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IR(endo,anti)-3-Bromocamphor-8-sulfonic acid ammonium salt [55870-50-3] M 328.2, m 284-285°(dec), $[\alpha]_D^{25} +84.8^\circ$ (c 4, H₂O). Passage of a hot aqueous soln through an alumina column removed water-soluble coloured impurities which remained on the column when the ammonium salt was eluted with hot water. The salt was crystd from water and dried over CaCl₂ [Craddock and Jones *J Am Chem Soc* 84 1098 1962; Kauffmann *J Prakt Chem* 33 295 1966].

Bromopyrogallol Red [16574-43-9] M 576.2, ϵ 5.45 x 10⁴ at 538nm (water pH 5.6-7.5). See Bromopyrogallol Red (5,5'-dibromopyrogallolsulfonephthalein) on p. 141 in Chapter 4.

Bromosulfalein (phenoltetrabromophthalein 3',3'-disulfonic acid disodium salt) [71-67-0] M 838.0. Purified by TLC on silica Gel G (Merck 250 μ particle size) in two solvent systems (BuOH-AcOH-H₂O 30:7.5:12.5 v/v; and BuOH-propionic acid-H₂O 30:20:7.5 v/v). When the solvent reached a height of 10cm the plate was removed, dried in air and developed with NH₃ vapour giving blue coloured spots. Also the dye was chromatographed on MN Silica Gel with *t*-BuOH-H₂O-*n*-BuOH (32:10:5 v/v and visualised with a dilute KOH (or NaOH if the Na salt is required) spray. The product corresponding to bromosulfalein was scraped off and eluted with H₂O, filtered and evap to dryness in a vacuum. It was dissolved in H₂O and filtered through Sephadex G-25 and evaporated to dryness. [UV and IR identification: *J Pharm Sci* 57 819 1968; NMR: *Chem Pharm Bull Jpn* 20 581 1972; *Anal Biochem* 83 75 1977.]

Bromtrimethylsilane (trimethylbromosilane, trimethylsilyl bromide) [2857-97-8] M 153.1, m -43.5° to -43.2°; b 40.5°/200mm, 77.3°/735mm, 79°/744mm, 79.8-79.9°/754mm, d_4^{20} 1.1805, d_4^{20} 1.190, n_D^{20} 1.422. Purified by repeated fractional distillation and stored in sealed ampoules in the dark. [*J Am Chem Soc* 75 1583 1953.] Also fractionally distd through a 15 plate column (0.8 x 32cm packed with 1/16in single turn helices from Pt-Ir wire). [*J Am Chem Soc* 68 1161 1946; 70 433 1948.]

***tert*-Butyldiphenylchlorosilane (TBDPSCI, *tert*-butylchlorodiphenylsilane)** [58479-61-1] M 274.9, b 90°/0.015mm, d 1.057, n 1.568. Purified by repeated fractional distn. It is soluble in DMF and pentane [Hanessian and Lavalee *Can J Chem* 53 2975 1975; Robl et al. *J Med Chem* 34 2804 1991].

***n*-Butylmercuric chloride** [543-63-5] M 293.1, m 130°. Crystd from EtOH.

***n*-Butylphenyl *n*-butylphosphonate** [36411-99-1] M 270.3. Crystd three times from hexane as its compound with uranyl nitrate. See *tri-n-butyl phosphate* below.

***tert*-Butyldimethylsilyl chloride (TBDMSCl)** [18162-48-6] M 150.7, m 87-89°, 92.5°, b 125°/760mm. Fractionally distd at atmospheric pressure. [*J Am Chem Soc* 76 1030 1954; 94 6190 1972.]

***p-tert*-Butylphenyl diphenyl phosphate** [981-40-8] M 382.4, b 261°/6mm, n^{25} 1.5522. Purified by vacuum distn, and percolation through an alumina column, followed by passage through a packed column maintained at 150° to remove residual traces of volatile materials in a counter-current stream of N₂ at reduced pressure [Dobry and Keller *J Phys Chem* 61 1448 1957].

***n*-Butylstannic acid [PhSn(OH)₃, trihydroxy-*n*-butylstannane]** [22719-01-3] M 208.8. Purified by adding excess KOH in CHCl₃ to remove *n*-BuSn(OH)Cl₂ and *n*-BuSn(OH)₂Cl, and isolated by acidification [Holmes et al. *J Am Chem Soc* 109 1408 1987].

Cacodylic acid (dimethylarsinic acid) [75-60-5] M 138.0, m 195-196°, pK²⁵ 6.15 [Me₂As(O)OH]. Crystd from warm EtOH (3mL/g) by cooling and filtering. Dried in vacuum desiccator over CaCl₂. Has also been twice recrystd from propan-2-ol. [Koller and Hawkrigde *J Am Chem Soc* 107 7412 1985.]

Cadion [1-(4-nitrophenyl)-3-(4-phenylazophenyl)-triazene] [5392-67-6] M 346, m 198°. Commercial cadion is purified by recrystn from 95% EtOH and dried. It is stable in 0.2 N KOH (in 20% aqueous EtOH) at 25°. It is a sensitive reagent for Cd, and the Cd complex has λ_{\max} (EtOH) 475nm. [*Aust Chem Inst J Proc* 4 26 1937 ; *Anal Chim Acta* 19 377 1958.]

Cadmium [7440-43-9] M 112.4, m 321.1°, b 767°. Oxide has been removed by filtering the molten metal, under vacuum through quartz wool.

Cadmium acetate (2H₂O) [5743-04-4] M 230.5, m 255°(anhydr), d 2.01 (hydr), 2.34 (anhydr), pK_1^{25} 9.7, pK_2^{25} ~11.0 (for Cd²⁺). Crystd twice from anhydrous acetic acid and dried under vacuum for 24h at 100°.

Cadmium bromide (4H₂O) [13464-92-1 (4H₂O); 7789-42-6 (anhydr)] M 344.2, m 566°, b 963, d 5.19°. Crystd from water (0.6mL/g) between 100° and 0°, and dried at 110°. Forms monohydrate below 36° and the 4H₂O above 36°.

Cadmium chloride [10108-64-2] M 183.3, m 568°, b 960°, d 4.06. Crystd from water (1mL/g) by addition of EtOH and cooling.

Cadmium fluoride [7790-79-6] M 150.4, m >1000°, b 1748°, d 6.35. Crystd by dissolving in water at room temperature (25mL/g) and heating to 60°.

Cadmium iodide [7790-80-9] M 366.2, m 388°, b 787°, d 5.66. Crystd from ethanol (2mL/g) by partial evaporation.

Cadmium ionophore I [*N,N,N',N'*-tetramethyl-3,6-dioxooctanedi-(thioamide)] [73487-00-0] M 432.7, m 35-36°. Wash well with pet ether, then several times with 2N HCl (if it has a slight odour of pyridine) then H₂O and dry in a vacuum over H₂SO₄. It is a polar selectrophore for Cd. [*Helv Chim Acta* 63 217 1980.]

Cadmium lactate [16039-55-7] M 290.6. Crystd from water (10mL/g) by partial evapn in a desiccator.

Cadmium nitrate (4H₂O) [10022-68-1] M 308.5, m 59.5°. Crystd from water (0.5mL/g) by cooling in ice-salt.

Cadmium potassium iodide [13601-63-3] M 532.2. Crystd from ethanol by partial evapn.

Cadmium salicylate [19010-79-8] M 248.5, 242°(dec). Crystd from distd H₂O by evapn in a desiccator.

Cadmium sulfate [7790-84-3 (for 3CdSO₄ 8H₂O); 10124-36-4 (anhydr)] M 208.4 (anhydr), 769.5 (hydr). Crystd from distd water by partial evapn in a desiccator. On heating gives monohydrate at 80°.

Calcein sodium salt [2',7'-bis-{*N,N*-di(carboxymethyl)aminomethyl}fluorescein Na salt, **Fluorexon**, **Fluorescein Complexon**] [1461-15-0] M 666.5, $pK_{\text{Est}(1)}$ ~ 1.9, $pK_{\text{Est}(2)}$ ~ 2.5, $pK_{\text{Est}(3)}$ ~ 8.0, $pK_{\text{Est}(4)}$ ~ 10.5 (all for N-CH₂COOH), and $pK_{\text{Est}(5)}$ ~ 3.5 (for benzoic COOH). Dissolve in distilled H₂O and acidify with dilute HCl to pH 3.5. Filter off the solid acid and wash well with H₂O. Redissolve ca 10g in 300mL H₂O containing 12g of NaOAc. Ppte again by adding HCl, filter and wash with H₂O. Add the solid to 200mL of EtOH stir for 1h and filter. Repeat the EtOH wash and dry the bright yellow solid in a vacuum. This acid decomposes on heating at ca 180°. See below for the prepn of the Na salt. [*Anal Chem* 28 882 1956].

Dissolve in H₂O and acidify with 3N HCl to pH 3.5. Collect the solid and wash with H₂O. The air-dried ppte is extracted with 70% aqueous EtOH, filtered hot and cooled slowly. Fine yellow needles of the acid crystallise out, are filtered and dissolved in the minimum quantity of 0.01N NaOH and reppted with N HCl to pH 3.5. It is then recrystd from 70% aqueous EtOH (3x). The final product (acid) is dried at 80° in a vacuum for 24h, m >300°dec. It contains one mol of water per mol of acid (C₃₀H₃₆N₄O₁₃·H₂O). The product is pure as revealed

by electrophoresis at pH 5.6 and 8.6, and by TLC in *i*-BuOH-*i*-PrOH-AcOH-H₂O (60:60:5:5 by vol) or *i*-PrOH or pH 8.0 borate buffer. [Wallach et al. *Anal Chem* 31 456 1959.]

The Na salt is prepared by dissolving the in H₂O containing 2 mols of NaOH per mol of acid reagent and lyophilising. It complexes with Ca and Mg ions.

Calcium [7440-70-2] **M 40.1, m 845°**. Cleaned by washing with ether to remove adhering paraffin, filed in an argon-filled glove box, and washed with ethanol containing 2% of conc HCl. Then washed with dry ethanol, dried in a vac and stored under pure argon [Addison, Coltreay and Halsstead, *J Chem Soc* 3868 1962].

Calcium acetate monohydrate [5743-26-0 (H₂O), 62-54-4 (xH₂O)] **M 176.2 (H₂O), m 150° (loses H₂O), pK²⁵ 12.7 (for Ca₂⁺)**. Crystd from water (3mL/g) by partial evapn in a desiccator.

Calcium benzoate (3H₂O) [2090-05-3] **M 336.4**. Crystd from water (10m/g) between 90° and 0°.

Calcium bromide (H₂O) [62648-72-0; 71626-99-8 (xH₂O); 7789-41-5 (anhydr)] **M 217.9, d 3.35**. Crystd from EtOH or Me₂CO. It loses H₂O on heating and is anhydrous at 750° then it loses Br. Deliquescent.

Calcium butyrate [5743-36-2] **M 248.2**. Crystd from water (5mL/g) by partial evapn in a desiccator.

Calcium carbamate [543-88-4] **M 160.1**. Crystd from aqueous ethanol.

Calcium chloride (anhydrous) [10043-52-4] **M 111.0, m 772°, b >1600°, d₄¹⁵ 2.15**. Available as fused granules or cubic crystals. It is very *hygroscopic*. Very soluble in H₂O (exothermic), and EtOH. Store in a tightly closed container.

Calcium chloride (2H₂O) [10035-04-8] **M 147.0, m 175°(dehydr), 772°(dec)**. Crystd from ethanol, and is hygroscopic. Loses H₂O at 200° so it can be dried at high temperatures to dehydrate. *Hexahydrate* [7774-34-7] has **m 30°** and **d 1.67**.

Calcium dithionite [13812-88-9] **M 168.2, m dec on heating**. Crystd from water, or water followed by acetone and dried in air at room temperature.

Calcium D-gluconate monohydrate [299-28-5] **M 448.4, m dec on heating, [α]₅₄₆²⁰ +11.0°, [α]_D²⁰ +9.0° (c 1.2, H₂O)**. It is sol in H₂O (3.5g in 100g at 25°). Dissolve in H₂O, filter and ppt by adding MeOH. Filter off solid and dry in a vacuum at 85°. Alternatively, dissolve in H₂O, filter (from insol inorganic Ca) and evaporate to dryness under vacuum at 85°. [*J Am Pharm Assoc* 41 366 1952.]

Calcium D-heptagluconate dihydrate [17140-60-2] **M 526.4, [α]₅₄₆²⁰ +5.2°, [α]_D²⁰ +4.4° (c 5, H₂O)**. Purified same as calcium D-gluconate.

Calcium formate [544-17-2] **M 130.1, m dec on heating, d 2.01**. Crystd from water (5mL/g) by partial evaporation in a desiccator.

Calcium hexacyanoferrate (II) (11H₂O) [13821-08-4] **M 490.3**. Recrystd three times from conductivity H₂O and air dried to constant weight over partially dehydrated salt. [*Trans Faraday Soc* 45 855 1949.] Alternatively the Ca salt can be purified by pptn with absolute EtOH in the cold (to avoid oxidation) from an air-free saturated aqueous soln. The pure lemon yellow crystals are centrifuged, dried in a vacuum desiccator first over dry charcoal for 24h, then over partly dehydrated salt and stored in a dark glass stoppered bottle. No deterioration occurred after 18 months. No trace of Na, K or NH₄ ions could be detected in the salt from the residue after decomposition of the salt with conc H₂SO₄. Analyses indicate 11mols of H₂O per mol of salt. The solubility in H₂O is 36.45g (24.9°) and 64.7g (44.7°) per 100g of solution. [*J Chem Soc* 50 1926.]

Calcium hydroxide [1305-62-0] **M 74.1, m loses H₂O on heating, pK²⁵ 12.7 (for Ca²⁺)**. Heat analytical grade calcium carbonate at 1000° during 1h. Allow the resulting oxide to cool and add slowly to

water. Heat the suspension to boiling, cool and filter through a sintered glass funnel of medium porosity (to remove soluble alkaline impurities). Dry the solid at 110° and crush to a uniformly fine powder.

Calcium iodate [7789-80-2 (H_2O)] **M 389.9, m >540°, pK²⁵ 0.79 (for HIO₃)**. Crystd from water (100mL/g).

Calcium iodide (xH_2O) [71626-98-7 (xH_2O); 10102-68-8 (*anhydr*)] **M 293.9 (for 4H₂O), m 740°, b 1100°**. Dissolved in acetone, which was then diluted and evaporated. This drying process was repeated twice, then the CaI₂ was crystd from acetone-diethyl ether and stored over P₂O₅. Very *hygroscopic* when anhydrous and is light sensitive [Cremllyn et al. *J Chem Soc* 528 1958]. *Hexahydrate* has m 42°.

Calcium ionophore I (ETH 1001) [58801-34-6] **M 685.0**. This is a neutral Ca selectophore. It can be purified by thick layer (2mm) chromatography (Kieselgel F₂₄₅) and eluted with Me₂CO-CHCl₃ (2:1). [*Helv Chim Acta* 56 1780 1973.]

Calcium ionophore II (ETH 129) [74267-27-9] **M 460.7, m 153-154°**. Recrystd from Me₂CO. It forms 1:2 and 1:3 metal/ligand complexes with Mg²⁺ and Ca²⁺ ions respectively, and induces selectivity in membranes for Ca²⁺ over Mg²⁺ by a factor of *ca* 10⁴. [*Helv Chim Acta* 63 191 1980.]

Calcium ionophore III [A23187 calcimycin] [52665-69-7] **M 523.6, m 181-182°, [α]_D²⁵ -56.0° (c 1, CHCl₃)**. Recrystallises from Me₂CO as colourless needles. Protect from light and moisture, store in a refrigerator. Soluble in Me₂SO or EtOH and can be stored for 3 months without loss of activity. Mg and Ca salts are soluble in organic solvents and cross biological membranes. It has a pK_a of 6.9 in 90% Me₂SO. The Ca complex cryst from 50% EtOH as colourless prisms. *Highly TOXIC* [*Ann Rev Biochem* 45 501 1976; *J Am Chem Soc* 96 1932 1974, *J Antibiotics* 29 424 1976.]

Calcium isobutyrate [533-90-4] **M 248.2**. Crystd from water (3mL/g) by partial evapn in a desiccator.

Calcium lactate (5H₂O) [814-80-2] **M 308.3, m anhydr at 120°**. Crystd from warm water (10mL/g) by cooling to 0°.

Calcium nitrate (4H₂O) [13477-34-4] **M 236.1, m 45°(dehydr), 560°(anhydr)**. Crystd four times from water (0.4mL/g) by cooling in a CaCl₂-ice freezing mixture. The tetrahydrate was dried over conc H₂SO₄ and stored over P₂O₅, to give the anhydrous salt. It is deliquescent. After 3 recrystns of ACS grade it had Co, Fe, Mg, Sr and Zn at 0.2, 1. 0, 0.02, 10 and 0.02 ppm resp.

Calcium nitrite (2H₂O) [13780-06-8 (30%w/w aq soln)] **M 150.1(hydr), m dec on heating, d 2.22**. Crystd from hot water (1.4mL/g) by adding ethanol and cooling to give the hydrate. It is deliquescent.

(+)-Calcium pantothenate (H₂O) (D(+)- 137-08-6; 63409-48-3] **M 476.5**. See R(+)-pantothenic acid calcium salt on p. 555 in Chapter 6.

Calcium permanganate (4H₂O) [10118-76-0 (*anhydr*)] **M 350.0 (for 4H₂O)**. Crystd from water (3.3mL/g) by partial evapn in a desiccator. It is deliquescent.

Calcium propionate [4075-81-4] **M 186.2, m dec on heating**. Crystd from water (2mL/g) by partial evapn in a desiccator.

Calcium salicylate (2H₂O) [824-35-1] **M 350.4**. Crystd from water (3mL/g) between 90° and 0°.

Calcium sulfate dihydrate [10101-41-4] **M 172.1, m 150(dec), d 2.32**. Loses only part of its H₂O at 100-150° (see below). Soluble in H₂O and very slowly soluble in glycerol. Insoluble in most organic solvents.

Calcium sulfate hemihydrate [10034-76-1] **M 145.2**. Sol in H₂O (0.2 parts/100 at 18.75°). Completely dehydrated >650°. Dry below 300° to give a solid with estimated pore size *ca* 38% of vol. Anhydrous CaSO₄ has high affinity for H₂O and will absorb 6.6% of its weight of H₂O to form the hemihydrate (gypsum). It sets to a hard mass with H₂O, hence should be kept in a tightly sealed container.

Calcium thiosulfate [10124-41-1] **M 152.2, m 43-49°, pK₁^{2.5} 0.6, pK₂^{2.5} 1.74 (for H₂S₂O₃)**. Recrystd from water below 60° in a N₂ atmosphere, followed by drying with EtOH and Et₂O. Stored in a refrigerator. [Pethybridge and Taba *J Chem Soc, Faraday Trans 1* **78** 1331 1982.]

(4-Carbamylphenylarsylenedithio)diacetic acid [531-72-6] **M 345.1, pK_{Est}~3.5**. Crystd from MeOH or EtOH.

Carbonate ionophore I [ETH 6010] (heptyl 4-trifluoroacetylbenzoate) [129476-47-7] **M 316.3, b 170°/0.02 Torr, d 0.909**. Purified by flash chromatography (2g of reagent with 30g of Silica Gel 60) and eluted with EtOAc/hexane (1:19). The fractions that absorbed at 260nm were pooled, evapd and dried at room temp (10.3 Torr). The oily residue was distd in a bubbled-tube apparatus (170°/0.02 Torr). Its IR (CHCl₃) had peaks at 1720, 1280, 940cm⁻¹ and its sol in tetrahydrofuran is 50mg/0.5mL. It is a lipophilic neutral ionophore selective for carbonate as well as being an optical humidity sensor. [*Anal Chim Acta* **233** 41 1990.]

Carbon dioxide [124-38-9] **M 44.0, sublimates at -78.5°, pK₁^{2.5} 6.35, pK₂^{2.5} 10.33 (for H₂CO₃)**. Passed over CuO wire at 800° to oxidise CO and other reducing impurities (such as H₂), then over copper dispersed on Kieselguhr at 180° to remove oxygen. Drying at -78° removed water vapour. Final purification was by vacuum distn at liquid nitrogen temperature to remove non-condensable gases [Anderson, Best and Dominey *J Chem Soc* 3498 1962]. Sulfur dioxide can be removed at 450° using silver wool combined with a plug of platinised quartz wool. Halogens are removed by using Mg, Zn or Cu, heated to 450°.

Carbon disulfide, see entry on p. 156 in Chapter 4.

Carbon monoxide [630-08-0] **M 28.0, b -191.5°**. Iron carbonyl is a likely impurity in CO stored under pressure in steel tanks. It can be decomposed by passage of the gas through a hot porcelain tube at 350-400°. Passage through alkaline pyrogallol soln removes oxygen (and CO₂). Removal of CO₂ and water are effected by passage through soda-lime followed by Mg(ClO₄)₂. Carbon monoxide can be condensed and distd at -195°. **HIGHLY POISONOUS gas.**

Carbonyl bromide [593-95-3] **M 187.8, b 64.5°/760mm**. Purified by distn from Hg and from powdered Sb to remove free bromine, then vacuum distd to remove volatile SO₂ (the major impurity) [Carpenter et al. *J Chem Soc, Faraday Trans 2* 384 1977]. **TOXIC**

Carbonyl sulfide [463-58-1] **M 60.1, m -138°, b -47.5°, -50°**. Purified by scrubbing through three consecutive fritted washing flasks containing conc NaOH at 0° (to remove HCN), and then through conc H₂SO₄ (to remove CS₂) followed by a mixture of NaN₃ and NaOH solution; or passed through traps containing satd aq lead acetate, then through a column of anhydrous CaSO₄. Then freeze-pumped repeatedly and distd through a trap packed with glass wool and cooled to -130° (using an *n*-pentane slurry). It liquefies at 0°/12.5mm. The gas is stored over conc H₂SO₄. **TOXIC**

Catecholborane (1,3,2-Benzodioxaborole) [274-07-7] **M 119.2, b 50°/50mm, 66°/80mm, 76-77°/100mm, 88°/165mm, d 1.125, n 1.507 (also available as a 1.0M soln in THF)**. A moisture sensitive flammable liquid which is purified by distn in a vacuum under a N₂ atmosphere and stored under N₂ at 0-4°. It liberates H₂ when added to H₂O or MeOH. A soln in THF after 25h at 25° has residual hydride of 95%(under N₂) and 80% (under air) [Brown and Gupta *J Am Chem Soc* **97** 5249 1975].

Celite 545 (diatomaceous earth) [12003-10-0]. Stood overnight in conc HCl after stirring well, then washed with distilled water until neutral and free of chloride ions. Washed with methanol and dried at 50°.

Ceric ammonium nitrate [16774-21-3] M 548.2, pK_1^{25} -1.15, pK_2^{25} -0.72, pK_3^{25} 1.68, pK_4^{25} 2.29 (for aquo Ce^{4+}). Ceric ammonium nitrate (125g) is warmed with 100mL of dilute HNO_3 (1:3 v/v) and 40g of NH_4NO_3 until dissolved, and filtered off on a sintered-glass funnel. The solid which separates on cooling in ice is filtered off on a sintered funnel (at the pump) and air is sucked through the solid for 1-2 h to remove most of the nitric acid. Finally, the solid is dried at 80-85°.

Cerous acetate [537-00-8] M 317.3, pK_1^{25} 8.1 (9.29), pK_2^{25} 16.3, pK_3^{25} 26.0 (for Ce^{3+}). Crystd twice from anhydrous acetic acid, then pumped dry under vacuum at 100° for 8h.

Cesium bromide [7787-69-1] M 212.8, m 636°, b ca 1300°, d 4.44. Very soluble in H_2O , soluble in EtOH but insoluble in Me_2CO . Dissolve in the minimum volume of H_2O , filter and ppte by adding Me_2CO . Filter solid and dry at 100°. Also recrystd from water (0.8mL/g) by partial evaporation in a desiccator.

Cesium carbonate [534-17-8] M 325.8, m 792°(at red heat). Crystd from ethanol (10mL/g) by partial evaporation.

Cesium chloride [7647-17-8] M 168.4, m 645°, b 1303°, d 3.99. Soluble in H_2O but can be purified by crystn from H_2O [sol in g per cent: 162.3(0.7°), 182.2(16.2°) and 290(at bp 119.4°)] and dried in high vac. Sol in EtOH and is deliquescent, keep in a tightly closed container. [*Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 951 1963.] For further purification of CsCl, a conc aqueous soln of the practically pure reagent is treated with an equivalent weight of I_2 and Cl_2 bubbled into the soln until pptn of $CsCl_2I$ ceased. Recrystn yields a salt which is free from other alkali metals. It is then decomposed to pure CsCl on heating. [*J Am Chem Soc* 52 3886 1930.] Also recrystd from acetone-water, or from water (0.5mL/g) by cooling in $CaCl_2$ /ice. Dried at 78° under vacuum.

Cesium chromate [56320-90-2] M 381.8, pK_1^{25} 0.74, pK_2^{25} 6.49 (for H_2CrO_4). Crystd from water (1.4mL/g) by partial evapn in a desiccator.

Cesium fluoride [13400-13-0] M 151.9, m 703°. Crystd from aqueous soln by adding ethanol.

Cesium iodide [7789-17-5] M 259.8, m 621°, b-1280°, d 4.5. Crystd from warm water (1mL/g) by cooling to -5°.

Cesium nitrate [7789-18-6] M 194.9, m 414°(dec), d 3.65. Crystd from water (0.6mL/g) between 100° and 0°. After 1 crystn of 99.9% grade it had K, Na and Se at 0.8, 0.4 and 0.2 ppm resp.

Cesium oleate [31642-12-3] M 414.4. Crystd from EtOAc, dried in an oven at 40° and stored over P_2O_5 .

Cesium perchlorate [13454-84-7] M 232.4, pK^{25} -2.4 to -3.1 (for $HClO_4$). Crystd from water (4mL/g) between 100° and 0°.

Cesium perfluoro-octanoate [17125-60-9] M 546.0. Recrystd from a butanol-petroleum ether mixture, dried in an oven at 40° and stored over P_2O_5 under vacuum.

Cesium sulfate [10294-54-9] M 361.9, m 1005°, d 4.24. Crystd from water (0.5mL/g) by adding ethanol and cooling.

Chloramine-T (*N*-chloro-*p*-toluenesulfonamide sodium salt) $3H_2O$ [7080-50-4] M 281.7, m 168-170°(dec). Crystd from hot water (2mL/g). Dried in a desiccator over $CaCl_2$ where it loses water. Protect from sunlight. Used for detection of bromate and halogens, and Co, Cr, Fe, Hg, Mn, Ni and Sb ions.

Chlorazol Sky Blue FF {6,6'-[(3,3'-dimethoxy[1,1'-biphenyl]-4,4'-diyl)bis(azo)bis(4-amino-5-hydroxy-1,3-naphthylenedisulfonic acid) tetra-Na salt [2610-05-1] M 996.9, m

>300°(dec). Freed from other electrolytes by adding aqueous sodium acetate to a boiling soln of the dye in distd water. After standing, the salted-out dye was filtered on a Büchner funnel, the process being repeated several times. Finally, the pptd dye was boiled several times with absolute EtOH to wash out any sodium acetate, then dried (as the sodium salt) at 105°. [McGregor, Peters and Petropolous *Trans Faraday Soc* **58** 1045 1962.]

Chlorine [7782-50-5] **M 70.9, m -101.5°, b -34.0°, d 2.898.** Passed in succession through aqueous KMnO₄, dilute H₂SO₄, conc H₂SO₄, and a drying tower containing Mg(ClO₄)₂. Or, bubbled through with water, dried over P₂O₅ and distd from bulb to bulb in a vac line. **HIGHLY TOXIC.**

Chlorine trifluoride [7790-91-2] **M 92.5, b 12.1°.** Impurities include chloryl fluoride, chlorine dioxide and hydrogen fluoride. Passed first through two U-tubes containing NaF to remove HF, then through a series of traps in which the liquid is fractionally distd. Can be purified *via* the KF complex, KClF₄, formed by adding excess ClF₃ to solid KF in a stainless steel cylinder in a dry-box and shaking overnight. After pumping out the volatile materials, pure ClF₃ is obtained by heating the bomb to 100-150° and condensing the evolved gas in a -196° trap [Schack, Dubb and Quaglino *Chem Ind (London)* 545 1967]. **HIGHLY TOXIC.**

Chlorodiphenylphosphine (diphenylphosphinous chloride) [1079-66-9] **M 220.6, m 15-16°, b 124-126°/0.6mm, 174°/5mm, 320°/atm, d 1.229, n 1.636.** Air sensitive, pale yellow lachrymatory liquid which is purified by careful fractional distn and discarding the lower boiling fraction which contains the main impurity PhPCl₂ (b 48-51°/0.7mm); and checking for impurities by NMR. [Weinberg *J Org Chem* **40** 3586 1975; Honer et al. *Chem Ber* **94** 2122 1961.]

4-(Chloromercuri)benzenesulfonic acid monosodium salt [14110-97-5] **M 415.2, dec on heating.** The free acid is obtained by acidifying an aq soln, filtering off the acid, washing it with H₂O and recrystallising from hot H₂O to give a colourless solid which is dried in a vacuum over P₂O₅ and should give negative Cl⁻ ions. The Na salt is made by dissolving in one equivalent of aqueous NaOH and evaporate to dryness. [*Chem Ber* **67** 130 1934; *J Am Chem Soc* **76** 4331 1954.] **HIGHLY TOXIC.**

4-Chloromercuribenzoic acid [59-85-8] **M 357.2, m >300°.** Its suspension in water is stirred with enough 1M NaOH to dissolve most of it: a small amount of insoluble matter is removed by centrifugation. The chloromercuribenzoic acid is then pptd by adding 1M HCl and centrifuged off. The pptn is repeated twice. Finally, the ppte is washed three times with distilled water (by centrifuging), then dried in a thin layer under vacuum over P₂O₅ [Boyer *J Am Chem Soc* **76** 4331 1954].

Chloromethylphosphonic acid dichloride [1983-26-2] **M 167.4, b 50°/0.5mm, 52-53(59)°/2mm, 63-65°/3mm, 78-79°/10mm, 87-88°/15mm, 102-103°/30mm, d₄²⁰ 1.638, n_D²⁰ 1.4971.** Fractionally distd using a short Claisen column and redistd. The *aniline salt* has m 199-201°. The ³¹P NMR has a line at -38±2 ppm from 85% H₃PO₄. [Kinneer and Perren *J Chem Soc* 3437 1952; NMR: *J Am Chem Soc* **78** 5715 1956; *J Org Chem* **22** 462 1957.]

2-Chloro-2-oxo-1,3,2-dioxaphospholane [6609-64-9] **M 142.5, m 12-14°, b 89-91°/0.8mm, d₄²⁰ 1.549, n_D²⁰ 1.448.** Should be distd at high vacuum as some polymerisation occurs on distn. It has IR bands at 3012, 2933, 1477, 1366, 1325, 1040, 924 and 858 cm⁻¹. In H₂O at 100° it is hydrolysed to HOCH₂CH₂OPO₃H₂ in 30min [IR: Cox and Westheimer *J Am Chem Soc* **80** 5441 1958].

2-Chlorophenyl diphenyl phosphate [115-85-5] **M 360.7, b 236°/4mm, n_D²⁵ 1.5707.** Purified by vacuum distn, percolated through a column of alumina, then passed through a packed column maintained by a countercurrent stream of nitrogen at reduced pressure [Dobry and Keller *J Phys Chem* **61** 1448 1957].

Chlorosulfonic (chlorosulfuric) acid [7790-94-5] **M 116.5, b 151-152°/750mm, d₄ 1.753, n 1.4929, pK -5.9 (aq H₂SO₄).** Distd in an all-glass apparatus, taking the fraction boiling at 156-158°. Reacts **EXPLOSIVELY** with water [Cremlyn *Chlorosulfonic acid: A Versatile Reagent*, Royal Society of Chemistry UK, 2002, 308 pp, ISBN 0854044981].

Chloro-(2,2':6',2'-terpyridine)platinum (II) chloride (2H₂O) [60819-00-3] **M 535.3**. Recrystd from hot dilute HCl and cooling to give the red dihydrate. The trihydrate crysts slowly from a cold aq soln and is air dried. The red dihydrate can be obtained from the trihydrate by desiccation over conc H₂SO₄, by washing with EtOH or by precipitating from a warm aq soln with HCl. The dihydrate is also formed by decomposing the black trihydrate form by heating in water (slowly), or more rapidly with hot 2N HCl. [*J Chem Soc* 1498 1934.]

Chloro-tri-isopropyl titanium [20717-86-6] **M 260.6, m 45-50°, b 61-65°/0.1mm**. Distd under vacuum and sets slowly to a solid on standing. Stock reagents are made by dissolving the warm liquid in pentane, toluene, Et₂O, THF, CH₂Cl₂, and can be stored in pure state or in soln under dry N₂ for several months. The reagent is *hygroscopic* and is hydrolysed by H₂O. [*Chem Ber* 118 1421 1985.]

Chlorotriphenylsilane (triphenylchlorosilane) [76-86-8] **M 294.9, m 90-92°, 91-93°, 94-95°, 97-99°, b 156°/1mm, 161°/0.6mm**. Likely impurities are tetraphenylsilane, small amounts of hexaphenyldisiloxane and traces of triphenylsilanol. Purified by distn at 2mm, then crystd from EtOH-free CHCl₃, and from pet ether (b 30-60°) or hexane by cooling in a Dry-ice/acetone bath. [*J Chem Soc* 3671 1957; *J Am Chem Soc* 72 4471 1958, 77 6395 1955, 79 1843 1957.]

Chlorotris(triphenylphosphine) rhodium I (Wilkinson's catalyst) [14694-95-2] **M 925.2, m 138°(dec), 140°(dec), 157-158°(dec)**. Forms dark burgundy crysts from hot EtOH after refluxing for 30min. When the soln is heated for only 5min orange crystals are formed, Heating the orange crystals in EtOH yields red crystals. Crystn from Me₂CO gives the orange crystals. The two forms have similar IR spectra but X-rays are slightly different. [Osborne et al. *J Chem Soc (A)* 1711 1966; Osborne and Wilkinson *Inorg Synth* 10 67 1967; Bennett and Donaldson *Inorg Chem* 16 655 1977.] Sol in CH₂Cl₂ ~2% (25°), in toluene 0.2% (25°), and less sol in Me₂CO, MeOH, BuOH and AcOH, but insol in pet ethers and cyclohexane. It reacts with donor solvents such as pyridine, DMSO and MeCN.

Chromazurol S [1667-99-8] **M 539.3, λ_{max} 540nm, ε 7.80 x 10⁴ (10M HCl), CI 43825, pK₁^{2.5} <0, pK₂^{2.5} 2.25, pK₃^{2.5} 4.88, pK₄^{2.5} 11.75**. Crude *phenolic triphenylmethanecarboxysulfonic acid triNa salt* (40g) is dissolved in water (250mL) and filtered. Then added conc HCl (50mL) to filtrate, with stirring. Ppte is filtered off, washed with HCl (2M) and dried. Redissolved in water (250mL) and pptn repeated twice more in water bath at 70°. Then dried under vacuum over solid KOH (first) then P₂O₅ [Martynov et al. *Zh Analyt Khim* 32 519 1977]. It has also been purified by paper chromatography using *n*-butanol, acetic acid and water (7:3:1). First and second spots were extracted. It chelates Al and Be. Used for estimating fluoride.

Chromic chloride (anhydrous) [10025-73-7] **M 158.4, m 1152°, pK₁^{2.5} 3.95, pK₂^{2.5} 5.55, pK₃^{2.5} 10.5 (for Cr³⁺)**. Sublimed in a stream of dry HCl. Alternatively, the impure chromic chloride (100g) was added to 1L of 10% aq K₂Cr₂O₇ and several millilitres of conc HCl, and the mixture was brought to a gentle boil with constant stirring for 10 min. (This removed a reducing impurity.) The solid was separated, and washed by boiling with successive 1L lots of distilled water until the wash water no longer gave a test for chloride ion, then dried at 110° [Poulsen and Garner *J Am Chem Soc* 81 2615 1959].

Chromium (III) acetylacetonate [21679-31-2] **M 349.3, m 212-216°, 216°, pK₁^{2.5} 4.0 (see chromic chloride)**. Purified by dissolving 6g in hot *C₆H₆ (20mL) and adding 75mL of pet ether slowly. Cool to room temp then chill on ice, filter off and dry in air to give 2.9g. Also crystallises from EtOH. Sol in heptane, *C₆H₆, toluene and pentane-2,4-dione at 20-40°. It forms a 1:2 complex with CHCl₃. [*Inorg Synth* 5 130 1957; *J Am Chem Soc* 80 1839 1958.]

Chromium ammonium sulfate (12H₂O) [34275-72-4 (*hydr*); 13548-43-1 (*anhydr*)] **M 478.4, m 94° loses 9H₂O then dehydr at 300°, d 1.72**. Crystd from a saturated aqueous soln at 55° by cooling slowly with rapid mechanical stirring. The resulting fine crystals were filtered on a Büchner funnel, partly dried on a porous plate, then equilibrated for several months in a vacuum desiccator over crude chromium ammonium sulfate (partially dehydrated by heating at 100° for several hours before use) [Johnson, Hu and Horton *J Am Chem Soc* 75 3922 1953].

Chromium (II) chloride (anhydrous) [10049-05-5] M 122.9, m 824°, d₄¹⁴ 2.75. Obtained from the dihydrate by heating *in vacuo* at 180°. It is a very *hygroscopic* white powder which dissolves in H₂O to give a sky blue solution. Stable in dry air but oxidises rapidly in moist air and should be stored in air tight containers. It sublimates at 800° in a current of HCl gas and cooled in the presence of HCl gas. Alternatively it can be washed with air-free Et₂O and dried at 110-120°. [*Inorg Synth* 3 150 1950.]

Chromium hexacarbonyl [13007-92-6] M 220.1, m 130°(dec), d 1.77. Wash with cold EtOH then Et₂O and allow to dry in air. Alternatively recrystallise from dry Et₂O. This is best accomplished by placing the hexacarbonyl in a Soxhlet extractor and extracting exhaustively with dry Et₂O. Pure Cr(CO)₆ is filtered off and dried in air. Completely colourless refracting crystals are obtained by sublimation at 40-50°/ <0.5mm in an apparatus where the collecting finger is cooled by Dry Ice and in which there is a wide short bore between the hot and cold sections to prevent clogging by the crystals. Loss of product in the crystn and sublimation is slight. It is important not to overdo the drying as the solid is appreciably volatile and **TOXIC** [vapour pressure is 0.04(8°), 1.0(48°) and 66.5(100°) mm]. Also do not allow the Et₂O solns to stand too long as a brown deposit is formed which is sensitive to light, and to avoid the possibility of violent decomposition. It sinters at 90°, dec at 130°, and **EXPLODES** at 210°. [*Inorg Synth* 3 156 1950; *J Am Chem Soc* 83 2057 1961.]

Chromium potassium sulfate (12H₂O) [7788-99-0] M 499.4, pK₁²⁵ 0.74, pK₂²⁵ 6.49 (for H₂CrO₄, chromic acid). Crystd from hot water (2mL/g) by cooling.

Chromium trioxide (chromic anhydride) [1333-82-0] M 100.0, m 197°, dec at 250° to Cr₂O₃, d 2.70 (pK₁²⁵ 0.74, pK₂²⁵ 6.49, for H₂CrO₄, chromic acid). Red crystals from water (0.5mL/g) between 100° and -5°, or from water/conc HNO₃ (1:5). It separates when potassium or sodium dichromate are dissolved in conc H₂SO₄. Dried in a vacuum desiccator over NaOH pellets; *hygroscopic*, powerful oxidant, can ignite with organic compounds. It is a skin and pulmonary **IRRITANT**.

§ Commercially available on polymer support. **CANCER SUSPECT**.

Chromium (III) tris-2,4-pentanedionate [21679-31-2] M 349.3, m 216°, pK²⁵ 4.0 (see **chromic chloride**). See chromium (III) acetylacetonate on p. 412.

Chromoionophore I [ETH 5294] [9-diethylamino-5-octadecanoyl-imino-5-*H*-benzo[*a*]-phenoxazine] [125829-24-5] M 583.9. Purified by flash chromatography (Silica Gel) and eluted with EtOAc. The coloured fractions are pooled, evaporated and recrystd from EtOAc. It is a lipophilic chromoionophore and is a selectophore for K and Ca ions. [*Anal Chem* 62 738 1990.]

Chromotropic acid (4,5-dihydroxynaphthalene-2,7-disulfonic acid di-Na salt) [5808-22-0 (2H₂O)] M 400.3, m >300°, pK₁ 0.61(SO₃⁻), pK₂ 0.7(SO₃⁻), pK₃ 5.45(OH), pK₄ 15.5(OH). See disodium 4,5(1,8)-dihydroxynaphthalene-2,7(3,6)-disulfonate (2H₂O) on p. 421.

Chromyl chloride [14977-61-8] M 154.9, b 115.7°, d 1.911. Purified by distn under reduced pressure. **TOXIC**.

Claisen alkali (alkali Claisen). Prepared from KOH (35g) in H₂O (25mL) and diluted to 100mL with MeOH. **STRONGLY CAUSTIC**.

Cobalt (II) meso-5,10,15,20-tetraphenylporphine complex [14172-90-8] M 671.7. Brown crystals from Et₂O or CHCl₃-MeOH (*cf iron chloride complex*). Recrystd by extraction (Soxhlet) with *C₆H₆. Sol in most organic solvents except MeOH and pet ether. [UV, IR: *J Am Chem Soc* 70 1808 1948; 81 5111 1959.]

Cobaltous acetate (4H₂O) [6147-53-1] M 249.1, pK₁²⁵ 9.85 (for Co²⁺). Crystd several times as the tetrahydrate from 50% aqueous acetic acid. Converted to the anhydrous salt by drying at 80°/1mm for 60h.

Cobaltous acetylacetonate [14024-48-7] M 257.2, m 172°. Crystd from acetone.

Cobaltous ammonium sulfate (6H₂O) [13596-46-8] M 395.5, d 1.90. Crystd from boiling water (2mL/g) by cooling. Washed with ethanol.

Cobaltous bromide (6H₂O) [85017-77-2 (*x*H₂O); 7789-43-7 (*anhydr*)] M 326.9 (6H₂O), m 47°(dec), b 100°(dec), d 4.9. Crystd from water (1mL/g) by partial evaporation in a desiccator.

Cobaltous chloride (6H₂O) [7791-13-1 (6H₂O); 7646-79-9 (*anhydr*)] M 237.9, m 87°(dec), d 1.92. A saturated aqueous soln at room temperature was fractionally crystd by standing overnight. The first half of the material that crystd in this way was used in the next crystn. The process was repeated several times, water being removed in a dry-box using air filtered through glass wool and dried over CaCl₂ [Hutchinson *J Am Chem Soc* 76 1022 1954]. Has also been crystd from dilute aq HCl.

Cobaltous nitrate (6H₂O) [10026-22-9] M 291.0, m ~55(6H₂O), 100-105°(dec), d 1.88. Crystd from water (1mL/g), or ethanol (1mL/g), by partial evapn. After 3 crystns (H₂O) it contains: metal(ppm) As (8), Fe (1.2), K (1), Mg (4), Mn (4), Mo (4), Na (0.6), Ni (18), Zn (1.6).

Cobaltous perchlorate (6H₂O) [13478-33-6] M 365.9, pK²⁵ -2.4 to -3.1 (for HClO₄). Crystd from warm water (0.7mL/g) by cooling.

Cobaltous potassium sulfate [13596-22-0] M 329.4. Crystd from water (1mL/g) between 50° and 0°, and dried in a vacuum desiccator over conc H₂SO₄.

Cobaltous sulfate (7H₂O) [10026-24-1 (7H₂O); 60459-08-7 (*x*H₂O); 10124-43-3 (*anhydr*)] M 281.1, m 41°(dec), d 2.03. Crystd three times from conductivity water (1.3mL/g) between 100° and 0°.

Copper (I) thiophenolate [1192-40-1] M 172.7, m *ca* 280°, pK₁²⁵ 6.62 (for PhS⁻). The Cu salt can be extracted from a thimble (Soxhlet) with boiling MeOH. It is a green-brown powder which gives a yellow-green soln in pyridine. Wash with EtOH and dry in a vacuum. It can be pted from a pyridine soln by addition of H₂O, collect ppte, wash with EtOH and dry in a vacuum. [*Synthesis* 662 1974; *J Am Chem Soc* 79 170 1957; *Chem Ber* 90 425 1957.]

12-Crown-4 (lithium ionophore V, 1,4,7,10-tetraoxacyclododecane) [294-93-9] M 176.2, m 17°. The distilled crude product had to be crystd from pentane at -20° to remove acyclic material. It is then dried over P₂O₅. [*Acta Chem Scand* 27 3395 1973.]

Cupferon ammonium salt (*N*-nitroso-*N*-phenylhydroxylamine ammonium salt) [135-20-6] M 155.2, m 150-155°(dec), 162.5-163.5°, 163-164°, pK²⁵ 4.16 (free base). Recrystd twice from EtOH after treatment with Norite and finally once with EtOH. The crystals are washed with diethyl ether and air dried then stored in the dark over solid ammonium carbonate. A standard soln (*ca* 0.05M prepared in air-free H₂O) is prepared daily from this material for analytical work and is essentially 100% pure. [*Anal Chem* 26 1747 1954.] It can also be washed with Et₂O, dried and stored as stated. In a sealed, dark container it can be stored for at least 12 months without deterioration. λ_{max} 260nm (CHCl₃). [*Org Synth Coll Vol I* 177 1948; *J Am Chem Soc* 78 4206 1956.] Possible **CARCINOGEN**.

Cupric acetate (H₂O) [6046-93-1 (H₂O); 142-71-2 (*anhydr*)] M 199.7, m 115°, 240°(dec), d 1.88, pK₁²⁵ 8.0, pK₂²⁵ 13.1 (for Cu²⁺). Crystd twice from warm dilute acetic acid solns (5mL/g) by cooling.

Cupric ammonium chloride (2H₂O) [10534-87-9 (*hydr*); 15610-76-1 (*anhydr*)] M 277.5, m 110-120°(anhydr) then dec at higher temp, d 2.0. Crystd from weak aqueous HCl (1mL/g).

Cupric benzoate [533-01-7] M 305.8. Crystd from hot water.

Cupric bromide [7789-45-9] M 223.4, m 498°, b 900°, d 4.7. Crystd twice by dissolving in water (140mL/g), filtering to remove any Cu_2Br_2 , and concentrating under vac at 30° until crystals appeared. The cupric bromide was then allowed to crystallise by leaving the soln in a vac desiccator containing P_2O_5 [Hope, Otter and Prue *J Chem Soc* 5226 1960].

Cupric chloride [7447-39-4] M 134.4, m 498°, 630°(dec). Crystd from hot dilute aq HCl (0.6mL/g) by cooling in a CaCl_2 -ice bath. Dehydrated by heating on a steam-bath under vacuum. It is deliquescent in moist air but efflorescent in dry air.

Cupric lactate (H_2O) [814-81-3] M 295.7. The monohydrate crystals from hot H_2O (3mL/g) on cooling.

Cupric nitrate ($3\text{H}_2\text{O}$) [10031-43-3 ($3\text{H}_2\text{O}$); 3251-23-8 (anhydr)] M 241.6, m 114°, b 170°(dec), d 2.0. Crystd from weak aqueous HNO_3 (0.5mL/g) by cooling from room temperature. The anhydrous salt can be prepared by dissolving copper metal in a 1:1 mixture of liquid NO_2 and ethyl acetate and purified by sublimation [Evans et al. *J Chem Soc, Faraday Trans 1* 75 1023 1979]. The hexahydrate dehydr to trihydrate at 26°, and the anhydrous salt sublimates between 150 and 225°, but melts at 255-256° and is deliquescent.

Cupric oleate [1120-44-1] M 626.5. Crystd from diethyl ether.

Cupric perchlorate ($6\text{H}_2\text{O}$) [10294-46-9 (hydr); 13770-18-8] M 370.5, m 230-240°, pK^{25} -2.4 to -3.1 (for HClO_4). Crystd from distilled water. The anhydrous salt is hygroscopic.

Cupric phthalocyanine [147-14-8] M 576.1. Precipitated twice from conc H_2SO_4 by slow dilution with water. Also purified by two or three sublimations at 580° in an argon flow at 300-400Pa.

Cupric sulfate [7758-98-7] M 159.6, m >560°. After adding 0.02g of KOH to a litre of nearly saturated aq soln, it was left for two weeks, then the ppte was filtered on to a fibreglass filter with pore diameter of 5-15 microns. The filtrate was heated to 90° and allowed to evaporate until some $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ had crystd. The soln was then filtered hot and cooled rapidly to give crystals which were freed from mother liquor by filtering under suction [Geballe and Giauque *J Am Chem Soc* 74 3513 1952]. Alternatively crystd from water (0.6mL/g) between 100° and 0°.

Cupric trifluoromethylsulfonate (copper II triflate) [34946-82-2] M 361.7, pK^{25} <-3.0 (for triflic acid). Dissolve in MeCN, add dry Et_2O until cloudy and cool at -20° in a freezer. The light blue ppte is collected and dried in a vacuum oven at 130°/20mm for 8h. It has λ_{max} 737nm (ϵ 22.4M⁻¹cm⁻¹) in AcOH. [*J Am Chem Soc* 95 330 1973]. It has also been dried in a vessel at 0.1Torr by heating with a Fischer burner [*J Org Chem* 43 3422 1978]. It has been dried at 110-120°/5mm for 1h before use and forms a *benzene complex which should be handled in a dry box because it is air sensitive [*Chem Pharm Bull Jpn* 28 262 1980; *J Am Chem Soc* 95 330 1973].

Cuprous bromide [7787-70-4] M 143.4, m 497°, b 1345°, d 4.72. Purified as for cuprous iodide but using aqueous NaBr.

Cuprous bromide dimethylsulfide complex [54678-23-8] M 205.6, m ca 135°(dec). Purified by recrystn in the presence of Me_2S . A soln of the complex (1.02g) in Me_2S (5mL) is slowly diluted with hexane (20mL) and the pure colourless prisms of the complex (0.96g) separate and are collected and dried, m 124-129°dec. The complex is insoluble in hexane, Et_2O , Me_2CO , CHCl_3 and CCl_4 . It dissolves in DMF and DMSO but the soln becomes hot and green indicating dec. It dissolves in * C_6H_6 , Et_2O , MeOH and CHCl_3 if excess of Me_2S is added a colourless soln is obtained. [*J Org Chem* 40 1460 1975.] Prior to use, the complex was dissolved in Me_2S and evaporated to dryness in the weighed reaction flask [*J Organomet Chem* 228 321 1983].

Cuprous chloride [7758-89-6] M 99.0, m 430°, b~1400°. Dissolved in strong HCl, pted by dilution with water and filtered off. Washed with ethanol and diethyl ether, then dried and stored in a vacuum desiccator [Österlöf *Acta Chem Scand* 4 375 1950]. Alternatively, to an aq. soln of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ was added, with stirring,

an aqueous soln of anhydrous sodium sulfite. The colourless product was dried at 80° for 30min and stored under N₂. CuCl₂ can be purified by zone-refining [Hall et al. *J Chem Soc, Faraday Trans 1* **79** 243 1983].

Cuprous cyanide [544-92-3] **M 89.6, m 474°**. Wash thoroughly with boiling H₂O, then with EtOH. Dry at 100° to a fine soft powder. [*J Chem Soc* **79** 1943.]

Cuprous iodide [7681-65-4] **M 190.5, m 605°, b 1336°, d₄²⁵ 5.63**. It can be freshly prepared by dissolving an appropriate quantity of CuI in boiling saturated aqueous NaI over 30min. Pure CuI is obtained by cooling and diluting the soln with water, followed by filtering and washing sequentially with H₂O, EtOH, EtOAc and Et₂O, pentane, then drying *in vacuo* for 24h [Dieter, *J Am Chem Soc* **107** 4679 1985]. Alternatively wash with H₂O then EtOH and finally with Et₂O containing a little iodine. Traces of H₂O are best removed first by heating at 110° and then at 400°. Excess of I₂ is removed completely at 400°. It dissolves in Et₂O if an amine is present to form the amine complex. [*Chem Ind (London)* 1180 1957.]

Cuprous iodide trimethylphosphite [34836-53-8] **M 314.5, m 175-177°, 192-193°**. Cuprous iodide dissolves in a *C₆H₆ soln containing trimethylphosphite to form the complex. The complex crystallises from *C₆H₆ or pet ether. [*Chem Ber* **38** 1171 1905; *Bull Chem Soc Jpn* **34** 1177 1961.]

Cuprous thiocyanate [18223-42-2] **M 121.6, pK²⁵ -1.85 (for HSCN)**. Purified as for cuprous iodide but using aq NaSCN.

Cyanamide [420-04-2] **M 42.0, m 43°, 45°, 46°, b 85-87°/0.5mm, pK₁²⁰ -0.36 (1.1 at 29°), pK₂²⁰ 10.27**. Purified by placing *ca* 15g in a Soxhlet thimble and extracting exhaustively (2-3h) with two successive portions of Et₂O (400mL, saturated with H₂O by shaking before use) containing two drops of 1N acetic acid. Two successive portions of Et₂O are used so that the NH₂CN is not heated for too long. Each extract is dried over Na₂SO₄(30g), then combined and evaporated under reduced pressure. The NH₂CN may be stored unchanged at 0° in Et₂O soln in the presence of a trace of AcOH. Extracts from several runs may be combined and evaporated together. The residue from evaporation of an Et₂O soln is a colourless viscous oil which sets to a solid, and can be recrystd from a mixture of 2 parts of *C₆H₆ and 1 part of Et₂O. Concentrating an aqueous soln of NH₂CN at high temps causes **EXPLOSIVE** polymerisation. [*Org Synth Coll Vol IV* 645 1963; *Inorg Synth* **3** 39 1950; *J Org Chem* **23** 613 1958.] *Hygroscopic*.

Cyanogen bromide [506-68-3] **M 105.9, m 49-51°, b 60-62°/atm**. *All operations with this substance should be performed in a very efficient fume cupboard - it is very POISONOUS and should be handled in small amounts. Fresh commercial material is satisfactory for nearly all purposes and does not need to be purified. It is a white crystalline solid with a strong cyanide odour. If it is reddish in colour and partly liquid or paste-like then it is too far gone to be purified, and fresh material should be sought.* It can be purified by distn using small amounts at a time, and using a short wide-bore condenser because it readily solidifies to a crystalline white solid and may clog the condenser. *An appropriate gas mask should be used when transferring the molten solid from one container to another and the operation should be done in an efficient fume cupboard.* The melting point (m 49-51°) should be measured in a sealed tube. [*Org Synth Coll Vol II* 150 1948.]

Cyanogen iodide [506-78-5] **M 152.9, m 146-147°**. *This compound is POISONOUS and the precautions for cyanogen bromide (above) apply here.* The reagent (*ca* 5.9g) is dissolved in boiling CHCl₃ (15mL), filtered through a plug of glass wool into a 25mL Erlenmeyer flask. Cool to room temperature for 15min, then place in an ice-salt bath and cool to -10°. This cooling causes a small aqueous layer to separate as ice. The ice is filtered with the CNI, but melts on the filter and is also removed with the CHCl₃ used as washing liquid. The CNI which is collected on a sintered glass funnel is washed 3x with CHCl₃ (1.5mL at 0°) and freed from last traces of solvent by being placed on a watch glass and exposed to the atmosphere in a good fume cupboard at room temp for 1h to give colourless needles (*ca* 4.5g), m 146-147° (sealed capillary totally immersed in the oil bath). The yield depends slightly on the rapidity of the operation, in this way loss by sublimation can be minimised. If desired, it can be sublimed under reduced pressure at temps at which CNI is only slowly decomposed into I₂ 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, CH₂Cl₂, or dry olefin-free-*n*-pentane, the solvent being subsequently removed by storing the crystals in a vacuum desiccator containing CaCl₂.

Deuterium [7782-39-0] M 4. Passed over activated charcoal at -195° [MacIver and Tobin *J Phys Chem* 64 451 1960]. Purified by diffusion through nickel [Pratt and Rogers, *J Chem Soc, Faraday Trans I* 92 1589 1976]. Always check deuterium for radioactivity to find out the amount of tritium in it (see D₂O below).

Deuterium oxide [7789-20-0] M 20, f 3.8°/760mm, b 101.4°/760mm, d 1.105. Distd from alkaline KMnO₄ [de Giovanni and Zamenhof *Biochem J* 92 79 1963]. NOTE that D₂O invariably contains tritiated water and will therefore be RADIOACTIVE; always check the radioactivity of D₂O in a scintillation counter before using.

cis-Diamminedichloroplatinum(II) (Cisplatin) [15663-27-1] M 300.1, m 270°(dec). Recrystd from dimethylformamide and the purity checked by IR and UV-VIS spectroscopy. [Raudaschl et al. *Inorg Chim Acta* 78 143 1983.] HIGHLY TOXIC, SUSPECTED CARCINOGEN.

Diammonium hydrogen orthophosphate [7783-28-0] M 132.1. Crystd from water (1mL/g) between 70° and 0°. After one crystn of ACS grade had Fe, Mo, Na, Se and Ti at 1, 0.2, 1.4, 0.2 and 0.8ppm resp.

Di-*n*-amyl *n*-amylphosphonate [6418-56-0] M 292.4, b 150-151°/2mm, n 1.4378. Purified by three crystns of its uranyl nitrate complex from hexane (see *tributyl phosphate*). Extracts Zr²⁺ from NaCl solns.

6,6-Dibenzyl-14-crown-4 (lithium ionophore VI; 6,6-dibenzyl-1,4,8,11-tetra-oxa-cyclo-tetradecane) [106868-21-7] M 384.5, m 102-103°. Dissolve in CHCl₃, wash with saturated aqueous NaCl, dry with MgSO₄, evaporate and purify by chromatography on silica gel and gradient elution with ¹³C₆H₆-MeOH followed by preparative reverse phase HPLC on an octadecyl silanised silica (ODS) column and eluting with MeOH. It can be crystd from MeOH (ν_{CBr} 1120 cm⁻¹, C-O-C). [*J Chem Soc Perkin Trans I* 1945 1986.]

Di-*n*-butyl boron triflate (di-*n*-butylboryl trifluoromethanesulfonate) [60669-69-4] M 274.1, b 37°/0.12mm, 60°/2mm, pK²⁵ <-3.0 (for triflic acid). 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 CCl₄ at ν 1405, 1380, 1320, 1200 and 1550cm⁻¹; and ¹³C NMR (CDCl₃) with δ at 118.1, 25.1, 21.5 and 13.6. [*Org Synth* 68 83 1990; *J Am Chem Soc* 103, 3099 1981.] TOXIC

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-*tert*-butyl dichlorosilane (DTBCl₂) [18395-90-9] 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. [*J Am Chem Soc* 70 2877 1948.]

Di-*n*-butyl *n*-butylphosphonate [78-46-6] M 250.3, b 150-151°/10mm, 160-162°/20mm, n²⁵ 1.4302. Purified by three recrystallisations of its compound with uranyl nitrate, from hexane. For method, see *tributyl phosphate*.

Di-*tert*-butyl silyl bis(trifluoromethanesulfonate) [85272-31-7] M 440.5, b 73.5-74.5°/0.35mm, d 1.36 (see pK for triflic acid). 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, (CHCl₃): *tert*-Bu₂Si(OTf)₂ [δ 1.25s]; but

impurities have δ 1.12s for *tert*-Bu₂Si(H)OTf and δ 1.19s for *tert*-Bu₂HSi(Cl)OTf. [*Tetrahedron Lett* **23** 487 1982.] **TOXIC.**

Di-*n*-butyltin oxide [818-08-6] **M 248.9, m >300°.** It is prepd by hydrolysis of di-*n*-butyltin dichloride with KOH. Hence wash with a little aq M KOH then H₂O and dry at -80°/10mm until the IR is free from OH bands. [Cummings *Aust J Chem* **18** 98 1965.]

Dicarbonyl(cyclopentadienyl)Co (I) [1207-25-0] **M 180.1, b 75°/22mm, b 139-140°(dec)/710mm.** Best distd in an atmosphere of CO in a vac. The red brown liquid decomposes slightly on distn even in a vac to liberate some CO. Operations should be performed in an efficient fume cupboard. It is sol in organic solvents and stable in air but decomposes slowly in sunlight and rapidly under UV. [Piper et al. *J Inorg Nucl Chem* **1** 165 1955.] **TOXIC.**

Dichlorodimethylsilane see dimethylchlorosilane p. 419.

2,6-Dichlorophenol-indophenol sodium salt (2H₂O) [620-45-1] **M 326.1, ϵ 2.1 x 10⁴ at 600nm and pH 8, pK³⁰ 5.7 (oxidised form), pK₁³⁰ 7.0, pK₂³⁰ 10.1 (reduced form).** Dissolved in 0.001M phosphate buffer, pH 7.5 (alternatively, about 2g of the dye was dissolved in 80mL of M HCl), and extracted into diethyl ether. The extract was washed with water, extracted with aqueous 2% NaHCO₃, and the sodium salt of the dye was ppted by adding NaCl (30g/100mL of NaHCO₃ soln), then filtered off, washed with dilute NaCl soln and dried.

Dicobalt octacarbonyl [10210-68-1] **M 341.9, m 51°.** Orange-brown crystals by recrystn from *n*-hexane under a carbon monoxide atmosphere [Ojima et al. *J Am Chem Soc* **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/diethyl ether.

Di-(2-ethylhexyl)phosphoric acid ('diisooctyl' phosphate) [27215-10-7; 298-07-7] **M 322.4, b 209°/10mm, d 0.965, pK_{Est} ~1.7.** 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 HNO₃, then with saturated (NH₄)₂CO₃ soln, with 3M HNO₃, and twice with water [Petrov and Allen *Anal Chem* **33** 1303 1961]. Similarly, the impure sodium salt, after scrubbing with pet ether, was acidified with HCl and the free organic acid was extracted into pet ether and purified as above. [Peppard et al. *J Inorg Nucl Chem* **7** 231 1958] or Stewart and Crandall [*J Am Chem Soc* **73** 1377 1951]. Purified also *via* the copper salt [McDowell et al. *J Inorg Nucl Chem* **38** 2127 1976].

Diethylmethylsilane [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 Nauk SSSR Otd Khim* 1416 1957; *J Am Chem Soc* **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 δ_p -128 ± 0.5 relative to H₃PO₄. [*J Org Chem* **46** 2097 1981; *J Gen Chem USSR (Engl Transl)* **45** 231 1975.]

***N,N*-Diethyltrimethylsilylamine** [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. [*J Am Chem Soc* **68** 241 1946; *J Org Chem* **23** 50 1958; *J Prakt Chem* **9** 315 1959.]

N,N'*-Diheptyl-*N,N'*-5,5-tetramethyl-3,7-dioxanonanedi-*amide [lithium ionophore I (ETH 149)] [58821-96-8] **M 442.7**. Purified by chromatography on Kieselgel using CHCl_3 as eluent (IR ν 1640cm^{-1}). [*Helv Chim Acta* **60** 2326 1977.]

Dihexadecyl phosphate [2197-63-9] **M 546.9**, **m 75°** , **$\text{pK}_{\text{Est}} \sim 1.2$** . Crystd from MeOH [Lukac *J Am Chem Soc* **106** 4387 1984].

1,2-Dihydroxybenzene-3,5-disulfonic acid, di-Na salt (TIRON) [149-45-1] **M 332.2**, ϵ **6.9×10^4** at 260nm, **pH 10.8**, **pK_1 and $\text{pK}_2 < 2$** (for SO_3^-), **pK_3 7.7**, **pK_4 12.6** (for OHs of disulfonate dianion). Recrystd from water [Hamaguchi et al. *Anal Chim Acta* **9** 563 1962]. Indicator color reagent for Fe, Mn, Ti and Mo ions and complexes with Al, Cd, Co, Co, Fe (III), Mn, Pd, UO_2^{2+} , VO^{2+} and Zn.

Diisooctyl phenylphosphonate [49637-59-4] **M 378.5**, **n^{25} 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 *J Phys Chem* **61** 1448 1957].

Diisopropyl chlorosilane (chlorodiisopropylsilane) [2227-29-4] **M 150.7**, **b $59^\circ/8\text{mm}$** , **$80^\circ/10\text{mm}$** , **$200^\circ/738\text{mm}$** , **d 0.9008**, **n 1.4518**. Impurities can be readily detected by ^1H NMR. Purified by fractional distn [*J Am Chem Soc* **69** 1499 1947; *J Chem Soc* 3668 1957; *J Organometal Chem* **282** 175 1985].

Dilongifolyl borane [77882-24-7] **M 422.6**, **m $169-172^\circ$** . Wash with dry Et_2O and dry in a vacuum under N_2 . It has **m $160-161^\circ$** in a sealed evacuated capillary. It is sparingly soluble in pentane, tetrahydrofuran, carbon tetrachloride, dichloromethane, and chloroform, but the suspended material is capable of causing asymmetric hydroboration. Disappearance of solid indicates that the reaction has proceeded. [*J Org Chem* **46** 2988 1981.]

Dimethyl carbonate [616-38-6] **M 90.1**, **b $89.5^\circ/755\text{mm}$** , **$90.2^\circ/\text{atm}$** , **d 1.0446**, **n 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. [*J Chem Soc* **78** 1939, 1847 1948.]

Dimethyl dicarbonate (dimethyl pyrocarbonate) [4525-33-1] **M 134.1**, **m 15.2°** , **b $45-46^\circ/5\text{mm}$** , **d 1.2585**, **n 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 $123-149^\circ$. It is readily hydrolysed by H_2O and is an **IRRITANT**. [*J Gen Chem USSR* **22** 1546 1952; see also *Chem Ber* **71** 1797 1938.]

Dimethyldichlorosilane [75-78-5] **M 129.1**, **m -75.5°** , **b $68.5-68.7^\circ/750\text{mm}$** , **$70.5^\circ/760\text{mm}$** , **d 1.0885**, **n 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 p. 441). See purification of MeSiCl_2 . [*J Am Chem Soc* **70** 3590 1948.]

2,6-Dimethyl-1,10-phenanthrolinedisulfonic acid, di-Na salt (H_2O) (bathocuproine-disulfonic acid di-Na salt) [52698-84-7] **M 564.5**, **$\text{pK}_{\text{Est}} \sim 0$** (for free acid). 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.

Dimethylphenylsilyl chloride (chlorodimethylphenylsilane) [768-33-2] **M 170.7**, **b $85-87^\circ/32\text{mm}$** , **$196^\circ/\text{atm}$** , **d 1.017**, **n 1.509**. See phenyl methyl chlorosilane on p. 449.

Dinitrogen tetroxide (nitrogen dioxide, 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. **TOXIC VAPOUR.**

Diocetyl phenylphosphonate [1754-47-8] M 378.8, d 1.485, n^{25} 1.4780. Purified as described under diisooctyl phenylphosphonate.

(1,3-Dioxalan-2-ylmethyl)triphenylphosphonium bromide [52509-14-5] M 429.3, m 191.5-193°, 193-195°. Wash the crystals with Et_2O , dry in a vac and recryst from CH_2Cl_2 -dry Et_2O to give prisms m 172-174°, which is raised to 191.5-193°, on drying at 56°/0.5mm. [Cresp et al. *J Chem Soc, Perkin Trans 1* 37 1974.]

Diphenyldiselenide [1666-13-3] M 312.1, m 62-64. Crystd twice from hexane [Kice and Purkiss *J Org Chem* 52 3448 1987].

Diphenyl hydrogen phosphate [838-85-7] M 250.2, m 99.5°, pK^{20} 0.26. 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 *J Chem Soc* 3174 1959]. **TOXIC.**

4,7-Diphenyl-1,10-phenanthroline-disulfonic acid, di-Na salt $3H_2O$ (bathophenanthroline-disulfonic acid di-Na salt) [52746-49-3] M 590.6, m 300°, $pK_{Est} \sim 0$ (for free acid). Dissolve crude sample in the minimum volume of water and add EtOH to ppt the contaminants. Carefully evaporate the filtrate to obtain pure material.

It forms a dark red complex with Fe^{2+} with λ_{max} 535nm (ϵ 2.23 x 10^4 mol $^{-1}$ cm $^{-1}$) [*Anal Chim Acta* 115 407 1980]. Prepared by sulfonating bathophenanthroline with $ClSO_3H$: to 100g of bathophenanthroline was added 0.5mL of Fe free $ClSO_3H$ and heated over a flame for 30sec. Cool and carefully add 10mL of pure distd H_2O and warm on a water bath with stirring till all solid dissolved. A stock soln is made by diluting 3mL of this reagent to 100mL with 45% aq NaOAc, filter off the solid and store in a dark bottle. In this way it is stable for several months. [*Am J Clinical Pathology* 29 590 1958.]

Diphenylphosphinic acid [1707-03-5] M 218.2, m 194-195°, pK^{20} 1.72. Recrystd from 95% EtOH and dried under vacuum at room temperature. [see 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_2O , mix slowly with ice-cold 10% AcOH. The Et_2O layer is then shaken with H_2O until the washings are neutral to litmus. Dry over Na_2SO_4 , evaporate the Et_2O and distil the residual oil under reduced pressure using a Claisen flask with the take-off head modified into a short column. Ph_2SiH_2 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_2O . A possible impurity is Ph_3SiH which has m 43-45° and would be found in the residue. [*J Org Chem* 18 303 1953 ; *J Am Chem Soc* 74 6481952, 81 5925 1959.]

Diphenylsilanediol [947-42-2] M 216.3, m 148°(dec). Crystd from $CHCl_3$ -methyl ethyl ketone.

Diphenyl tolyl phosphate [26444-49-5] M 340.3, n^{25} 1.5758. Vac distd, then percolated through a column of alumina. Finally, passed through a packed column maintained at 150° to remove traces of volatile

impurities in a countercurrent stream of nitrogen at reduced pressure. [Dobry and Keller *J Phys Chem* **61** 1448 1947.]

Disodium calcium ethylenediaminetetraacetate [39208-14-5] **M 374.3**, (see pKs for EDTA in entry below). Dissolved in a small amount of water, filtered and ppted with excess EtOH. Dried at 80°.

Disodium dihydrogen ethylenediaminetetraacetic acid (2H₂O) [6381-92-6] **M 372.2**, **m 248°(dec)**, **pK₁^{2.5} 0.26**, **pK₂^{2.5} 0.96**, **pK₃^{2.5} 2.60**, **pK₄^{2.5} 2.67**, **pK₅^{2.5} 6.16**, **pK₆^{2.5} 10.26** (see EDTA Cha 4). 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 ppt is formed, filtering, and adding an equal volume of ethanol. The ppt is filtered off on a sintered-glass funnel, is washed with acetone, followed by diethyl ether, and dried in air overnight to give the dihydrate. Drying at 80° for at least 24h converts it to the anhydrous form.

Disodium 4,5(1,8)-dihydroxynaphthalene-2,7(3,6)-disulfonate (2H₂O) [5808-22-0] **M 400.3**, **m >300°**, **pK₁ 0.61(SO₃⁻)**, **pK₂ 0.7(SO₃⁻)**, **pK₃ 5.45(OH)**, **pK₄ 15.5(OH)**. Crystd from H₂O or H₂O by addition of EtOH. Complexes with Ag, ClO₃⁻, Cr, Hg, NO₂⁻, NO₃⁻ and Ti. [cf Chromotropic acid p. 413.]

Disodium ethylenebis[dithiocarbamate] [142-59-6] **M 436.5**, **pK_{Est} ~ 3.0**. Crystd (as hexahydrate) from aqueous ethanol.

Disodium-β-glycerophosphate [819-83-0 (4H₂O)] **M 216.0**, **m 102-104°**, **pK₂^{2.5} 6.66** (free acid). Crystd from water.

Disodium hydrogen orthophosphate (anhydrous) [7558-79-4] **M 142.0**, (see pK of H₃PO₄). 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**, **pK₁^{2.5} 0.26**, **pK₂^{2.5} 0.96**, **pK₃^{2.5} 2.60**, **pK₄^{2.5} 2.67**, **pK₅^{2.5} 6.16**, **pK₆^{2.5} 10.26** (see EDTA on p. 237 in Chapter 4). Dissolved in a small amount of water, filtered and ppted with an excess of MeOH. Dried at 80°.

Disodium naphthalene-1,5-disulfonate [1655-29-4] **M 332.3**, **pK_{Est} ~0**. Recrystd from aqueous acetone [Okahata et al. *J Am Chem Soc* **108** 2863 1986].

Disodium 4-nitrophenylphosphate (6H₂O) [4264-83-9] **M 371.1** Dissolve in hot aqueous MeOH, filter and ppt by adding Me₂CO. Wash the solid with Me₂CO and repeat the purification. Aq MeOH and Et₂O can also be used as solvents. The white fibrous crystals contain less than 1% of free 4-nitrophenol [assay: *J Biol Chem* **167** 57 1947].

Disodium phenylphosphate (2H₂O) [3279-54-7] **M 254.1**, **pK₁^{2.5} 1.46**, **pK₂^{2.5} 6.29** [for PhPO(OH)₂]. Dissolved in a minimum amount of methanol, filtering off an insoluble residue of inorganic phosphate, then ppted by adding an equal volume of diethyl ether. Washed with diethyl ether and dried [Tsuboi *Biochim Biophys Acta* **8** 173 1952].

Disodium succinate [150-90-3] **M 162.1**. Crystd twice from water (1.2mL/g) and dried at 125°. Freed from other metal ions by passage of a 0.1M soln through a column of Dowex resin A-1 (Na form).

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**, **n²⁵ 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 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 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 *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, pK_2^{25} 5.81, pK_3^{25} 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, pK_2^{25} 7.0, pK_3^{25} 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].