polemical against Cohen and Urey (A., 1939, I, 515). Differences in  $C_p$  and v.p. of *o*- and *p*- $H_2$ , incompletely described previously (A., 1939, I, 311), are explained by the temp.-dependence of the internal field and not by a variation of intermol. forces. This temp. effect is calc., and the difference in latent heat of vaporisation is const., in agreement with experimental data, whereas, on the theory of Cohen and Urey, it should be dependent on temp.

W. R. A.

**Propagation of a thermal disturbance.** B. Baule (*Z. physikal. Chem.*, 1941, B, 49, 102—106).—The propagation of a momentary burst of heat is obtained by consideration of the equation for thermal conductivity. The thermal disturbance is propagated with decreasing velocity and rapidly decreasing intensity. The time taken for the disturbance to travel a distance  $r \propto r^2$ .

A. J. M.

**Nature of the covalent binding.** T. Berlin and K. Fajans (*J. Chem. Physics*, 1942, 10, 691—692).—Calculations of binding energy, made with the same assumptions as used in Bohr's treatment of  $H_2$ , indicate not only that binding in the covalent mols.  $H_2^+$ ,  $H_2$ , and  $Li_2$  is caused by Coulombic forces but that part of the potential energy is due to the temporary polarity of the mols.

C. R. H.

**Elementary association and dissociation processes on crystal surfaces and equilibrium conditions for finite crystals.** R. Kaishev (*Z. physikal. Chem.*, 1940, B, 48, 82—90).—The average life of a vacant lattice space on a cryst. surface at a given vapour concn. is determined, not only by the work of separation of a lattice unit, but also by its average vibration-vol., which must be the same for all faces at equilibrium.

L. J. J.

**Crystal theory of metals: calculation of the elastic constants.** K. Fuchs and H. W. Peng (*Proc. Roy. Soc.*, 1942, A, 180, 451—476).—Approx. equations of motion for the electrons in a cyclic lattice of a metal are set up with the help of the self-consistent field. The displacements of the ions are then considered as perturbations of the motion of the electrons. The change of potential energy of the lattice due to a deformation is calc. The vals. of the elastic consts. are in satisfactory agreement with observed vals.

G. D. P.

### III.—CRYSTAL STRUCTURE.

**Effect of thermal vibrations on the scattering of X-rays. III.** M. Born (*Proc. Roy. Soc.*, 1942, A, 180, 397—413; cf. A., 1942, I, 135, 389).—Raman's explanation of diffuse scattering is compared with the thermal theory and it is shown that the modern forms of the latter are based on quantum principles. The relation between the dynamical matrix (describing the mechanical properties of the lattice) and the scattering matrix (describing the scattering power) is investigated. The background intensity determines the dynamics of the lattice in the same way that the Laue-Bragg patterns determine the geometry of the lattice. The influence of the mutual deformation of the atoms on the background scattering is considered.

G. D. P.

**Diffuse scattering of X-rays by crystals. II. Detailed calculation of the surfaces of isodiffusion for the (002), (112), (222), and (110) reflexions of sodium single crystals.** H. A. Jahn (*Proc. Roy. Soc.*, 1942, A, 180, 476—483; cf. A., 1942, I, 198).—Based on the theory of previous publications, detailed calculations are made for the diffuse reflexions of Na single crystals. The results are in excellent agreement with the experiments of Lonsdale and Smith. The Faxén-Waller theory of diffuse scattering receives decisive confirmation.

G. D. P.

**Structure of a metal during deformation.** D. Harker (*J. Chem. Physics*, 1942, 10, 692).—X-Ray diffraction patterns of a Cu ribbon obtained during and after distortion at room temp. are indistinguishable. This is taken to indicate that  $\frac{2}{3}$  of the Cu was non-cryst. and that the cryst. material was of the same particle size and state of strain 0.01 sec. or several min. after deformation, i.e., a metal recrystallises as fast as it is deformed.

C. R. H.

**Structure of silver chlorate,  $AgClO_3$ .** S. von Náray-Szabó and J. Pöczá (*Z. Krist.*, 1942, 104, 28—38).—Approx. goniometric data, verifying  $c/a = 0.932$ , are recorded. The class  $C_{4h}$ , assigned from morphological features, is confirmed by Laue photographs and by the absence of piezoelectric properties. Oscillation X-radiograms give  $a$  8.486,  $c$  7.894  $\text{Å}$ ,  $c/a$  0.930;  $\rho_{\text{obs.}}$  4.37; 8 mols. per unit cell; space-group  $C_{4h}^1$ — $I4/m$ . Intensity data obtained with an ionisation spectrometer are recorded. The at. parameters are derived, partly

by assuming the symmetry of the  $\text{ClO}_3$  group. The Ag atoms are of two co-ordination types [(a) 4 O at 2.47 Å.; (b) 4 O at 2.52 and 4 O at 2.55 Å.].  $\text{AgBrO}_3$  and  $\text{AgClO}_3$  are isomorphous. A. J. E. W.

**Structure of  $\gamma\text{-Ca}_2\text{SiO}_4$  and  $\text{Na}_2\text{BeF}_4$ .** H. O'Daniel and L. Tschischwili (*Z. Krist.*, 1942, 104, 124—141).— $\gamma\text{-Ca}_2\text{SiO}_4$  is of the  $\text{Si}_2$ -olivine type, with  $a$  5.06,  $b$  11.28,  $c$  6.78 Å.  $\text{Na}_2\text{BeF}_4$  has  $a$  4.89,  $b$  10.90,  $c$  6.56 Å. A hexagonal modification of  $\text{Na}_2\text{BeF}_4$  is formed at high temp. The isomorphism of  $\text{X}_2\text{SiO}_4$  ( $\text{X} = \text{Mg, Ca, Fe}^{II}, \text{ or Mn}$ ) is discussed. A. J. M.

**Crystal structure of rubber hydrochloride.** C. W. Bunn and (Mrs.) E. V. Garner (*J.C.S.*, 1942, 654—658).—X-Ray fibre photographs of rubber hydrochloride taken with the fibre axis both perpendicular to the beam and oscillating with respect to it, using specimens oriented by extension, give a cell spacing ( $c$ ) 8.95 Å. along the fibre axis, with  $a$  5.83 Å. and  $b$  10.38 Å. This gives a cell containing 4  $\cdot\text{CMeCl}\cdot\text{CH}_2$  units, with a true density 1.255 for the single crystal. The space-group is the monoclinic (pseudo-orthorhombic)  $P2_1/c$ , with two chains passing through the unit cell. The chain-form involves staggered C bonds. All C—C distances are 1.54 Å. and C—Cl 1.79 Å.; C—C bond angles are all  $112 \pm 4^\circ$  in the chains, but Me groups are distorted with respect to adjacent C—C bonds. The structure confirms Markovnikov addition of HCl to rubber. Successive  $\text{CMeCl}\cdot\text{CH}_2$  groups are alternately right- and left-handed. L. J. J.

**Structure of tetramethylhaematoporphyrin.** C. H. O'Daniel and A. Damaschke (*Z. Krist.*, 1942, 104, 114—123).—The substance has  $a$  31.3,  $c$  19.56 Å.; 6 mols. in elementary rhombohedral cell. Space-group  $C_{2h}^3$ —R3. As in phthalocyanine, the porphyrin ring is planar. A. J. M.

**Co-ordinate sexavalency in amines of cupric halides.** G. Peyronel (*Gazzetta*, 1941, 71, 363—375).—The Debye spectra of the amines  $[\text{Cu}(\text{NH}_3)_6]\text{Br}_2$  (I) and  $[\text{Cu}(\text{NH}_3)_4]\text{I}_2$  (II) are determined. These have octahedral symmetry (fluorite type) with lattice consts.  $a_{(001)}$  10.30 Å.,  $a_{(100)}$  10.72 Å. Optical anisotropy is slight (not seriously altering the cubic lattice) in (I), but great in  $[\text{Cu}(\text{NH}_3)_4]\text{Cl}_2$  (III), which cannot be assigned cubic structure. The lattice is retained during thermal decomp. of (I), (II), and (III) down to  $(\text{NH}_3)_{5.05}$ , at which a lattice of low symmetry is formed, persisting down to  $(\text{NH}_3)_{2.2}$ . (I) prepared in liquid  $\text{NH}_3$  shows slight lattice distortion (hysteresis), which is lost after gentle heating. Magnetic susceptibility of (I), (II), and (III) is that normal for complex salts of  $\text{Cu}^{II}$ . E. W. W.

**Structures of thuringite, bavalite, and chamosite, and their place in the chlorite group.**—See A., 1943, I, 44.

**Structures of gallium and indium trihalides.** D. P. Stevenson and V. Schomaker (*J. Amer. Chem. Soc.*, 1942, 64, 2514).—Interat. distances, recalculated by applying the radial distribution method to Brode's data (*Ann. Physik*, 1940, 37, 344), differ from his vals. and the correction he applied is considered unsuitable. The new vals. agree with those obtained by using the usual correlation method. Further, the application of the radial distribution method supports Brode's conclusion that  $\text{GaI}_3$  is monomeric and coplanar, but  $\text{GaCl}_3$ ,  $\text{GaBr}_3$ ,  $\text{InCl}_3$ ,  $\text{InBr}_3$ , and  $\text{InI}_3$  are dimeric. W. R. A.

**Etching patterns on aluminium surfaces.** I. H. Mahl and I. N. Stranski (*Z. physikal. Chem.*, 1942, B, 51, 319—346).—Etching of Al foils by HCl and HCl—HF mixtures in  $\text{H}_2\text{O}$  and EtOH, and by electrolysis, gives a cubic relief, sometimes concealed (e.g., in purest Al) by a pseudo-pyramidal relief. The etched surface is first covered by a stable oxide film, which is then released by placing in aq.  $\text{HgCl}_2$ . Adhering metal is removed by dil. acid and the film is photographed in an electron microscope. The form of the relief depends on the nature of the impurities in the Al, and its formation on the reaction of Al in the media and on dissolution of reaction products. Observed results are attributed to initial formation of an oxide layer. W. R. A.

**Investigation of the fine structure of macromolecular substances with the electron microscope. I. Structure of  $\beta$ -polyoxymethylene crystals.** M. von Ardenne and D. Beischer (*Z. physikal. Chem.*, 1940, B, 45, 465—473).— $\beta$ -Polyoxymethylene crystals on mechanical disintegration yield fibres of 0.04—0.07  $\mu$ . diameter varying periodically along the axis, and finer fibrils of 50—100 Å. diameter. Crystals etched with 2.5N-NaOH show a sieve-like structure, with large parallel transverse fibres, 0.36  $\mu$ . apart, and fine longitudinal fibres, 50—100 Å. in diameter. The fine fibrils have a diameter corresponding with 100 parallel-packed mols. The techniques of preparing and photographing specimens are discussed. W. R. A.

**X-Ray and optical measurements on  $\beta$ -lactoglobulin.** I. Fankuchen (*J. Amer. Chem. Soc.*, 1942, 64, 2504—2505).—Orthorhombic crystals of  $\beta$ -lactoglobulin are tabular. The smallest refractive index,  $a$ , lies in the main prism face perpendicular to the prism length. The other vibration directions,  $\beta$  and  $\gamma$ , are parallel to the thickness and length of the crystal. Vals. of  $c$ ,  $a$ , and  $b$  are 111, 60, and 62 Å., in good agreement with Crowfoot's vals. (*Chem. Rev.*, 1941, 28, 215). W. R. A.

**Metamictic state.** A. Faessler (*Z. Krist.*, 1942, 104, 81—113).—A series of rare-earth minerals exists in the metamictic state, i.e., in

a vitreous, amorphous form, although preserving their external crystal appearance. A list of known examples is given. The transformation from the metamictic to the crystal state has been investigated in gadolinite (I) and fergusonite. It takes place rapidly at high temp. with the emission of light, but also occurs slowly at lower temp. without light emission. When investigated by the Debye-Scherrer method the very brightly glowing (I) gave no interference pattern, but after being heated it gave a crystal pattern. The sp. heat of metamictic (I) is 0.006 > that of crystal (I). The heat of transformation is  $41,700 \pm 1700$  g.-cal. per g.-mol. The transformation to the metamictic state is probably brought about in nature by  $\alpha$ -rays, and is associated with a comparatively unstable lattice, such as would be present in a substance which, like (I), shows great tendency towards isomorphism. A. J. M.

## IV.—PHYSICAL PROPERTIES OF PURE SUBSTANCES.

**Physical constants of  $N$ -octyl-, -dodecyl-, and -cetyl-piperidine.** F. H. Stross and R. J. Evans (*J. Amer. Chem. Soc.*, 1942, 64, 2511).—Vals. are given for b.p.,  $\rho_4^{20}$ ,  $n_D^{20}$ , and  $pK_{\text{H}^+}$ . W. R. A.

**Heat capacity of organic vapours. IV. Benzene, fluorobenzene, toluene, cyclohexane, methylcyclohexane, and cyclohexene.** J. B. Montgomery and T. De Vries (*J. Amer. Chem. Soc.*, 1942, 64, 2375—2377).—Vals. of  $C_p$  at 1 atm. and from their b.p. to  $410^\circ \text{K}$ . have been measured for vapours of  $\text{C}_6\text{H}_6$ , PhF, PhMe, cyclohexane, methylcyclohexane, and cyclohexene and are compared with semi-empirically calc. vals. W. R. A.

**Heat capacity of hexadeuterobenzene.** W. T. Ziegler and D. H. Andrews (*J. Amer. Chem. Soc.*, 1942, 64, 2482—2485).—Vals. of  $C_p$  for  $\text{C}_6\text{D}_6$  have been determined from 101.9° to 322.6° K. with an accuracy of  $\sim 2\%$  and are compared with semi-theoretically calc. vals. The agreement indicates that the low  $\nu$  of  $\text{C}_6\text{D}_6$  are correctly assigned. It is suggested that some of the medium-valued  $\nu$  have been assigned too low vals. W. R. A.

**Empirical heat-capacity equations of gases.** H. M. Spencer and G. N. Flannagan (*J. Amer. Chem. Soc.*, 1942, 64, 2511—2513).—Consts. of empirical equations of the form  $C_p = a + bT + cT^2$  have been evaluated from spectroscopic and thermochemical data on some 60 polyat. (including some triat.) mols. W. R. A.

**Molar heat, heats of transition, fusion, and vaporisation, moment of inertia, and entropy of monogermane,  $\text{GeH}_4$ .** K. Clusius and G. Faber (*Z. physikal. Chem.*, 1942, B, 51, 352—370).— $\text{GeH}_4$  has three solid phases, and the  $C_p$ - $T$  curve shows anomalies at 62.9°, 73.2°, and 76.5° K., the two latter representing transitions. Phase III exists from 0° to 73° K., phase II from 73° to  $\sim 76.5^\circ \text{K}$ ., and phase I from  $\sim 76.5^\circ$  to 107.26° K. (m.p.). B.p. 184.80° K.; heats of transition: III  $\rightarrow$  II 130.7; II  $\rightarrow$  I 129.6 g.-cal. per mol.; heats of fusion and vaporisation are 199.7 and 3361 g.-cal. per mol. Optically phase I has a more symmetrical lattice than II or III. Moment of inertia is  $8.3 \times 10^{-40}$  g.-cm.<sup>2</sup>. Entropy at 184.80° K. is  $46.56 \pm 0.20$  g.-cal. per degree per mol. from thermochemical data, in good agreement with the val. 46.60 derived statistically. W. R. A.

**Equilibrium and nucleus formation in melting and freezing.** U. Dehlinger (*Physikal. Z.*, 1941, 42, 197—203).—The range of accuracy of the Lennard-Jones theory of melting is discussed. Theory and experiment indicate that it requires modification in the case of metals. A differential equation for the formation of nuclei in a supercooled melt is obtained. It agrees qualitatively with experiment on the effect of temp. Another equation holds for crystal growth. A. J. M.

**Definition of mol. wt., the mol., and Loschmidt's number.** W. H. Westphal (*Physikal. Z.*, 1942, 43, 329—331).—Pohl's interpretation of mol. wt. as a dimensionless no. (cf. A., 1942, I, 318) is questioned, mol. wt. being regarded as the true mass of one mol. On this basis Loschmidt's no. remains dimensionless, but certain units applied to mol. quantities must be revised. A. J. E. W.

**Specific gravity of deuterium oxide at different temperatures.** K. Wirtz (*Naturwiss.*, 1942, 30, 330—332).—The ratio of the sp. gr. of  $\text{D}_2\text{O}$  at different temp. has been determined, based on the val. of  $d_{20}^{20}$  obtained by Tronstad *et al.* (A., 1938, I, 364). From the  $d$  of  $\text{D}_2\text{O}$  and ordinary  $\text{H}_2\text{O}$  it is calc. that  $\text{D}_2\text{O} : \text{H}_2\text{O} = 1 : 5960$ . A. J. M.

**Vapour pressures of indene, styrene, and dicyclopentadiene.** P. F. Burchfield (*J. Amer. Chem. Soc.*, 1942, 64, 2501).—V.p. data for indene (56.2—181.8°), styrene (33.5—116.3°), and dicyclopentadiene (I) (40.1—90.8°) are given. At 100° (I) is noticeably depolymerised. W. R. A.

**Premelting anomalies of some long-chain normal paraffin hydrocarbons.** A. Van Hook and L. Silver (*J. Chem. Physics*, 1942, 10, 686—690).—The coeffs. of thermal expansion of  $\text{C}_{14}\text{H}_{30}$ ,  $\text{C}_{18}\text{H}_{38}$ ,  $\text{C}_{18}\text{H}_{38}$ , and  $\text{C}_{24}\text{H}_{50}$ , determined over a wide range below and above the m.p., show increases above the standard vals. in the immediate neighbourhood of the m.p. This suggests an order-disorder mechan-

ism for the solid-liquid transition which may be more effective on the liquid than on the solid side of the m.p. Small amounts (<2%) of likely impurities have little effect on expansion, but large amounts lead to lower m.p. of the solid and to an increased slope of the expansion curve of the liquid. C. R. H.

**Nomograph for calculating reduced temperatures.** D. S. Davis (*Ind. Eng. Chem.*, 1942, **34**, 1174).—The nomograph includes crit. and other temp. for gases and vapours ranging from  $H_2$  to  $C_6H_{18}$ . D. F. R.

**Surface tension-viscosity nomograph for organic liquids.** D. S. Davis (*Ind. Eng. Chem.*, 1942, **34**, 1231).—The nomograph is constructed from Buehler's equation (A., 1939, I, 63) and data for 32 org. liquids. D. F. R.

**Surface determinations and diffusion measurements by means of radioactive inert gases. Technique and quantitative applications of the emanation method. I. Practical. II. Evaluation of results.** K. E. Zimens (*Z. physikal. Chem.*, 1942, **A**, 191, 1—53, 95—128).—I. A comprehensive review of methods using the radioactive inert gases as indicators is given. The production of the radioactive prep. and the estimation of its emanation capacity are discussed. The variation of emanation capacity with temp. is considered, and work with artificial radioactive inert gases is described. A comprehensive bibliography is given.

II. Conclusions that can be drawn from emanation curves are considered. The investigation of processes occurring within solids by the determination of changes in emanation capacity is described. The determination of surface sizes and diffusion consts. of inert gases by analysis of emanation curves is discussed. A. J. M.

## V.—SOLUTIONS AND MIXTURES (INCLUDING COLLOIDS).

**Activity of highly diluted ("potenziert") substances.** K. F. Luft (*Naturwiss.*, 1942, **33**, 505—506).—The effects reported by Heintz (A., 1942, I, 393) could not be confirmed with aq.  $NaNO_3$  or with  $AcOH$  in  $C_6H_6$ . F. L. U.

**Rate of dissolution of crystals.**—See B., 1943, I, 3.

**Potentiometric determination of solubility product of manganese hydroxide.** R. Nasänen (*Z. physikal. Chem.*, 1942, **A**, 191, 54—64).—The solubility product of  $Mn(OH)_2$  in  $NaCl$  and  $KCl$  solutions has been determined potentiometrically. The thermodynamic solubility product is  $1.89 \times 10^{-13}$ . The titration curve for  $MnCl_2$  and alkali hydroxide does not correspond with theory if there is excess of  $MnCl_2$ , owing to the formation of basic chlorides, but a reproducible curve can be obtained if the  $MnCl_2$  is added to the alkali hydroxide. The effect of addition of alkali carbonate on the titration curves can be explained by the attraction of  $HCO_3^-$  and  $MnHCO_3^+$  ions. The dissociation const. of  $MnHCO_3^+$  is  $3 \times 10^{-4}$ . A. J. M.

**Adsorption of water vapour by cellulose.** J. D. Babbitt (*Canad. J. Res.*, 1942, **20**, A, 143—172).—The form of the adsorption isotherm for  $H_2O$  on wood is discussed. The Langmuir isotherm fits this curve for R.H. 0—20%, whilst Brunauer, Emmett, and Teller's relation (A., 1938, I, 190) covering multimol. adsorbed films is in accord with observation at R.H. > 70%, above which range capillary condensation probably occurs. It is pointed out that the latter relation would also apply to unimol. adsorption if a repulsion exists between adsorbed mols. The adsorption on cotton is similar to that on wood but the internal surface area is less. The heats of adsorption—R.H. curves for various forms of wood and cotton are also derived and are shown to be almost coincident. The relationship between the heats of adsorption and of condensation is developed and there is shown to be an excess of internal energy in the adsorbed state over that in the liquid state. The reduction in total vol. when  $H_2O$  vapour is adsorbed on cellulose is attributed to the attachment of the  $H_2O$  mols. by two H-bonds. J. W. S.

**Adsorption of indicator dyes on micelles of paraffin chain salts.** J. Stauff (*Z. physikal. Chem.*, 1942, **A**, 191, 69—94).—Some indicator dyes show colour changes on addition to aq. colloidal solutions of soaps, but electrometric investigations show no change in pH. The colour change is due to adsorption of the dye mols. on the micelles of the soaps. The adsorption, and therefore the colour change, are dependent on  $[H^+]$  and  $[OH^-]$  on the micelle surfaces, and the dissociation const. of the indicator. The equilibrium between adsorbed indicator mols. and  $H^+$  and  $OH^-$  is satisfactorily governed by the law of mass action. A. J. M.

**Surface tension of micelle-forming solutions.** A. E. Alexander (*J. Chem. Physics*, 1942, **10**, 691).—The author criticises Cassel's statement (cf. A., 1942, I, 264) that a cryst. or liquid-cryst. structure can be assigned to adsorbed surface films, and suggests that a superposition of surface tension ( $\sigma$ ) and surface stress is responsible for time effects before the min.  $\sigma$  is reached. C. R. H.

**Resemblance between surface films on solids and on water.** S. J. Gregg (*J.C.S.*, 1942, 696—708).— $FA-FS$  curves ( $F$  = surface pressure of adsorbed film,  $A$  = area occupied per adsorbed mol.,  $S$  =

"sp. surface" of adsorbent), which have been drawn from published isotherms of gases adsorbed on solids, show a close resemblance to those for surface films on  $H_2O$ . Four film states have been considered, viz., gaseous, liquid-expanded and -condensed, and a transition state intermediate between the last two. From the linear portions of a liquid-condensed branch the no. of mols. adsorbable in a completed monolayer ( $x_0$ ) can be calc.  $x_0$  for a given gas and adsorbent is nearly independent of temp., theory predicting only a very slight increase with rise of temp. The formation of an adsorbed layer > 1 mol. in thickness on non-porous adsorbents is marked by a fall in the  $FA-FS$  curve which is connected with a reduction in the differential heat of adsorption. In the case of porous adsorbents the curves show that the adsorbed layer is unimol. throughout the region covered by a hysteresis loop, and that hysteresis originates in the transformation of the liquid-expanded to the liquid-condensed state occurring within the monolayer. C. R. H.

**Physico-chemical metastases.** W. Ostwald (*Kolloid-Z.*, 1942, **100**, 2—57).—The claim of the study of colloid science to an independent status is argued on various grounds, in particular because this study cannot be conveniently accommodated within the framework of classical physical chemistry, which is predominantly concerned with systems in equilibrium. Colloid systems are mostly not in equilibrium, nor do they often consist of phases in the classical sense. Retaining the term "phase" for systems in equilibrium, the author proposes the adoption of "stase" (cf. Friedel, A., 1931, 898) to denote a state of aggregation, formation, or orientation, and "metastase" to denote an intermediate condition (e.g., protoplasm or a jelly). A classification on this basis is outlined. F. L. U.

**Effect of grinding on properties of high polymerides (cellulose and polystyrene) and the nature of its action.**—See B., 1943, II, 9.

**Chain-length differences between celluloses and cellulose nitrates.**—See B., 1943, II, 9.

**Soluble and insoluble forms of gelatin.**—See B., 1943, II, 33.

**Molecular kinetic and electrophoretic studies on carbonic anhydrase.** M. L. Petermann and N. V. Hakala (*J. Biol. Chem.*, 1942, **145**, 701—705).—Carbonic anhydrase (I) has a sedimentation const. of 2.8S, a diffusion const. of  $9 \times 10^{-7}$  sq. cm. per sec., and a mol. wt. of 30,000. The electrophoretic mobility of the enzyme is determined over the pH range 5—9 at const. ionic strength. Its isoelectric point is at pH 5.3. Both amorphous and cryst. preps. of (I), with activities of 9000 or more units per mg. of solids, contain ~15% of impurity. A. T. P.

## VI.—KINETIC THEORY. THERMODYNAMICS.

**Existence and basicity of ammonium hydroxide.** G. Briegleb (*Naturwiss.*, 1942, **33**, 506—508).—It is inferred on thermochemical and spectroscopic evidence that if  $NH_4OH$  is considered as a base in aq.  $NH_3$ , it must be a strong base. It is better, however, to adopt Brønsted's point of view and regard  $NH_4^+$  as a weak acid. It is probable that  $NH_3$  in  $H_2O$  exists only as a hydrate, and not even partly as  $NH_4OH$ . F. L. U.

**New method of graphical representation. Diagrams for binary mixtures involving only straight lines and circles.**—See B., 1943, I, 3.

**Changes in the partial pressures of binary systems with temperature.** K. Fredenhagen and E. Tramitz (*Z. Elektrochem.*, 1942, **48**, 353—361).—When no chemical reaction occurs between the phases, the  $\log p-1/T$  curve ( $p$  = partial pressure) for one component of a binary mixture is parallel to the corresponding curve for the pure component, since the vol. concn. is almost independent of temp. When reaction occurs between the components or intermol. forces are set up between the mols., however, the effective concn. is modified and may vary considerably with temp., thereby giving rise to an anomalous  $\log p$  curve. J. W. S.

**Phase equilibria in hydrocarbon systems. Composition of the dew-point gas of the methane-water system.** R. H. Olds, B. H. Sage, and W. N. Lacey (*Ind. Eng. Chem.*, 1942, **34**, 1223—1227).—The composition was determined experimentally from 100° to 460° F. and up to 10<sup>4</sup> lb. per sq. in. The results agree within 4% with Lauther and Briscoe's vals. for the system natural gas— $H_2O$ . The amount of  $H_2O$  in the gas phase at the higher pressures and lower temp. is ~5 times that expected from simple v.p. considerations. Within the temp. range investigated the crit. pressures for the  $CH_4-H_2O$  system appear to be  $\geq 10^4$  lb. per sq. in. D. F. R.

**Vapour-liquid equilibrium of methanol-ethanol-water. Mechanism of ethanol dehydration.** J. Griswold and J. A. Dinwiddie (*Ind. Eng. Chem.*, 1942, **34**, 1188—1191).—Equilibrium data are given. The system has no ternary azeotrope. The dehydration of EtOH by the addition of  $C_6H_6$ ,  $Et_2O$ , or  $C_2HCl_3$ , followed by an aq. azeotropic distillation, is discussed. Another possible method consists of adding an agent to destroy the  $EtOH-H_2O$  azeotrope and to separate the EtOH as an overhead binary mixture. MeOH is unsuitable for this purpose. D. F. R.

**Affinity and heat changes accompanying mixed crystal formation in the system chromium-nickel.** G. Grube and M. Flad (*Z. Elektrochem.*, 1942, 48, 377—389).—The dissociation pressure of  $\text{Cr}_2\text{O}_3$  in contact with Cr has been determined at 780—1300° and from the results the mean heat of formation of  $\text{Cr}_2\text{O}_3$  in that temp. range is calc. as 265.6 kg.-cal. per g.-mol. The sp. heat of  $\text{Cr}_2\text{O}_3$  between 1000° and room temp. has also been measured and by combining the results with sp. heat data for Cr and  $\text{O}_2$  the heat of formation of  $\text{Cr}_2\text{O}_3$  at room temp. is calc. as 269.1 kg.-cal. per g.-mol. The dissociation pressure of  $\text{Cr}_2\text{O}_3$  in equilibrium with Cr-Ni mixed crystals has also been studied at 1100° and 1200°, and the free energy changes associated with the formation of the alloy are deduced. The results confirm the observation that the reduction of oxides of reactive metals is facilitated by the presence of a less active metal with which mixed crystals can be formed. J. W. S.

## VII.—ELECTROCHEMISTRY.

**Electrical resistance of manganese-copper alloys.**—See B., 1943, I, 37.

**The system aluminium chloride in liquid sulphur dioxide. I. Electrolytic conductivity.** U. Tesei (*Gazzetta*, 1941, 71, 351—363).—The molar conductivity  $\Lambda$  of  $\text{AlCl}_3$  in liquid  $\text{SO}_2$  at low concns. falls, and at high concns. rises, as temp. increases, from  $-10^\circ$  to  $40^\circ$ ; at intermediate concns.  $\Lambda$  rises to a max. and then falls with increasing temp. E. W. W.

**Automatic adjustment of solute concentration in the "moving-boundary" method for the determination of transport numbers.** R. Wright (*J.C.S.*, 1942, 678—680).—The change in concn. of electrolytes (0.5N-HCl + 0.5—0.125N-NaCl, -KCl, or -LiCl) has been determined by direct analysis of solutions withdrawn from different parts of the transport apparatus. For NaCl-HCl transference there is a fall in  $[\text{NaCl}]$  at the boundary in the case of 0.5N-NaCl. With 0.25N-NaCl  $[\text{NaCl}]$  at the boundary remains const., and with 0.125N-NaCl it increases. For KCl and LiCl the salt concns. of the unchanging solutions are 0.3N, and 0.2N, respectively. Application of these concns. to the relation  $T_a/T_b = C_a/C_b$  ( $T$  = transport no.,  $C$  = cation concn. at the boundary, and  $a$  and  $b$  refer to leading and indicator cation respectively) results in fair agreement. C. R. H.

**Is there a film theory of passivity?** R. Weiner and F. Halla (*Z. Elektrochem.*, 1942, 48, 361—377).—Some of the assumptions made in Müller's theory of passivity are shown to be invalid, whilst the quant. inferences arising from the theory are not in accord with observation. It is suggested that the passification is the primary and the coating with a film of salt a secondary process. J. W. S.

**Polarography of naphthaquinone derivatives.** C. Sartori and C. Cattaneo (*Gazzetta*, 1941, 71, 713—722).—The method of Brdička (*Z. Elektrochem.*, 1941, 47, 314) for the polarographic study of reduction of org. substances ( $\text{M} \rightleftharpoons \text{M}'' \rightleftharpoons \text{MH}_2$ ) through an intermediate semiquinonoid phase ( $\text{M}'$ ), with or without reversible dimerisation (to  $\text{M}_2''$ ) is applied to 3-hydroxy-2-methyl- (I) ("phthiocol"; cf. Anderson *et al.*, A., 1934, 77), 2-hydroxy-, 2-methyl-, 2-ethyl-, and 2:3-dimethyl-1:4-naphthaquinone, and to 4-hydroxy-1:2-naphthaquinone. Reduction of (I) is approx. of second order up to pH 10.4, semiquinone and dimeride being produced, the former in greater proportion at greater pH, and of first order from pH 10.4 to 13.08, semiquinone only being obtained. With the other compounds, results are similar: introduction of substituents into the naphthaquinone nucleus displaces the depolarisation potential towards the more negative, in the following order:  $\text{Me} < \text{Et} < \text{Me}_2 < \text{OH} < (\text{OH})\text{Me}$ . E. W. W.

**Oxidation-reduction potentials measured with the dropping mercury electrode. IV. Polarographic study of  $\alpha$ -hydroxyphenazine.** O. H. Müller (*J. Biol. Chem.*, 1942, 145, 425—441).— $\alpha$ -Hydroxyphenazine (I) can exist as a tautomeride the half-wave potential of which is more positive than that of the normal form. The tautomeride does not form a semiquinone over the pH range 1—13 and is not altered by the presence of small quantities of EtOH. It is the only form in which (I) exists below a concn. of  $10^{-4}\text{M}$ . and never exceeds this concn., but diminishes slightly with increasing concn. of (I). The polarographic wave of this tautomeride decreases with increase in the drop time of the electrode so that its presence will not be indicated by a stationary electrode. Below a concn. of  $10^{-4}\text{M}$ ., the potentiometric and polarographic methods show divergent results, but above  $10^{-4}\text{M}$ . the effect of the tautomeride becomes negligible. J. E. P.

## VIII.—REACTIONS.

**Condition of freshly burnt gases.** (A) W. T. David. (B) A. G. Gaydon (*Nature*, 1942, 150, 636; cf. A., 1942, I, 400).—Polemical. A. A. E.

**Upper ignition limits of sulphur in oxygen and in mixtures of oxygen and inert gases.** B. M. Gugel (*J. Phys. Chem. Russ.*, 1941, 15, 31—39).—If  $P_1$  is the pressure of  $\text{O}_2$ , the lowest pressure  $P_2$  of

S which at 180—206° produces a flame is given by  $P_1 = 1.11 \times 10^4 P_2^{0.687}$  mm. Hg. If  $n$  mols. of  $\text{N}_2$ ,  $\text{CO}_2$ , or  $\text{H}_2\text{O}$  are present for 1 mol. of  $\text{O}_2$ ,  $P_1$  is reduced by the factor  $(1 + kn)^{0.5}$ , where  $k = 68.5/M^{0.5}$ ,  $M$  being the mol. wt. of the addendum. The exponent at  $P_2$  is different from the theoretical val. 0.5 probably because of the presence of  $\text{SO}_2$  in the gas mixture before ignition. Lachs and Piekelný's results (cf. A., 1938, I, 146) are not confirmed.

J. J. B.  
**Kinetics of autoxidation of inorganic reducing agents. I. Titanous chloride.** H. A. E. Mackenzie and F. C. Tompkins (*Trans. Faraday Soc.*, 1942, 38, 465—473).—The rate of oxidation of aq.  $\text{TiCl}_3$  by  $\text{O}_2$  is given by  $k[\text{TiCl}_3][\text{O}_2]/[\text{HCl}] + k'[\text{TiCl}_3]^2[\text{O}_2]$ . The rate is largely controlled by hydrolysis and complex formation. A mechanism is suggested. F. L. U.

**Energy-level treatment of reaction data.** J. W. Baker (*Nature*, 1942, 150, 551).—By the use of Audsley and Goss' vals. for the primary, induced, and mesomeric dipole moments of halogenobenzenes (A., 1942, I, 353) relative energy-levels in the reaction  $p\text{-X}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\text{Br} + \text{C}_5\text{H}_5\text{N} \rightarrow \text{X}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2\cdot\text{N}^+\text{C}_5\text{H}_5\text{Br}^-$  have been calc. The vals. predict the same order for the velocities as that previously indicated in agreement with the experimental sequence. A. A. E.

**Kinetics of the oxidation by permanganate by side-chains to the benzene nucleus. I. Oxidation of monochlorotoluenes.** G. Speroni and R. Barchielli (*Gazzetta*, 1941, 71, 765—773).— $\text{KMnO}_4$ , which in presence of  $\text{H}_2\text{SO}_4$  oxidises chlorotoluenes to  $\text{CO}_2$ , in AcOH yields only chlorotoluic acids. The velocities of the (second-order) reactions at 30.2° and 40.2° do not differ greatly as between isomerides; in oxidisability  $p > o > m$ . Heats of activation are:  $o$ , 14,300;  $m$ , 12,600;  $p$ , 14,100 g.-cal. E. W. W.

**Kinetics of oxidation of aldehydes by chromic acid. III. Oxidation of tolualdehydes. IV. Oxidation of bromobenzaldehydes.** E. Lucchi (*Gazzetta*, 1941, 71, 729—736, 752—761; cf. *Boll. Scient. Fac. Chim. Ind. Bologna*, 1940, 1, 208, 333).—III. The velocity of oxidation of  $o$ -,  $m$ -, and  $p$ - $\text{C}_6\text{H}_4\text{Me}\cdot\text{CHO}$  in  $\text{K}_2\text{Cr}_2\text{O}_7\text{-H}_2\text{SO}_4\text{-AcOH}$  is determined at 20°, 30°, 40°, and 50°. In comparison with PhCHO, substitution of Me reduces oxidation velocity, which is greatest in (I). Respective heats of activation are: 12,550, 13,200, 13,050 g.-cal.; activity consts. 2.2, 7.2, and  $5.6 \times 10^8$  (time in min.).

II. In velocity of oxidation by  $\text{K}_2\text{Cr}_2\text{O}_7\text{-H}_2\text{SO}_4\text{-AcOH}$  at 20°, 30°, 40°, and 50°,  $m$ - greatly  $> p$ -  $> o$ - $\text{C}_6\text{H}_4\text{Br}\cdot\text{CHO}$ ; at 20° the last  $>$  PhCHO, at 50° slightly  $<$  PhCHO. Heats of activation are:  $o$ , 12,150;  $m$ , 12,200;  $p$ , 13,200 g.-cal.; activity consts. 1.7, 4.8,  $14.3 \times 10^8$  (time in min.). E. W. W.

**Halogenation of phenolic ethers and anilides. XIII. Arrhenius activation energies for di- and poly-substituted aromatic ethers.** B. Jones (*J.C.S.*, 1942, 676—678; cf. A., 1941, II, 287).—The kinetic study of the nuclear chlorination of aromatic ethers at 15—35° is extended to di- and poly-substituted ethers containing activating as well as deactivating substituents. The  $P$  term of the expression  $k = PZ e^{-E/RT}$  is const. within limits of experimental error, whilst  $E$  varies from 9900 to 14,350 g.-cal. Results confirm views stated previously. Velocity coeffs. for chlorination in 99% AcOH of 2:4:1- $\text{C}_6\text{H}_3\text{Cl}_2\cdot\text{OR}$  ( $R = \text{Me, Et, Pr, Pr}^i$ ), 4:2:1- $\text{C}_6\text{H}_3\text{BrMe}\cdot\text{OR}$  ( $R = \text{Me, CH}_2\text{Ph}$ ), 2-chloro-4-tert-butylphenyl  $p$ -nitrobenzyl ether, m.p. 90°, and 2:4:3:5:1- $\text{C}_6\text{HCl}_2\text{Me}_2\cdot\text{OR}$  ( $R = \text{Me, CH}_2\text{Ph, CH}_2\text{-C}_6\text{H}_4\text{Cl-}p$  or  $-m, \text{CH}_2\text{-CO}_2\text{H}$ ), are recorded. 2:4-Dichloro-3:5-dimethylphenoxyacetic acid has m.p. 146°. A. T. P.

**Kinetics and mechanism of the conversion of crotyl and methylvinylcarbinyl chlorides into acetates and ethyl ethers.**—See A., 1943, II, 21.

**Exchange experiments with radioactive tracers.** S. Ruben, M. D. Kamen, M. B. Allen, and P. Nahinsky (*J. Amer. Chem. Soc.*, 1942, 64, 2297—2298).—Using radioactive tracers exchange occurred at a slow but measurable rate for  $\text{Fe}^{2+}$  and  $\text{Fe}^{2+}$   $o$ -phenanthroline,  $\text{Fe}^{2+}$  and  $\text{Fe}^{2+}$  2:2'-dipyridyl, and  $\text{Mg}^{2+}$  and  $\text{Mg}^{2+}$  8-hydroxyquinoline salt. No exchange occurred between  $\text{Fe}^{2+}$  and ferrihaem, ferrihaemoglobin,  $\text{Fe}^{3+}$  phaeophytin, or  $\text{Fe}^{3+}$  tetraphenylporphyrin, or between  $\text{Cu}^{2+}$  and  $\text{Cu}^{2+}$  phaeophytin. Structural factors appear to be more important than bond character in determining the readiness of the metallic ion in an organo-metallic compound to undergo exchange. W. R. A.

**Absorption of oxygen by glutathione in alkaline solutions. I. Kinetics of the reaction at pH 9—11.** M. B. Young and H. A. Young (*J. Amer. Chem. Soc.*, 1942, 64, 2282—2287; cf. A., 1941, II, 283).—The rate of absorption of  $\text{O}_2$  by glutathione (I), catalysed by  $\text{CuSO}_4$ , in the pH range 9—11 has been measured under varying conditions of pressure of  $\text{O}_2$ ,  $[\text{OH}^-]$ ,  $[\text{Cu}^{2+}]$ , and concn. of (I). (I) is the reactive species. The autocatalytic reaction is independent of concn. of (I) but depends on  $[\text{Cu}^{2+}]$ . A rate law and partial mechanism are proposed. W. R. A.

**Hydrogen exchange of aromatic amines with  $\text{D}_2\text{O}$  and  $\text{T}_2\text{O}$ .** B. J. Fontana (*J. Amer. Chem. Soc.*, 1942, 64, 2503—2504).—Quant. exchange data for  $\text{D}_2\text{O}$  and  $\text{T}_2\text{O}$  with crystal-violet (I), methylene-blue (II), Me-orange, Congo-red, benzidine, and benzidine-HCl (1:1 and 1:2) mixtures are recorded. Vals. of partition ratios ( $k$ ) for

(I) and (II) have been corr. for zero D content and the average val. is 1.04. Vals. of  $k$  for T and D and for T and H are 1.2 and 2.0. W. R. A.

**Kinetics and equilibria of the carbinol formation of phenolphthalein.** (Miss) M. D. Barnes and V. K. La Mer (*J. Amer. Chem. Soc.*, 1942, **64**, 2312—2316).—The kinetics of the reaction at 25° between phenolphthalein (I) and NaOH (0.006—0.020M.) with and without addition of NaCl have been investigated spectrophotometrically. No perceptible secondary salt effect exists. The limiting slope of  $\log k$  (carbinol formation const.) against  $\mu^{\frac{1}{2}}$  ( $\mu$  = ionic strength) indicates that carbinol formation involves reaction between a doubly-charged negative ion of (I) with OH':  $R'' + OH' \rightarrow ROH'''$  (I), but  $\log K$  (equilibrium const.) against  $\mu^{\frac{1}{2}}$  gives a limiting slope half that predicted by equation (1) and this suggests that ROH''' is partly hydrolysed,  $ROH''' + H_2O \rightleftharpoons RHOH'' + OH'$ . W. R. A.

**Tracer studies with radioactive carbon. Synthesis and oxidation of three-carbon acids.** P. Nahinsky, C. N. Rice, S. Ruben, and M. D. Kamen (*J. Amer. Chem. Soc.*, 1942, **64**, 2299—2302).—Syntheses, using  $^{14}CO_2$ , are described for:  $Et^{14}CO_2H$ ,  $OH\cdot CHMe^{14}CO_2H$ , and  $OH\cdot [CH_2]_2^{14}CO_2H$ . On oxidation with alkaline  $KMnO_4$  each anion yields 1 mol. of  $CO_3''$  and 1 mol. of  $C_2O_4''$ . The % of  $^{14}C$  in the  $CO_3''$  and  $C_2O_4''$  have been measured for different concns. of NaOH and ratios of  $\alpha$ -C- $\beta$ -C bond rupture to  $\alpha$ -C- $CO_2H$  bond rupture have been calc. The  $\alpha$ - $\beta$  carbon linking at  $[OH'] > 2N$ . is broken  $2\frac{1}{2}$  times as often as the  $\alpha$ -C- $CO_2H$  for all three anions, and this suggests that lactate or  $OH\cdot [CH_2]_2^{14}CO_2'$  is an intermediate in the oxidation of propionate. At  $[OH'] > 2N$ .  $\alpha$ - $\beta$  rupture in  $EtCO_2'$  is greatly increased but in the other two it is unaltered. Therefore the  $\alpha$ - $\beta$  linking in  $EtCO_2'$  is broken before either position is hydroxylated. A tentative scheme for oxidation of  $EtCO_2'$  is suggested. W. R. A.

**Influence of reaction mechanism on size distribution in polymerides.** E. F. G. Herington and A. Robertson (*Trans. Faraday Soc.*, 1942, **38**, 490—501).—Kinetic study of a polymerisation in conjunction with size distribution data, when growth occurs by addition of a monomeride, enables the reaction to be classified with respect to its mechanism, and affords information about the dependence of the ratio of the velocity coeff. of propagation to that of the termination reaction on the chain length. The relations indicated are developed for some simple cases. F. L. U.

**Catalytic effect of electrolytes on solvolytic reactions.** L. F. Audrieth, L. D. Scott, and O. F. Hill (*J. Amer. Chem. Soc.*, 1942, **64**, 2498—2499).—The influence of salts on the aminolysis of  $CH_2Ph\cdot CO_2Et$  in  $NH_3\cdot Bu^a$  at 25° and on the ammonolysis of  $EtOBz$  in liquid  $NH_3$  at 0° has been investigated. Small amounts of neutral salts speed up the solvolytic reaction. The catalytic effect of equimol. concn. of these salts is  $\ll$  that for the corresponding "onium" salts. What has previously been considered as evidence of acid catalysis in basic solvents by the solvated proton may be a special case of electrolyte catalysis. W. R. A.

**Acid-catalysed hydrolysis of phenyl-substituted aliphatic esters.** H. A. Smith and R. R. Myers (*J. Amer. Chem. Soc.*, 1942, **64**, 2362—2365).—The acid-catalysed hydrolysis in 70%  $CO_2Me_2$  has been investigated for  $Et$  phenylacetate,  $\beta$ -phenylpropionate,  $\gamma$ -phenylbutyrate,  $\delta$ -phenylvalerate, hydratropate, phenylethylacetate, diphenylacetate, and cyclohexylacetate. Reaction rates are decreased by introducing Ph into an aliphatic ester, probably on account of steric effects. The effects of Ph substitution on acid hydrolysis, saponification, and esterification are compared. W. R. A.

**Tracer studies with radioactive hydrogen. (A) Synthesis of labelled methyl iodide. (B) Menschutkin reaction.** D. Harman, T. D. Stewart, and S. Ruben (*J. Amer. Chem. Soc.*, 1942, **64**, 2293—2294, 2294—2296).—(A) Prep. of  $^{14}CH_3I$  (from  $H^{14}CO_2H$  via Na salt, Me ester, and  $^{14}CH_3\cdot OH$ , using  $CuO\cdot CrO_3$  catalyst, then HI) and of  $C^3H^3I$  (I) (from  $HCO_2Me$  and  $^3H^3H$ ; then as for  $^{14}CH_3I$ ) is described.

(B) Reaction between excess of  $NMe_3$  or  $NPhMe_2$  and (I) in  $EtOH$  or  $C_6H_6$  yields a quaternary salt in which all the  $^3H$  from (I) is found, indicating that there is no reaction intermediate reversibly formed in which the  $I'$  ion is detached from the  $C^3H^3H_2$ . W. R. A.

**Catalytic properties of charcoal. IV. Factors influencing the indophenol reaction.** C. Schwob [with J. E. Biegner, K. J. Carson, and G. V. Scott] (*J. Amer. Chem. Soc.*, 1942, **64**, 2276—2279).—The production of indophenol (I) by oxidation of a mixture of  $\alpha$ - $C_{10}H_7\cdot OH$  (II) and  $p$ - $C_6H_4(NH_2)_2$  (III) by  $O_2$  or  $H_2O_2$  in presence of C  $\propto$  the amount of C used, decreases as (II) concn. increases, is not sensibly affected by the concn. of (III), and increases as  $[H^+]$  decreases. The production appears to involve several simultaneous and successive processes and does not constitute an adequate test of the activity of the C. W. R. A.

[Catalytic] cyclisation of hydrocarbon mixtures.—See B., 1943, II, 1.

Catalytic hydrogenation of sulphonated lignin.—See B., 1943, II, 12.

Citrate additions to cuprous cyanide plating solutions.—See B., 1943, I, 33.

Electrodeposition of lead from acetate solution.—See B., 1943, I, 35.

Electrolytic deposition of silver-lead alloys.—See B., 1943, I, 35.

Hard chromium-plating of aluminium alloys.—See B., 1943, I, 38.

[Electrodeposition of chromium.]—See B., 1943, I, 35.

Cathode films in tungstate-containing plating baths.—See B., 1943, I, 36.

Electrodeposition of gold alloys.—See B., 1943, I, 36.

Rhodium plating.—See B., 1943, I, 36.

Electrolysis of Grignard reagents. Short-lived free radicals in ethyl ether.—See A., 1943, II, 49.

**Photo-combination of hydrogen and chlorine in oxygen-free systems.** M. Ritchie and D. Taylor (*Proc. Roy. Soc.*, 1942, **A**, 180, 423—451).—The photo-synthesis of HCl was studied under varied conditions of light intensity and of  $H_2$ ,  $Cl_2$ , HCl,  $N_2$ , and A pressures. The inhibiting actions of  $Cl_2$  and HCl are confirmed. With increasing pressure of HCl the quantum efficiency decreases, but with  $Cl_2$ ,  $N_2$ , and A max. are observed at intermediate pressures. A scheme for the initiation and propagation of reaction chains is proposed. G. D. P.

**Photo-oxidation of methyl iodide.** W. J. Blaedel, R. A. Ogg, jun., and P. A. Leighton (*J. Amer. Chem. Soc.*, 1942, **64**, 2500—2501).—A new mechanism for the photo-oxidation of MeI, involving formation of a peroxide as an intermediate but not formation of OH, has been proposed. In this mechanism the reaction  $Me + I \rightarrow MeI$  is negligible compared with  $Me + I_2 = MeI + I$ . W. R. A.

**Attempted detection of free hydroxyl as an intermediate in photochemical reactions.** W. J. Blaedel, R. A. Ogg, jun., and P. A. Leighton (*J. Amer. Chem. Soc.*, 1942, **64**, 2499—2500).—The attempted detection by absorption spectral methods of free OH formation proved unsuccessful for each of the following:  $MeI + O_2 + hv$ ;  $H_2O_2 + hv$ ;  $H_2O_2 + Hg + hv$ ;  $H_2O + Hg + hv$ ;  $H_2 + O_2 + hv$ ;  $MeOH + Hg + hv$ . Therefore, in the last five reactions, the steady state concn. of OH was  $< 5 \times 10^{-6}$  M. W. R. A.

**Photo-enolisation of ketones.** T. Iredale (*Nature*, 1942, **150**, 579).—In absence of  $O_2$  anhyd.  $CO_2Me_2$  reacts with I after absorption of ultra-violet light, presumably owing to a shift of the equilibrium in the direction of the enol form. The effect is much less pronounced if I is added in the dark to pre-irradiated  $CO_2Me_2$ ; hence catalytic hastening of the attainment of normal equilibrium is unlikely. Irradiated liquid  $CO_2Me_2$  gave no detectable amount of MeI or Ac, (cf. A., 1939, I, 330). A. A. E.

## IX.—METHODS OF PREPARATION.

**Action of fatty acids on copper.** R. Dubrisay (*Compt. rend.*, 1941, **213**, 837—839; cf. A., 1939, I, 254).—A Cu wire is immersed in buffer solutions covered with a PhMe solution of palmitic or stearic acid. Cu stearate or palmitate is pptd. at the interface. Pptn. is more rapid in solutions of pH  $> 6.95$  whilst in the buffer solutions alone Cu ions appear in the aq. solution most rapidly at pH  $< 6.95$ . O. D. S.

**Tervalent salts of copper and silver. II.** L. Malatesta (*Gazzetta*, 1941, **71**, 580—584).—Boiling aq.  $TeO_2$ , KOH, and  $CuSO_4$  (or  $Ag_2SO_4$ ) with  $K_2S_2O_8$  give a solution which with  $Na_2SO_4$  yields the *Na cupri* ( $Cu^{III}$ ) and *argenti* ( $Ag^{III}$ ) tellurates,  $Na_2H_4[Cu^{III}(TeO_6)_2] \cdot 18H_2O$ ,  $Na_2H_2[Cu^{III}(TeO_6)_2] \cdot 12H_2O$ ,  $Na_2H_3[Ag^{III}(TeO_6)_2] \cdot 18H_2O$ , and  $Na_2H_2[Ag^{III}(TeO_6)_2] \cdot 14H_2O$ .  $HAuCl_4$ , KOH, and  $K_2TeO_4$ , followed by  $Na_2SO_4$ , give *Na auritellurate*,  $Na_2H_3[Au^{III}(TeO_6)_2] \cdot 14H_2O$ . E. W. W.

**Preparation and properties of trimeric phosphonitric chloride.** R. Steinman, F. B. Schirmer, jun., and L. F. Audrieth (*J. Amer. Chem. Soc.*, 1942, **64**, 2377—2378).—Phosphonitric chlorides have been prepared by heating a mixture of  $PCl_5$  and (usually excess of)  $NH_4Cl$  for 4—6 hr. at 145—160°. Predominantly the tri- and tetramers were formed and were separated by fractional distillation under 12—14 mm. at 140°. The trimeride, recryst. from hot glacial AcOH and fractionally sublimed at 100°/1 mm., has  $v.p.$  given by  $\log p = -3.978/\theta + 11.187$  ( $\theta = 75.2—114.9^\circ$ ) and  $-2880/\theta + 8.357$  ( $\theta = 114.9—189.3^\circ$ ). W. R. A.

**Formation of complex tungsto-tartrates.** (Miles.) M. Cordier and M. Murgier (*Compt. rend.*, 1941, **213**, 836—837).—A study of the optical rotation of solutions of Na tungstate, tartaric acid (I), and HCl indicates the formation of a complex  $WO_3 \cdot C_4H_6O_6$  with rotation 15—30 times that of (I) and showing mutarotation. The salt  $Na_2[WO_3 \cdot C_4H_6O_6]$  is formed by neutralisation. O. D. S.

**Alkali pyroiodates: their existence and mechanism of formation.** U. Croatto and G. Bryk (*Gazzetta*, 1941, **71**, 590—596).— $I_2O_5$  (from I in  $CCl_4$  and  $N_2O_5$ ; method described) with  $KIO_3$  and  $RbIO_3$  at 170° gives the *pyroiodates*,  $KI_3O_8$ , m.p. 316°, and  $RbI_3O_8$ , m.p. 340°, also obtained by heating  $KH_2I_3O_8$  (at 160°) and  $RbH_2I_3O_8$  (at 150°) in vac. in presence of  $P_2O_5$ . E. W. W.

**Study of corrosion products of iron by means of X-rays and electron diffraction.** N. A. Schischakov and P. S. Moiseev (*J. Phys. Chem.*



*Russ.*, 1941, 15, 101—108).—The oxide film on Fe abraded under H<sub>2</sub>O consists of  $\gamma$ -FeOOH. The rust on Fe kept under H<sub>2</sub>O for 1—3 months consists of  $\gamma$ -FeOOH and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. J. J. B.

**Basic indium salicylates.**—See A., 1943, II, 33.

## X.—ANALYSIS.

**Methods of chromatography.** F. A. Robinson and A. L. Bacharach (*Ind. Chem.*, 1942, 13, 456—460).—A review.

**Determination of mol. wt. by precipitation-titration.** B. Jirgensons (*J. pr. Chem.*, 1942, [ii], 161, 30—48).—This new method of mol. wt. determination is applicable to the members of various series of homologous polymerides of mol. wt.  $10^2$ — $5 \times 10^6$ . It depends on the fact that in such a series the solubility decreases with increasing mol. wt. The weight of pptg. agent required to start pptn. of members of the series is determined. The theory of the method is given. The error is 5—30%, and the range of greatest accuracy is for mol. wt.  $5 \times 10^2$ — $5 \times 10^4$ . A. J. M.

**Absorption spectrophotometry in pharmaceutical analysis.**—See B., 1943, III, 19.

**Determination of chlorine in fat of flour.**—See B., 1943, III, 13.

**Alkalimetric standardisation of iodine solutions.** F. L. Hahn (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 773).—The reaction  $\text{SO}_2'' + \text{H}_2\text{O} + \text{I}_2 = \text{SO}_4'' + 2\text{HI}$  is utilised. The I solution is decolorised by means of aq. Na<sub>2</sub>SO<sub>3</sub> or K<sub>2</sub>SO<sub>3</sub> made slightly alkaline to thymolphthalein. The HI is then titrated with standard NaOH (phenolphthalein). L. S. T.

**Gas analysis with mass spectrometer.** J. A. Hipple (*J. Appl. Phys.*, 1942, 13, 551—560).—Published work on determination of traces of O<sub>2</sub> in N<sub>2</sub>, analysis of hydrocarbon mixtures, gas quantities <0.1 c.c., and tracer analysis is reviewed. A 90°-deflexion instrument is described. L. J. J.

**Volumetric determination of sulphates. Tetrahydroxybenzoquinone as an external indicator.** S. W. Lee, J. H. Wallace, jun., W. C. Hand, and N. B. Hannay (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 839—840).—A drop of 10% aq. solution of the Na salt of tetrahydroxybenzoquinone on filter-paper is used as indicator, and the first change from yellow to pink taken as the end-point. An indicator blank must be applied. H<sub>2</sub>SO<sub>4</sub> solutions are first neutralised with aq. NH<sub>3</sub>. Al<sup>+++</sup> is pptd. by means of aq. NH<sub>3</sub>, and large amounts of NaCl do not interfere. L. S. T.

**Standardisation and stability of 0.1N-sodium thiosulphate solutions in hot weather.** S. O. Rue (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 802—805).—An improved K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> method for standardising 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, particularly at 30—40°, is described. The method is accurate to 0.07% between 23° and 40°. The Vosburgh (A., 1922, ii, 863) and the Willard-Furman methods give, at 40°, high and slightly erratic results. The accuracy of the different K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> methods was checked against pure I. Data for 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solutions treated in various ways and stored at 20° and 40° for ~4 months are recorded. Untreated solutions showed a max. variation in normality of 0.3—0.4%. CHCl<sub>3</sub> and HgI<sub>2</sub> are the best stabilisers, but are effective for only 2 months at 40°. NaOH and Na<sub>2</sub>CO<sub>3</sub> accelerate the decomp. of aq. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> at this temp. L. S. T.

**Source of error in determination of nitrogen.**—See B., 1943, I, 23.

**Perchloric acid in micro-Kjeldahl digestions.** L. F. Wicks and H. I. Firminger (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 760—762).—Results obtained by well-known digestion procedures on samples of serum, milk, urine, protein-free blood filtrates, and h molyse d blood show that the use of HClO<sub>4</sub> can cause serious loss of N<sub>2</sub>. This is particularly significant in micro-determinations. Addition of HClO<sub>4</sub> to hot (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>SO<sub>4</sub> solution removes N<sub>2</sub>. The use of HClO<sub>4</sub> in these digestions is deprecated. L. S. T.

**Kjeldahl distillation without absorbing acid.** J. A. Bradley (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 705—706).—NH<sub>3</sub> is distilled into H<sub>2</sub>O, and titrated directly with standard acid (Me-red or -orange). Loss during distillation is prevented by using a closed still fitted with a small rubber balloon to relieve excess pressure. Test data show that the results obtained are slightly more accurate than those given by the usual method in which absorbing acid is used. With a closed still and absorbing acid even better results are obtained. Different excesses of acid have no effect on accuracy. L. S. T.

**Determination of ammonia by a diffusion method.** A. N. Prater, E. J. Cowles, and R. P. Straka (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 703—705).—NH<sub>3</sub> or other volatile base is liberated in a Petri dish by means of 4% aq. NaOH or of a solution saturated with NaBO<sub>2</sub> and KCl, and absorbed in drops of a saturated solution of H<sub>3</sub>BO<sub>3</sub> in glycerol suspended from the cover of the dish. After absorption is complete (3 hr. at room temp.) the drops are washed into a flask and titrated electrometrically, or by means of 0.01N-HCl (Me-red-bromocresol-green). Data for standard aq. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and a meat extract are given. The method is suitable for 0.1—9 mg. of NH<sub>3</sub>-N. L. S. T.

**Micro-analysis of gases. Nitric oxide-hydrogen, nitric oxide-nitrogen, and nitrous oxide-ammonia mixtures.** R. N. Smith and P. A. Leighton (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 758—759).—NO is removed by adding O<sub>2</sub> in presence of KOH, and determined by difference from analysis of the remaining gas. NH<sub>3</sub> is separated from N<sub>2</sub>O by absorption on a fused bead of CH<sub>2</sub>Cl-CO<sub>2</sub>H. An improved combustion coil is described. L. S. T.

**Colorimetric micro-determination of phosphorus.** C. P. Sideris (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 762—764).—The Berenblum-Chain method (A., 1938, I, 211) is improved by using Sn + HCl instead of SnCl<sub>2</sub> in HCl, replacing EtOH by Bu<sup>o</sup>OH for washing the funnel and diluting the blue phosphomolybdate compound, and heating the unknown with 2N-H<sub>2</sub>SO<sub>4</sub> and NH<sub>4</sub> molybdate. L. S. T.

**Adaptation of an indirect method for potassium to the photoelectric colorimeter.** C. P. Sideris (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 821—822).—The method described previously (A., 1937, I, 264) is improved by using a photoelectric colorimeter with appropriate light filters. Optimum ranges are 0.5—15  $\mu$ g. of K and 1.0—30  $\mu$ g. of Co. L. S. T.

**Solubility of strontium chromate and the detection of strontium.** T. W. Davis (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 709—711).—Data for the solubility of SrCrO<sub>4</sub> in H<sub>2</sub>O and in aq. EtOH at 25°, 50°, and 75° are recorded. Solubility diminishes slightly with a rise in temp. SrCrO<sub>4</sub> in contact with H<sub>2</sub>O undergoes a very slow hydration at room temp., and this, like the formation of a saturated solution at the same temp., is incomplete after 1 year. The Noyes method for the alkaline earths is accelerated by pptg. and filtering SrCrO<sub>4</sub> in hot solution. Cooling the mixture neither eliminates possible pptn. of CaCrO<sub>4</sub> nor promotes more complete removal of Sr. L. S. T.

**Relation of alkaloidal to inorganic chemistry. Bromoauric acid as reagent for inorganic microcrystal tests.** C. C. Fulton (*J. Amer. Pharm. Assoc.*, 1942, 31, 177—182).—HauBr<sub>4</sub> in H<sub>3</sub>PO<sub>4</sub>, which is a valuable reagent for the detection of feebly basic amines (e.g., NH<sub>2</sub>Me), may also be used for identification of Mg, Zn, Cd, Na, Li, K, NH<sub>4</sub>, Rb, and Cs, by application to the dry salt. Typical crystal forms are obtained with Mg and Na. P. G. M.

**Determination of zinc and cadmium by precipitation with sodium anthranilate.** H. Funk (*Z. anal. Chem.*, 1942, 123, 241—244).—After removal of acid by evaporation and addition of dil. aq. Na<sub>2</sub>CO<sub>3</sub> (Me-red), Zn is pptd. hot (A., 1933, 244), and the ppt. dried at 105—110° (factor, 0.1936). Cd is pptd. hot, and filtered cold (factor 0.2922). Interference by Cl<sup>-</sup> is much more marked with Cd than with Zn. A. A. E.

**Evaluation of metallic zinc in zinc dust.**—See B., 1943, I, 34.

**Analytical use of sodium rhodizonate [detection of lead].** F. Feigl and H. A. Suter (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 840—842).—The reactions between Na rhodizonate and neutral or tartaric acid-tartrate (pH 2.8) solutions containing Ag<sup>+</sup>, Hg<sup>+</sup>, Tl<sup>+</sup>, Pb<sup>++</sup>, Cu<sup>++</sup>, Hg<sup>++</sup>, Cd<sup>++</sup>, Bi<sup>+++</sup>, Zn<sup>++</sup>, UO<sub>2</sub><sup>++</sup>, Ba<sup>++</sup>, Sr<sup>++</sup>, Sn<sup>++</sup>, Fe<sup>++</sup>, and Fe<sup>+++</sup> are described. The prep. of blue-violet coloured PbC<sub>6</sub>O<sub>6</sub>.Pb(OH)<sub>2</sub>.H<sub>2</sub>O from neutral solution, and of scarlet-red coloured 2PbC<sub>6</sub>O<sub>6</sub>.Pb(OH)<sub>2</sub>.H<sub>2</sub>O from a tartrate-tartaric acid buffer (pH 2.8), is detailed. The use of these compounds for the detection of Pb in presence of TiCl<sub>3</sub>, Hg<sub>2</sub>Cl<sub>2</sub>, and AgCl, in presence of Ba<sup>++</sup>, in Pb ores and minerals, in alloys, in Pb pigments, and in glass is described. As a drop reaction in a neutral solution of a Pb salt the limit of identification is 0.1  $\mu$ g. (1 : 5  $\times 10^6$ ). The detection of Ba and Sr in the carbonate ppt. of the alkaline-earth metals is described. L. S. T.

**Detection of lead in power petrol.**—See B., 1943, I, 18.

**Conversion of iron into ferrocyanide. Precipitation of vanadium ferrocyanide.** B. S. Evans (*Analyst*, 1942, 67, 355—356; cf. B., 1928, 786).—Sufficient Na<sub>2</sub>CO<sub>3</sub> is added to the HCl-HNO<sub>3</sub> solution of the steel in the presence of citric acid to dissolve any pptd. WO<sub>3</sub>. KCN is then added and SO<sub>2</sub> passed until the reduction is complete and before any tendency towards formation of a blue tinge is apparent. The solution is boiled for 10 min. and should give no blue ppt. with a few drops of HCl. V is then pptd. as a dusty yellow ppt. of VFe(CN)<sub>6</sub> on adding more HCl. S. B.

**Determination of iron, manganese, and magnesium in pharmaceutical preparations by means of 8-hydroxyquinoline.**—See B., 1943, III, 19.

**Zinc-reduction method for determination of iron.**—See B., 1943, I, 33.

**Analysis of chromic acid anodising baths.**—See B., 1943, I, 37.

## XI.—APPARATUS ETC.

**Heating device for micro-beakers, flasks, and centrifuge tubes.** C. R. Noller (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 834). L. S. T.

**Helium liquefaction without pre-cooling with liquid hydrogen.** W. Meissner (*Physikal. Z.*, 1942, 43, 261—274).—A liquefier similar in principle to that of Kapitza (cf. A., 1935, 57) is described in detail.

The expansion engine, operated by He at 30 atm. pre-cooled by liquid air, cools the gas to 14° K. A new inversion curve for the differential Joule-Thomson effect in He supports changes from the pressure and temp. relationships in Kapitza's liquefier. When steady conditions are reached the liquefier requires ~3 kg. of liquid air, and produces 2—2.8 l. of liquid He per hr. A. J. E. W.

**Colour pyrometry.** H. Schmidt (*Z. tech. Physik*, 1942, 23, 88—89).—A modified colour pyrometer is described and data obtained by it for a W band lamp are compared with existing data.

W. R. A.

**Heat capacity of organic vapours. III. Comparison of flow calorimeters.** J. B. Montgomery and T. De Vries (*J. Amer. Chem. Soc.*, 1942, 64, 2372—2375).—From an examination of the utility of various flow calorimeters in determining  $C_p$  for org. vapours, the simple apparatus of Callendar is preferred. A recycling vaporiser for producing a const. rate of flow of vapour has been designed.

W. R. A.

**Dispersion of liquids and solutions. I. New spectro-interferometric method for the accurate measurement of refractive index between 1.0 and 0.2  $\mu$ .** W. Geffcken and A. Kruis (*Z. physikal. Chem.*, 1940, B, 45, 411—437).—A Rayleigh interferometer, interposed between the collimator and prism of a spectrograph, produces three or four interference lines for every spectral line.  $n$  can thus be measured for each separate  $\lambda$  over a range limited only by the material of the apparatus. The accuracy of measurement with a 1-mm. cell is  $\pm 1 \times 10^{-6}$ , provided that correction consts. for the apparatus are applied. Constructional and operational details of the apparatus are given.

W. R. A.

**Fluorescent microscope lamp.** L. H. Berkelhamer (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 833—834).—The lamp described gives sufficient light for high-power microscopy and low-magnification photomicrography without undue heat radiation.

L. S. T.

**Development of the prism spectroscope since the time of Kirchhoff and Bunsen.** G. Hansen (*Physikal. Z.*, 1942, 43, 213—217).—A review of developments in design.

A. J. E. W.

**Comparison of the intensities of X-ray interferences of different objects. Construction of a comparison camera for fibre photographs.** O. Kratky, F. Schosberger, and A. Sekora (*Z. Elektrochem.*, 1942, 48, 409—418).—The conditions necessary for obtaining comparable records of X-ray interference patterns are discussed. A comparison camera which brings the two preps. into the X-ray beam for short periods alternately and throws the images on the same film is described. The intensities of the images are compared photometrically. The application of the method to the comparison of the structures of alkali- and  $CS_2$ -treated cellulose is described.

J. W. S.

**Rapid X-ray diffraction method.** A. T. McCord (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 793—796).—The powdered sample is pressed into a rigid rod 3 mm. in diameter, and is inserted in the camera to form one side of the defining slit. Cryst. powders can be identified irrespective of their coeffs. of absorption, and an optimum set of conditions can be defined for any mixture of two or more substances.

L. S. T.

**Microradiography by reflexion.** J. J. Trillat (*Compt. rend.*, 1941, 213, 833—836).—The structure of alloys containing atoms of widely different at. wts. is photographed by applying a film insensitive to X-rays to a plane surface of a macro-specimen and illuminating with X-rays. The image is formed by the emitted photoelectrons.

O. D. S.

**Electrical ignition of the spectrographic arc.** F. G. Brockman and F. P. Hochgesang (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 796).—A circuit that enables an arc to be struck instantaneously with the electrodes separated at the working distance is described.

L. S. T.

**Measurement of relative abundance with mass spectrometer.** E. B. Jordan and N. D. Coggeshall (*J. Appl. Physics*, 1942, 13, 539—550).—Isotopic abundance measurements are subject to small errors due to the effect of the crossed electric and magnetic fields on the electron beam, and to space-charge effects on ionic paths. With both sector and  $\pi$  radian type instruments measurements are subject to a max. error of 1—2%. Curves giving crit. consts. for mass spectrometers are given.

L. J. J.

**Linearity of the mass scale of Aston's mass spectrograph.** J. B. Seth and C. V. H. Rao (*Indian J. Physics*, 1942, 16, 219—227).—Mathematical. The most linear portion of the mass-distance curve has been investigated using considerations of pure geometry and of co-ordinate geometry. The results agree.

W. R. A.

**All-glass burette system.** T. F. Lavine (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 739).—The side-arm is connected to the storage bottle by means of ball-and-socket joints.

L. S. T.

**Apparatus for iodometric determination of tin.** W. M. Rumberger (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 801).

L. S. T.

**Location of the end-point on certain graphical titration curves.** A. Langer and D. P. Stevenson (*Ind. Eng. Chem. [Anal.]*, 1942, 14,

770—773).—Equations for certain types of titration curves are derived. Possible methods for finding the end-point graphically are described.

L. S. T.

**Steam-distillation apparatus for micro-Kjeldahl analysis.**—See A., 1943, III, 76.

**Pyrex brand glass wool as a filtering medium.** G. B. Heisig (*Ind. Eng. Chem. [Anal.]*, 1942, 14, 766).—Dil. HCl extracts  $Al^{+++}$  from glass wool, Pyrex brand glass No. 719. After repeated extraction and thorough washing, traces of  $Al^{+++}$  are still removable.

L. S. T.

**Device for use in making powder specimens for a high-temperature X-ray camera.** H. Lipson and O. S. Edwards (*J. Sci. Instr.*, 1942, 19, 186).—A holder for use in sealing a  $SiO_2$  rod into a thin-walled  $SiO_2$  tube is figured.

A. A. E.

**Laboratory-type air separator.** G. D. Joglekar (*J. Indian Chem. Soc., Ind. Ed.*, 1942, 5, 102—106).—The construction of a laboratory cyclone-type air separator is described. The ground mass is taken by the air-blast into a cylindrical chamber in which coarse particles are removed. The chamber is connected by an outlet pipe to a dust box and filter bags.

R. J. W. R.

**Cutting wide-bore glass tubing and repairing partly broken chemical glassware.** H. F. Taylor (*Chem. and Ind.*, 1942, 531—532).—The scratch flanked by wet paper bands need be only 0.5 in. long, and the tube is rotated rapidly in a batswing flame. Winchester quart bottles and Pyrex beakers may be cut.

A. A. E.

**Laboratory shaking machine.**—See A., 1943, III, 75.

**Simple test-tube rack and comparator. Nomographic chart for colorimeter. Simplification of fluorescence microscopy. Mercury vacuum release for ultrafiltration etc.**—See A., 1943, III, 76.

[Review of] laboratory apparatus progress. R. H. Powell (*Paint Manuf.*, 1942, 12, 128—129, 155).

D. R. D.

### XIII.—GEOCHEMISTRY.

**Why the sea is salt.** C. H. White (*Amer. J. Sci.*, 1942, 240, 714—724).—A discussion.

L. S. T.

**Geology and metamorphism in the McVeigh Lake area, N. Manitoba.** J. D. Bateman (*Amer. J. Sci.*, 1942, 240, 789—808).

L. S. T.

**Pyrrhotite; melting relationships and composition.** E. Jensen (*Amer. J. Sci.*, 1942, 240, 695—709).—The field of stability of pyrrhotite is reproduced from existing data and from new data obtained for the system FeS-Fe by the differential heating-cooling curve method. The mixtures were sealed in specially-designed  $SiO_2$  glass containers to prevent oxidation and loss of S at high temp. A prep. corresponding with the formula FeS melts over a large temp. range with dissociation into a solid richer in S and a liquid richer in Fe. An increase in S content raises melting temp. to a max. where the composition  $Fe_{12}S_{13}$  melts sharply like a single compound. Mixtures with more Fe than corresponds with FeS show eutectic melting.

L. S. T.

**Structures of thuringite, bavalite, and chamosite, and their place in the chlorite group.** W. von Engelhardt (*Z. Krist.*, 1942, 104, 142—159).—Thuringite (I) from Schmiedefeld and Zirmsee, and bavalite from Brittany, belong to the chlorite group, and may be considered to be the Fe-rich terminal members of the series. The lattice consts. of these minerals have been determined. Chamosite (II) from Schmiedefeld has a structure very similar to that of chlorite, but there are deviations. The fact that the elementary cell of (II) is of greater vol. than that of (I) indicates that (I) is probably a metamorphic product of (II).

A. J. M.

**Crystal structure of  $\alpha$ - $AlOOH$  (diaspore). II. Fourier analysis.** W. Hoppe (*Z. Krist.*, 1942, 104, 11—17; cf. *ibid.*, 1940, 103, 73—89).—A Fourier projection of the structure on (001) is constructed from photometric intensity measurements on Weissenberg-Böhm X-ray diagrams (Cu  $K\alpha$  radiation). The recorded intensity vals. are derived by comparison with spectrometric abs. intensities for selected reflexions. Supplementary data for Mo  $K\alpha$  radiation are given. The at. parameters for Al and O agree closely with those obtained by successive approximation.

A. J. E. W.

**Structure of leucite,  $KAlSi_3O_8$ .** S. von Náráy-Szabó (*Z. Krist.*, 1942, 104, 39—44).—Oscillation and powder X-ray diagrams, data for which are recorded, give  $a$  13.01,  $c$  13.82 Å; 16 mols. per unit cell; space-group  $C_{4v}^2-14_1/a$ ; the crystals are not piezoelectric. Comparison of the observed intensities with those given by analcime and pollucite confirm that the Si-Al-O structures are similar in the three minerals; the  $K^+$  ions in leucite occupy similar positions to  $Cs^+$  ions in pollucite, although their smaller size causes deformation of the lattice.

A. J. E. W.

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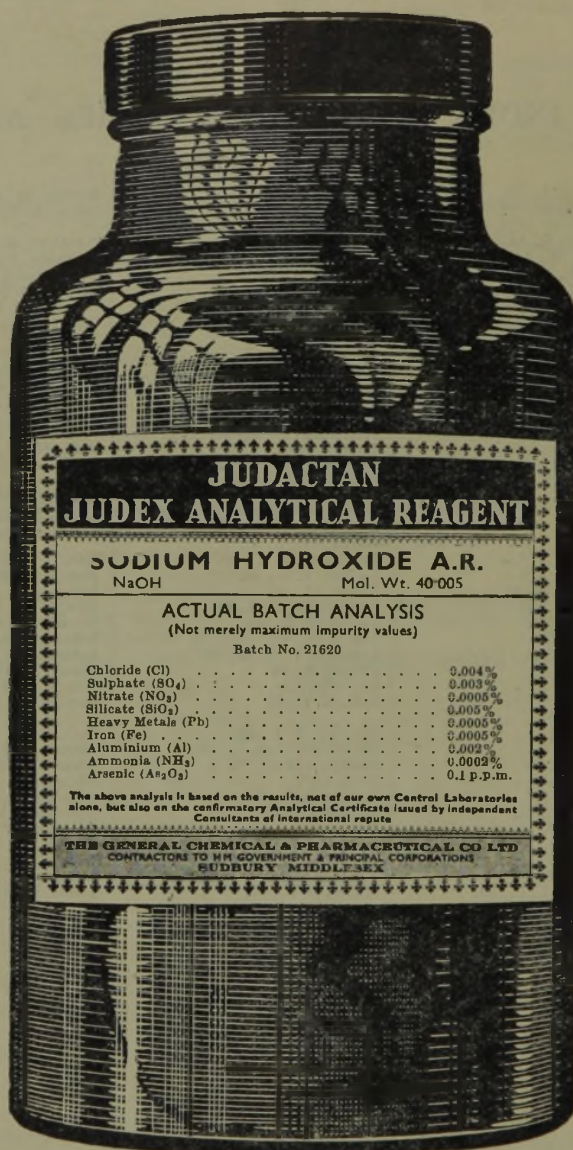
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