

only slowly decomposed into  $I_2$  and  $(CN)_2$ . The vacuum will need to be renewed constantly due to the volatility of CNI. [*Org Synth* 32 29 1952].

**Decaborane** [17702-41-9] **M 122.2, m 99.7-100°**. Purified by vacuum sublimation at 80°/0.1mm, followed by crystn from methylcyclohexane, methylene chloride, or dry olefin-free-*n*-pentane, the solvent being subsequently removed by storing the crystals in a vacuum desiccator containing  $CaCl_2$ .

**Deuterium** [7782-39-0] **M 4**. Passed over activated charcoal at -195° [MacIver and Tobin *JPC* 64 451 1960]. Purified by diffusion through nickel [Pratt and Rogers, *JCSFT* 192 1589 1976].

**Deuterium oxide** [7789-20-0] **M 20, fp 3.8°/760mm, b 101.4°/760mm, d 1.105**. Distd from alkaline  $KMnO_4$  [de Giovanni and Zamenhof *BJ* 92 79 1963]. **NOTE that  $D_2O$  invariably contains tritiated water and will therefore be RADIOACTIVE; always check the radioactivity of  $D_2O$  in a scintillation counter before using.**

**cis-Diamminedichloroplatinum(II)** [15663-27-1] **M 300.1**. Recrystd from dimethylformamide and the purity checked by IR and UV-VIS spectroscopy. [Raudaschl et al. *Inorg Chim Acta* 78 143 1983].

**Diammonium hydrogen orthophosphate** [7783-28-0] **M 132.1**. Crystd from water (1ml/g) between 70° and 0°.

**Di-*n*-amyl *n*-amylphosphonate** [6418-56-0] **M 292.4**. Purified by three crystns of its compound with uranyl nitrate from hexane. For method see *tributyl phosphate*.

**6,6-Dibenzyl-14-crown-4, (lithium ionophore VI; 6,6-dibenzyl-1,4,8,11-tetra-oxa-cyclotetradecane)** [106868-21-7] **M 384.5, m 102-103°**. Dissolve in  $CHCl_3$ , wash with saturated aqueous NaCl, dry with  $MgSO_4$ , evaporate and purify by chromatography on silica gel and gradient elution with  $C_6H_6$ -MeOH followed by preparative reverse phase HPLC on an octadecyl silanised silica (ODS) column and eluting with MeOH. It can be crystd from MeOH ( $\nu_{KBr}$  1120  $cm^{-1}$ , C-O-C). [*JCS Perk I* 1945 1986].

**Di-*n*-butyl boron triflate (di-*n*-butylboryl trifluorosulphonate)** [60669-69-4] **M 274.1, b 37°/0.12mm, 60°/2mm**. Distil in vacuum under argon and store under argon. Should be used within 2 weeks of purchase or after redistn. Use a short path distn system. It has IR bands in  $CCl_4$  at  $\nu$  1405, 1380, 1320, 1200 and 1550 $cm^{-1}$ ; and  $^{13}C$  NMR( $CDCl_3$ ) with  $\delta$  at 118.1, 25.1, 21.5 and 13.6ppm. [*Org Synth* 68 83 1990; *JACS* 103, 3099 1981].

**Di-*n*-butyl cyclohexylphosphonate** [1085-92-3] **M 245.4**. The compound with uranyl nitrate was crystd three times from hexane. For method see *tributyl phosphate*.

**Di-*ter*-butyl dichlorosilane (DTBCl<sub>2</sub>)** [18395-90-0] **M 213.2, m -15°, b 190°/729mm, 195-197°/atm, d 1.01**. Purified by fractional distn. It is a colourless liquid with a pleasant odour and does not fume in moist air, but does not titrate quantitatively with excess of dil alkali. [*JACS* 70 2877 1948].

**Di-*n*-butyl *n*-butylphosphonate** [78-46-6] **M 250.3, b 150-151°/10mm, 160-162°/20mm, n<sup>25</sup> 1.4302**. Purified by three recrystallisations of its compound with uranyl nitrate, from hexane. For method, see *tributyl phosphate*.

**Di-*ter*-butyl silyl bis(trifluoromethanesulphonate)** [85272-31-7] **M 440.5, b 73.5-74.5°/0.35mm, d 1.36**. Purified by fractional distillation. It is a pale yellow liquid which should be stored under argon. It is less reactive than the diisopropyl analogue. The presence of the intermediate monochloro compound can be detected by  $^1H$  NMR, ( $CHCl_3$ ):  $t-Bu_2Si(OTf)_2$  [ $\delta$  1.25s];  $t-Bu_2Si(H)OTf$  [ $\delta$  1.12s] and  $t-Bu_2HSi(Cl)OTf$  [ $\delta$  1.19s]. [*TET LETT* 23 487 1982].

**Dichlorodimethylsilane** [75-78-5] **M 129.1**, **m -76°**, **b 70°**, **d 1.064**, **n 1.404**. Purified by fractional distillation. *Irritant and TOXIC*.

**Dichloro methyl phenylsilane** see **methylphenyl dichlorosilane**.

**Dichloro methylsilane** see **methyl dichlorosilane**.

**Dichloro methyl vinyl silane** see **methylvinyl dichlorosilane**.

**Dicobalt octacarbonyl** [15226-74-1] **M 341.9**, **m 51°**. Orange-brown crystals by recrystn from *n*-hexane under a carbon monoxide atmosphere [Ojima et al. *JACS* **109** 7714 1987; see also Hileman in *Preparative Inorganic Reactions*, Jolly ed, vol 1 101 1987].

**Diethyl aluminium chloride** [96-10-6] **M 120.6**, **m -75.5°**, **b 106.5-108°/24.5mm**, **d 0.96**. Distd from excess dry NaCl (to remove ethyl aluminium dichloride) in a 50-cm column containing a heated nichrome spiral.

***O,O*-Diethyl-*S*-2-diethylaminoethyl phosphorothiolate** [78-53-5] **M 269.3**, **m 98-99°**. Crystd from isopropanol/ethyl ether.

**Di-(2-ethylhexyl)phosphoric acid ('diisooctyl' phosphate)** [27215-10-7] [298-07-7] **M 322.4**. Contaminants of commercial samples include the monoester, polyphosphates, pyrophosphate, 2-ethylhexanol and metal impurities. Dissolved in *n*-hexane to give an 0.8M soln. Washed with an equal volume of M HNO<sub>3</sub>, then with saturated (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> soln, with 3M HNO<sub>3</sub>, and twice with water [Petrov and Allen *AC* **33** 1303 1961]. Similarly, the impure sodium salt, after scrubbing with pet ether, has been acidified with HCl and the free organic acid has been extracted into pet ether and purified as above. For purification *via* the copper salt see McDowell et al. [*JINC* **38** 2127 1976].

**Diethyl methylsilane** [760-32-7] **M 102.3**, **b 78.4°/760mm**, **77.2-77.6°/atm**, **d 0.71**. Fractionally distilled through a *ca* 20 plate column and the fraction boiling within a range of less than 0.5° is collected. [*Izv Akad SSSR Otd Chim* 1416 1957; *JACS* **69** 2600 1947].

**Diethyl trimethylsilyl phosphite** [13716-45-5] **M 210.3**, **b 61°/10mm**, **66°/15mm**, **d 0.9476**, **n 1.4113**. Fractionated under reduced pressure and has  $\delta_P$  -128  $\pm$  0.5 ppm relative to H<sub>3</sub>PO<sub>4</sub>. [*JOC* **46** 2097 1981; *J Gen Chem USSR (Eng edn)* **45** 231 1975].

***N,N*-Diethyltrimethyl silylamine** [996-50-9] **M 145.3**, **b 33°/26mm**, **126.8-127.1°/738mm**, **126.1-126.4°**, **d 0.763**, **n 1.411**. Fractionated through a 2ft vac-jacketed column containing Helipak packing with a reflux ratio of 10:1. [*JACS* **68** 241 1946; *JOC* **23** 50 1958; *J prakt Chem* **9** 315 1959].

***N,N'*-Diheptyl-*N,N'*-5,5-tetramethyl-3,7-dioxanonanediamide**, [lithium ionophore I (ETH 149) [58821-96-8] **M 442.7**. Purified by chromatography on Kieselgel using CHCl<sub>3</sub> as eluent (IR  $\nu$  1640cm<sup>-1</sup>). [*HCA* **60** 2326 1977].

**Dihexadecyl phosphate** [2197-63-9] **M 546.9**, **m 75°**. Crystd from MeOH [Lukac *JACS* **106** 4387 1984].

**1,2-Dihydroxybenzene-3,5-disulphonic acid, disodium salt (TIRON)** [149-45-1] **M 332.2**,  $\epsilon$  **6.9 x 10<sup>4</sup>** at 260nm, **pH 10.8**. Recrystd from water [Hamaguchi et al. *Anal Chim Acta* **9** 563 1962].

**Diiron nonacarbonyl** see **Iron enne carbonyl**.

**Diisooctyl phenylphosphonate** [49637-59-4] **M 378.5**, **n<sup>25</sup> 1.4780**. Vacuum distilled, percolated through a column of alumina, then passed through a packed column maintained at 150° to remove residual

traces of volatile materials in a countercurrent stream of  $N_2$  at reduced pressure [Dobry and Keller *JPC* **61** 1448 1957].

'Diisooctyl' phosphate see **di-(2-ethylhexyl)phosphoric acid**.

**Diisopropyl chlorosilane (chlorodiisopropylsilane)** [2227-29-4] **M 150.7, b 59°/8mm, 80°/10mm, 200°/738mm, d 0.9008, n<sub>D</sub> 1.4518**. Impurities can be readily detected by  $^1H$  NMR. Purified by fractional distn [*JACS* **69** 1499 1947; *JCS* 3668 1957; *J Organometal Chem* **282** 175 1985].

**Dilongifolyl borane** [77882-24-7] **M 422.6, m 169-172°**. Wash with dry  $Et_2O$  and dry in a vacuum under  $N_2$ . It has **m** 160-161° in a sealed evacuated capillary. It is sparingly soluble in pentane, THF,  $CCl_4$ ,  $CH_2Cl_2$ , and  $CHCl_3$ , but the suspended material is capable of causing asymmetric hydroboration. Disappearance of solid indicates that the reaction has proceeded. [*JOC* **46** 2988 1981].

**Dimethyl carbonate** [616-38-6] **M 90.1, b 89.5°/755mm, 90.2°/atm, d 1.0446, n<sub>D</sub> 1.3687**. If the reagent has broad intense bands at  $3300cm^{-1}$  and above (i.e. OH stretching) then it should be purified further. Wash successively with 10%  $Na_2CO_3$  soln, saturated  $CaCl_2$ ,  $H_2O$  and dried by shaking mechanically for 1h with anhydrous  $CaCl_2$ , and fractionated. [*JCS* 78 1939, 1847 1948].

**Dimethyl dicarbonate (dimethyl pyrocarbonate)** [4525-33-1] **M 134.1, m 15.2°, b 45-46°/5mm, d 1.2585, n<sub>D</sub> 1.3950**. Dissolve in  $Et_2O$ , shake with a small vol of 0.1N HCl, dry  $Et_2O$  with  $Na_2SO_4$  and distil in vac below 100° to give a clear liquid. It dec to  $CO_2$  and dimethyl carbonate on heating at 123-149°. It is readily hydrolysed by  $H_2O$ . [*J Gen Chem USSR* **22** 1546 1952; see also *B* **71** 1797 1938].

**Dimethyl dichlorosilane** [75-78-5] **M 129.1, m -75.5°, b 68.5-68.7°/750mm, 70.5°/760mm, d 1.0885, n<sub>D</sub> 1.4108**. Other impurities are chlorinated silanes and methylsilanes. Fractionated through a 3/8in diameter 7ft Stedman column rated at 100 theoretical plates at almost total reflux. See purification of  $MeSiCl_2$ . [*JACS* **70** 3590 1948].

**2,6-Dimethyl-1,10-phenanthrolinedisulphonic acid, disodium salt ( $H_2O$ )** [52698-84-7] **M 564.5**. Inorganic salts and some coloured species can be removed by dissolving the crude material in the minimum volume of water and precipitating by adding EtOH. Purified reagent can be obtained by careful evapn of the filtrate.

**Dinitrogen tetroxide,  $N_2O_4$**  [10544-72-6] **M 92.0 m -11.2°, b 21.1°**. Purified by oxidation at 0° in a stream of oxygen until the blue colour changed to red-brown. Distd from  $P_2O_5$ , then solidified on cooling in a deep-freeze (giving nearly colourless crystals). Oxygen can be removed by alternate freezing and melting.

***N,N*-Dioctadecyl methylamine** see **hydrogen ionophore III**

**Diocetyl phenylphosphonate** [1754-47-8] **M 378.8, d 1.485, n<sub>D</sub><sup>25</sup> 1.4780**. Purified as described under diisooctyl phenylphosphonate.

**Diphenyl hydrogen phosphate** [838-85-7] **M 250.2, m 99.5°**. Crystd from  $CHCl_3$ /pet ether.

**Diphenylmercury** [587-85-9] **M 354.8, m 125.5-126°**. Sublimed, then crystd from nitromethane or ethanol. If phenylmercuric halides are present they can be converted to phenylmercuric hydroxide which, being much more soluble, remains in the alcohol or benzene used for crystn. Thus, crude material (10g) is dissolved in warm ethanol (ca 150ml) and shaken with moist  $Ag_2O$  (ca 10g) for 30min, then heated under reflux for 30min and filtered hot. Concentration of the filtrate by evaporation gives diphenylmercury, which is recrystd from benzene [Blair, Bryce-Smith and Pengilly *JCS* 3174 1959]. **TOXIC**.

**4,7-Diphenyl-1,10-phenanthrolinedisulphonic acid, disodium salt** [52746-49-3] **M 536.5**. Dissolve crude sample in the minimum volume of water and add EtOH to ppt the contaminants. Carefully evaporate the filtrate to obtain pure material.

**Diphenylphosphinic acid** [1707-03-5] **M 218.2, m 194-195°**. Recrystd from 95% EtOH and dried under vacuum at room temperature. [see entries in Kosolapoff *Organophosphorus Compounds* J Wiley, NY, 1950; Kosolapoff and Maier *Organic Phosphorus Compounds* Wiley-Interscience, NY, 1972-1976].

**Diphenylsilane** [775-12-2] **M 184.3, b 75-76°/0.5mm, 113-114°/9mm, 124-126°/11mm, 134-135°/16mm, d 1.0027, n 1.5802, 1.5756**. Dissolve in Et<sub>2</sub>O, mix slowly with ice-cold 10% AcOH. The Et<sub>2</sub>O layer is then shaken with H<sub>2</sub>O until the washings are neutral to litmus. Dry over Na<sub>2</sub>SO<sub>4</sub>, evaporate the Et<sub>2</sub>O and distil the residual oil under reduced pressure using a Claisen flask with the take-off head modified into a short column. Ph<sub>2</sub>SiH<sub>2</sub> boils at 257°/760mm but it cannot be distd at this temp because exposure to air leads to flashing, decomposition and formation of silica. It is a colourless, odourless oil, miscible with organic solvents but not H<sub>2</sub>O. A possible impurity is Ph<sub>3</sub>SiH which has **m 43-45°** and would be found in the residue. [*JOC* **18** 303 1953 ; *JACS* **74** 6481952, **81** 5925 1959].

**Diphenylsilanediol** [947-42-2] **M 216.3, m 148°(dec)**. Crystd from CHCl<sub>3</sub>-methyl ethyl ketone.

**Disodium anthraquinone-2,6-disulphonate** [853-693-9] **M 412.3**. Crystd from water.

**Disodium calcium ethylenediaminetetraacetate** [62-33-9] **M 374.3**. Dissolved in a small amount of water, filtered and ppted with excess EtOH. Dried at 80°.

**Disodium dihydrogen ethylenediaminetetraacetic acid (2H<sub>2</sub>O)** [6381-92-6] **M 372.2, m 248°(dec)**. Analytical reagent grade material can be used as primary standard after drying at 80°. Commercial grade material can be purified by crystn from water or by preparing a 10% aqueous soln at room temperature, then adding ethanol slowly until a slight permanent ppte is formed, filtering, and adding an equal volume of ethanol. The ppte is filtered off on a sintered-glass funnel, is washed with acetone, followed by ethyl ether, and dried in air overnight to give the dihydrate. Drying at 80° for at least 24h converts it to the anhydrous form.

**Disodium 1,8-dihydroxynaphthalene-3,6-disulphonate (2H<sub>2</sub>O)** [2808-22-0] **M 400.3, m >300°**. Crystd from water.

**Disodium ethylenebis[dithiocarbamate]** [142-59-6] **M 436.5**. Crystd (as hexahydrate) from aqueous ethanol.

**Disodium-β-glycerophosphate** [819-83-0] **M 216.0, m 102-104°**. Crystd from water.

**Disodium hydrogen orthophosphate (anhydrous)** [7558-79-4] **M 142.0**. Crystd twice from warm water, by cooling. Air dried, then oven dried overnight at 130°. *Hygroscopic*: should be dried before use.

**Disodium magnesium ethylenediaminetetraacetate** [14402-88-1] **M 358.5**. Dissolved in a small amount of water, filtered and ppted with an excess of methanol. Dried at 80°.

**Disodium naphthalene-1,5-disulphonate** [1655-29-4] **M 332.3**. Recrystd from aqueous acetone [Okahata et al. *JACS* **108** 2863 1986].

**Disodium 4-nitrophenylphosphate (6H<sub>2</sub>O)** [4264-83-9] **M 371.1** Dissolve in hot aqueous MeOH, filter and ppte by adding Me<sub>2</sub>CO. Wash the solid with Me<sub>2</sub>CO and repeat the purification. Aq MeOH and Et<sub>2</sub>O can also be used as solvents. The white fibrous crystals contain less than 1% of free 4-nitrophenol [assay: *JBC* **167** 57 1947].

**Disodium phenylphosphate (2H<sub>2</sub>O)** [3279-54-7] **M 254.1**. Dissolved in a minimum amount of methanol, filtering off an insoluble residue of inorganic phosphate, then ppted by adding an equal volume of ethyl ether. Washed with ethyl ether and dried [Tsuboi *Biochim Biophys Acta* **8** 173 1952].

**Disodium succinate** [150-90-3] **M 162.1**. Crystd twice from water. Freed from other metal ions by passage of an 0.1M soln through a column of Dowex ion-exchange resin A-1, sodium form.

**Disodium 4-nitrophenylphosphate**  $6\text{H}_2\text{O}$  [4264-83-9] **M 371.1** Dissolve in hot aqueous MeOH, filter and ppte by adding  $\text{Me}_2\text{CO}$ . Wash the solid with  $\text{Me}_2\text{CO}$  and repeat the purification. Aqueous MeOH and  $\text{Et}_2\text{O}$  can also be used as solvents. The white fibrous crystals contain less than 1% of free 4-nitrophenol [assay: *JBC* 167 57 1947].

**Di-*p*-tolylmercury** [50696-65-6] **M 382.8, m 244-246°**. Crystd from xylene.

**Di-*p*-tolyl phenylphosphonate** [94548-75-1] **M 388.3, n<sup>25</sup> 1.5758**. Purified as described under diisooctyl phenylphosphonate.

**1,3-Divinyl-1,1,3,3-tetramethyldisiloxane** [2627-95-4] **M 186.4, m -99.7°; b 128-129°/atm, 139°/760mm, d 0.811, n 1.4122**. Dissolve in  $\text{Et}_2\text{O}$ , wash with  $\text{H}_2\text{O}$ , dry over  $\text{CaCl}_2$  and distil. [*JACS* 77 1685 1955; *Coll Czech Chem Comm* 24 3758 1959].

**Eosin (as disodium salt)** [548-26-5] **M 624.1**. Dissolved in water and ppted by addition of dilute HCl. The ppte was washed with water, crystd from ethanol, then dissolved in the minimum amount of dilute NaOH soln and evaporated to dryness on a water-bath. The purified disodium salt was then crystd twice from ethanol [Parker and Hatchard *TFS* 57 1894 1961].

**Ethylarsonic acid** [507-32-4] **M 154.0, m 99.5°**. Crystd from ethanol.

**Ethylmercuric chloride** [107-27-7] **M 265.1, m 193-194°**. Mercuric chloride can be removed by suspending ethylmercuric chloride in hot distilled water, filtering with suction in a sintered-glass crucible and drying. Then crystd from ethanol and sublimed under reduced pressure. It can also be crystd from water.

**Ethylmercuric iodide** [2440-42-8] **M 356.6, m 186°**. Crystd once from water (50ml/g).

**Ethyl trimethylsilylacetate** [4071-88-9] **M 160.3, b 74.5°/41mm, 75.5°/42mm, (157°/730mm), d 0.8762, n 1.4149**. Purified by distilling *ca* 10g of reagent through a 15cm, Vigreux column and then redistilling through a 21cm glass helices-packed column [*JACS* 75 994 1953]. Alternatively, dissolve in  $\text{Et}_2\text{O}$ , wash with  $\text{H}_2\text{O}$ , dilute  $\text{Na}_2\text{CO}_3$ , dry over  $\text{Na}_2\text{CO}_3$ , evaporate  $\text{Et}_2\text{O}$ , and distil through a column of 15 theoretical plates [*JACS* 70 2874 1948].

**Ethyl 3-(trimethylsilyl)propionate** [17728-88-0] **M 174.3, b 93°/40mm, 178°-180°/atm, d 0.8763, n 1.4198**. Dissolve in  $\text{Et}_2\text{O}$ , wash with  $\text{H}_2\text{O}$ , dilute  $\text{Na}_2\text{CO}_3$ , dry over  $\text{Na}_2\text{SO}_4$ , evaporate  $\text{Et}_2\text{O}$  and fractionally distil. [*JACS* 72 1935 1950].

**Ethynyl tributylstannane** [994-81-8] **M 315.1, b 76°/0.2mm, 130-135°/0.7mm, 200°/2mm, d 1.1113, n 1.4770**. Purified by dissolving the reagent (*ca* 50g) in heptane (250ml), washing with  $\text{H}_2\text{O}$  (100ml), drying ( $\text{MgSO}_4$ ), evaporating and distilling in a vacuum. It has IR  $\nu$  3280 ( $\equiv\text{C-H}$ ), 2950, 2850, 2005 ( $\text{C}\equiv\text{C}$ ), 1455, 1065 and  $865\text{cm}^{-1}$ . [*JOC* 46 5221 1981; *JACS* 109 2138 1987; *J Gen Chem USSR (Engl Edn)* 37 1469 1967].

**Ethynyl trimethylsilane** [1066-54-2] **M 98.2, b 53°/atm, 52.5°/atm, d 0.71, n 1.3871**. Distil through an efficient column. The IR has bands at 2041 ( $\text{C}\equiv\text{C}$ ) and 3289 ( $\equiv\text{C-H}$ )  $\text{cm}^{-1}$ . [*B* 92 30 1959].

**Ethyl triphenylphosphonium bromide** [1530-32-1] **M 371.3, m 203-205°**. Recrystd from  $\text{H}_2\text{O}$  and dried in high vacuum at 100°. IR has bands at 6.90, 6.99 and  $10.03\mu$ . [*A* 606 1 1957; *JOC* 23 1245 1958].

**Europium (III) acetate ( $2\text{H}_2\text{O}$ )** [101953-41-7] **M 383.1**. Recrystd several times from water [Ganapathy et al. *JACS* 108 3159 1986].

**Europium shift reagents** see **lanthanide shift reagents** in Chapter 3.

**Ferric acetylacetonate** [14024-18-1] **M 353.2, m 181.3-182.3°**. Recrystd twice from benzene-pet ether **m 181.3-182.3°** corr [JCS 1256 1938]. Recrystd from EtOH or Et<sub>2</sub>O, **m 179°** [A 323 13 1902]. Recrystd from absolute EtOH, **m 159.5°** [B 67 286 1934].

**Ferric acetylacetonate** [14024-18-1] **M 353.2, m 179°**. Crystd from 95% EtOH and dried for 1h at 120°.

**Ferric chloride (anhydrous)** [7705-08-0] **M 162.2, m >300°(dec)**. Sublimed at 200° in an atmosphere of chlorine. Stored in a weighing bottle inside a desiccator.

**Ferric chloride (6H<sub>2</sub>O)** [10025-77-1] **M 270.3**. An aqueous soln, saturated at room temperature, was cooled to -20° for several hours. Pptn was slow, even with scratching and seeding, and it was generally necessary to stir overnight. The presence of free HCl retards the pptn [Linke JPC 60 91 1956].

**Ferric nitrate (9H<sub>2</sub>O)** [7782-61-8] **M 404.0**. Cryst from aqueous solutions of moderately strong HNO<sub>3</sub> as the violet nonhydrate. With more concentrated aqueous solns (containing some HNO<sub>3</sub>), the hexahydrate crystals out. The anhydrous salt is slightly deliquescent and decomposes at 47°.

**Ferric perchlorate (9H<sub>2</sub>O)** [13537-24-1] **M 516.3**. Crystd twice from conc HClO<sub>4</sub>, the first time in the presence of a small amount of H<sub>2</sub>O<sub>2</sub> to ensure that the iron is fully oxidised [Sullivan JACS 84 4256 1962]. Extreme care should be taken with this preparation because it is potentially **DANGEROUS**.

**Ferric sulphate (xH<sub>2</sub>O)** [10028-22-5] **M 399.9 + xH<sub>2</sub>O**. Dissolve in the minimum volume of dilute aqueous H<sub>2</sub>SO<sub>4</sub> and allow to evaporate at room temp until crystals start to form. Do not concentrate by boiling off the H<sub>2</sub>O as basic salts will be formed. Various *hydrates* are formed the common ones are the *dodeca* and *nona hydrates* which are violet in colour. The anhydrous salt is colourless and very *hygroscopic* but dissolves in H<sub>2</sub>O slowly unless ferrous sulphate is added.

**Ferrocene** [102-54-5] **M 186.0, m 173-174°**. Purified by crystn from pentane or cyclohexane (also C<sub>6</sub>H<sub>6</sub> or MeOH can be used). Moderately soluble in Et<sub>2</sub>O. Sublimes readily above 100°. Crystallisation from EtOH gave **m 172.5-173°**. [Org Synth Col Vol IV 473 1963; JCS 632 1952]. Also crystd from methanol and sublimed *in vacuo*. [Saltiel et al. JACS 109 1209 1987].

**Ferrocene carboxaldehyde** [12093-10-6] **M 214.1, m 117-120°, 118-120°, 121°, 124.5°**. Red crystals from EtOH or pet ether and sublimed at 70°/1mm. *Semicarbazone* **m 217-219°(dec)** cryst from aqueous EtOH. *O-Acetyloxime* **m 80-81°** cryst from hexane [JOC 22 355 1957]. *2,4-Dinitrophenylhydrazone* **m 248°(dec)**. [Beilstein 16 IV 1798; JACS 79 3416 1957; JCS 650 1958].

**Ferrocene carboxylic acid** [1271-42-7] **M 230.1, m 210°(dec), 225-230°(dec)**. Yellow crystals from pet ether. Also crystd from aqueous ethanol. [Matsue et al. JACS 107 3411 1985]. *Acid chloride* **m 49°** crystallises from pentane, λ<sub>max</sub> 458nm [JOC 24 280 1959]. *Methyl ester* crystallises from aq MeOH **m 70-71°**. *Anhydride* **m 143-145°** from pet ether [JOC 24 1487 1959]. *Amide* **m 168-170°** from CHCl<sub>3</sub>-Et<sub>2</sub>O or **m 167-169°** from C<sub>6</sub>H<sub>6</sub>-MeOH. [JACS 77 6295 1955; 76 4025 1954].

**Ferrocene-1,1'-dicarboxylic acid** [1293-87-4] **M 274.1, m >250°(dec), >300°**. Orange-yellow crystals from AcOH. Sublimes above 230°. *Monomethyl ester* **m 147-149°** [Dokl Acad Nauk USSSR 115, 518 1957]. *Dimethyl ester* **m 114-115°** [JACS 74, 3458 1958]. *Diacid chloride* **m 92-93°** from pet ether. [Dokl Acad Nauk USSSR 120 1267 1958; 127 333 1959].

**Ferrocene-1,1,-dimethanol** [1291-48-1] **M 246.1, m 107-108°**. Obtained from the diacid with  $\text{LiAlH}_4$  reduction and recrystd from  $\text{Et}_2\text{O}$ -pet ether. [JACS 82 4111 1960]

**Ferrous bromide** [20049-65-4] **M 215.7 + xH<sub>2</sub>O, m 684°, d<sup>25</sup> 4.63**. Crystn from air-free  $\text{H}_2\text{O}$  provides the *hexahydrate* as pale green to bluish-green rhombic prisms. On heating at 49°  $\text{H}_2\text{O}$  is lost and the *tetrahydrate* is formed. Further heating at 83° more  $\text{H}_2\text{O}$  is lost and the *dihydrate* is formed as a light yellow to dark brown *hygroscopic* powder. The ferrous iron in the aqueous solns of these salts readily oxidises to ferric iron. The salts should be stored over  $\text{H}_2\text{SO}_4$  under  $\text{N}_2$  in tightly closed containers. They have some solubility in EtOH. [B 38 236 1904].

**Ferrous chloride (4H<sub>2</sub>O)** [13478-10-9] **M 198.8**. A 550ml round-bottomed Pyrex flask was connected, via a glass tube fitted with a medium porosity sintered-glass disc, to a similar flask. To 240g of  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  in the first flask was added conductivity water (200ml), 38% HCl (10ml), and pure electrolytic iron (8-10g). A stream of purified  $\text{N}_2$  was passed through the assembly, escaping through a mercury trap. The salt was dissolved by heating which was continued until complete reduction had occurred. By inverting the apparatus and filtering (under  $\text{N}_2$  pressure) through the sintered glass disc, unreacted iron was removed. After cooling and crystn, the unit was again inverted and the crystals of ferrous chloride were filtered free from mother liquor by applied  $\text{N}_2$  pressure. Partial drying by overnight evacuation at room temperature gave a mixed hydrate which, on further evacuation on a water bath at 80°, lost water of hydration and its absorbed HCl (with vigorous effervescence) to give a white powder,  $\text{FeCl}_2 \cdot 2\text{H}_2\text{O}$  [Gayer and Wootner JACS 78 3944 1956].

**Ferrous chloride** [7758-94-3] **M 126.8, m 674°, b 1023°, d<sup>25</sup> 3.16**. Sublimes in a stream of HCl at ca 700°, or in  $\text{H}_2$  below 300°. Its vapour pressure at 700° is 12mm. Anhydrous  $\text{FeBr}_2$  can be obtained by carefully dehydrating the *tetrahydrate* in a stream of HBr and  $\text{N}_2$ , and it can be sublimed under  $\text{N}_2$ . White *hygroscopic* rhombohedral crystals with a green tint. They oxidise in air to  $\text{FeCl}_3 + \text{Fe}_2\text{O}_3$ . Sol in  $\text{H}_2\text{O}$ , EtOH  $\text{Me}_2\text{CO}$  but insol in  $\text{Et}_2\text{O}$ . The *tetrahydrate* is pale green to pale blue in colour and loses  $2\text{H}_2\text{O}$  at 105-115°. The *dihydrate* loses  $\text{H}_2\text{O}$  at 120°. The ferrous iron in the aqueous solns of these salts readily oxidises to ferric iron. [Inorg Synth 6 172 1960; Handbook of Preparative Inorganic Chemistry (Ed Brauer) Vol II 1491 1965].

**Ferrous perchlorate (6H<sub>2</sub>O)** [13933-23-8] **M 362.9**. Crystd from  $\text{HClO}_4$ .

**Ferrous sulphate (7H<sub>2</sub>O)** [7782-63-0] **M 278.0**. Crystd from 0.4M  $\text{H}_2\text{SO}_4$ .

**Flophemesyl chloride** see **pentafluorophenyl dimethylchlorosilane**.

**Fluorine** [7782-41-4] **M 38.0, b -129.2°**. Passed through a bed of NaF at 100° to remove HF and  $\text{SiF}_4$ . [For description of stills used in fractional distn, see Greenberg et al. JPC 65 1168 1961; Stein, Rudzitis and Settle Purification of Fluorine by Distillation, Argonne National Laboratory, ANL-6364 1961 (from Office of Technical Services, US Dept of Commerce, Washington 25)]. **HIGHLY TOXIC**.

**Fluoroboric acid** [16872-11-0] **M 87.8**. Crystd several times from conductivity water.

**Gallium** [7440-55-3] **M 69.7, m 29.8°**. Dissolved in dilute HCl and extracted into  $\text{Et}_2\text{O}$ . Pptn with  $\text{H}_2\text{S}$  removed many metals, and a second extraction with  $\text{Et}_2\text{O}$  freed Ga more completely, except for Mo, Th(III) and Fe which were largely removed by pptn with NaOH. The soln was then electrolysed in 10% NaOH with a Pt anode and cathode (2-5A at 4-5V) to deposit Ga, In, Zn and Pb, from which Ga was obtained by fractional crystn of the melt [Hoffman J Res Nat Bur Stand 13 665 1934]. Also purified by heating to boiling in 0.5-1M HCl, then heating to 40° in water and pouring the molten Ga with water under vacuum onto a glass filter (30-50  $\mu$  pore size), to remove any unmelted metals or oxide film. The Ga was then fractionally crystd from the melt under water.

**Gallium (III) Chloride** [13450-90-3] **M 176.1, m 77.8°; b 133°/100mm, 197.7°/700mm, d 2.47.** Pure compound can be obtained by redistn in a stream of  $\text{Cl}_2$  or  $\text{Cl}_2/\text{N}_2$  followed by vacuum sublimation or zone refining. Colourless needles which give *gallium dichloride* [ $\text{Ga}(\text{GaCl}_4)$ , **m 172.4°**] on heating. Dissolves in  $\text{H}_2\text{O}$  with liberation of heat. Soluble in  $\text{Et}_2\text{O}$ . [*Handbook of Preparative Inorganic Chemistry (Ed Brauer) Vol I 846 1963*].

**Gallium (III) nitrate (9H<sub>2</sub>O)** [63462-65-7] **M 417.9, m ca 65°.** Recrystd from  $\text{H}_2\text{O}$  (sol: 295g/100ml at 20°). White deliquescent colourless powder soluble in  $\text{H}_2\text{O}$ , absolute  $\text{EtOH}$  and  $\text{Et}_2\text{O}$ . Loses  $\text{HNO}_3$  upon heating at 40°. Addition of  $\text{Et}_2\text{O}$  to a warm ethanolic soln (40-50°) of  $\text{Ga}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  precipitates  $\text{Ga}(\text{OH})_2\text{NO}_3 \cdot \text{Ga}(\text{OH})_3 \cdot 2\text{H}_2\text{O}$ . If the salt has partly hydrolysed, dissolve in conc  $\text{HNO}_3$ , reflux, dilute with  $\text{H}_2\text{O}$  and concentrate on a sand bath. Wash several times by adding  $\text{H}_2\text{O}$  and evaporate until there is no odour of acid. Dilute the residue to a Ga concentration of 26g/100ml. At this concentration, spongy  $\text{Ga}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$  separates from the viscous soln. After standing for several days the crystals are collected and dried in a stream of dry air first at room temp then at 40°. Dehydration is complete after 2 days. Recrystallise from  $\text{H}_2\text{O}$  and dry on a water pump at room temperature. [*Z Naturforsch 20B 71 1965; Handbook of Preparative Inorganic Chemistry (ed Brauer) Vol I 856/1963*].

**Gallium (III) sulphate** [34781-33-4] **M 427.6.** Recrystn from  $\text{H}_2\text{O}$  gives the 16-18 $\text{H}_2\text{O}$  hydrate (sol at 20° is 170g/100ml). Alternatively dissolve in 50%  $\text{H}_2\text{SO}_4$  and evaporate (60-70°), cool and ppt by adding  $\text{EtOH}/\text{Et}_2\text{O}$ . On heating at 165° it provides the *anhydrous* salt which is a white *hygroscopic* solid. [*Z Naturforsch 20B 71 1965*].

**Germanium** [7440-56-4] **M 72.6.** Copper contamination on the surface and in the bulk of single crystals of Ge can be removed by immersion in molten alkali cyanide under  $\text{N}_2$ . The Ge was placed in dry cyanide powder in a graphite holder in a quartz or porcelain boat. The boat was then inserted into a heated furnace which, after a suitable time, was left to cool to room temperature. At 750°, a 1mm thickness requires about 1min, whereas 0.5cm needs about half hour. The boat was removed and the samples were taken out with plastic-coated tweezers, carefully rinsed in hot water and dried in air [Wang *JPC 60 45 1956*].

**Germanium (IV) oxide** [1310-53-8] **M 104.6, m 1080°(soluble form), d<sup>25</sup> 6.239; m 1116°(insoluble form) d<sup>25</sup> 4.228.** The oxide is usually prepared by hydrolysing redistd  $\text{GeCl}_4$  and igniting in order to remove  $\text{H}_2\text{O}$  and chloride. It can be further purified by dissolving in hot  $\text{H}_2\text{O}$  (sol: 4g/L cold) evaporating and drying the residual crystalline solid. When the *soluble* form (which is produced in  $\text{H}_2\text{O}$  at 355°) is heated for 100h it is converted to the *insoluble* form. This form is stable at temperatures up to 1033°, and fusion at 1080° for 4h causes complete devitrification and it reverts to the *soluble* form. [*JACS 46 2358 1924, 47 1945 1925, 54 2303 1032*].

**Germanium tetrachloride** [10038-98-9] **M 214.4, m -49.5° ( $\alpha$ ), -52.0° ( $\beta$ ), b 83.1°/760mm, 86.5°/760mm corr, d<sub>4</sub><sup>20</sup> 1.84.** Traces of  $\text{Cl}_2$  and  $\text{HCl}$  can be removed from the liquid by blowing dry air through it for a few hours at room temperature or shake it with  $\text{Hg}$  or  $\text{Hg}_2\text{Cl}_2$  and then fractionally distil in a vacuum. It decomposes on heating at 950°. It has a sharp penetrating odour and fumes in moist air to give a chalky coat of  $\text{GeO}_2$ . It is slowly hydrolysed by  $\text{H}_2\text{O}$  to give  $\text{GeO}_2$ . [*JACS 44 306 1922*].

**Germanium tetraethoxide** [14165-55-0] **M 252.8, m -72°; b 54.5°/5mm, 71-72°/11mm, 188-190°/722mm, d<sup>25</sup> 1.1288.** Distil through a 10cm Vigreux column under reduced pressure. Alternatively distil through a Fenske glass helices column fitted with a total condensation variable take-off stillhead. Fractionate under reduced pressure using a reflux ratio of 10:1. [*JACS 75 718 1953; JCS 4916 1956*].

**Glass powder** (100-300 mesh). Washed with 10%  $\text{HNO}_3$ , water and dried.

**Gold (III) bromide (gold tribromide)** [10294-28-7] **M 436.7, m 150°(dec).** Purified by adding pure  $\text{Br}_2$  to the dark powder, securely stopper the container, warm a little and shake while keeping away from light for ca 48h. Remove the stopper and place over  $\text{NaOH}$  until free  $\text{Br}_2$  is no longer in the apparatus (48-60h). The bright yellow needles of the tribromide are stable over  $\text{NaOH}$  in the dark. It is sol in  $\text{H}_2\text{O}$  and in  $\text{EtOH}$  where it is slowly reduced. Keep in a cooled closed container and protect from light as decomposition



causes gold to be formed. *Aurobromic acid* can be obtained by adding the calculated amount of conc HBr to  $\text{AuBr}_3$  (actually  $\text{Au}_2\text{Br}_6$ ) until all dissolves, whereby the acid crystallises out as  $\text{HAuBr}_4 \cdot 5\text{H}_2\text{O}$ , deliquescent solid soluble in EtOH with  $m$  ca 27°, and store as above. [*JCS* 2410 1931, 217, 219 1935].

**Gold (III) chloride (hydrate)** [16903-35-8]  $M$  339.8 +  $x\text{H}_2\text{O}$ ,  $m$  229°,  $b$  354°dec,  $d$  3.9. Obtained as a dark red crystalline mass by dissolving Au in aqua regia and evaporating. When sublimed at 180° the crystals are ruby red. The anhydrous salt is *hygroscopic* sol in  $\text{H}_2$  but sparingly soluble in EtOH and  $\text{Et}_2\text{O}$ . *Aurochloric acid* is formed when  $\text{AuCl}_3$  is dissolved in HCl. [*JACS* 35 553 1913; *Handbook of Preparative Inorganic Chemistry* (ed Brauer) Vol II 1056 1965].

**Gold (I) cyanide** [506-65-0]  $M$  223.0. The lemon yellow powder is sparingly soluble in  $\text{H}_2\text{O}$  and EtOH but soluble in aqueous  $\text{NH}_3$ . It is obtained by heating  $\text{H}[\text{Au}(\text{CN})_2]$  at 110°. Wash well with  $\text{H}_2\text{O}$  and EtOH and dry at 110°. It has an IR band at  $\nu$  2239 $\text{cm}^{-1}$  typical for  $\text{C}\equiv\text{N}$  stretching vibration. [*Handbook of Preparative Inorganic Chemistry* (ed Brauer) Vol II 1064 1965].

**Gold (I) iodide** [13453-24-2]  $M$  577.7. It has been prepared by heating gold and iodine in a tube at 120° for 4 months. Since it decomposes to Au and  $\text{I}_2$  in the presence of UV light and heat then the main impurity is Au. The salt is therefore purified by heating at 120° with  $\text{I}_2$  for several weeks. The crystals should be kept dry and in a cool place in the dark. [*Z Naturforsch* 11B 604 1956].

**Gold (III) oxide hydrate** [1303-58-8]  $M$  441.9 +  $x\text{H}_2\text{O}$ . Most probable impurities are  $\text{Cl}^-$  ions. Dissolve in strong boiling KOH soln (ca 5M) and precipitate (care) with excess of 3N  $\text{H}_2\text{SO}_4$ . Then shake and centrifuge, resuspend in  $\text{H}_2\text{O}$  and repeat wash several times until free from  $\text{SO}_4$  and  $\text{Cl}$  ions. This gives a *wet* oxide which is dried in air. It is best to keep it wet as it decomposes on drying (analyse wet sample). Store away from light in the presence of  $\text{H}_2\text{O}$  vapour. It evolves  $\text{O}_2$  at 110°. It is insoluble in  $\text{H}_2\text{O}$  but soluble in HCl and conc  $\text{HNO}_3$ . [*JACS* 49 1221 1927].

**Graphite** [7782-42-5]. Treated with hot 1:1 HCl. Filtered, washed, dried, powdered and heated in an evacuated quartz tube at 1000° until a high vacuum was obtained. Cooled and stored in an atmosphere of helium [Craig, Van Voorhis and Bartell *JPC* 60 1225 1956].

## Haematoporphyrin IX [14459-29-1] $M$ 598.7. Recrystd from MeOH.

**Helium** [7440-59-7]  $M$  4.0. Dried by passage through a column of Linde 5A molecular sieves and  $\text{CaSO}_4$ , then passed through an activated-charcoal trap cooled in liquid  $\text{N}_2$ , to adsorb  $\text{N}_2$ , argon, xenon and krypton. Passed over  $\text{CuO}$  pellets at 300° to remove hydrogen and hydrocarbons, over Ca chips at 600° to remove oxygen, and then over titanium chips at 700° to remove  $\text{N}_2$  [Arnold and Smith *JCSFT* 2 77 861 1981].

**Heptyl-4-trifluoroacetylbenzoate** see **carbonate ionophore I**.

**Hexachlorocyclotriphosphazene** [940-71-6]  $M$  354.0,  $m$  113-114°, 113-115°. Purified by sublimation and twice crystd from hexane [Meirovitch et al. *JPC* 88 1522, 1984; Alcock et al. *JACS* 106 5561 1984; Winter and van de Grampel *JCSDT* 1269 1986].

**2,2,4,4,6,6-Hexamethylcyclotrisiloxane** [1009-93-4]  $M$  219.5,  $m$  -10°; 81-82°/19mm, 111-112°/85mm, 188°/756mm,  $d$  0.9196,  $n$  1.448. Purified by fractional distillation at atmospheric pressure until the temperature reaches 200° The residue in the flask is mostly octamethylcyclotetrasilazane. [*JACS* 70 3888 1948].

**Hexamethyldisilane** [1450-14-2]  $M$  164.4,  $m$  9-12°, 113.1°/750mm,  $d$  0.7272,  $n$  1.4229. Most likely impurity is trimethylchlorosilane (*cf* boiling point). Wash with  $\text{H}_2\text{O}$ , cold conc  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{O}$  again then aqueous  $\text{NaHCO}_3$ , dry over  $\text{CaSO}_4$  and fractionate at atmospheric pressure. [*JCS* 2811 1958].

Grossly impure sample (25% impurities) was purified by repeated spinning band distn. This lowered the impurity level to 500 ppm. The main impurity was identified as 1-hydroxypentamethyldisilane.

**Hexamethyldisilazane** [999-97-3] **M 161.4, b 125-125.6°/atm, 126°/760mm, d 0.7747, n 1.407.** Possible impurity is Me<sub>3</sub>SiCl. Wash well with pet ether and fractionate through a vacuum jacketed column packed with Helipac using a reflux ratio of 10:1. [JOC 23 50 1958].

**Hexaethyldisiloxane** [924-49-0] **M 246.5, b 114-115°/16mm, 235.5°/760mm, d 0.8443, n 1.4330.** Distil in a vacuum, but can be distilled at atmospheric pressure without decomposition. It is characterised by completely dissolving in conc H<sub>2</sub>SO<sub>4</sub>. [JCS 3077 1950].

**Hexamethyldisiloxane** [107-46-0] **M 162.4, b 99.4°/760mm, 100.4°/764mm, d 0.7633, n 1.3777.** Fractionally distilled through a column packed with glass helices with *ca* 15 theoretical plates. [JACS 76 2672 1954; J Gen Chem USSR (Engl ed) 25 469 1955].

**Hexamethyldisilthiane (bis-trimethylsilyl sulphide)** [3385-94-2] **M 178.5, b 65-67°/16mm, 162.5-163.5°/750mm corr, 164°/760mm, d 0.85, n 1.4598.** Dissolve in pet ether (b *ca* 40°), remove solvent and distilled. Redistilled under atmospheric pressure of dry N<sub>2</sub>. It is collected as a colourless liquid which solidifies to a white solid in Dry-ice. On standing for several days it turns yellow possibly due to liberation of sulphur. Store below 4° under dry N<sub>2</sub>. [JCS 3077 1950].

**Hexamethyl ditin (hexamethyldistannane)** [661-69-8] **M 327.6, m 23.5°, b 85-88°/45mm, 182°/756mm, d<sup>25</sup> 1.57.** Wash with H<sub>2</sub>O and extract with C<sub>6</sub>H<sub>6</sub>, dry by filtering through powdered Na<sub>2</sub>SO<sub>4</sub>, remove C<sub>6</sub>H<sub>6</sub> on a rotary evaporator and fractionally dist the oily residue under vacuum (b 85-88°/45mm). *It boils at ca 182° at atmospheric press but it cannot be distilled in air because the hot vapours flash in the condenser.* [JACS 47 2361 1925, 63 2509 1941; TFS 53 1612 1957].

**Hexamethylphosphoric triamide (HMPT)** [680-31-9] **M 179.2, f.p. 7.2°, b 68-70°/1mm, 235°/760mm, d 1.024, n 1.460.** The industrial synthesis is usually by treatment of POCl<sub>3</sub> with excess of dimethylamine in isopropyl ether. Impurities are water, dimethylamine and its hydrochloride. It is purified by refluxing over BaO or CaO at about 4mm pressure in an atmosphere of nitrogen for several hours, then distd from sodium at the same pressure. The middle fraction (b *ca* 90°) is collected, refluxed over sodium under reduced pressure under nitrogen and distd. It is kept in the dark under nitrogen, and stored in solid CO<sub>2</sub>. Can also be stored over 4A molecular sieves.

Alternatively, it is distd under vacuum from CaH<sub>2</sub> at 60° and crystd twice in a cold room at 0°, seeding the liquid with crystals obtained by cooling in liquid nitrogen. After about two-thirds frozen, the remaining liquid is drained off [Fujinaga, Izutsu and Sakara PAC 44 117 1975]. For tests of purity see Fujinaga et al. in *Purification of Solvents*, ed Coetzee, Pergamon Press, Oxford, 1982. For efficiency of desiccants in drying HMPT see Burfield and Smithers [JOC 43 3966 1978; Sammes et al. JCSFT 1 281 1986].

**CARCINOGEN.**

**Hexamminecobalt(III) chloride** [10534-89-1] **M 267.5.** Crystd from warm water (8ml/g) by cooling.

**Hexammineruthenium(III) chloride** [14282-91-8] **M 309.6.** Crystd twice from 1M HCl.

**Hexarhodium hexadecacarbonyl** [28407-51-4] **M 1065.6, m 220°(dec, in air), d 2.87.** Slowly loses CO when heated in air; may be regenerated by heating at 80-200° in the presence of CO at 200atm pressure for 15h, preferably in the presence of Cu. Forms black crystals which are insoluble in hexane. It has bands at 2073, 2026 and 1800cm<sup>-1</sup> in the IR. [Z Anorg Allgem Chem 251 96 1963; JACS 85 1202 1963; TET LETT 22 1783 1981].

**Hydrazine (anhydrous)** [302-01-2] **M 32.1, fp 1.5-2.0°, b 113-113.5°, n 1.470, d 1.91.** Hydrazine hydrate is dried by refluxing with an equal weight of NaOH pellets for 3h, then distilled from fresh NaOH or BaO in a current of dry N<sub>2</sub>.

**Hydrazine dihydrochloride** [5341-61-7] **M 105.0**. Crystd from aqueous EtOH and dried under vacuum over CaSO<sub>4</sub>.

**Hydrazine monohydrochloride** [2644-70-4] **M 68.5, m 89°**. Prepared by dropwise addition of cold conc HCl to cold liquid hydrazine in equimolar amounts. The crystals were harvested from water and were twice recrystd from absolute MeOH and dried under vacuum. [Kovack et al. *JACS* **107** 7360 1985].

**Hydriodic acid** [10034-85-2] **M 127.9, b 127°, d 1.701**. Iodine can be removed from aqueous HI, probably as the amine hydrogen triiodide, by three successive extractions using a 4% soln of Amberlite LA-2 (a long-chain aliphatic amine) in CCl<sub>4</sub>, toluene or pet ether (10ml per 100ml of acid). [Davidson and Jameson *Chemistry & Industry (London)* 1686 1963]. Extraction with tributyl phosphate in CHCl<sub>3</sub> or other organic solvents is also suitable. Alternatively, a De-acidite FF anion-exchange resin column in the OH<sup>-</sup>-form using 2M NaOH, then into its I<sup>-</sup>-form by passing dilute KI soln, can be used. Passage of an HI solution under CO<sub>2</sub> through such a column removes polyiodide. The column can be regenerated with NaOH. [Irving and Wilson *Chemistry & Industry (London)* 653 1964]. The earlier method was to reflux with red phosphorus and distil in a stream of N<sub>2</sub>. The colourless product was stored in ampoules in the dark [Bradbury *JACS* **74** 2709 1952]. Fumes in moist air.

**Hydrobromic acid** [10035-10-6] **M 80.9**. A soln of aqueous HBr *ca* 48% (w/w, constant boiling) was distilled twice with a little red phosphorus, and the middle half of the distillate was taken. (The azeotrope at 760mm contains 47.8% (w/w) HBr.) [Hetzer, Robinson and Bates *JPC* **66** 1423 1962]. Free bromine can be removed by Irvine and Wilson's method for HI (see above), except that the column is regenerated by washing with an ethanolic solution of aniline or styrene. Hydrobromic acid can also be purified by aerating with H<sub>2</sub>S distilling and collecting the fraction boiling at 125-127°.

**Hydrochloric acid** [7647-01-0] **M 36.5, d 1.20**. Readily purified by fractional distillation as constant boiling point acid, following dilution with H<sub>2</sub>O. The constant-boiling fraction contains 1 mole of HCl in the following weights of distillate at the stated pressures: 179.555g (730mm), 179,766g (740mm), 179,979 (750mm), 180.193 (760mm), 180.407 (770mm) [Foulk and Hollingsworth *JACS* **45** 1220 1923].

**Hydrofluoric acid** [7664-36-3] **M 20.0, d 1.150**. Freed from lead (Pb *ca* 0.002ppm) by co-precipitation with SrF<sub>2</sub>, by addition of 10ml of 10% SrCl<sub>2</sub> soln per kilogram of the conc acid. After the ppte has settled, the supernatant is decanted through a filter in a hard-rubber or paraffined-glass vessel [Rosenqvist *Amer J Sci* **240** 358 1942. Pure aqueous HF solutions (up to 25M) can be prepared by isothermal distn in polyethylene, polypropylene or platinum apparatus [Kwestroo and Visser *Analyst* **90** 297 1965]. **HIGHLY TOXIC**.

**Hydrogen** [1333-75-0] **M 2.0, m -259.1°, -252.9°**. Usually purified by passage through suitable absorption train. Carbon dioxide is removed with KOH pellets, soda-lime or NaOH pellets. Oxygen is removed with a "De-oxo" unit or by passage over Cu heated to 450-500°, Cu on Kieselguhr at 250°. Passage over a mixture of MnO<sub>2</sub> and CuO (Hopcalite) oxidises any CO to CO<sub>2</sub> (which is removed as above). Hydrogen can be dried by passage through dried silica-alumina at -195°, through a dry-ice trap followed by a liquid-N<sub>2</sub> trap packed with glass wool, through CaCl<sub>2</sub> tubes, or through Mg(ClO<sub>4</sub>)<sub>2</sub> or P<sub>2</sub>O<sub>5</sub>. Other purification steps include passage through a hot palladium thimble [Masson *JACS* **74** 4731 1952], through an activated-charcoal trap at -195°, and through non-absorbent cotton-wool filter or small glass spheres coated with a thin layer of silicone grease. *Potentially EXPLOSIVE in air*.

**Hydrogen bromide (anhydrous)** [10035-10-6] **M 80.9**. Dried by passage through Mg(ClO<sub>4</sub>)<sub>2</sub> towers. This procedure is **hazardous**, see Stoss and Zimmermann [*Ind Eng Chem* **17** 70 1939]. Shaken with mercury, distd through a -78° trap and condensed at -195°/10<sup>-5</sup>mm. Fumes in moist air.

**Hydrogen chloride** [7647-01-0] **M 36.5**. Passed through conc H<sub>2</sub>SO<sub>4</sub>, then over activated charcoal and silica gel. Fumes in moist air. Hydrogen chloride in gas cylinder include ethylene, 1,1-dichloroethane and ethyl chloride. The latter two may be removed by fractionating the HCl through a trap cooled to -112°. Ethylene is difficult to remove. Fumes in moist air.

**Hydrogen cyanide (anhydrous)** [74-90-8] **M 27.0, b 25.7°**. Prepared from NaCN and H<sub>2</sub>SO<sub>4</sub>, and dried by passage through H<sub>2</sub>SO<sub>4</sub> and over CaCl<sub>2</sub>, then distilled in a vacuum system and degassed at 77°K before use [Arnold and Smith *JCSFT* 2 77 861 1981]. Cylinder HCN may contain stabilisers against explosive polymerisation, together with small amounts of H<sub>3</sub>PO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, SO<sub>2</sub>, and water. It can be purified by distn over P<sub>2</sub>O<sub>5</sub>, then frozen in Pyrex bottles at Dry-ice temperature for storage. It has a pK<sub>a</sub><sup>25</sup> of 9.22 in water. **HIGHLY POISONOUS.**

**Hydrogen fluoride (anhydrous)** [7664-39-3] **M 20.0, b 19.4°**. Can be purified by trap-to-trap distn, followed by drying over CoF<sub>2</sub> at room temperature and further distn. Alternatively, it can be absorbed on NaF to form NaHF<sub>2</sub> which is then heated under vacuum at 150° to remove volatile impurities. The HF is regenerated by heating at 300° and stored with CoF<sub>3</sub> in a nickel vessel, being distilled as required. (Water content *ca* 0.01%.) To avoid contact with base metal, use can be made of nickel, polychlorotrifluoroethylene and gold-lined fittings [Hyman, Kilpatrick and Katz *JACS* 79 3668 1957]. **HIGHLY TOXIC.**

**Hydrogen iodide (anhydrous)** [10034-85-2] **M 127.9, b -35.5°**. After removal of free iodine from aqueous HI (q.v.), the solution is frozen, then covered with P<sub>2</sub>O<sub>5</sub> and allowed to melt under vacuum. The gas evolved is dried by passage through P<sub>2</sub>O<sub>5</sub> on glass wool. It can be freed from iodine contamination by repeated fractional distillation at low temperatures. Fumes in moist air.

**Hydrogen ionophore II (ETH 1907) (4-nonadecylpyridine - Proton ionophore)** [70268-36-9] **M 345.6, b 180°/0.07mm**. Dissolve the waxy solid (*ca* 60g) in CHCl<sub>3</sub> (200ml), wash with H<sub>2</sub>O (3 X 200ml), dry and evaporate to dryness then distil in vacuum. A waxy solid is formed on cooling the distillate. UV, 257nm ( $\epsilon$  1.86 x 10<sup>3</sup> M<sup>-1</sup>cm<sup>-1</sup>), 308nm ( $\epsilon$  1.7 x 10<sup>2</sup> M<sup>-1</sup>cm<sup>-1</sup>). [IR, NMR UV: *Inorg Chem* 18 2160 1979].

**Hydrogen ionophore III (N,N-dioctadecyl methylamine)** [4088-22-6] **M 536.0, m 40°, 44-46°, 48-49°, b 252-259°**. It can be distd at high vacuum; but dissolving in C<sub>6</sub>H<sub>6</sub>, filtering and evaporating gives a waxy solid suitable for electrode use. It recrystallises from Me<sub>2</sub>CO. [*B* 69 60 1936; *Talanta* 34 435 1987].

**Hydrogen ionophore IV ETH 1778 (octadecyl isonicotinate)** [103225-02-1] **M 375.6, m 57.5°**. Dissolve in Et<sub>2</sub>O and wash 3 times with H<sub>2</sub>O. Dry, evaporate, and recrystallise the residue from EtOAc/hexane (4:1). The pK<sub>a</sub> of the short chain homologue methyl isonicotinate is 3.6. [*AC* 58 2285 1986].

**Hydrogen peroxide** [7722-84-1] **M 34.0, d 1.110**. The 30% material has been steam distilled using distilled water. Gross and Taylor [*JACS* 72 2075 1950] made 90% H<sub>2</sub>O<sub>2</sub> approximately 0.001M in NaOH and then distilled under its own vapour pressure, keeping the temperature below 40°, the receiver being cooled with a Dry-ice/isopropyl alcohol mush. The 98% material has been rendered anhydrous by repeated fractional crystn in all-quartz vessels. **EXPLOSIVE IN CONTACT WITH ORGANIC MATERIAL.**

**Hydrogen sulphide** [7783-06-4] **M 34.1, b -59.6°**. Washed, then passed through a train of flasks containing saturated Ba(OH)<sub>2</sub> (two), water (two), and dilute HCl [Goates et al. *JACS* 73 707 1951]. **HIGHLY POISONOUS.**

**Hydroxylamine** [7803-49-8] **M 33.0, m 33.1°, b 56.5°/22mm**. Crystd from *n*-butanol at -10°, collected by vacuum filtration and washed with cold ethyl ether.

**Hydroxylamine hydrochloride** [5470-11-1] **M 69.5, m 151°**. Crystallised from aqueous 75% ethanol or boiling methanol, and dried under vacuum over CaSO<sub>4</sub> or P<sub>2</sub>O<sub>5</sub>. Has also been dissolved in a minimum of water and saturated with HCl; after three such crystns it was dried under vacuum over CaCl<sub>2</sub> and NaOH.

**Hydroxylamine sulphate** [10039-54-0] **M 164.1, m 170°(dec)**. Crystallised from boiling water (1.6ml/g) by cooling to 0°.

**Hydroxynaphthol Blue, disodium salt, M 620.5.** Crude material was treated with hot EtOH to remove soluble impurities, then dissolved in 20% aqueous MeOH and chromatographed on a cellulose powder column with propanol:EtOH:water (5:5:4) as eluent. The upper of three zones was eluted to give the pure dye which was pptd as the monosodium salt trihydrate by adding conc HCl to the concentrated eluate [Ito and Ueno *Analyst* **95** 583 1970].

**4-Hydroxy-3-nitrobenzenearsonic acid [121-19-7] M 263.0.** Crystd from water.

**Hydroxyurea [127-07-1] M 76.1, m 133-136°, 140°dec, 139-141°(dec).** Recrystallise from absolute EtOH (10g in 150ml). Note that the rate of solution in boiling EtOH is slow (15-30 min). It should be stored in a cool dry place but some decomposition could occur after several weeks. (*Org Synth Col Vol V* 645 1973). It is very soluble in H<sub>2</sub>O and can be crystd from Et<sub>2</sub>O. It has a pKa of 10.6. [*Acta Chem Scand* **10** 256 1956].

**Hypophosphorous acid (Phosphinic acid) [6303-21-5] M 66.0, m 26.5°, d<sub>4</sub><sup>30</sup> 1.217, 1.13 and 1.04 for 50, 30-32, and 10% aq solns resp.** Phosphorous acid is a common contaminant of commercial 50% hypophosphorous acid. Jenkins and Jones [*JACS* **74** 1353 1952] purified this material by evaporating about 600ml in a 1L flask at 40°, under reduced pressure (in N<sub>2</sub>), to a volume of about 300ml. After the soln was cooled, it was transferred to a wide-mouthed Erlenmeyer flask which was stoppered and left in a Dry-ice/acetone bath for several hours to freeze (if necessary, with scratching of the wall). When the flask was then left at ca 5° for 12h, about 30-40% of it liquefied, and again filtered. This process was repeated, then the solid was stored over Mg(ClO<sub>4</sub>)<sub>2</sub> in a vacuum desiccator in the cold. Subsequent crystns from *n*-butanol by dissolving it at room temperature and then cooling in an ice-salt bath at -20° did not appear to purify it further. The free acid forms deliquescent crystals m 26.5°, and is soluble in H<sub>2</sub>O and EtOH. It has a pKa of 1.1, and the NaH<sub>2</sub>PO<sub>3</sub> salt can be purified through an anion exchange resin [*Z Anorg Allgem Chem* **260** 267 1949].

**Hydroxylamine-O-sulphonic acid [2950-43-8] M 113.1, m 210-211°, 215°(dec).** Stir the solid vigorously with anhydrous Et<sub>2</sub>O and filter off using large volumes of dry Et<sub>2</sub>O. Drain dry at the pump for 5min and then for 12-14h in a vacuum. Store in a vacuum desiccator/conc H<sub>2</sub>SO<sub>4</sub>. Determine the purity by oxidation of iodide to I<sub>2</sub>. Must be stored in a dry atmosphere at 0-4°. It decompose slowly in H<sub>2</sub>O at 25° and more rapidly above this temperature. [*Inorg Synth* **5** 122 1957].

**Indium [7440-74-6] M 114.8.** Before use, the metal surface can be cleaned with dilute HNO<sub>3</sub>, followed by a thorough washing with water and an alcohol rinse.

**Indium (III) chloride [10025-82-8] M 211.2, m 586°, d 4.0.** The anhydrous salt forms yellow deliquescent crystals which can be sublimed at 600° in the presence of Cl<sub>2</sub>/N<sub>2</sub> (1:1) {does not melt}. It is resublimed in the presence of Cl<sub>2</sub>/N<sub>2</sub> (1:10) and finally heated to 150° to expel excess Cl<sub>2</sub>. It is soluble in H<sub>2</sub>O and should be stored in a tightly closed container. [*JACS* **55** 1943 1933].

**Indium (III) oxide [1313-43-2] M 277.6, d 7.18.** Wash with H<sub>2</sub>O and dry below 850°. Volatilises at 850° and dissolves in hot mineral acids to form salts. Store away from light because it darkens due to formation of In.

**Indium sulphate [13464-82-9] M 517.8.** Crystd from dilute aqueous H<sub>2</sub>SO<sub>4</sub>.

**Indium (III) sulphate (5H<sub>2</sub>O) [17069-79-3] M 607.9, d 3.44.** Dissolve in strong H<sub>2</sub>SO<sub>4</sub> and slowly evaporate at ca 50°. Wash crystals with glacial AcOH and then heat in a furnace at a temperature of 450-500° for 6h. Sol in H<sub>2</sub>O is 5%. The pentahydrate is converted to an anhydrous *hygroscopic* powder on heating at 500° for 6h; but heating above this temperature over N<sub>2</sub> yields the oxide sulphate. Evaporation of neutral aqueous solutions provides basic sulphates. [*JACS* **55** 1943 1933, **58** 2126 1936].

**Iodic acid** [7782-68-5] **M 175.9, m 118°(dec), d 4.628.** Dissolve in the minimum volume of hot dilute HNO<sub>3</sub>, filter and evaporate in a vacuum desiccator until crystals are formed. Collect crystals and wash with a little cold H<sub>2</sub>O and dry in air in the dark. Soluble in H<sub>2</sub>O: 269g/100ml at 20° and 295g/100ml at 40°. Soluble in dilute EtOH and darkens on exposure to light. It is converted to HIO<sub>3</sub>.I<sub>2</sub>O<sub>5</sub> on heating at 70°, but at 220° complete conversion to HIO<sub>3</sub> occurs. [*JACS* **42** 1636 1920, **53** 44 1931].

**Iodine** [7553-56-2] **M 253.8, m 113.6°.** Usually purified by vacuum sublimation. Preliminary purifications include grinding with 25% by weight of KI, blending with 10% BaO and subliming; subliming with CaO; grinding to a powder and treating with successive portions of H<sub>2</sub>O to remove dissolved salts, then drying; and crystn from benzene. Barrer and Wasilewski [*TFS* **57** 1140 1961] dissolved I<sub>2</sub> in conc KI and distilled it, then steam distilled three times, washing with distilled H<sub>2</sub>O. Organic material was removed by sublimation in a current of O<sub>2</sub> over platinum at about 700°, the iodine being finally sublimed under vacuum.

**Iodine monobromide** [7789-33-5] **M 206.8, m 42°,**

**Iodine monochloride** [7790-99-0] **M 162.4, m 27.2°.** Purified by repeated fractional crystallisation from its melt.

**Iodine pentafluoride** [7783-66-6] **M 221.9, m -8.0°, b 97°.** Rogers et al. [*JACS* **76** 4843 1954] removed dissolved iodine from IF<sub>5</sub> by agitating with a mixture of dry air and ClF<sub>3</sub> in a fluorothene beaker using a magnetic stirrer. The mixture was transferred to a still and the more volatile impurities were pumped off as the pressure was reduced below 40mm. The still was gradually heated (kept at 40mm) to remove the ClF<sub>3</sub> before IF<sub>5</sub> distilled. Stevens [*JOC* **26** 3451 1961] pumped IF<sub>5</sub> under vacuum from its cylinder, trapping it at -78°, then allowing it to melt in a stream of dry N<sub>2</sub>.

**Iodine trichloride** [22520-96-3] **M 233.3, m 33°, b 77°(dec).** Purified by sublimation at room temperature.

**Iodomethyl trimethylsilane** [4206-67-1] **M 214.1, b 139.5°/744mm, d 1.44, n<sub>D</sub><sup>25</sup> 1.4917.** If slightly violet in colour wash with aqueous 1% sodium metabisulphite, H<sub>2</sub>O, dry over Na<sub>2</sub>SO<sub>4</sub> and fractionally distil at atmospheric pressure. [*JACS* **68** 481 1946].

**Iodotrimethylsilane** [16029-98-4] **M 200.1, b 106.8°/742mm, 107.5°/760mm, d 1.470.** Add a little antimony powder and fractionate with this powder in the still. Stabilise with 1% wt of Cu powder. [*JCS* 3077 1950].

**Iridium** [7439-88-5] **M 192.2, m 2450°, b ~4500°, d 22.65.** It is a silver white hard solid which oxidises superficially in air. Scrape the outer tarnished layer until silver clear and store under paraffin. Stable to acids but dissolves in aqua regia. [*Chem Reviews* **32** 277 1943].

**Iridium (IV) chloride hydrate (hexachloroiridic acid)** [16941-92-7] **M 515.1.** If it contains nitrogen then repeatedly concentrate a conc HCl solution until free from nitrogen, and dry free from HCl in a vacuum over CaO until crystals are formed. The solid is very *hygroscopic*. [*JACS* **53** 884 1931; *Handbook of Preparative Inorganic Chemistry* (ed Brauer) Vol II 1592 1965].

**Iron (wire)** [7439-89-6] **M 55.9, m 1535°.** Cleaned in conc HCl, rinsed in de-ionised water, then reagent grade acetone and dried under vacuum.

**Iron enneacarbonyl (di-iron nonacarbonyl)** [15321-51-4] **M 363.7, m 100°(dec).** Wash with EtOH and Et<sub>2</sub>O and dry in air. Sublimes at 35° at high vacuum. Dark yellow plates stable for several days when kept in small amounts. Large amounts, especially when placed in a desiccator spontaneously *ignite* in a period of one day. It decomposes in moist air. It is insoluble in hydrocarbon solvents but forms complexes with several organic compounds. [*JACS* **72** 1107 1950; *B* **60** 1424 1424 ].

**Iron (III) meso-5,10,15,20-tetraphenylporphine chloride complex** [16456-81-8] **M 704.0.** Crystallise by extraction from a thimble (Soxhlet) with CHCl<sub>3</sub>. Concentrate the extract to ca 10ml and add ca

80ml of hot MeOH. Dark blue crystals separate on cooling. It can be recrystallised several times from  $\text{CHCl}_3$ -MeOH. Avoid prolonged heating. It is quite soluble in organic solvents but insoluble in pet ether. [JACS 70 1808 1948; UV: 73 4315 1951].

**Iron pentacarbonyl** [13463-40-6] M 195.9, b 102.5°, n 1.520, d 1.490. Distilled under vacuum, the middle cut being redistd twice and stored in a bulb protected from light (*photosensitive*).

**Isopentyloxy trimethylsilane** [1833-53-0] M 130.3, b 93-95°, d 0.786. Can contain up to 5% of hexamethyldisiloxane (b 99-101°) but is generally non-reactive and need not be removed. It can be removed by efficient fractional distillation at atmospheric pressure.

**Isopropenyloxy trimethylsilane** [1833-53-0] M 130.3, b 93-95°/atm, d 0.786. Purified by fractional distillation using a very efficient column at atmospheric pressure. Usually contains 5% of hexamethyldisiloxane which boils at 99-101°, but is generally non-reactive and need not be removed. [JACS 71 5091 1952]. It has been distilled under  $\text{N}_2$  through a 15cm column filled with glass helices. Fraction b 99-104° is further purified by gas chromatography through a Carbowax column (Autoprep A 700) at a column temperature of 87°, retention time is 9.5min. [J Organometal Chem 1 476 1963-4].

**Isopropyl dimethyl chlorosilane** [3634-56-8] M 140.7, b 109.8-110.0°/738mm, d 0.88, n 1.4158. Probable impurity is  $\text{Me}_3\text{SiCl}$  (b 56.9°/783mm) which can be removed by efficient fractional distillation. [JACS 76 801 1954].

## Lanthanide shift reagents see Chapter 3.

**Lanthanum** [7439-91-0] M 138.9, m 920°, b 3470°, d 6.16. White metal that slowly tarnishes in air due to oxidation. Slowly decomposed by  $\text{H}_2\text{O}$  in the cold and more rapidly on heating to form the hydroxide. The metal is cleaned by scraping off the tarnished areas until the shiny metal is revealed and stored under oil or paraffin. It burns in air at 450°.

**Lanthanum triacetate** [917-70-4] M 316.0  $\times \text{H}_2\text{O}$ . Boil with redistilled  $\text{Ac}_2\text{O}$  for 10min (does not dissolve and is a white solid). Cool, filter, wash with  $\text{Ac}_2\text{O}$  and keep in a vacuum desiccator (NaOH) till free from solvent. [JICS 33 877 1956].

**Lead [II] acetate** [301-04-2] M 325.3, m 280°. Crystallised twice from anhydrous acetic acid and dried under vacuum for 24h at 100°.

**Lead (II) bromide** [10031-22-8] M 367.0, m 373°. Crystallised from water containing a few drops of HBr (25ml of water per gram  $\text{PbBr}_2$ ) between 100° and 0°. A neutral solution was evaporated at 110° and the crystals that separated were collected by rapid filtration at 70°, and dried at 105° (to give the *monohydrate*). To prepare the anhydrous bromide, the hydrate is heated for several hours at 170° and then in a Pt boat at 200° in a stream of HBr and  $\text{H}_2$ . Finally fused [Clayton et al. JCSFT 176 2362 1980].

**Lead (II) chloride** [7758-95-4] M 278.1, m 501°. Crystallised from distilled water at 100° (33ml/g) after filtering through sintered-glass and adding a few drops of HCl, by cooling. After three crystns the solid was dried under vacuum or under anhydrous HCl vapour by heating slowly to 400°.

**Lead diethyldithiocarbamate** [17549-30-3] M 503.7. Wash with  $\text{H}_2\text{O}$  and dry at 60-70°, or dissolve in the min vol of  $\text{CHCl}_3$  and add the same vol of EtOH. Collect the solid that separates and dry as before. Alternatively, recryst by slow evaporation of a  $\text{CHCl}_3$  soln at 70-80°. Filter the crystals, wash with  $\text{H}_2\text{O}$  until all  $\text{Pb}^{++}$  ions are eluted (check by adding chromate) and then dry at 60-70° for at least 10h. [A 49 1146 1977].

**Lead (II) formate** [811-54-4] M 297.3. Crystd from aqueous formic acid.

**Lead (II) iodide** [10101-63-0] **M 461.0, m 402°**. Crystd from a large volume of water.

**Lead monoxide** [1317-36-8] **M 223.2, m 886°**. Higher oxides were removed by heating under vacuum at 550° with subsequent cooling under vacuum. [Ray and Ogg *JACS* **78** 5994 1956].

**Lead nitrate** [10099-74-8] **M 331.2**. Ppted twice from hot (60°) conc aqueous soln by adding HNO<sub>3</sub>. The ppte was sucked dry in a sintered-glass funnel, then transferred to a crystallising dish which was covered by a clock glass and left in an electric oven at 110° for several hours [Beck, Singh and Wynne-Jones *TFS* **55** 331 1959].

**Lead (biscyclopentadienyl)** [1294-74-2] **M 337.4**. Purified by vacuum sublimation. Handled and stored under N<sub>2</sub>.

**Lead tetraacetate** [546-67-8] **M 443.4**. Dissolved in hot glacial acetic acid, any lead oxide being removed by filtration. White crystals of lead tetraacetate separated on cooling. Stored in a vacuum desiccator over P<sub>2</sub>O<sub>5</sub> and KOH for 24h before use.

**Lissapol C (mainly sodium salt of cetyl oleyl alcohol sulphate)** [2425-51-6],  
**Lissapol LS (mainly sodium salt of anisidine sulphate)** [28903-20-0]. Refluxed with 95% EtOH, then filtered to remove insoluble inorganic electrolytes. The alcohol solution was then concentrated and the residue was poured into dry acetone. The ppte was filtered off, washed in acetone and dried under vacuum. [Biswas and Mukerji *JPC* **64** 1 1960].

**Lithium (metal)** [7439-93-2] **M 6.9**. After washing with pet ether to remove storage oil, lithium was fused at 400° and then forced through a 10-micron stainless-steel filter with argon pressure. It was again melted in a dry-box, skimmed, and poured into an iron distillation pot. After heating under vacuum to 500°, cooling and returning to the dry-box for a further cleaning of its surface, the lithium was distilled at 600° using an all-iron distillation apparatus [Gunn and Green *JACS* **80** 4782 1958].

**Lithium acetate (2H<sub>2</sub>O)** [6108-17-4] **M 102.0, m 54-56°**. Crystallised from EtOH (5ml/g) by partial evaporation.

**Lithium aluminium hydride** [16853-85-3] **M 37.9, m 125°(dec)**. Extracted with Et<sub>2</sub>O, and, after filtering, the solvent was removed under vacuum. The residue was dried at 60° for 3h, under high vacuum [Ruff *JACS* **83** 1788 1961]. **Ignites in the presence of a small amount of water.**

**Lithium amide** [7782-89-0] **M 23.0, m 380-400°, d<sup>17.5</sup> 1.178**. Purified by heating at 400° while NH<sub>3</sub> is passed over it in the upper of two crucibles (the upper crucible is perforated). The LiNH<sub>2</sub> will drip into the lower crucible through the holes in the upper crucible. The product is cooled in a stream of NH<sub>3</sub>. Protect it from air and moisture, store under N<sub>2</sub> in a clear glass bottle sealed with paraffin. Store small quantities so that all material is used once the bottle is opened. If the colour of the amide is yellow it should be destroyed as it is likely to have oxidised and to **EXPLODE**. On heating above 450° it is decomposed to Li<sub>2</sub>NH which is stable up to 750-800°. [*Handbook of Preparative Inorganic Chemistry* (ed Brauer) Vol I 463 1963; *Inorg Synth* **2** 135 1953].

**Lithium benzoate** [553-54-8] **M 128.1**. Crystd from EtOH (13ml/g) by partial evaporation.

**Lithium borohydride** [16949-15-8] **M 21.8**. Crystd from Et<sub>2</sub>O, and pumped free of ether at 90-100° during 2h [Schaeffer, Roscoe and Stewart *JACS* **78** 729 1956].

**Lithium bromide** [7550-35-8] **M 86.8, m 550°**. Crystd several times from water or EtOH, then dried under high vacuum for 2 days at room temperature, followed by drying at 100°.

**Lithium carbonate** [554-13-2] **M 73.9, m 618°**. Crystd from water. Its solubility decreases as the temperature is raised



**Lithium chloride** [7447-47-8] **M 42.4, m 600°**. Crystd from water (1ml/g) or MeOH and dried for several hours at 130°. Other metal ions can be removed by preliminary crystallisation from hot aqueous 0.01M disodium EDTA. Has also been crystallised from conc HCl, fused in an atmosphere of dry HCl gas, cooled under dry N<sub>2</sub> and pulverised in a dry-box. Kolthoff and Bruckenstein [*JACS* **74** 2529 1952] pptd with ammonium carbonate, washed with Li<sub>2</sub>CO<sub>3</sub> five times by decantation and finally with suction, then dissolved in HCl. The LiCl solution was evaporated slowly with continuous stirring in a large evaporating dish, the dry powder being stored (while still hot) in a desiccator over CaCl<sub>2</sub>.

**Lithium diisopropylamide** [4111-54-0] **M 107.1, b 82-84°/atm, 84°/atm, d<sup>22</sup> 0.722, flash point -6°**. It is purified by refluxing over Na wire or NaH for 30min and then distilled into a receiver under N<sub>2</sub>. Because of the low boiling point of the amine a dispersion of NaH in mineral oil can be used directly in this purification without prior removal of the oil. It is *highly flammable*, and is decomposed by air and moisture. [*Org Synth* **50** 67 1970].

**Lithium dodecylsulphate** [2044-56-6] **M 272.3**. Recrystd twice from absolute EtOH and dried under vacuum.

**Lithium fluoride** [7789-24-4] **M 25.9, m 842°, 848°, b 1676°, 1681°, d 2.640**. Possible impurities are LiCO<sub>3</sub>, H<sub>2</sub>O and HF. These can be removed by calcining at red heat, then pulverised with a Pt pestle and stored in a paraffin bottle. Solubility in H<sub>2</sub>O is 0.27% at 18°. It volatilises between 1100-1200°. [*Handbook of Preparative Inorganic Chemistry* (ed Brauer) Vol I 235 1963].

**Lithium formate (H<sub>2</sub>O)** [556-63-8] **M 70.0**. Crystd from hot water (0.5ml/g) by chilling.

**Lithium hydride**. [7580-67-8] **M 7.95, m 680°, d 0.76-0.77**. It should be a white powder otherwise replace it. It darkens rapidly on exposure to air and is decomposed by H<sub>2</sub>O to give H<sub>2</sub> and LiOH, and reacts with lower alcohols. One gram in H<sub>2</sub>O liberates 2.8L of H<sub>2</sub>.

**Lithium hydroxide (H<sub>2</sub>O)** [1310-66-3] **M 42.0**. Crystd from hot water (3ml/g) as the monohydrate. Dehydrated at 150° in a stream of CO<sub>2</sub>-free air.

**Lithium iodate** [13765-03-2] **M 181.9**. Crystd from water and dried in a vacuum oven at 60°.

**Lithium iodide** [10377-51-2] **M 133.8**. Crystd from hot water (0.5ml/g) by cooling in CaCl<sub>2</sub>-ice, or from acetone. Dried under vacuum over P<sub>2</sub>O<sub>5</sub> for 1h at 60° and then at 120°.

**Lithium ionophore I (ETH 149)** see *N,N'*-diheptyl-*N,N'*-5,5-tetramethyl-3,7-dioxanonanediamide.

**Lithium ionophore V** see 12-crown-4, (1,4,7,10-tetraoxacyclododecane).

**Lithium ionophore VI** see 6,6-dibenzyl-14-crown-4, (6,6-dibenzyl-1,4,8,11-tetraoxacyclotetradecane).

**Lithium methylate (lithium methoxide)** [865-34-9] **M 38.0**. Most probable impurity is LiOH due to hydrolysis by moisture. It is important to keep the sample dry. It can be dried by keeping in a vacuum at 60-80° under dry N<sub>2</sub> using an oil pump for a few hours. Store under N<sub>2</sub> in the cold. It should not have bands above 3000cm<sup>-1</sup>; IR has  $\nu_{\text{KBr}}$  1078, 2790, 2840 and 2930cm<sup>-1</sup>. [*JOC* **21** 156 1956].

**Lithium nitrate** [7790-69-4] **M 68.9**. Crystd from water or EtOH. Dried at 180° for several days by repeated melting under vacuum. If it is crystallised from water keeping the temperature above 70°, formation of trihydrate is avoided. The anhydrous salt is dried at 120° and stored in a vacuum desiccator over CaSO<sub>4</sub>.

**Lithium nitrite (H<sub>2</sub>O)** [13568-33-7] **M 71.0**. Crystd from water by cooling from room temperature.