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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 Et₂O, wash with H₂O, dry over CaCl₂ and distil. [*J Am Chem Soc* 77 1685 1955; *Collect Czech Chem Comm* 24 3758 1959.]

Eosin B (Bluish, Eosin Scarlet, 4',5'-dibromo-2',7'-dinitrofluorescein disodium salt) [548-24-3] M 624.1, λ_{\max} 514nm, CI 45400. Freed from inorganic halides by repeated crystn from butan-1-ol.

Eosin Y (as di-Na salt) [17372-87-1] M 691.9. Dissolved in water and ppted by addition of dilute HCl. The ppt 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 *Trans Faraday Soc* 57 1894 1961].

Eosin YS (Eosin Yellowish, 2',4',5'7'-tetrabromofluorescein di-Na salt) [17372-87-1] M 691.9, CI 45380. Dissolve in the minimum vol of H₂O (1g/mL), filter and add EtOH until separation of salt is complete. Filter off, wash with abs EtOH, then Et₂O and dry first in air, then at 100°. Used for staining blood cells and for estimating traces of Ag. [Selsted and Becker *Anal Biochem* 155 270 1986; El-Ghamry and Frei *Anal Chem* 40 1986 1968.]

Eriochrome Black T [1787-61-7] M 416.4, $A_{1\text{cm}}^{\%}$ (λ_{\max}) 656(620nm) at pH 10, using the dimethylammonium salt, $\text{pK}_2^{2.5}$ 5.81, $\text{pK}_3^{2.5}$ 11.55. The sodium salt (200g) was converted to the free acid by stirring with 500mL of 1.5M HCl, and, after several minutes, the slurry was filtered on a sintered-glass funnel. The process was repeated and the material was air dried after washing with acid. It was extracted with *benzene for 12h in a Soxhlet extractor, then the *benzene was evaptd and the residue was air dried. A further desalting with 1.5M HCl (1L) was followed by crystn from dimethylformamide (in which it is very soluble) by forming a saturated soln at the boiling point, and allowing to cool slowly. The crystalline dimethylammonium salt so obtained was washed with *benzene and treated repeatedly with dilute HCl to give the insoluble free acid which, after air drying, was dissolved in alcohol, filtered and evaporated. The final material was air dried, then dried in a vacuum desiccator over Mg(ClO₄)₂ [Diehl and Lindstrom, *Anal Chem* 31 414 1959]. Indicator for complexometry of alkaline earth metals.

Eriochrome Blue Black R (Palatine Chrome Black 6BN, Calcon, 3-hydroxy-4-(2-hydroxy-1-naphthylazo)naphthalene-1-sulfonic acid Na salt) [2538-85-4] M 416.4, $\text{pK}_2^{2.5}$ 7.0, $\text{pK}_3^{2.5}$ 13.5. Freed from metallic impurities by three pptns from aqueous soln by addn of HCl. The ppted dye was dried at 60° under vacuum. Indicator for complexometry of Al, Fe and Zr.

Ethoxycarbonylmethylene triphenylphosphonium bromide [1530-45-6] M 429.3, m 155-155.5°, 158°(dec). Wash with pet ether (b 40-50°) and recryst from CHCl₃/Et₂O and dry in high vac at 65°. [Isler et al. *Helv Chim Acta* 40 1242 1957; Wittig and Haag *Chem Ber* 88 1654, 1664 1955.]

(Ethoxycarbonylmethylene)triphenylphosphorane [ethyl (triphenylphosphoranylidene)-acetate] [1099-45-2] M 348.4, m 116-117°, 128-130°. Cryst by dissolving in AcOH and adding pet ether (b 40-50°) to give colorless plates. UV λ_{\max} ($A_{1\text{mm}}^1$): 222nm (865) and 268nm (116) [Isler et al. *Helv Chim Acta* 40 1242 1957].

Ethylarsonic acid [507-32-4] M 154.0, m 99.5°, pK_1 4.72 (As(OH)O⁻), pK_2 8.00 [AsO₂²⁻]. Crystd from ethanol.

2-Ethyl-1,2-benzisoxazolium tetrafluoroborate [4611-62-5] M 235.0, m 107-109°, 109.5-110.2°. Recrystd from MeCN-EtOAc to give magnificent crystals. It is not hygroscopic but on long exposure to moisture it etches glass. It is light-sensitive and should be stored in brown glass bottles. UV (H₂O), λ_{\max} 258nm (ϵ 13 100) and λ_{\max} 297nm (ϵ 2 900); IR (CH₂Cl₂): 1613 (C=N) and 1111-1000 (BF₄⁻) [UV, IR, NMR: Kemp and Woodward *Tetrahedron* 21 3019 1965].

Ethylene bis(diphenylphosphine) [1,2-bis(diphenylphosphino)ethane] [1663-45-2] **M 398.4, m 139-140°**. See 1,2-bis-(diphenylphosphino)ethane (DIPHOS) on p. 402.

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 Orange (sodium 4,4'-diethylaminophenylazobenzenesulfonate) [62758-12-7] **M 355.4, pK_{Est} ~ 3.8**. Recrystd twice from water.

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 [J Am Chem Soc 75 994 1953]. Alternatively, dissolve in Et₂O, wash with H₂O, dilute Na₂CO₃, dry over Na₂CO₃, evaporate Et₂O, and distil through a column of 15 theoretical plates [J Am Chem Soc 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 Et₂O, wash with H₂O, dilute Na₂CO₃, dry over Na₂SO₄, evaporate Et₂O and fractionally distil. [J Am Chem Soc 72 1935 1950.]

Ethynyl tributylstannane [994-89-8] **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 H₂O (100mL), drying (MgSO₄), evaporating and distilling in a vacuum. It has IR ν 3280 (\equiv C-H), 2950, 2850, 2005 (C \equiv C), 1455, 1065 and 865cm⁻¹. [J Org Chem 46 5221 1981; J Am Chem Soc 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 (C \equiv C) and 3289 (\equiv C-H) cm⁻¹. [Chem Ber 92 30 1959.]

Ethyl triphenylphosphonium bromide [1530-32-1] **M 371.3, m 203-205°**. Recrystd from H₂O and dried in high vacuum at 100°. IR has bands at 1449, 1431 and 997cm⁻¹. [Justus Liebigs Ann Chem 606 1 1957; J Org Chem 23 1245 1958.]

Europium (III) acetate (2H₂O) [62667-64-5] **M 383.1, pK₁^{2.5} 8.31 (for aquo Eu³⁺)**. Recrystd several times from water [Ganapathy et al. J Am Chem Soc 108 3159 1986]. For europium shift reagents see lanthanide shift reagents in Chapter 4.

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 [J Chem Soc 1256 1938]. Recrystd from EtOH or Et₂O, **m 179°** [Justus Liebigs Ann Chem 323 13 1902]. Recrystd from absolute EtOH, **m 159.5°** [Chem Ber 67 286 1934]. Dry for 1hr at 120°.

Ferric Bromide [10031-26-2] **M 395.6, m >130°(dec)**. Sublimed in a sealed tube with Br₂ at 120°-200°. [Lux in Handbook of Preparative Inorganic Chemistry (Ed. Brauer) Vol II, p 1494 1963.]

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 (6H₂O) [10025-77-1] **M 270.3, m 37°(dec), pK₁²⁵ 2.83, pK₂²⁵ 4.59** (for hydrolysis of Fe³⁺). 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 *J Phys Chem* **60** 91 1956].

Ferric nitrate (9H₂O) [7782-61-8] **M 404.0, m 47°(dec)**. Cryst from aqueous solutions of moderately strong HNO₃ as the violet nonahydrate. With more concentrated aqueous solns (containing some HNO₃), the hexahydrate crystals out. The anhydrous salt is slightly deliquescent and decomposes at 47°.

Ferric perchlorate (9H₂O) [13537-24-1] **M 516.3, pK²⁵ -2.4 to -3.1 (for HClO₄)**. Crystd twice from conc HClO₄, the first time in the presence of a small amount of H₂O₂ to ensure that the iron is fully oxidised [Sullivan *J Am Chem Soc* **84** 4256 1962]. Extreme care should be taken with this preparation because it is potentially **DANGEROUS**.

Ferric sulfate (xH₂O) [10028-22-5] **M 399.9 + xH₂O**. Dissolve in the minimum volume of dilute aqueous H₂SO₄ and allow to evaporate at room temp until crystals start to form. Do not concentrate by boiling off the H₂O 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 H₂O slowly unless ferrous sulfate is added.

Ferrocene [102-54-5] **M 186.0, m 173-174°**. Purified by crystn from pentane or cyclohexane (also *C₆H₆ or MeOH can be used). Moderately soluble in Et₂O. Sublimes readily above 100°. Crystallisation from EtOH gave **m 172.5-173°**. [*Org Synth Coll Vol IV* 473 1963; *J Chem Soc* 632 1952.] Also crystd from methanol and sublimed *in vacuo*. [Saltiel et al. *J Am Chem Soc* **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 [*J Org Chem* **22** 355 1957]. *2,4-Dinitrophenylhydrazone* **m 248°(dec)**. [*Beilstein* **16 4th Suppl** 1798; *J Am Chem Soc* **79** 3416 1957; *J Chem Soc* 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. *J Am Chem Soc* **107** 3411 1985.] *Acid chloride* **m 49°** crystallises from pentane, λ_{max} 458nm [*J Org Chem* **24** 280 1959]. *Methyl ester* crystallises from aq MeOH **m 70-71°**. *Anhydride* **m 143-145°** from pet ether [*J Org Chem* **24** 1487 1959]. *Amide* **m 168-170°** from CHCl₃-Et₂O or **m 167-169°** from *C₆H₆-MeOH. [*J Am Chem Soc* **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°** [*J Am Chem Soc* **74**, 3458 1958]. *Diacid chloride* **m 92-93°** from pet ether. [*Dokl Acad Nauk SSSR* **120** 1267 1958; **127** 333 1959.]

Ferrocene-1,1,-dimethanol [1291-48-1] **M 246.1, m 107-108°**. Obtained from the diacid with LiAlH₄ reduction and recrystd from Et₂O-pet ether. [*J Am Chem Soc* **82** 4111 1960.]

Ferrous bromide [20049-65-4] **M 215.7 + xH₂O, m 684°, d²⁵ 4.63**. Crystn from air-free H₂O provides the *hexahydrate* as pale green to bluish-green rhombic prisms. On heating at 49° H₂O is lost and the *tetrahydrate* is formed. Further heating at 83° more H₂O 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 H₂SO₄ under N₂ in tightly closed containers. They have some solubility in EtOH. [*Chem Ber* **38** 236 1904.]

Ferrous chloride (4H₂O) [13478-10-9] **M 198.8, m 105°(dec), pK₁²⁵ 6.7, pK₂²⁵ 9.3** (for aquo Fe²⁺). 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 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 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 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 *J Am Chem Soc* **78** 3944 1956].

Ferrous chloride [7758-94-3] **M 126.8, m 674° , b 1023° , d^{25} 3.16.** Sublimes in a stream of HCl at *ca* 700° , or in H_2 below 300° . Its vapour pressure at 700° is 12mm. Anhydrous FeBr_2 can be obtained by carefully dehydrating the *tetrahydrate* in a stream of HBr and N_2 , and it can be sublimed under 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 H_2O , EtOH Me_2CO but insol in Et_2O . The *tetrahydrate* is pale green to pale blue in colour and loses $2\text{H}_2\text{O}$ at 105 - 115° . The *dihydrate* loses H_2O 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 ($6\text{H}_2\text{O}$) [13933-23-8] **M 362.9, pK^{25} -2.4 to -3.1 (for HClO_4).** Crystd from HClO_4 .

Ferrous sulfate ($7\text{H}_2\text{O}$) [7782-63-0] **M 278.0, m $\sim 60^\circ$ (dec).** Crystd from 0.4M H_2SO_4 .

Fluorine [7782-41-4] **M 38.0, b -129.2° .** Passed through a bed of NaF at 100° to remove HF and SiF_4 . [For description of stills used in fractional distn, see Greenberg et al. *J Phys Chem* **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, pK -4.9.** Crystd several times from conductivity water.

Fluorotrimethylsilane (trimethylsilyl fluoride, TMSF) [420-56-4] **M 92.2, m -74° , b $16^\circ/760\text{mm}$, $19^\circ/730\text{mm}$, d^0 0.793.** It is a **FLAMMABLE** gas which is purified by fractional distn through a column at low temperature and with the exclusion of air [Booth and Suttle *J Am Chem Soc* **68** 2658 1946; Reid and Wilkins *J Chem Soc* 4029 1955].

Gallium [7440-55-3] **M 69.7, m 29.8° .** Dissolved in dilute HCl and extracted into Et_2O . Pptn with H_2S removed many metals, and a second extraction with Et_2O 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 μ 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 $133^\circ/100\text{mm}$, $197.7^\circ/700\text{mm}$, d 2.47, pK_1^{25} 2.91, pK_2^{25} 3.70, pK_3^{20} 4.42 (for Ga^{3+}).** Pure compound can be obtained by redistn in a stream of Cl_2 or Cl_2/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 H_2O with liberation of heat. Soluble in Et_2O . [*Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 846 1963.]

Gallium (III) nitrate ($9\text{H}_2\text{O}$) [63462-65-7] **M 417.9, m *ca* 65° .** Recrystd from H_2O (sol: 295g/100mL at 20°). White deliquescent colourless powder soluble in H_2O , absolute EtOH and Et_2O . Loses

HNO₃ upon heating at 40°. Addition of Et₂O to a warm ethanolic soln (40-50°) of Ga(NO₃)₃·9H₂O precipitates Ga(OH)₂NO₃·Ga(OH)₃·2H₂O. If the salt has partly hydrolysed, dissolve in conc HNO₃, reflux, dilute with H₂O and concentrate on a sand bath. Wash several times by adding H₂O and evaporate until there is no odour of acid. Dilute the residue to a Ga concentration of 26g/100mL. At this concentration, spongy Ga(NO₃)₃·xH₂O 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 H₂O 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) sulfate [13494-91-2 (*anhydr*); 13780-42-2 (*hydr*)] **M 427.6**. Recrystn from H₂O gives the 16-18H₂O hydrate (sol at 20° is 170g/100mL). Alternatively dissolve in 50% H₂SO₄ and evaporate (60-70°), cool and ppt by adding EtOH/Et₂O. 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, m 937°, 925-975°, b 2700°, d 5.3**. Copper contamination on the surface and in the bulk of single crystals of Ge can be removed by immersion in molten alkali cyanide under N₂. 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 *J Phys Chem* **60** 45 1956].

Germanium (IV) oxide [1310-53-8] **M 104.6, m 1080°(soluble form), d²⁵ 6.239; m 1116°(insoluble form) d²⁵ 4.228, pK₁²⁵ 9.02, pK₂²⁵ 12.82 (for germanic acid H₂GeO₃)**. The oxide (GeO₂) is usually prepared by hydrolysing redistd GeCl₄ and igniting in order to remove H₂O and chloride. It can be further purified by dissolving in hot H₂O (sol: 4g/L cold) evaporating and drying the residual crystalline solid. When the *soluble* form (which is produced in H₂O 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. [*J Am Chem Soc* **46** 2358 1924, **47** 1945 1925, **54** 2303 1032.]

Germanium tetrachloride [10038-98-9] **M 214.4, m -49.5° (α), -52.0° (β), b 83.1°/760mm, 86.5°/760mm corr, d₄²⁰ 1.84**. Traces of Cl₂ and HCl can be removed from the liquid by blowing dry air through it for a few hours at room temperature or shake it with Hg or Hg₂Cl₂ 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 GeO₂. It is slowly hydrolysed by H₂O to give GeO₂. [*J Am Chem Soc* **44** 306 1922.]

Germanium tetraethoxide [14165-55-0] **M 252.8, m -72°; b 54.5°/5mm, 71-72°/11mm, 188-190°/722mm, d²⁵ 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. [*J Am Chem Soc* **75** 718 1953; *J Chem Soc* 4916 1956.]

Glass powder (100-300 mesh). Washed with 10% HNO₃, water and dried.

Gold (III) bromide (gold tribromide) [10294-28-7] **M 436.7, m 150°(dec)**. Purified by adding pure Br₂ 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 NaOH until free Br₂ is no longer in the apparatus (48-60h). The bright yellow needles of the tribromide are stable over NaOH in the dark. It is sol in H₂O and in 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 AuBr₃ (actually Au₂Br₆) until all dissolves, whereby the acid crystallises out as H₂AuBr₄·5H₂O, deliquescent solid soluble in EtOH with **m ca 27°**, and store as above. [*J Chem Soc* 2410 1931, 217, 219 1935.]

Gold (III) chloride (hydrate) [16903-35-8] **M 339.8 + xH₂O, 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 H₂O but sparingly soluble in EtOH and Et₂O. *Aurochloric acid* is formed when AuCl₃ is dissolved in HCl. [*J Am Chem Soc* **35** 553 1913; *Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol II 1056 1965.]

Gold (I) cyanide [506-65-0] **M 223.0, m dec on heating.** The lemon yellow powder is sparingly soluble in H₂O and EtOH but soluble in aqueous NH₃. It is obtained by heating H[Au(CN)₂] at 110°. Wash well with H₂O and EtOH and dry at 110°. It has an IR band at ν 2239cm⁻¹ typical for C≡N stretching vibration. [*Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol II 1064 1965.] CARE: may evolve HCN.

Gold (I) iodide [10294-31-2] **M 323.9, m 120°(dec), d 8.25.** It has been prepared by heating gold and iodine in a tube at 120° for 4 months. Since it decomposes to Au and I₂ in the presence of UV light and heat then the main impurity is Au. The salt is therefore purified by heating at 120° with I₂ 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 + xH₂O, evolves O₂ at 110°, pK₁²⁵ < 11.7, pK₂²⁵ 13.36, pK₃²⁵ > 15.3 [for Au(OH)₃].** Most probable impurities are Cl⁻ ions. Dissolve in strong boiling KOH soln (ca 5M) and precipitate (care) with excess of 3N H₂SO₄. Then shake and centrifuge, resuspend in H₂O and repeat wash several times until free from SO₄ and Cl ions. This gives a *wet* oxide which is dried in air, and dec to Au in sunlight. It is best to keep it wet as it decomposes on drying (analyse wet sample). Store away from light in the presence of H₂O vapour. It evolves O₂ at 110°. It is insoluble in H₂O but soluble in HCl and conc HNO₃. [*J Am Chem Soc* **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 *J Phys Chem* **60** 1225 1956].

Haematoporphyrin IX [8,13-bis(1-hydroxyethyl)-3,7,12,17-tetramethyl-21H-23H-porphin-2,18-dipropionic acid [14459-29-1] **M 598.7, pK_{Est} ~ 4.8 (-CH₂CH₂COOH).** See haematoporphyrin on p. 541 in Chapter 6.

Helium [7440-59-7] **M 4.0.** Dried by passage through a column of Linde 5A molecular sieves and CaSO₄, then passed through an activated-charcoal trap cooled in liquid N₂, to adsorb N₂, argon, xenon and krypton. Passed over 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 N₂ [Arnold and Smith *J Chem Soc, Faraday Trans 2* **77** 861 1981].

Hexabutyl-distannane [hexabutyl-ditin, bis(tributyl)tin] [813-19-4] **M 580.4, b 160-162°/0.3mm, d 1.148, n 1.512.** Purified by distn in a vacuum and stored in the dark. [Shirai et al. *Yakugaku Zasshi* **90** 59 1970, *Chem Abstr* **72** 90593 1970.]

Hexachlorocyclotriphosphazene [940-71-6] **M 347.7, m 113-114°, 113-115°.** See phosphonitric chloride trimer on p. 450.

Hexachloroplatinic acid hydrate (H₂PtCl₆, chloroplatinic acid, platinum IV chloride solution) [16941-12-1] **M 409.8 + H₂O, m 60° (deliquescent solid).** If it is to be purified, or regenerated from Pt recovered from catalytic hydrogenations, it was dissolved in aqua regia followed by evaporation to dryness and dissolution in the minimum vol of H₂O. Then the aqueous solution was treated with saturated ammonium chloride until all the ammonium hexachloroplatinate separated. The (NH₄)₂PtCl₆ was filtered off and dried at 100°. Ignite the salt to give Pt sponge, dissolve the Pt sponge in aqua regia, boil to

dryness, dissolve in concentrated HCl, boil to dryness again and repeat the process. Protect from light. [Hickers *J Am Chem Soc* **43** 1268 1921; *Org Synth Coll Vol I* 463, 466 1941; Bruce *J Am Chem Soc* **58** 687 1936.]

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 H₂SO₄. [*J Chem Soc* 3077 1950.]

2,2,4,4,6,6-Hexamethylcyclotrisiloxane [1009-93-4] **M 219.5, m -10°, b 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. [*J Am Chem Soc* **70** 3888 1948.]

Hexamethyldisilane [1450-14-2] **M 164.4, m 9-12°, b 113.1°/750mm, d 0.7272, n 1.4229.** Most likely impurity is trimethylchlorosilane (*cf* boiling point). Wash with H₂O, cold conc H₂SO₄, H₂O again then aqueous NaHCO₃, dry over CaSO₄ and fractionate at atmospheric pressure. [*J Chem Soc* 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 (HMDS) [999-97-3] **M 161.4, b 125-125.6°/atm, 126°/760mm, d 0.7747, n 1.407.** Possible impurity is Me₃SiCl. Wash well with pet ether and fractionate through a vacuum jacketed column packed with Helipac using a reflux ratio of 10:1. [*J Org Chem* **23** 50 1958.]

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. [*J Am Chem Soc* **76** 2672 1954; *J Gen Chem USSR (Engl ed)* **25** 469 1955.]

Hexamethylditin (hexamethyldistannane) [661-69-8] **M 327.6, m 23.5°, b 85-88°/45mm, 182°/756mm, d²⁵ 1.57.** Wash with H₂O and extract with *C₆H₆, dry by filtering through powdered Na₂SO₄, remove *C₆H₆ 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.* [*J Am Chem Soc* **47** 2361 1925, **63** 2509 1941; *Trans Faraday Soc* **53** 1612 1957.]

Hexamethylphosphoric triamide (HMPA) [680-31-9] **M 179.2, f 7.2°, b 68-70°/1mm, 235°/760mm, d 1.024, n 1.460.** The industrial synthesis is usually by treatment of POCl₃ 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 CO₂. Can also be stored over 4A molecular sieves.

Alternatively, it is distd under vacuum from CaH₂ 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 *Pure Appl Chem* **44** 117 1975]. For tests of purity see Fujinaga et al. in *Purification of Solvents*, Coetzee Ed., Pergamon Press, Oxford, 1982. For efficiency of desiccants in drying HMPT see Burfield and Smithers [*J Org Chem* **43** 3966 1978; Sammes et al. *J Chem Soc, Faraday Trans 1* 281 1986]. **CARCINOGEN.**

Hexamethylphosphorous triamide (HMPT) [1608-26-0] **M 163.2, m 7.2°, b 49-51°/12mm, 162-164°/12mm, d 0.989, n 1.466.** It may contain more than 1% of phosphoric triamide. The yellow oil is first distd at atm press then under reduced press and stored under N₂. It is air sensitive, **TOXIC**, should not be inhaled and is absorbed through the skin. [Mark *Org Synth Coll Vol V* 602 1973.]

Hexamminecobalt(III) chloride [10534-89-1] **M 267.5.** Crystd from warm water (8mL/g) by cooling. [Bjerrum and McReynolds *Inorg Synth* **2** 217 1946.]

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⁻¹ in the IR. [*Z Anorg Allg Chem* **251** 96 1963; *J Am Chem Soc* **85** 1202 1963; *Tetrahedron Lett* **22** 1783 1981.]

Hydrazine (anhydrous) [302-01-2] **M 32.1, f 1.5-2.0°, b 47°/26mm, 56°/71mm, 113-113.5°/atm, n 1.470, d 1.91, pK₁²⁵ -0.88, pK₂²⁵ 8.11.** Hydrazine hydrate is dried by refluxing with an equal weight of KOH pellets for 3h, then distilled from fresh NaOH or BaO in a current of dry N₂.

Hydrazine dihydrochloride [5341-61-7] **M 105.0, m 198°, d 1.42.** Crystd from aqueous EtOH and dried under vacuum over CaSO₄.

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. *J Am Chem Soc* **107** 7360 1985.]

Hydriodic acid [10034-85-2] **M 127.9, b 127°(aq azeotrope), d 1.701, pK²⁵ -8.56.** 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 CCl₄, toluene or pet ether (10mL per 100mL of acid). [Davidson and Jameson *Chem Ind (London)* 1686 1963.] Extraction with tributyl phosphate in CHCl₃ or other organic solvents is also suitable. Alternatively, a De-acidite FF anion-exchange resin column in the OH⁻-form using 2M NaOH, then into its I⁻-form by passing dilute KI soln, can be used. Passage of an HI solution under CO₂ through such a column removes polyiodide. The column can be regenerated with NaOH. [Irving and Wilson *Chem Ind (London)* 653 1964]. The earlier method was to reflux with red phosphorus and distil in a stream of N₂. The colourless product was stored in ampoules in the dark [Bradbury *J Am Chem Soc* **74** 2709 1952; *Inorg Synth* **1** 157 1939]. Fumes in moist air. **HARMFUL VAPOURS.**

Hydrobromic acid [10035-10-6] **M 80.9, b 125°(aq azeotrope, 47.5% HBr), d 1.38 (34% HBr), pK²⁵ -8.69.** 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 *J Phys Chem* **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 H₂S, distilling and collecting the fraction boiling at 125-127°. [*Inorg Synth* **1** 155 1939.] **HARMFUL VAPOURS.**

Hydrochloric acid [7647-01-0] **M 36.5, b 108.6°(aq azeotrope, 20.2% HCl), d 1.09(20%), pK²⁵ -6.1.** Readily purified by fractional distillation as constant boiling point acid, following dilution with H₂O. 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 *J Am Chem Soc* **45** 1220 1923..] **HARMFUL VAPOURS.**

Hydrofluoric acid [7664-39-3] **M 20.0, b 112.2°(aq azeotrope, 38.2% HF), d 1.15 (47-53% HF), pK²⁵ 3.21.** Freed from lead (Pb ca 0.002ppm) by co-precipitation with SrF₂, by addition of 10mL of 10% SrCl₂ soln per kilogram of the conc acid. After the ppte has settled, the supernatant is decanted through a filter in a hard-rubber or paraffin lined-glass vessel [Rosenqvist *Am 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 [1333-74-0] **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 MnO₂ and CuO (Hopcalite) oxidises any CO to CO₂ (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-N₂ trap

packed with glass wool, through CaCl_2 tubes, or through $\text{Mg}(\text{ClO}_4)_2$ or P_2O_5 . Other purification steps include passage through a hot palladium thimble [Masson *J Am Chem Soc* **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 VERY EXPLOSIVE in air.*

Hydrogen bromide (anhydrous) [10035-10-6] **M 80.9**. Dried by passage through $\text{Mg}(\text{ClO}_4)_2$ 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 $-195^\circ/10^{-5}\text{mm}$. Fumes in moist air. **HARMFUL VAPOURS**.

Hydrogen chloride [7647-01-0] **M 36.5**. Passed through conc H_2SO_4 , 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. **HARMFUL VAPOURS**.

Hydrogen cyanide (anhydrous) [74-90-8] **M 27.0, b 25.7°, pK²⁵ 9.21 (aq acid)**. Prepared from NaCN and H_2SO_4 , and dried by passage through H_2SO_4 and over CaCl_2 , then distilled in a vacuum system and degassed at 77°K before use [Arnold and Smith *J Chem Soc, Faraday Trans 2* **77** 861 1981]. Cylinder HCN may contain stabilisers against explosive polymerisation, together with small amounts of H_3PO_4 , H_2SO_4 , SO_2 , and water. It can be purified by distn over P_2O_5 , then frozen in Pyrex bottles at Dry-ice temperature for storage. **EXTREMELY 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_2 at room temperature and further distn. Alternatively, it can be absorbed on NaF to form NaHF_2 which is then heated under vacuum at 150° to remove volatile impurities. The HF is regenerated by heating at 300° and stored with CoF_3 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 *J Am Chem Soc* **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, the solution is frozen, then covered with P_2O_5 and allowed to melt under vacuum. The gas evolved is dried by passage through P_2O_5 on glass wool. It can be freed from iodine contamination by repeated fractional distillation at low temperatures. Fumes in moist air. **HARMFUL VAPOURS**.

Hydrogen ionophore II (ETH 1907) (4-nonadecylpyridine - Proton ionophore) [70268-36-9] **M 345.6, b 180°/0.07mm, pK_{Est}~ 6.0**. Dissolve the waxy solid (*ca* 60g) in CHCl_3 (200mL), wash with H_2O (3 x 200mL), dry and evaporate to dryness then distil in vacuum. A waxy solid is formed on cooling the distillate. UV: 257nm (ϵ $1.86 \times 10^3 \text{ M}^{-1}\text{cm}^{-1}$), 308nm (ϵ $1.7 \times 10^2 \text{ M}^{-1}\text{cm}^{-1}$). [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°, pK_{Est}~ 10**. It can be distd at high vacuum; but dissolving in $^*\text{C}_6\text{H}_6$, filtering and evaporating gives a waxy solid suitable for electrode use. It recrystallises from Me_2CO . [*Chem Ber* **69** 60 1936; *Talanta* **34** 435 1987.]

Hydrogen ionophore IV ETH 1778 (octadecyl isonicotinate) [103225-02-1] **M 375.6, m 57.5°, pK_{Est}~ 3.5**. Dissolve in Et_2O and wash 3 times with H_2O . Dry, evaporate, and recrystallise the residue from EtOAc /hexane (4:1). [*Anal Chem* **58** 2285 1986.]

Hydrogen peroxide [7722-84-1] **M 34.0, d 1.110, pK²⁵ 11.65**. The 30% material has been steam distilled using distilled water. Gross and Taylor [*J Am Chem Soc* **72** 2075 1950] made 90% H_2O_2 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 sulfide [7783-06-4] **M 34.1, b -59.6°, pK₁²⁵ 7.05, pK₂²⁵ 12.89.** Washed, then passed through a train of flasks containing saturated Ba(OH)₂ (2x), water (2x), and dilute HCl [Goates et al. *J Am Chem Soc* 73 707 1951]. **HIGHLY POISONOUS.**

Hydroquinone-2-sulfonic acid K salt [21799-87-1] **M 228.3, m 250°(dec), pK_{Est(1)}~1, pK_{Est(2)}~8.5, pK_{Est(3)}~11.** Recrystd from water.

Hydroxylamine [7803-49-8] **M 33.0, m 33.1°, b 56.5°/22mm, d 1.226, pK²⁰ 5.96.** Crystd from *n*-butanol at -10°, collected by vacuum filtration and washed with cold diethyl ether. **Harmful vapours.**

Hydroxylamine hydrochloride [5470-11-1] **M 69.5, m 151°.** Crystallised from aqueous 75% ethanol or boiling methanol, and dried under vacuum over CaSO₄ or P₂O₅. Has also been dissolved in a minimum of water and saturated with HCl; after three such crystns it was dried under vacuum over CaCl₂ and NaOH.

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

Hydroxylamine-O-sulfonic acid [2950-43-8] **M 113.1, m 210-211°, 215°(dec), pK⁴⁵ 1.48.** Stir the solid vigorously with anhydrous Et₂O and filter off using large volumes of dry Et₂O. Drain dry at the pump for 5min and then for 12-14h in a vacuum. Store in a vacuum desiccator/conc H₂SO₄. Determine the purity by oxidation of iodide to I₂. Must be stored in a dry atmosphere at 0-4°. It decompose slowly in H₂O at 25° and more rapidly above this temperature. [*Inorg Synth* 5 122 1957.]

Hydroxynaphthol Blue tri-Na salt [63451-35-4] **M 620.5, m dec on heating, pK_{Est} <0.** 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 ppted 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.** See 2-nitrophenol-4-arsonic acid on p. 446.

Hydroxyurea [127-07-1] **M 76.1, m 70-72° (unstable form), m 133-136°, 141° (stable form), pK 10.6.** 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 Coll Vol V* 645 1973.] It is very soluble in H₂O and can be crystd from Et₂O. [*Acta Chem Scand* 10 256 1956.]

Hypophosphorous acid (Phosphinic acid) [6303-21-5] **M 66.0, m 26.5°, d₄³⁰ 1.217, 1.13 and 1.04 for 50, 30-32, and 10% aq solns resp, pK²⁵ 1.31 (H₃PO₂).** Phosphorous acid is a common contaminant of commercial 50% hypophosphorous acid. Jenkins and Jones [*J Am Chem Soc* 74 1353 1952] purified this material by evaporating about 600mL in a 1L flask at 40°, under reduced pressure (in N₂), 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(ClO₄)₂ 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 H₂O and EtOH. The NaH₂PO₂ salt can be purified through an anion exchange resin [*Z Anorg Allg Chem* 260 267 1949.]

Indigocarmine (2[1,3-dihydro-3-oxo-5-sulfo-2H-indol-2-ylidene]-2,3-dihydro-3-oxo-1H-indole-5-sulfonic acid di-Na salt) [860-22-0] **M 466.4, pK₁²⁰ 2.8, pK₂²⁰ 12.3.** Its

solubility in H₂O is 1g/100mL at 25°. Could be purified by dissolving in H₂O, filtering and adding EtOH to cause the salt to separate. Wash the solid with EtOH, Et₂O and dry *in vacuo*. [Vörländer and Schubert *Chem Ber* **34** 1860 1901; UV: Smit et al. *Anal Chem* **27** 1159 1955; Preisler et al. *J Am Chem Soc* **81** 1991 1959.]

Indium [7440-74-6] **M 114.8, m 156.6°, b 2000°, d 7.31**. Before use, the metal surface can be cleaned with dilute HNO₃, 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, pK₁²⁵ 3.54, pK₂²⁵ 4.28, pK₃²⁵ 5.16 (for aquo In³⁺)**. The anhydrous salt forms yellow deliquescent crystals which can be sublimed at 600° in the presence of Cl₂/N₂ (1:1) {does not melt}. It is resublimed in the presence of Cl₂/N₂ (1:10) and finally heated to 150° to expel excess Cl₂. It is soluble in H₂O and should be stored in a tightly closed container. [*J Am Chem Soc* **55** 1943 1933.]

Indium (III) oxide [1312-43-2] **M 277.6, d 7.18, m sublimes at 850°**. Wash with H₂O 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 sulfate [13464-82-9] **M 517.8**. Crystd from dilute aqueous H₂SO₄.

Indium (III) sulfate (5H₂O) [17069-79-3] **M 607.9, d 3.44**. Dissolve in strong H₂SO₄ 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 H₂O is 5%. The pentahydrate is converted to an anhydrous *hygroscopic* powder on heating at 500° for 6h; but heating above this temperature over N₂ yields the oxide sulfate. Evaporation of neutral aqueous solutions provides basic sulfates. [*J Am Chem Soc* **55** 1943 1933, **58** 2126 1936.]

Iodic acid [7782-68-5] **M 175.9, m 118°(dec), d 4.628, pK²⁵ 0.79**. Dissolve in the minimum volume of hot dilute HNO₃, filter and evaporate in a vacuum desiccator until crystals are formed. Collect crystals and wash with a little cold H₂O and dry in air in the dark. Soluble in H₂O: 269g/100mL at 20° and 295g/100mL at 40°. Soluble in dilute EtOH and darkens on exposure to light. It is converted to HIO₃.I₂O₅ on heating at 70°, but at 220° complete conversion to HIO₃ occurs. [*J Am Chem Soc* **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 H₂O to remove dissolved salts, then drying; and crystn from *benzene. Barrer and Wasilewski [*Trans Faraday Soc* **57** 1140 1961] dissolved I₂ in conc KI and distilled it, then steam distilled three times, washing with distilled H₂O. Organic material was removed by sublimation in a current of O₂ over platinum at about 700°, the iodine being finally sublimed under vacuum. **HARMFUL VAPOURS.**

Iodine monobromide [7789-33-5] **M 206.8, m 42°**. Purified by repeated fractional crystallisation from its melt.

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. [*J Am Chem Soc* **76** 4843 1954] removed dissolved iodine from IF₅ by agitating with a mixture of dry air and ClF₃ 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 ClF₃ before IF₅ distilled. Stevens [*J Org Chem* **26** 3451 1961] pumped IF₅ under vacuum from its cylinder, trapping it at -78°, then allowing it to melt in a stream of dry N₂. **HARMFUL VAPOURS.**

Iodine trichloride [865-44-1] **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_D²⁵ 1.4917.** If slightly violet in colour wash with aqueous 1% sodium metabisulfite, H₂O, dry over Na₂SO₄ and fractionally distil at atmospheric pressure. [*J Am Chem Soc* **68** 481 1946.]

Iodotrimethylsilane (trimethylsilyl iodide, TMSI) [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. [*J Chem Soc* 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 on the surface in air. Scrape the outer tarnished layer until silver clear and store under paraffin. Stable to acids but dissolves in aqua regia. [*Chem Rev* **32** 277 1943.]

Iridium (IV) chloride hydrate (hexachloroiridic acid) [16941-92-7 (6H₂O); 207399-11-9 (xH₂O)] **M 334.0+H₂O.** If it contains nitrogen then repeatedly concentrate a conc HCl solution until free from nitrogen, and dry free from HCl in a vac over CaO until crystals are formed. The solid is very *hygroscopic*. [*J Am Chem Soc* **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 Et₂O 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. [*J Am Chem Soc* **72** 1107 1950; *Chem Ber* **60** 1424 1927.]

Iron (III) meso-5,10,15,20-tetraphenylporphine chloride complex [16456-81-8] **M 704.0.** Crystallise by extraction from a thimble (Soxhlet) with CHCl₃. 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 CHCl₃-MeOH. Avoid prolonged heating. It is quite soluble in organic solvents but insoluble in pet ether. [*J Am Chem Soc* **70** 1808 1948; UV: **73** 4315 1951.]

Iron pentacarbonyl (pentacarbonyl iron) [13463-40-6] **M 195.9, m -20°, b 102.8°/749mm, 103°/760mm, n 1.520, d 1.490.** It is a pale yellow viscous liq which is PYROPHORIC and readily absorbed by the skin. **HIGHLY TOXIC (protect from light and air).** It should be purified in a vacuum line by distilling and collecting in a trap at -96° (toluene-Dry ice slush). It has been distd at atm pressure (use a very efficient fume cupboard). At 180°/atm it decomp to give Fe and CO. In UV light in pet ether it forms Fe₂(CO)₉. [Hagen et al. *Inorg Chem* **17** 1369 1978; Ewens et al. *Trans Faraday Soc* **35** 6811 1939.]

Isopropyldimethyl chlorosilane [3634-56-8] **M 140.7, b 109.8-110.0°/738mm, d 0.88, n 1.4158.** Probable impurity is Me₃SiCl (b 56.9°/783mm) which can be removed by efficient fractional distillation. [*J Am Chem Soc* **76** 801 1954.]

(2,3-O-Isopropylidene)-2,3-dihydroxy-1,4-bis-(diphenylphosphino)butane (DIOP) [4R,5S(-)-32305-98-9; 4S,5R(+)-37002-48-5] **M 498.5, m 88-90°, [α]_D¹⁹ (-) and (+) 26° (c 2.3, CHCl₃), pK_{Est} ~ 4.5.** It has been recrystd from *C₆H₆-pet ether. After 2 recrystns from EtOH it was pure by TLC on silica gel using Me₂CO-hexane as solvent. [Kagan and Dang *J Am Chem Soc* **94** 6429 1972.]

Lanthanide shift reagents see p. 277 in Chapter 4.

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 H₂O 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, pK₁²⁵ 9.06 (for aquo La³⁺)**. Boil with redistilled Ac₂O for 10min (does not dissolve and is a white solid). Cool, filter, wash with Ac₂O and keep in a vacuum desiccator (NaOH) till free from solvent. [*J Indian Chem Soc* **33** 877 1956.]

***N*-Lauroyl-*N*-methyltaurine sodium salt (sodium *N*-decanoyl-*N*-methyl-2-aminoethane sulfonate)** [4337-75-1] **M 343.5, pK_{Est} ~1.5**. Prepared from methyldecanoate (at 180° under N₂) or decanoyl chloride and sodium *N*-methyltaurine sulfonate and purified by dissolving in H₂O and precipitating by addition of Et₂O. Decomposes on heating. [*Desseigne and Mathian Mém Services Chim Etat Paris* **31** 359 1944, *Chem Abstr* **41** 705 1947.]

Lead II acetate [301-04-2 (anhydr); 6080-56-4 (3H₂O)] **M 325.3, m 280°, pK₁²⁵ 7.1 (for Pb²⁺), pK₂²⁵ 10.1 (HPbO₂⁻), pK₃²⁵ 10.8 (PbO₂²⁻)**. Crystallised twice from anhydrous acetic acid and dried under vacuum for 24h at 100°.

Lead (bis-cyclopentadienyl) [1294-74-2] **M 337.4**. Purified by vacuum sublimation. Handled and stored under N₂.

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 PbBr₂) 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 H₂. Finally fused [Clayton et al. *J Chem Soc, Faraday Trans 1* **76** 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 crystals the solid was dried under vacuum or under anhydrous HCl vapour by heating slowly to 400°.

Lead diethyldithiocarbamate [17549-30-3] **M 503.7, pK₁²⁵ 3.36 (for *N,N*-diethyldithiocarbamate)**. Wash with H₂O and dry at 60-70°, or dissolve in the min vol of CHCl₃ and add the same vol of EtOH. Collect the solid that separates and dry as before. Alternatively, recryst by slow evaporation of a CHCl₃ soln at 70-80°. Filter the crystals, wash with H₂O until all Pb²⁺ ions are eluted (check by adding chromate) and then dry at 60-70° for at least 10h. [*Justus Liebigs Ann Chem* **49** 1146 1977.]

Lead (II) formate [811-54-4] **M 297.3, m 190°**. 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 *J Am Chem Soc* **78** 5994 1956.]

Lead nitrate [10099-74-8] **M 331.2, m 470°**. Pptd twice from hot (60°) conc aqueous soln by adding HNO₃. 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 *Trans Faraday Soc* **55** 331 1959]. After 2 recrystns of ACS grade no metals above 0.001 ppm were detected.

Lead tetraacetate [546-67-8] **M 443.4, m 175-180°, pK₁ 1.8, pK₂ 3.2, pK₃ 5.2, pK₄ 6.7**. 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 P₂O₅ and KOH for 24h before use.

Lissamine Green B {1-[bis-(4,4'-dimethylaminophenyl)methyl]-2-hydroxynaphthalene-3,6-disulfonic acid sodium salt, Acid Green 50} [3087-16-9] **M 576.6, m >200°(dec), CI 44090, λ_{max} 633nm**. Crystd from EtOH/water (1:1, v/v).

Lissapol C (mainly sodium salt of 9-octadecene-1- sulfate) [2425-51-6]. 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 ppt was filtered off, washed in acetone and dried under vacuum. [Biswas and Mukerji *J Phys Chem* **64** 1 1960].

Lissapol LS (mainly sodium salt of anisidine sulfate) [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 ppt was filtered off, washed in acetone and dried under vacuum. [Biswas and Mukerji *J Phys Chem* **64** 1 1960].

Lithium (metal) [7439-93-2] **M 6.9, m 180.5°, b 1342°, d 0.534**. 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 distd at 600° using an all-iron distn apparatus [Gunn and Green *J Am Chem Soc* **80** 4782 1958].

Lithium acetate (2H₂O) [546-89-4] **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 Et₂O, and, after filtering, the solvent was removed under vacuum. The residue was dried at 60° for 3h, under high vacuum [Ruff *J Am Chem Soc* **83** 1788 1961]. **IGNITES in the presence of a small amount of water and reacts EXPLOSIVELY.**

Lithium amide [7782-89-0] **M 23.0, m 380-400°, d^{17.5} 1.178**. Purified by heating at 400° while NH₃ is passed over it in the upper of two crucibles (the upper crucible is perforated). The LiNH₂ will drip into the lower crucible through the holes in the upper crucible. The product is cooled in a stream of NH₃. Protect it from air and moisture, store under N₂ 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 Li₂NH 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**. from EtOH (13mL/g) by partial evaporation.

Lithium borohydride [16949-15-8] **M 21.8, mCrystd 268°, b 380°(dec), d 0.66**. Crystd from Et₂O, and pumped free of ether at 90-100° during 2h [Schaeffer, Roscoe and Stewart *J Am Chem Soc* **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 552°, 618°**. Crystd from water. Its solubility decreases as the temperature is raised.

Lithium chloride [7447-41-8] **M 42.4, m 600°, 723°**. 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 N₂ and pulverised in a dry-box. Kolthoff and Bruckenstein [*J Am Chem Soc* **74** 2529 1952] pptd with ammonium carbonate, washed with Li₂CO₃ 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 CaCl₂.

Lithium diisopropylamide [4111-54-0] **M 107.1, b 82-84°/atm, 84°/atm, d²² 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_2 . 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 dodecylsulfate [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 $LiCO_3$, H_2O 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_2O is 0.27% at 18°. It volatilises between 1100-1200°. [*Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 235 1963].

Lithium formate (H_2O) [6108-23-2 (H_2O); 556-63-8 (anhydr)] M 70.0, d 1.46. 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_2O to give H_2 and LiOH, and reacts with lower alcohols. One gram in H_2O liberates 2.8L of H_2 (Could be explosive).

Lithium hydroxide (H_2O) [1310-66-3 (H_2O); 1310-65-2 (anhydr)] M 42.0, m 471°, d 1.51, pK^{25} 13.82. Crystd from hot water (3mL/g) as the monohydrate. Dehydrated at 150° in a stream of CO_2 -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, m 469°, b 1171°, d 4.06. Crystd from hot water (0.5mL/g) by cooling in $CaCl_2$ -ice, or from acetone. Dried under vacuum over P_2O_5 for 1h at 60° and then at 120°.

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_2 using an oil pump for a few hours. Store under N_2 in the cold. It should not have bands above $3000cm^{-1}$; IR has ν_{KBr} 1078, 2790, 2840 and $2930cm^{-1}$. [*J Org Chem* 21 156 1956.]

Lithium nitrate [7790-69-4] M 68.9, m 253°, d 2.38. 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 vac desiccator over $CaSO_4$. After 99% salt was recrystd 3 times it contained: metal (ppm) Ca (1.6), K (1.1), Mo (0.4), Na (2.2).

Lithium nitrite (H_2O) [13568-33-7] M 71.0. Crystd from water by cooling from room temperature.

Lithium picrate [18390-55-1] M 221.0. Recrystd three times from EtOH and dried under vacuum at 45° for 48h [D'Aprano and Sesta *J Phys Chem* 91 2415 1987]. The necessary precautions should be taken in case of **EXPLOSION**.

Lithium perchlorate [7791-03-9] M 106.4, pK^{25} -2.4 to -3.1 (for $HClO_4$). Crystd from water or 50% aq MeOH. Rendered anhydrous by heating the trihydrate at 170-180° in an air oven. It can then be recrystd twice from acetonitrile and again dried under vacuum [Mohammad and Kosower *J Am Chem Soc* 93 2713 1971].

Lithium salicylate [552-38-5] M 144.1. Crystd from EtOH (2mL/g) by partial evaporation.

Lithium sulfate (anhydrous) [10377-48-7] M 109.9, loses H_2O at 130° and m 859°, d 2.21. Crystd from H_2O (4mL/g) by partial evaporation.

Lithium tetrafluoroborate [14283-07-9] M 93.7, pK^{25} 13.82 (Li^+), pK^{25} -4.9 (for HBF_4). Dissolve in THF just below its solubility, filter from insol material and evap to dryness in a vacuum below

50°. Wash the residue with dry Et₂O, and pass dry N₂ gas over the solid and finally heat in an oven at 80-90°. Solubility in Et₂O: 1.9 (1.3)g in 100mL at 25°, in THF: 71g in 100mL at 25°. It is *hygroscopic* and is an **IRRITANT**. [*J Am Chem Soc* 74 5211 1952, 75 1753 1953.]

Lithium thiocyanate (lithium rhodanide) [556-65-0] M 65.0, pK²⁵ -1.85 (for HSCN). It crystallises from H₂O as the dihydrate but on drying at 38-42° it gives the monohydrate. It can be purified by allowing an aqueous soln to crystallise in a vac over P₂O₅. The crystals are collected, dried out in vacuum at 80°/P₂O₅ in a stream of pure N₂ at 110°. [*J Chem Soc* 1245 1936.]

Lithium trimethylsilanolate (trimethylsilanol Li salt) [2004-14-0] M 96.1, m 120°(dec in air). Wash with Et₂O and pet ether. Sublimes at 180°/1mm as fine transparent needles. [*J Org Chem* 17 1555 1952.]

Magnesium [7439-95-4] M 24.3, m 651°, b 1100°, d 1.739. Slowly oxidises in moist air and tarnishes. If dark in colour do not use. Shiny solid should be degreased by washing with dry Et₂O, dry and keep in a N₂ atmosphere. It can be activated by adding a crystal of I₂ in the Et₂O before drying and storing.

Magnesium acetate [142-72-3 (anhydr); 16674-78-5 (4H₂O)] M 214.5, m 80°. Crystd from anhydrous acetic acid, then dried under vacuum for 24h at 100°

Magnesium benzoate (3H₂O) [553-70-8] M 320.6, m ~200°. Crystd from water (6mL/g) between 100° and 0°.

Magnesium bromide (anhydrous) [7789-48-2] M 184.1, m 711°, d 3.72. Crystd from EtOH.

Magnesium chloride (6H₂O) [7791-18-6] M 203.3, m ~100°(dec), pK₁²⁵ 10.3, pK₂²⁵ 12.2 (for Mg²⁺ hydrolysis). Crystd from hot water (0.3mL/g) by cooling.

Magnesium dodecylsulfate [3097-08-3] M 555.1. Recrystd three times from EtOH and dried in a vacuum.

Magnesium ethylate (magnesium ethoxide) [2414-98-4] M 114.4. Dissolve *ca* 1g of solid in 12.8mL of absolute EtOH and 20mL of dry xylene and reflux in a dry atmosphere (use CaCl₂ in a drying tube at the top of the condenser). Add 10mL of absolute EtOH and cool. Filter solid under dry N₂ and dry in a vacuum. Alternatively dissolve in absolute EtOH and pass through molecular sieves (40 mesh) under N₂, evap under N₂, and store in a tightly stoppered container. [*J Am Chem Soc* 68 889 1964.]

Magnesium D-gluconate [3632-91-5] M 414.6, [α]₅₄₆²⁰ +13.5°, [α]_D²⁰ +11.3° (c 1, H₂O). Cryst from dilute EtOH to give *ca* trihydrate, and then dry at 98° in high vacuum. Insol in EtOH and solubility in H₂O is 16% at 25°.

Magnesium iodate (4H₂O) [7790-32-1] M 446.2. Crystd from water (5mL/g) between 100° and 0°.

Magnesium iodide [10377-58-9] M 278.1, m 634°. Crystd from water (1.2mL/g) by partial evapn in a desiccator.

Magnesium ionophore I (ETH 1117), (N,N'-diheptyl-N,N'-dimethyl-1,4-butanediamide) [75513-72-3] M 340.6. Purified by flash chromatography (at 40 kPa) on silica and eluting with EtOH-hexane (4:1). IR has ν(CHCl₃) 1630cm⁻¹. [*Helv Chim Acta* 63 2271 1980.] It is a good magnesium selectophore compared with Na, K and Ca [*Anal Chem* 52 2400 1980].

Magnesium ionophore II (ETH 5214), [*N,N''*-octamethylene-bis(*N'*-heptyl-*N''*-methyl methylmalonamide)] [119110-37-1] **M 538.8**. Reagent (*ca* 700mg) can be purified by flash chromatography on Silica Gel 60 (30g) and eluting with CH₂Cl₂-Me₂CO (4:1). [*Anal Chem* **61** 574 1989.]

Magnesium lactate [18917-37-1] **M 113.4**. Crystd from water (6mL/g) between 100° to 0°.

Magnesium nitrate (6H₂O) [13446-18-9] **M 256.4, m ~95°(dec)**. Crystd from water (2.5mL/g) by partial evapn in a desiccator. After 2 recrystns ACS grade has: metal (ppm) Ca (6.2), Fe (8.4), K (2), Mo (0.6), Na (0.8), Se (0.02).

Magnesium perchlorate (Anhydron, Dehydrite) [10034-81-8 (*anhydr*)] **M 259.2, m >250°, pK²⁵ -2.4 to -3.1 (for HClO₄)**. Crystd from water to give the *hexahydrate* **M 331.3 [13346-19-0]**. Coll, Nauman and West [*J Am Chem Soc* **81** 1284 1959] removed traces of unspecified contaminants by washing with small portions of Et₂O and drying in a vac (**CARE**). The anhydrous salt is commercially available as an ACS reagent, and is as efficient a dehydrating agent as P₂O₅ and is known as "Dehydrite" or "Anhydron". [Smith et al. *J Am Chem Soc* **44** 2255 1922 and *Ind Eng Chem* **16** 20 1924.] Hygroscopic, Keep in a tightly closed container. **EXPLOSIVE in contact with organic materials, and is a SKIN IRRITANT.**

Magnesium succinate [556-32-1] **M 141.4**. Crystd from water (0.5mL/g) between 100° and 0°.

Magnesium sulfate (anhydrous) [7487-88-9] **M 120.4, m 1127°**. Crystd from warm H₂O (1mL/g) by cooling. Dry heptahydrate at ~250° until it loses 25% of its wt. Store in a sealed container.

Magnesium trifluoromethanesulfonate [60871-83-2] **M 322.4, m >300°**. Wash with CH₂Cl₂ and dry at 125°/2h and 3mmHg. [*Tetrahedron Lett* **24** 169 1983.]

Magon [3-hydroxy-4-(hydroxyphenylazo)-2-naphthoyl-2,4-dimethylanilide; Xylidyl Blue II] [523-67-1] **M 411.5, m 246-247°**. Suspend in H₂O and add aqueous NaOH until it dissolves, filter and acidify with dil HCl. Collect the dye, dissolve in hot EtOH (sol is 100mg/L at *ca* 25°) concentrate to a small volume and allow to cool. Sol in H₂O of the Na salt is 0.4mg/mL. [*Anal Chim Acta* **16** 155 1957; *Anal Chem* **28** 202 1956.]

Manganese (III) acetate (2H₂O) [19513-05-4] **M 268.1, pK²⁵ 0.06 (for Mn³⁺ hydrolysis)**. Wash the acetate with AcOH then thoroughly with Et₂O and dry in air to obtain the dihydrate. The *anhydrous* salt can be made by stirring vigorously a mixt of the hydrated acetate (*ca* 6g) and Ac₂O (22.5mL) and heat carefully (if necessary) until the mixture is clear. It is set aside overnight for the material to crystallise. Filter the solid, wash with Ac₂O and dry over P₂O₅. The dihydrate can also be obtained from the *di-* and *tetra-* hydrate mixture of the divalent acetate by adding 500mL of Ac₂O and 48g of the hydrated acetate and refluxing for 20min, then add slowly 8.0g of KMnO₄. After refluxing for an additional 30min, the mixture was cooled to room temperature and 85mL of H₂O added. It should be noted that larger amounts of H₂O change the yield and nature of the manganese acetate and the yields of reactions that use this reagent, e.g. formation of lactones from olefines. The Mn(OAc)₃·2H₂O is then filtered off after 16h, washed with cold AcOH and air dried. [*J Am Chem Soc* **90** 5903, 5905 1968, Heiba et al. **91** 138 1969.]

Alternatively dissolve the salt (30g) in glacial acetic acid (200mL) by heating and filter. If crystals do not appear, the glass container should be rubbed with a glass rod to induce crystn which occurs within 1h. If not, allow to stand for a few days. Filter the cinnamon brown crystals which have a silky lustre and dry over CaO. Keep away from moisture as it is decomposed by cold H₂O. [Lux in *Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol II, p 1469 1963; Williams and Hunter *Can J Chem* **54** 3830 1976.]

Manganese (II) acetylacetonate [14024-58-9] **M 253.2, m ~250°**. Purify by stirring 16g of reagent for a few min with 100mL absolute EtOH and filter by suction as rapidly as possible through coarse filter paper. Sufficient EtOH is added to the filtrate to make up for the loss of EtOH and to redissolve any solid that separates. Water (15mL) is added to the filtrate and the solution is evaporated with a stream of N₂ until reduced to half its vol. Cool for a few min and filter off the yellow crystals, dry under a stream of N₂, then in a

vacuum at room temp for 6-8h. These conditions are important for obtaining the *dihydrate*. A vacuum to several mm of Hg or much lower pressure for several days produces the anhydrous complex. The degree of hydration can be established by determining the loss in weight of 100g of sample after heating for 4h at 100° and <20mmHg. The theoretical loss in weight for 2H₂O is 12.5%. Material sublimates at 200°/2mm. It is soluble in heptane, MeOH, EtOH or *C₆H₆ at 30°. [*Inorg Synth* 6 164 1960, 5 105 1957.]

Manganese decacarbonyl Mn₂(CO)₁₀ [10170-69-1] M 390.0, m 151-152°, 154-155°(sealed tube), d²⁵ 1.75. Golden yellow crystals which in the absence of CO begin to decompose at 110°, and on further heating yield a metallic mirror. In the presence of 3000psi of CO it does not decompose on heating to 250°. It is soluble in common organic solvents, insoluble in H₂O, not very stable in air, to heat or UV light. Dissolves in a lot of *C₆H₆ and can be crystallised from it. It distils with steam at 92-100°. It can be purified by sublimation under reduced pressure (<0.5mm) at room temperature to give well formed golden yellow crystals. If the sample is orange coloured this sublimation leads to a mixture of golden-yellow and dark red crystals of the carbonyl and carbonyl iodide respectively which can be separated by hand picking under a microscope. Separate resublimations provide the pure compounds. **POISONOUS** [*J Am Chem Soc* 76 3831 1954, 80 6167 1958, 82 1325 1960].

Manganous acetate (4H₂O) [6156-78-1 (4H₂O); 638-38-0 (anhydr)] M 245.1, m 80°, d 1.59, pK²⁵ 10.59 (for Mn²⁺ hydrolysis). Crystd from water acidified with acetic acid.

Manganous bromide (anhydrous) [13446-03-2] M 214.8, m 695°; 4H₂O [10031-20-6] M 286.8, m 64°(dec). Rose-red deliquescent crystals soluble in EtOH. The H₂O is removed by heating at 100° then in HBr gas at 725° or dry in an atmosphere of N₂ at 200°.

Manganous chloride (4H₂O) [13446-34-9; 7773-01-5 (anhydr)] M 197.9, m 58°, 87.5°, d 2.01. Crystd from water (0.3mL/g) by cooling.

Manganous ethylenebis(dithiocarbamate) [12427-38-2] M 265.3, pK_{Est} ~ 3.0 (for —NCSSH). Crystd from EtOH.

Manganous lactate (3H₂O) [51877-53-3] M 287.1. Crystd from water.

Manganous sulfate (H₂O) [10034-96-5 (H₂O); 15244-36-7 (xH₂O)] M 169.0, d 2.75. Crystd from water (0.9mL/g) at 54-55° by evaporating about two-thirds of the water. Dehydr at >400°.

2-Mercaptopyridine N-oxide sodium salt (pyridinethione or pyrithione sodium salt) [3811-73-2] M 149.1, m ~250°(dec), pK₁ -1.95, pK₂ 4.65. When recrystd from water it assayed as 98.7% based on AgNO₃ titration [Krivis et al. *Anal Chem* 35 966 1963, see also Krivis et al. *Anal Chem* 48 1001 1976; and Barton and Crich *J Chem Soc. Perkin Trans 1* 1603, 1613 1986].

Mercuric acetate [1600-27-7] M 318.7, pK₁²⁵ 2.47, pK₂²⁵ 3.49 (for Hg²⁺ hydrolysis). Crystd from glacial acetic acid. **POISONOUS**.

Mercuric bromide [7789-47-1] M 360.4, m 238.1°. Crystd from hot saturated ethanolic soln, dried and kept at 100° for several hours under vacuum, then sublimed. **POISONOUS**.

Mercuric chloride [7487-94-7] M 271.5, m 276°, b 304°, d 5.6. Crystd twice from distilled water, dried at 70° and sublimed under high vacuum. **POISONOUS**.

Mercuric cyanide [592-04-1] M 252.6, m 320°(dec), d 4.00. Crystd from water. **POISONOUS**.

Mercuric iodide (red) [7774-29-0] M 454.4, m 259°(yellow >130°), b ~350°(subl), d 6.3. Crystd from MeOH or EtOH, and washed repeatedly with distilled water. Has also been mixed thoroughly with excess 0.001M iodine solution, filtered, washed with cold distilled water, rinsed with EtOH and Et₂O, and dried in air. **POISONOUS**.

Mercuric oxide (yellow) [21908-53-2] **M 216.6, m 500°(dec)**. Dissolved in HClO_4 and ppted with NaOH soln.

Mercuric thiocyanate [592-85-8] **M 316.8, m 165°(dec), $\text{pK}^{25} -1.85$ (for HSCN)**. Recryst from H_2O , and can form various crystal forms depending on conditions. Solubility in H_2O is 0.069% at 25°, but is more soluble at higher temps. Decomposes to Hg above 165°. **Poisonous**. [*J Phys Chem* **35** 1128 1931; *Chem Ber* **68** 919 1935.]

Mercurous nitrate ($2\text{H}_2\text{O}$) [7782-86-7 ($2\text{H}_2\text{O}$); 7783-34-8 (H_2O); 10415-75-5 (anhydr)] **M 561.2, m 70°(dec), d 4.78, $\text{pK}^{25} 2.68$ (for Hg_2^{2+} hydrolysis)**. Solubility in H_2O containing 1% HNO_3 is 7.7%. Recrystd from a warm saturated soln of dilute HNO_3 and cool to room temp slowly to give elongated prisms. Rapid cooling gives plates. Colourless crystals to be stored in the dark. **POISONOUS**. [*J Chem Soc* 1312 1956.]

Mercurous sulfate [7783-36-0] **M 497.3, d 7.56**. Recrystallise from dilute H_2SO_4 , and dry in a vacuum under N_2 and store in the dark. Solubility in H_2O is 0.6% at 25°. **POISONOUS**.

Mercury [7439-97-6] **M 200.6, m -38.9°, b 126°/1mm, 184°/10mm, 261°/100mm, 356.9°/atm, d 13.534**. After air had been bubbled through mercury for several hours to oxidise metallic impurities, it was filtered to remove coarser particles of oxide and dirt, then sprayed through a 4-ft column containing 10% HNO_3 . It was washed with distilled water, dried with filter paper and distilled under vacuum.

Mercury(II) bis(cyclopentadienyl) [18263-08-6] **M 330.8**. Purified by low-temp recrystn from Et_2O .

Mercury dibromofluorescein {mercurochrome, merobromin, [2',7'-dibromo-4'-(hydroxy-mercurio)-fluorescein di-Na salt]} [129-16-8] **M 804.8, m >300°**. The Na salt is dissolved in the minimum vol of H_2O , or the free acid suspended in H_2O and dilute NaOH added to cause it to dissolve, filter and acidify with dilute HCl . Collect the ppte wash with H_2O by centrifugation and dry in vacuum. The di Na salt can be purified by dissolving in the minimum volume of H_2O and ppted by adding EtOH , filter, wash with EtOH or Me_2CO and dry in a vacuum. Solubility in 95% EtOH is 2% and in MeOH it is 16%. [*J Am Chem Soc* **42** 2355 1920.]

Mercury orange [1-(4-chloromercuriophenylazo)-2-naphthol] [3076-91-3] **M 483.3, m 291.5-293°(corr) with bleaching**. Wash several times with boiling 50% EtOH and recrystallise from 1-butanol (0.9g/L of boiling alcohol). Fine needles insoluble in H_2O but slightly soluble in cold alcohols, CHCl_3 and soluble in aqueous alkalis. [*J Am Chem Soc* **70** 3522 1948.]

Mercury(II) trifluoroacetate [13257-51-7] **M 426.6**. Recrystd from trifluoroacetic anhydride/trifluoroacetic acid [Lan and Kochi *J Am Chem Soc* **108** 6720 1986]. Very **TOXIC** and *hygroscopic*.

Metanil Yellow (3[{4-phenylamino}phenylazo]-benzenesulfonic acid) [587-98-4] **M 375.4, $\text{pK}_{\text{Est}} < 0$** . Salted out from water three times with sodium acetate, then repeatedly extracted with EtOH [McGrew and Schneider, *J Am Chem Soc* **72** 2547 1950].

(Methoxycarbonylmethyl)triphenylphosphorane [methyl (triphenylphosphoranylidene)-acetate] [2605-67-6] **M 334.4, m 162-163°, 169-171°**. Cryst by dissolving in AcOH and adding pet ether (b 40-50°) to give colorless plates. UV λ_{max} ($A_{1\text{mm}}^{1\%}$): 222nm (865) and 268nm (116) [Isler et al. *Helv Chim Acta* **40** 1242 1957].

Methoxycarbonylmethyltriphenylphosphonium bromide [1779-58-4] **M 415.3, m 163°, 165-170°(dec)**. Wash with pet ether (b 40-50°) and recryst from $\text{CHCl}_3/\text{Et}_2\text{O}$ and dry in high vac at 65°. [Isler et al. *Helv Chim Acta* **40** 1242 1957; Wittig and Haag *Chem Ber* **88** 1654, 1664 1955.]

Methoxymethyl trimethylsilane (trimethylsilylmethyl methyl ether) [14704-14-4] M 118.3, b 83°/740mm, d_4^{25} 0.758, n_D^{25} 1.3878. Forms an azeotrope with MeOH (b 60°). If it contains MeOH (check IR for bands above 3000cm⁻¹) then wash with H₂O and fractionate. A possible impurity could be chloromethyl trimethylsilane (b 97°/740mm). [*J Am Chem Soc* 70 4142 1948.]

1-Methoxy-2-methyl-1-trimethylsiloxypropene (dimethyl ketene methyl trimethylsilyl acetal) [31469-15-5] M 174.3, b 121-122°/0.35mm, 125-126°/0.4mm, 148-150°/atm, d 0.86. Add Et₂O, wash with cold H₂O, dry (Na₂SO₄), filter, evaporate Et₂O, and distil oily residue in a vacuum. [*J Organometal Chem* 46 59 1972.]

trans-1-Methoxy-3-(trimethylsilyloxy)-1,3-butadiene (Danishefsky's diene) [54125-02-9] M 172.3, b 68-69°/14mm, 70-72°/16mm, d 0.885, n 1.4540. It may contain up to 1% of the precursor 4-methoxybut-4-ene-2-one. Easily distd through a Vigreux column in a vac and taking the middle fraction. [Danishefsky and Kitihara *J Am Chem Soc* 96 7807 1974; Danishefsky *Acc Chem Res* 14 400 1981.]

Methylarsonic acid [124-58-3] M 137.9, m 161°, pK₁²⁵ 1.54, pK₂²⁵ 6.31 [As(OH)₂]. Crystd from abs EtOH.

Methyl dichlorosilane (dichloro methylsilane) [75-54-7] M 115.0, m -92.5°, b 41°/748mm, 40.9°/760mm, 40-45°/atm, d_{27}^{27} 1.105. Impurities are generally other chloromethyl silanes. Distilled through a conventional Stedman column of 20 theoretical plates or more. [Stedman column. A plain tube containing a series of wire-gauze discs stamped into flat, truncated cones and welded together, alternatively base-to-base and edge-to-edge, with a flat disc across each base. Each cone has a hole, alternately arranged, near its base, vapour and liquid being brought into intimate contact on the gauze surfaces (Stedman *Can J Res B* 15 383 1937)]. It should be protected from H₂O by storing over P₂O₅. [*Chem Ber* 52 695 1919; *J Am Chem Soc* 68 9621946.]

Methylmercuric chloride [115-09-3] M 251.1, m 167°. Crystd from absolute EtOH (20mL/g).

Methyl Orange (sodium 4,4'-dimethylaminophenylazobenzenesulfonate) [547-58-0] M 327.3, pK 3.47. Crystd twice from hot water, then washed with a little EtOH followed by diethyl ether. Indicator: pH 3.1 (red) and pH 4.4 (yellow).

Methylphenyl dichlorosilane (dichloro methyl phenylsilane) [149-74-6] M 191.1, b 114-115°/50mm, 202-205°/atm, d 1.17. Purified by fractionation using an efficient column. It hydrolyses *ca* ten times more slowly than methyltrichlorosilane and *ca* sixty times more slowly than phenyltrichlorosilane. [*J Phys Chem* 61 1591 1957].

Methylphosphonic acid [993-13-5] M 96.0, m 104-106°, 105-107°, 108°, pK₁²⁵ 2.12, pK₂²⁵ 7.29. If it tests for Cl⁻, add H₂O and evaporate to dryness; repeat several times till free from Cl⁻. The residue solidifies to a wax-like solid. Alternatively, dissolve the acid in the minimum volume of H₂O, add charcoal, warm, filter and evaporate to dryness in a vacuum over P₂O₅. [*J Am Chem Soc* 75 3379 1953.] The *di-Na salt* is prepared from 24g of acid in 50mL of dry EtOH and a solution of 23g Na dissolved in 400mL EtOH is added. A white ppt is formed but the mixture is refluxed for 30min to complete the reaction. Filter off and recrystallise from 50% EtOH. Dry crystals in a vacuum desiccator. [*J Chem Soc* 3292 1952.]

Methylphosphonic dichloride [676-97-1] M 132.9, m 33°, 33-37°, b 53-54°/10mm, 64-67°/20.5mm, 86°/44mm, 162°/760mm, d_4^{40} 1.4382. Fractionally redistd until the purity as checked by hydrolysis and acidimetry for Cl⁻ is correct and should solidify on cooling. [*J Chem Soc* 3437 1952; *J Am Chem Soc* 75 3379 1952; for IR see *Can J Chem* 34 1611 1956.]

Methyl Thymol Blue, sodium salt [1945-77-3] M 844.8, ϵ 1.89 x 10⁴ at 435nm, pH 5.5. Starting material for synthesis is Thymol Blue. Purified as for Xylenol Orange on p. 387 in Chapter 4.

Methyl trichlorosilane [75-79-6] *M* 149.5, *b* 13.7°/101mm, 64.3°/710.8mm, 65.5°/745mm, 66.1°/atm, *d* 1.263, *n* 1.4110. If very pure distil before use. Purity checked by ²⁹Si nmr, δ in MeCN is 13.14 with respect to Me₄Si. Possible contaminants are other silanes which can be removed by fractional distillation through a Stedman column of >72 theoretical plates with total reflux and 0.35% take-off (see p. 441). The apparatus is under N₂ at a rate of 12 bubbles/min fed into the line using an Hg manometer to control the pressure. Sensitive to H₂O. [*J Am Chem Soc* 73 4252 1951; *J Org Chem* 48 3667 1983.]

Methyl triethoxysilane [2031-67-6] *M* 178.31, *b* 142-144.5°/742mm, 141°/765mm, 141.5°/775mm, *d* 0.8911, *n* 1.3820. Repeated fractionation in a stream of N₂ through a 3' Heligrad packed Todd column (see p. 174). Hydrolysed by H₂O and yields cyclic polysiloxanes on hydrolysis in the presence of acid in *C₆H₆. [*J Am Chem Soc* 77 1292, 3990 1955.]

Methyl trimethoxysilane [1185-55-3] *M* 136.2, *b* 102°/760mm, *d* 1.3687, *n* 1.3711. Likely impurities are 1,3-dimethyltetramethoxy disiloxane (*b* 31°/1mm) and cyclic polysiloxanes, see methyl triethoxysilane. [*J Org Chem* 16 1400 1952, 20 250 1955.]

***N*-Methyl-*N*-trimethylsilylacetamide** [7449-74-3] *M* 145.3, *b* 48-49°/11mm, 84°/13mm, 105-107°/35mm (solid at room temp), *d* 0.90, *n* 1.4379. Likely impurity is Et₃N.HCl which can be detected by its odour. If it is completely soluble in *C₆H₆, then redistil, otherwise dissolve in this solvent, filter and evaporate first in a vacuum at 12mm then fractionate, all operations should be carried out in a dry N₂ atmosphere. [*J Am Chem Soc* 88 3390 1966; *Chem Ber* 96 1473 1963.]

Methyl trimethylsilylacetate [2916-76-9] *M* 146.3, *b* 65-68°/50mm, *d* 0.89. Dissolved in Et₂O, shaken with 1M HCl, washed with H₂O, aqueous saturated NaHCO₃, H₂O again, and dried (a ppte may be formed in the NaHCO₃ soln and should be drawn off and discarded). The solvent is distd off and the residue is fractionated through a good column. IR (CHCl₃) ν 1728cm⁻¹. [*J Org Chem* 32 3535 1967, 45 237 1980.]

Methyl 2-(trimethylsilyl)propionate [55453-09-3] *M* 160.3, *b* 155-157°/atm, *d* 0.89. Dissolve in Et₂O, wash with aqueous NaHCO₃, H₂O, 0.1M HCl, H₂O again, dry (MgSO₄), evaporated and distil. [*J Chem Soc Perkin Trans 1* 541 1985; *Tetrahedron* 39 3695 1983.]

Methyl triphenoxyphosphonium iodide [17579-99-6] *M* 452.2, *m* 146°. Gently heat the impure iodide with good grade Me₂CO. The saturated solution obtained is decanted rapidly from undissolved salt and treated with an equal volume of dry Et₂O. The iodide separates as beautiful flat needles which are collected by centrifugation, washed several times with dry Et₂O, and dried in a vacuum over P₂O₅. For this recrystn it is essential to minimise the time of contact with Me₂CO and to work rapidly and with rigorous exclusion of moisture. If the crude material is to be used, it should be stored under dry Et₂O, and dried and weighed *in vacuo* immediately before use. [*J Chem Soc Perkin Trans 1* 982 1974; *J Chem Soc* 224 1953.]

Methyl triphenylphosphonium bromide [1779-49-3] *M* 357.3, *m* 229-230°(corr), 227-229°, 230-233°. If the solid is sticky, wash with *C₆H₆ and dry in a vacuum over P₂O₅. [Marvel and Gall *J Org Chem* 24 1494 1959; *Chem Ber* 87 1318 1954; Milas and Priesing *J Am Chem Soc* 79 6295 1957; Wittig and Schöllkopf *Org Synth* 40 66 1960..] The iodide crystd from H₂O has *m* 187.5-188.5° [*J Chem Soc* 1130 1953; *Justus Liebigs Ann Chem* 580 44 1953].

***N*-Methyl-*N*-trimethylsilyl trifluoroacetamide** [24589-78-4] *M* 199.3, *b* 78-79°/130mm. Fractionate through a 40mm Vigreux column. Usually it contains *ca* 1% of methyl trifluoroacetamide and 1% of other impurities which can be removed by gas chromatography or fractionating using a spinning band column. [*J Chromatogr* 42 103 1969, 103 91 1975.]

Methyl vinyl dichlorosilane (dichloro methyl vinyl silane) [124-70-9] *M* 141.1, *b* 43-45.5°/11-11.5mm, 91°/742mm, 92.5°/743.2mm, 92.5-93°/atm, *d* 1.0917, *n* 1.444. Likely impurities are dichloromethylsilane, butadienyl-dichloromethylsilane. Fractionate through a column packed

with metal filings (20 theoretical plates) at atmospheric pressure. [*Izv Akad Nauk SSSR Ser Khim* 1474 1957 and 767 1958.]

Milling Red SWB {1-[4-[4-[4-toluenesulfonyloxy]phenylazo](3,3'-dimethyl-1,1'-biphenyl)-4'-azo]-2-hydroxynaphthalene-6,8-disulfonic acid di-Na salt, Acid Red 114} [6459-94-5] M 830.8, m dec >250°, CI 23635, λ_{\max} ~514nm. Salted out three times with sodium acetate, then repeatedly extracted with EtOH. [McGrew and Schneider *J Am Chem Soc* 72 2547 1950.] See Solochrome Violet R on p. 352 in Chapter 4.

Milling Yellow G [51569-18-7]. Salted out three times with sodium acetate, then repeatedly extracted with EtOH. [McGrew and Schneider *J Am Chem Soc* 72 2547 1950.] See Solochrome Violet R on p. 352 in Chapter 4.

Molybdenum hexacarbonyl [13939-06-5] M 264.0, m 150°(dec), b 156°. Sublimed in a vacuum before use [Connor et al. *J Chem Soc, Dalton Trans* 511 1986].

Molybdenum hexafluoride [7783-77-9] M 209.9, b 35°/760mm. Purified by low-temperature trap-to-trap distillation over predried NaF. [Anderson and Winfield *J Chem Soc, Dalton Trans* 337 1986.]
Poisonous vapours.

Molybdenum trichloride [13478-18-7] M 202.3, m 1027°, d 3.74. Boiled with 12M HCl, washed with absolute EtOH and dried in a vacuum desiccator.

Molybdenum trioxide (molybdenum IV oxide, MoO₃) [1313-27-5] M 143.9, m 795°, d 4.5. Crystd from water (50mL/g) between 70° and 0°.

Monocalcium phosphate (2H₂O) (monobasic) [7789-77-7 (2H₂O); 7757-93-9 (anhydr)] M 154.1, m 200°(dec, loses H₂O at 100°), d 2.2. Crystd from a near-saturated soln in 50% aqueous reagent grade phosphoric acid at 100° by filtering through fritted glass and cooling to room temperature. The crystals were filtered off and this process was repeated three times using fresh acid. For the final crystn the solution was cooled slowly with constant stirring to give thin plate crystals that were filtered off on fritted glass, washed free of acid with anhydrous acetone and dried in a vacuum desiccator [Egan, Wakefield and Elmore, *J Am Chem Soc* 78 1811 1956].

Monoperoxyphthalic acid magnesium salt 6H₂O. (MMPP) [84665-66-7] M 494.7, m ~93°(dec). MMPP is a safer reagent than *m*-chloroperbenzoic acid because it is not as explosive and has advantages of solubility. It is sol in H₂O, low mol wt alcohols, *i*-PrOH and DMF. The product of reaction, Mg phthalate, is sol in H₂O. It has been used in aq phase to oxidise compds in e.g. CHCl₃ and using a phase transfer catalyst e.g. methyltrioctylammonium chloride [Brougham *Synthesis* 1015 1987]. The oxidising activity can be checked (as for perbenzoic acid in Silbert et al. *Org Synth Coll Vol V* 906 1973), and if found to be low it would be best to prepare afresh from phthalic anhydride (1mol), H₂O₂ (1mol) and MgO at 20-25° to give MMPP. [Hignett, European Pat Appl 27 693 1981, *Chem Abstr* 95 168810 1981.]

Naphthalene Scarlet Red 4R [1-(4-sulfonaphthalene-1-azo)-2-hydroxynaphthalene-6,8-disulfonic acid tri-Na salt, New Coccine, Acid Red 18] [2611-82-7] M 604.5, m >250°(dec), CI 16255, λ_{\max} 506nm. Dissolved in the minimum quantity of boiling water, filtered and enough EtOH was added to ppt ca 80% of the dye. This process was repeated until a soln of the dye in aqueous 20% pyridine had a constant extinction coefficient.

Naphthol Yellow S (citronin A, flavianic acid sodium salt, 8-hydroxy-5,7-dinitro-2-naphthalene sulfonic acid disodium salt) [846-70-8] M 358.2, m dec on heating. Greenish yellow powder soluble in H₂O. The *free sulfonic acid* can be recrystd from dil HCl (m 150°) or AcOH-EtOAc (m 148-149.5°). The disodium salt is then obtained by dissolving the acid in two equivalents of aqueous NaOH