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**Rubidium perchlorate** [13510-42-4] M 184.9, d 2.80,  $pK_1^{25}$  -2.4 to -3.1 (for  $HClO_4$ ). Crystd from hot water (1.6mL/g) by cooling to 0°.

**Rubidium sulfate** [7488-54-2] M 267.0, m 1050°, d 6.31. Crystd from water (1.2mL/g) between 100° and 0°.

**Ruthenium (III) acetylacetonate** [14284-93-6] M 398.4, m 240°(dec). Purified by recrystn from \*benzene. [*J Am Chem Soc* 74 6146 1952.]

**Ruthenium (III) chloride (2H<sub>2</sub>O) (β-form)** [14898-67-0] M 207.4 + H<sub>2</sub>O, m >500°(dec), d 3.11,  $pK_1^{25}$  3.40 (for aquo  $Rh^{3+}$  hydrolysis). Dissolve in H<sub>2</sub>O, filter and concentrate to crystallise in the absence of air to avoid oxidation. Evaporate the solution in a stream of HCl gas while being heated just below its boiling point until a syrup is formed and finally to dryness at 80-100° and dried in a vacuum over H<sub>2</sub>SO<sub>4</sub>. When heated at 700° in the presence of Cl<sub>2</sub> the insoluble α-form is obtained [*Handbook of Preparative Inorganic Chemistry (Ed. Brauer)* Vol II 1598 1965; *J Org Chem* 46 3936 1981].

**Ruthenium (IV) oxide** [12036-10-1] M 133.1, d 6.97. Freed from nitrates by boiling in distilled water and filtering. A more complete purification is based on fusion in a KOH-KNO<sub>3</sub> mix to form the soluble ruthenate and perruthenate salts. The melt is dissolved in water, and filtered, then acetone is added to reduce the ruthenates to the insoluble hydrate oxide which, after making a slurry with paper pulp, is filtered and ignited in air to form the anhydrous oxide [Campbell, Ortner and Anderson *Anal Chem* 33 58 1961].

**Ruthenocene [bis-(cyclopentadienyl)ruthenium]** [1287-13-4] M 231.2, m 195.5°, 199-210°. Sublime in high vacuum at 120°. Yellow crystals which can be recrystallised from CCl<sub>4</sub> as transparent plates. [*J Am Chem Soc* 74 6146 1952].

**Samarium (II) iodide** [32248-43-4] M 404.2, m 520°, b 1580. Possible impurity is SmI<sub>3</sub> from which it is made. If present, grind solid to a powder and heat in a stream of pure H<sub>2</sub>. The temperature (~ 500-600°) should be below the m (~ 628°) of SmI<sub>3</sub>, since the molten compounds react very slowly. [Wetzel in *Handbook of Preparative Inorganic Chemistry (Ed. Brauer)* Vol II pp. 1149, 1150 1965.]

**Selenious acid** [7783-00-8] M 129.0, m 70°(dec), d 3.0,  $pK_1^{25}$  2.62,  $pK_2^{25}$  8.32 (H<sub>2</sub>SeO<sub>3</sub>). Crystd from water. On heating it loses water and SeO<sub>2</sub> sublimes.

**Selenium** [7782-49-2] M 79.0, m 217.4°, d 4.81. Dissolved in small portions in hot conc HNO<sub>3</sub> (2mL/g) filtered and evaporated to dryness to give selenious acid which was then dissolved in conc HCl. Passage of SO<sub>2</sub> into the solution pptd selenium (but not tellurium) which was filtered off and washed with conc HCl. This purification process was repeated. The selenium was then converted twice to the selenocyanate by treating with a 10% excess of 3M aqueous KCN, heating for half an hour on a sand-bath and filtering. Addition of an equal weight of crushed ice to the cold solution, followed by an excess of cold, conc HCl, with stirring (in a well ventilated fume hood because HCN is evolved) pptd selenium powder, which, after washing with water until colourless, and then with MeOH, was heated in an oven at 105°, then by fusion for 2h under vacuum. It was cooled, crushed and stored in a desiccator [Tideswell and McCullough *J Am Chem Soc* 78 3036 1956].

**Selenium dioxide** [7446-08-4] M 111.0, m 340°. Purified by sublimation, or by solution in HNO<sub>3</sub>, pptn of selenium which, after standing for several hours or boiling, is filtered off, then re-oxidised by HNO<sub>3</sub> and cautiously evaporated to dryness below 200°. The dioxide is dissolved in water and again evaporated to dryness.

**Selenopyronine** [85051-91-8] M 365.8,  $\lambda_{max}$  571nm ( $\epsilon$  81,000). Purified as the hydrochloride from hydrochloric acid [Fanghanel et al. *J Phys Chem* 91 3700 1987].

**Selenourea** [630-10-4] M 123.0, m 214-215°(dec). Recrystd from water under nitrogen.

**Silica** [7631-86-9 (*colloidal*); 112945-52-5 (*fumed*)]. Purification of silica for high technology applications uses isopiestic vapour distillation from conc volatile acids and is absorbed in high purity water. The impurities remain behind. Preliminary cleaning to remove surface contaminants uses dip etching in HF or a mixture of HCl, H<sub>2</sub>O<sub>2</sub> and deionised water [Phelan and Powell *Analyst* 109 1299 1984].

**Silica gel** [63231-67-4; 112926-00-8]. Before use as a drying agent, silica gel is heated in an oven, then cooled in a desiccator. Conditions in the literature range from heating at 110° for 15h to 250° for 2-3h. Silica gel has been purified by washing with hot acid (in one case successively with aqua regia, conc HNO<sub>3</sub>, then conc HCl; in another case digested overnight with hot conc H<sub>2</sub>SO<sub>4</sub>), followed by exhaustive washing with distilled water (one week in a Soxhlet apparatus has also been used), and prolonged oven drying. Alternatively, silica gel has been extracted with acetone until all soluble material was removed, then dried in a current of air, washed with distilled water and oven dried. Silica gel has also been washed successively with water, M HCl, water, and acetone, then activated at 110° for 15h.

**Silicon monoxide** [10097-28-6] M 44.1, m > 1700°, d 2.18. Purified by sublimation in a porcelain tube in a furnace at 1250° (4h) in a high vacuum (10<sup>-4</sup>mm) in a stream of N<sub>2</sub>. It is obtained as brownish black scales. [*Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 696 1963.]

**Silicon tetraacetate** [562-90-3] M 264.3, m 110-111°, b 148°/5-6mm, pK<sub>1</sub><sup>25</sup> 9.7, pK<sub>2</sub><sup>25</sup> 11.9 (for H<sub>4</sub>SiO<sub>4</sub> free acid). It can be crystallised from mixtures of CCl<sub>4</sub> and pet ether or Et<sub>2</sub>O, or from acetic anhydride and then dried in a vacuum desiccator over KOH. Ac<sub>2</sub>O adheres to the crystals and is removed first by drying at room temp then at 100° for several hours. It is soluble in Me<sub>2</sub>CO, is very *hygroscopic* and effervesces with H<sub>2</sub>O. It decomposes at 160-170°. [*Z Obshch Khim (Engl Transl)* 27 985 1957; *Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 701 1963.]

**Silicon tetrachloride** [10026-04-7] M 169.9, m -70°, b 57.6°, d 1.483. Distd under vacuum and stored in sealed ampoules under N<sub>2</sub>. Very sensitive to moisture.

**12-Silicotungstic acid (tungstosilicic acid; H<sub>4</sub>SiW<sub>12</sub>O<sub>40</sub>)** [12027-43-9] M 2914.5. Extracted with diethyl ether from a solution acidified with HCl. The diethyl ether was evaporated under vacuum, and the free acid was crystallised twice [Matijevic and Kerker *J Phys Chem* 62 1271 1958].

**Silver (metal)** [7440-22-4] M 107.9, m 961.9°, b 2212°, d 10.5. For purification by electrolysis, see Craig et al. [*J Res Nat Bur Stand* 64A 381 1960].

**Silver acetate** [563-63-3] M 166.9, pK<sup>25</sup> >11.1 (for aquo Ag<sup>+</sup> hydrolysis). Shaken with acetic acid for three days, the process being repeated with fresh acid, the solid then being dried in a vacuum oven at 40° for 48h. Has also been recrystallised from water containing a trace of acetic acid, and dried in air.

**Silver bromate** [7783-89-3] M 235.8, m dec on heating, d 5.21. Crystd from hot water (80mL/g).

**Silver bromide** [7785-23-1] M 187.8, m 432°, d 6.47. Purified from Fe, Mn, Ni and Zn by zone melting in a quartz vessel under vacuum.

**Silver chlorate** [7783-92-8] M 191.3, m 230°, b 270°(dec), d 4.43. Recrystd three times from water (10mL/g at 15°; 2mL/g at 80°).

**Silver chloride** [7783-90-6] M 143.3, m 455°, b 1550°, d 5.56. Recrystd from conc NH<sub>3</sub> solution.

**Silver chromate** [7784-01-2] M 331.8, d<sup>25</sup> 5.625, pK<sub>1</sub><sup>25</sup> 0.74, pK<sub>2</sub><sup>25</sup> 6.49 (for H<sub>2</sub>CrO<sub>4</sub>). Wash the red-brown powder with H<sub>2</sub>O, dry in a vacuum, then powder well and dry again in a vacuum at 90°/5h. Solubility in H<sub>2</sub>O is 0.0014% at 10°. [*J Org Chem* 42 4268 1977.]

**Silver cyanide** [506-64-9] M 133.9, m dec at 320°, d 3.95. **POISONOUS** white or grayish white powder. Stir thoroughly with H<sub>2</sub>O, filter, wash well with EtOH and dry in air in the dark. It is very insoluble in H<sub>2</sub>O (0.000023g in 100mL H<sub>2</sub>O) but is soluble in HCN or aqueous KCN to form the soluble Ag(CN)<sub>2</sub><sup>-</sup> complex. [Chem Ber 72 299 1939; J Am Chem Soc 52 184 1930.]

**Silver diethyldithiocarbamate** [1470-61-7] M 512.3, pK<sub>1</sub><sup>25</sup> 3.36 (for *N,N*-diethyldithiocarbamate). Purified by recrystn from pyridine. Stored in a desiccator in a cool and dark place.

**Silver difluoride** [7783-95-1] M 145.9, m 690°, d 4.7. Highly **TOXIC** because it liberates HF and F<sub>2</sub>. Very *hygroscopic* and reacts violently with H<sub>2</sub>O. It is a powerful oxidising agent and liberates O<sub>3</sub> from dilute acids, and I<sub>2</sub> from I<sup>-</sup> soln. Store in quartz or iron ampoules. White when pure, otherwise it is brown-tinted. Thermally stable up to 700°. [Handbook of Preparative Inorganic Chemistry (Ed. Brauer) Vol I 241 1963.]

**Silver fluoride** [7775-41-9] M 126.9, m 435°, b ca 1150°, d 5.852. *Hygroscopic* solid with a solubility of 135g/100mL of H<sub>2</sub>O at 15°, and forms an insoluble basic fluoride in moist air. Purified by washing with AcOH and dry \*C<sub>6</sub>H<sub>6</sub>, then kept in a vacuum desiccator at room temperature to remove \*benzene and stored in opaque glass bottles. Flaky *hygroscopic* crystals which darken on exposure to light. It *attacks* bone and teeth. [J Chem Soc 4538 1952; Handbook of Preparative Inorganic Chemistry (Ed. Brauer) Vol I 240 1963.]

**Silver iodate** [7783-97-3] M 282.8, m >200°, d 5.53. Washed with warm dilute HNO<sub>3</sub>, then H<sub>2</sub>O and dried at 100°, or recrystd from NH<sub>3</sub> soln by adding HNO<sub>3</sub>, filtering, washing with H<sub>2</sub>O and drying at 100°.

**Silver lactate** [128-00-7] M 196.9, m ~ 100°. Recrystd from H<sub>2</sub>O by adding EtOH. The solid was collected washed with EtOH then Et<sub>2</sub>O and dried at 80° to give the dihydrate. White powder soluble in 15 parts of H<sub>2</sub>O but only slightly soluble in EtOH. [Justus Liebigs Ann Chem 63 89 1847; Helv Chim Acta 2 251 1919.]

**Silver nitrate** [7761-88-8] M 169.9, m 212°, b 444°(dec), d 4.35. Purified by recrystn from hot water (solubility of AgNO<sub>3</sub> in water is 992g/100mL at 100° and 122g/100mL at 0°). It has also been purified by crystn from hot conductivity water by slow addition of freshly distilled EtOH.

**CAUTION: avoid using EtOH for washing the ppte; and avoid concentrating the filtrate to obtain further crops of AgNO<sub>3</sub> owing to the risk of EXPLOSION (as has been reported to us) caused by the presence of silver fulminate. When using EtOH in the purification the apparatus should be enveloped in a strong protective shield.** [Tully, News Ed (Am Chem Soc) 19 3092 1941; Garin and Henderson J Chem Educ 47 741 1970; Bretherick, Handbook of Reactive Chemical Hazards 4th edn, Butterworths, London, 1985, pp 13-14.] Before being used as a standard in volumetric analysis, analytical reagent grade AgNO<sub>3</sub> should be finely powdered, dried at 120° for 2h, then cooled in a desiccator.

Recovery of silver residues as AgNO<sub>3</sub> [use protective shield during the whole of this procedure] can be achieved by washing with hot water and adding 16M HNO<sub>3</sub> to dissolve the solid. Filter through glass wool and concentrate the filtrate on a steam bath until precipitation commences. Cool the solution in an ice-bath and filter the precipitated AgNO<sub>3</sub>. Dry at 120° for 2h, then cool in a desiccator in a vacuum. Store over P<sub>2</sub>O<sub>5</sub> in a vacuum in the dark. *AVOID contact with hands due to formation of black stains.*

**Silver nitrite** [7783-99-5] M 153.9, m 141°(dec), d 4.45. Crystd from hot conductivity water (70mL/g) in the dark. Dried in the dark under vacuum.

**Silver(I) oxide** [20667-12-3] M 231.7, m ~200°(dec), d 7.13. Leached with hot water in a Soxhlet apparatus for several hours to remove any entrained electrolytes.

**Silver (II) oxide** [1301-96-8] M 123.9, m >100°(dec), d<sup>25</sup> 7.22. Soluble in 40,000 parts of H<sub>2</sub>O, and should be protected from light. Stir with an alkaline solution of potassium peroxydisulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) at 85-

90°. The black AgO is collected, washed free from sulfate with H<sub>2</sub>O made slightly alkaline and dried in air in the dark. [*Inorg Synth* 4 12 1953.]

**Silver perchlorate (H<sub>2</sub>O)** [14242-05-8 (H<sub>2</sub>O); 7783-93-9 (anhydr)] M 207.3, pK<sup>25</sup> -2.4 to -3.1 (for HClO<sub>4</sub>). Refluxed with \*benzene (6mL/g) in a flask fitted with a Dean and Stark trap until all the water was removed azeotropically (ca 4h). The soln was cooled and diluted with dry pentane (4mL/g of AgClO<sub>4</sub>). The pptd AgClO<sub>4</sub> was filtered off and dried in a desiccator over P<sub>2</sub>O<sub>5</sub> at 1mm for 24h [Radell, Connolly and Raymond *J Am Chem Soc* 83 3958 1961]. It has also been recrystallised from perchloric acid. [Caution due to **EXPLOSIVE** nature in the presence of organic matter.]

**Silver permanganate** [7783-98-4] M 226.8, d 4.49. Violet crystals which can be crystallised from hot H<sub>2</sub>O (sol is 9g/L at 20°). Store in the dark. Oxidising agent, decomposed by light.

**Silver sulfate** [10294-26-5] M 311.8, m 652°, b 1085°(dec), d 5.45. Crystd form hot conc H<sub>2</sub>SO<sub>4</sub> contg a trace of HNO<sub>3</sub>, cooled and diluted with H<sub>2</sub>O. The ppte was filt'd off, washed and dried at 120°.

**Silver thiocyanate** [1701-93-5] M 165.9, m 265°(dec), d 3.746, pK<sup>25</sup> -1.85 (for HSCN). Digest the solid salt with aqueous NH<sub>4</sub>NCS, wash thoroughly with H<sub>2</sub>O and dry at 110° in the dark. Soluble in dilute aqueous NH<sub>3</sub>. Dissolve in strong aqueous NH<sub>4</sub>NCS solution, filter and dilute with large volume of H<sub>2</sub>O when the Ag salt separates. The solid is washed with H<sub>2</sub>O by decantation until free from NCS<sup>-</sup> ions, collected, washed with H<sub>2</sub>O, EtOH and dried in an air oven at 120°. Alternatively dissolve in dilute aqueous NH<sub>3</sub> and single crystals are formed by free evaporation of the solution in air. [*J Chem Soc* 836, 2405 1932; IR and Raman: *Acta Chem Scand* 13 1607 1957; *Acta Cryst* 10 29 1957.]

**Silver tosylate** [16836-95-6] M 279.1. The anhydrous salt is obtained by recrystn from H<sub>2</sub>O. [*Chem Ber* 12 1851 1879.]

**Silver trifluoroacetate** [2966-50-9] M 220.9, m 251-255°. Extract the salt (Soxhlet) with Et<sub>2</sub>O. The extract is filtered and evaporated to dryness, then the powdered residue is completely dried in a vacuum desiccator over silica gel. Solubility in Et<sub>2</sub>O is 33.5g in 750mL. It can be recrystd from \*C<sub>6</sub>H<sub>6</sub> (sol: 1.9g in 30mL of \*C<sub>6</sub>H<sub>6</sub>; and 33.5g will dissolve in 750mL of anhydrous Et<sub>2</sub>O). [*J Org Chem* 23 1545 1958; *J Chem Soc* 584 1951.] It is also soluble in trifluoroacetic acid (15.2% at 30°), toluene, *o*-xylene and dioxane [*J Am Chem Soc* 76 4285 1954].

**Silver trifluoromethanesulfonate** [2923-28-6] M 256.9. Recrystd twice from hot CCl<sub>4</sub> [Alo et al. *J Chem Soc, Perkin Trans 1* 805 1986].

**Sodium (metal)** [7440-23-5] M 23.0, m 97.5°, d 0.97. The metal was placed on a coarse grade of sintered-glass filter, melted under vacuum and forced through the filter using argon. The Pyrex apparatus was then re-evacuated and sealed off below the filter, so that the sodium could be distilled at 460° through a side arm and condenser into a receiver bulb which was then sealed off [Gunn and Green *J Am Chem Soc* 80 4782 1958]. **EXPLODES and IGNITES in water.**

**Sodium acetate** [127-09-3] M 82.0, m 324°, d 1.53. Crystd from acetic acid and pumped under vacuum for 10h at 120°. Alternatively, crystd from aqueous EtOH, as the trihydrate. This material can be converted to the anhydrous salt by heating slowly in a porcelain, nickel or iron dish, so that the salt liquefies. Steam is evolved and the mass again solidifies. Heating is now increased so that the salt melts again. (NB: if it is heated too strongly, the salt chars.) After several minutes, the salt is allowed to solidify and cooled to a convenient temperature before being powdered and bottled (water content should now less than 0.02%).

**Sodium acetylide** [1066-26-8] M 48.0. It disproportionates at ca 180° to sodium carbide. It sometimes contains diluents, e.g. xylene, butyl ether or dioxane which can be removed by filtration followed by a vacuum at 65-60°/5mm. Alternatively the acetylide is purged with HC≡CH at 100-125° to remove diluent. NaC<sub>2</sub>H adsorbs 2.2x, 2.0x and 1.6x its wt of xylene, butyl ether and dioxane respectively. Powdered NaC<sub>2</sub>H is yellow or yellow-gray in colour and is relatively stable. It can be heated to ca 300° in the absence of air. Although no

explosion or evolution of gas occurs, it turns brown due to disproportionation. At 170-190° in air it ignites slowly and burns smoothly. At 215-235° in air it flash-ignites and burns quickly. It can be dropped into a *slight* excess of H<sub>2</sub>O without flashing or burning but vigorous evolution of HC≡CH (**HIGHLY FLAMMABLE IN AIR**) occurs. The sample had been stored in the absence of air for one year without deterioration. Due to the high flammability of HC≡CH the salt should be stored dry, and treated with care. After long storage, NaC≡CH can be redissolved in liquid NH<sub>3</sub> and used for the same purposes as the fresh material. However it may be slightly turbid due to the presence of moisture. [*J Org Chem* **22** 649 1957; *J Am Chem Soc* **77** 5013 1955; *Inorg Synth* **2** 76, 81 1946; *Org Synth* **30** 15 1950.] See p. 89, Chapter 4 for preparation.

**Sodium alginate** [9005-38-3]. Freed from heavy metal impurities by treatment with ion-exchange resins (Na<sup>+</sup>-form), or with a dilute solution of the sodium salt of EDTA. Also dissolved in 0.1M NaCl, centrifuged and fractionally pptd by gradual addition of EtOH or 4M NaCl. The resulting gels were centrifuged off, washed with aq EtOH or acetone, and dried under vacuum. [Büchner, Cooper and Wassermann *J Chem Soc* 3974 1961.]

**Sodium *n*-alkylsulfates.** Crystd from EtOH/Me<sub>2</sub>CO [Hashimoto and Thomas *J Am Chem Soc* **107** 4655 1985].

**Sodium amide** [7782-92-5] **M 39.0, m 210°.** It reacts *violently* with H<sub>2</sub>O and is soluble in liquid NH<sub>3</sub> (1% at 20°). It should be stored in wax-sealed container in small batches. It is very *hygroscopic* and absorbs CO<sub>2</sub> and H<sub>2</sub>O. If the solid is discoloured by being yellow or brown in colour then it should be destroyed as it can be highly **EXPLOSIVE**. It should be replaced if discoloured. It is best destroyed by covering with much toluene and slowly adding dilute EtOH with stirring until all the ammonia is liberated (FUME CUPBOARD). [*Inorg Synth* **1** 74 1939; *Handbook of Preparative Inorganic Chemistry* (Ed. Brauer) Vol I 465 1963; *Org Synth Coll Vol III*, 778 1955.]

**Sodium 4-aminobenzoate** [555-06-6] **M 159.1.** Crystd from water.

**Sodium 4-aminosalicylate (2H<sub>2</sub>O)** [6018-19-5] **M 175.1.** Crystd from water at room temperature (2mL/g) by adding acetone and cooling.

**Sodium ammonium hydrogen phosphate** [13011-54-6] **M 209.1, m 79°(dec), d 1.55.** Crystd from hot water (1mL/g).

**Sodium amylpenicillin** [575-47-3] **M 350.4.** Crystd from moist acetone or moist ethyl acetate.

**Sodium anthraquinone-1,5-disulfonate (H<sub>2</sub>O)** [853-35-0] **M 412.3.** Separated from insoluble impurities by continuous extraction with water. Crystd twice from hot water and dried under vacuum.

**Sodium anthraquinone-1-sulfonate (H<sub>2</sub>O)** [107439-61-2] **M 328.3.** Crystd from hot water (4mL/g) after treatment with active charcoal, or from water by addition of EtOH. Dried under vacuum over CaCl<sub>2</sub>, or in an oven at 70°. Stored in the dark.

**Sodium anthraquinone-2-sulfonate (H<sub>2</sub>O)** [131-08-8] **M 328.3.** See 9,10-anthraquinone-2-sulfonic acid disodium salt on p. 395.

**Sodium antimonyl tartrate** [34521-09-0] **M 308.8.** Crystd from water.

**Sodium arsenate (7H<sub>2</sub>O)** [10048-95-0] **M 312.0, m 50 (loses 5H<sub>2</sub>O), m 130°, d 1.88 pK<sub>1</sub><sup>2.5</sup> 2.22, pK<sub>2</sub><sup>2.5</sup> 6.98 (for H<sub>3</sub>AsO<sub>4</sub>).** Crystd from water (2mL/g).

**Sodium azide** [26628-22-8] **M 65.0, m 300°(dec, explosive), pK<sup>2.5</sup> 4.72 (for HN<sub>3</sub>).** Crystd from hot water or from water by the addition of absolute EtOH or acetone. Also purified by repeated crystn from an aqueous solution saturated at 90° by cooling it to 10°, and adding an equal volume of EtOH. The crystals were washed with acetone and the azide dried at room temperature under vacuum for several hours in an

Abderhalden pistol. [Das et al. *J Chem Soc, Faraday Trans 1* 78 3485 1982.] **HIGHLY POISONOUS and potentially explosive.**

**Sodium barbitone (sodium 5,5-diethylbarbiturate)** [144-02-5] **M 150.1,  $pK_1^{25}$  3.99,  $pK_2^{25}$  12.5 (barbituric acid).** Crystd from water (3mL/g) by adding an equal volume of EtOH and cooling to 5°. Dried under vacuum over P<sub>2</sub>O<sub>5</sub>.

**Sodium benzenesulfinate** [873-55-2] **M 164.2, m >300°.** See benzenesulfinic acid sodium salt on p. 400.

**Sodium benzenesulfonate** [515-42-4] **M 150.1,  $pK_1^{25}$  0.70 (2.55) (for PhSO<sub>3</sub>H<sub>2</sub>).** Crystd from EtOH or aqueous 70-100% MeOH, and dried under vacuum at 80-100°.

**Sodium benzoate** [532-32-1] **M 144.1.** Crystd from EtOH (12mL/g).

**Sodium benzylpenicillin** see *N*-benzylpenicillin sodium salt on p. 514 in Chapter 6.

**Sodium bicarbonate** [144-55-8] **M 84.0, m ~50°(dec, -CO<sub>2</sub>).** Crystd from hot water (6mL/g). The solid should not be heated above 40° due to the formation of carbonate.

**Sodium bis(trimethylsilyl)amide (hexamethyl disilazane sodium salt)** [1070-89-9] **M 183.4, m 165-167°(sintering at 140°).** It can be sublimed at 170°/2 Torr (bath temp 220-250°) onto a cold finger, and can be recrystd from \*C<sub>6</sub>H<sub>6</sub> (sol: 10g in 100mL at 60°). It is slightly soluble in Et<sub>2</sub>O and is decomposed by H<sub>2</sub>O. [*Chem Ber* 94 1540 1961.]

**Sodium bisulfite** [7631-90-5] **M 104.1, d 1.48.** Crystd from hot H<sub>2</sub>O (1mL/g). Dried at 100° under vac for 4h.

**Sodium borate (borax)** [1330-43-4] **M 201.2, m 741°, d 2.37.** Most of the water of hydration was removed from the decahydrate by evacuation at 25° for three days, followed by heating to 100° and evacuation with a high-speed diffusion pump. The dried sample was then heated gradually to fusion (above 966°), allowed to cool gradually to 200°, then transferred to a desiccator containing P<sub>2</sub>O<sub>5</sub> [Grenier and Westrum *J Am Chem Soc* 78 6226 1956].

**Sodium borate (decahydrate, hydrated borax)** [1303-96-4] **M 381.2, m 75°(loses 5H<sub>2</sub>O at 60°), d 1.73.** Crystd from water (3.3mL/g) keeping below 55° to avoid formation of the pentahydrate. Filtered at the pump, washed with water and equilibrated for several days in a desiccator containing an aqueous solution saturated with respect to sucrose and NaCl. Borax can be prepared more quickly (but its water content is somewhat variable) by washing the recrystd material at the pump with water, followed by 95% EtOH, then Et<sub>2</sub>O, and air dried at room temperature for 12-18h on a clock glass.

**Sodium borohydride** [16940-66-2] **M 37.8, m ~400°(dec), d 1.07.** After adding NaBH<sub>4</sub> (10g) to freshly distilled diglyme (120mL) in a dry three-necked flask fitted with a stirrer, nitrogen inlet and outlet, the mixture was stirred for 30min at 50° until almost all of the solid had dissolved. Stirring was stopped, and, after the solid had settled, the supernatant liquid was forced under N<sub>2</sub> pressure through a sintered-glass filter into a dry flask. [The residue was centrifuged to obtain more of the solution which was added to the bulk.] The solution was cooled slowly to 0° and then decanted from the white needles that separated. The crystals were dried by pumping for 4h to give anhydrous NaBH<sub>4</sub>. Alternatively, after the filtration at 50° the solution was heated at 80° for 2h to give a white ppte of substantially anhydrous NaBH<sub>4</sub> which was collected on a sintered-glass filter under N<sub>2</sub>, then pumped at 60° for 2h [Brown, Mead and Subba Rao *J Am Chem Soc* 77 6209 1955].

NaBH<sub>4</sub> has also been crystd from isopropylamine by dissolving it in the solvent at reflux, cooling, filtering and allowing the solution to stand in a filter flask connected to a Dry-ice/acetone trap. After most of the solvent was passed over into the cold trap, crystals were removed with forceps, washed with dry diethyl ether and dried under vacuum. [Kim and Itoh *J Phys Chem* 91 126 1987.] Somewhat less pure crystals were obtained more rapidly by using Soxhlet extraction with only a small amount of solvent and extracting for about 8h. The

crystals that formed in the flask were filtered off, then washed and dried as before. [Stockmayer, Rice and Stephenson *J Am Chem Soc* **77** 1980 1955.] Other solvents used for crystallisation include water and liquid ammonia.

**Sodium bromate** [7789-38-0] **M 150.9, m 381°**, **d 3.3**. Crystd from hot water (1.1mL/g) to decrease contamination by NaBr, bromine and hypobromite. [Noszticzius et al. *J Am Chem Soc* **107** 2314 1985.]

**Sodium bromide** [7647-15-6] **M 102.9, m 747°**, **b 1390°**, **d 3.2**. Crystd from water (0.86mL/g) between 50° and 0°, and dried at 140° under vacuum (this purification may not eliminate chloride ion).

**Sodium 4-bromobenzenesulfonate** [5015-75-8] **M 258.7**. Crystd from MeOH, EtOH or distd water.

**Sodium tert-butoxide** [865-48-5] **M 96.1**. It sublimes at 180°/1 Torr. Its solubility in *tert*-BuOH is 0.208M at 30.2° and 0.382M at 60°, and is quite soluble in tetrahydrofuran (32g/100g). It should not be used if it has a brown colour. [*J Am Chem Soc* **78** 4364, 3614 1956, *Inorg Synth* **1** 87 1939; IR: *J Org Chem* **21** 156 1956.]

**Sodium butyrate** [156-54-7] **M 110.1**. Prepared by neutralisation of the acid and recrystn from EtOH.

**Sodium cacodylate (3H<sub>2</sub>O)** [124-65-2] **M 214.0, m 60°**. Crystd from aqueous EtOH.

**Sodium carbonate** [497-19-8] **M 106.0, m 858°**, **d 2.5**. Crystd from water as the decahydrate which was redissolved in water to give a near-saturated soln. By bubbling CO<sub>2</sub>, NaHCO<sub>3</sub> was pptd. It was filtered, washed and ignited for 2h at 280° [MacLaren and Swinehart *J Am Chem Soc* **73** 1822 1951]. Before being used as a volumetric standard, analytical grade material should be dried by heating at 260-270° for 0.5h and allowed to cool in a desiccator. For preparation of primary standard sodium carbonate, see *Pure Appl Chem* **25** 459 1969. After 3 recrystns tech grade had Cr, Mg, K, P, Al, W, Sc and Ti at 32, 9.4, 6.6, 3.6, 2.4, 0.6, 0.2 and 0.2 ppm resp; another technical source had Cr, Mg, Mo, P, Si, Sn and Ti at 2.6, 0.4, 4.2, 13.4, 32, 0.6, 0.8 ppm resp.

**Sodium carboxymethylcellulose** [9004-32-4]. Dialysed for 48h against distilled water.

**Sodium cetyl sulfate** [1120-01-0] **M 344.5**. See sodium hexadecylsulfate on p. 471.

**Sodium chlorate** [7775-09-9] **M 106.4, m 248°**, **b >300°(dec)**, **d 2.5**. Crystd from hot water (0.5mL/g).

**Sodium chloride** [7647-14-5] **M 58.4, m 800.7°**, **b 1413°**, **d 2.17**. Crystd from saturated aqueous solution (2.7mL/g) by passing in HCl gas, or by adding EtOH or acetone. Can be freed from bromide and iodide impurities by adding chlorine water to an aqueous solution and boiling for some time to expel free bromine and iodine. Traces of iron can be removed by prolonged boiling of solid NaCl in 6M HCl, the crystals then being washed with EtOH and dried at *ca* 100°. Sodium chloride has been purified by sublimation in a stream of pre-purified N<sub>2</sub> and collected by electrostatic discharge [Ross and Winkler *J Am Chem Soc* **76** 2637 1954]. For use as a primary analytical standard, analytical reagent grade NaCl should be finely ground, dried in an electric furnace at 500-600° in a platinum crucible, and allowed to cool in a desiccator. For most purposes, however, drying at 110-120° is satisfactory.

**Sodium chlorite** [7758-19-2] **M 90.4, m ~180°(dec)**. Crystd from hot water and stored in a cool place. Has also been crystd from MeOH by counter-current extraction with liquid ammonia [Curti and Locchi *Anal Chem* **29** 534 1957]. Major impurity is chloride ion; can be recrystallised from 0.001M NaOH.

**Sodium 4-chlorobenzenesulfonate** [5138-90-9] **M 214.6, pK<sub>Est</sub> <0 (for SO<sub>3</sub>H)**. Crystd twice from MeOH and dried under vacuum.

**Sodium 3-chloro-5-methylbenzenesulfonate** [5138-92-1] M 228.7,  $pK_{\text{Est}} < 0$  (for  $\text{SO}_3\text{H}$ ). Crystd twice from MeOH and dried under vacuum.

**Sodium chromate ( $4\text{H}_2\text{O}$ )** [10034-82-9] M 234.0, m  $\sim 20^\circ$  (for  $10\text{H}_2\text{O}$ ), d, 2.7,  $pK_1^{25} 0.74$ ,  $pK_2^{25} 6.49$  (for  $\text{H}_2\text{CrO}_4$ ). Crystd from hot water (0.8mL/g).

**dl-Sodium creatine phosphate ( $4\text{H}_2\text{O}$ )** [922-32-7] M 327.1. See creatine phosphate di-Na salt on p. 523 in Chapter 6.

**Sodium cyanate** [917-61-3] M 65.0, m  $550^\circ$ ,  $d_4^{20} 1.893$ ,  $pK^{25} 3.47$  (for HCNO). Colourless needles from EtOH. Solubility in EtOH is 0.22g/100g at  $0^\circ\text{C}$ . Soluble in  $\text{H}_2\text{O}$  but can be recrystallised from small volumes of it.

**Sodium cyanoborohydride** [25895-60-7] M 62.8, m  $240\text{-}242^\circ(\text{dec})$ ,  $d^{28} 1.20$ . Very hygroscopic solid, soluble in  $\text{H}_2\text{O}$  (212% at  $29^\circ$ , 121% at  $88^\circ$ ), tetrahydrofuran (37% at  $28^\circ$ , 42.2% at  $62^\circ$ ), very soluble in EtOH but insoluble in  $\text{Et}_2\text{O}$ ,  $^*\text{C}_6\text{H}_6$  and hexane. It is stable to acid up to pH 3 but is hydrolysed in 12N HCl. The rate of hydrolysis at pH 3 is  $10^{-8}$  that of  $\text{NaBH}_4$ . The fresh commercially available material is usually sufficiently pure. If very pure material is required one of the following procedures must be used [*Synthesis* 135 1975]: (a) The  $\text{NaBH}_3\text{CN}$  is dissolved in tetrahydrofuran (20% w/v), filtered and the filtrate is treated with a fourfold volume of  $\text{CH}_2\text{Cl}_2$ . The solid is collected and dried in a vacuum [*Inorg Chem.* 9 2146 1970].

Dissolve the  $\text{NaBH}_3\text{CN}$  in dry  $\text{MeNO}_2$ , filter, and pour the filtrate into a 10-fold volume of  $\text{CCl}_4$  with vigorous stirring. The white ppt is collected, washed several times with  $\text{CCl}_4$  and dried in a vacuum [*Inorg Chem* 9 624 1970]. (b) When the above procedures fail to give a clean product then dissolve the  $\text{NaBH}_3\text{CN}$  (10g) in tetrahydrofuran (80mL) and add N MeOH/HCl until the pH is 9. Pour the solution with stirring into dioxane (250mL). The solution is filtered, and heated to reflux. A further volume of dioxane (150mL) is added slowly with swirling. The solution is cooled slowly to room temp then chilled in ice and the crystalline dioxane complex is collected, dried in a vacuum for 4h at  $25^\circ$ , then 4h at  $80^\circ$  to yield the amorphous dioxane-free powder is 6.7g with purity  $>98\%$  [*J Am Chem Soc* 93 2897 1971]. The purity can be checked by iodometric titration [*Anal Chem* 91 4329 1969].

**Sodium p-cyrenesulfonate** [77060-21-0] M 236.3. Dissolved in water, filtered and evaporated to dryness. Crystd twice from absolute EtOH and dried at  $110^\circ$ .

**Sodium decanoate (sodium caproate)** [1002-62-6] M 194.2. Neutralised by adding a slight excess of the free acid, recovering the excess acid by  $\text{Et}_2\text{O}$  extraction. The salt is crystd from solution by adding pure acetone, repeating the steps several times, then dried in an oven at  $ca 110^\circ$  [Chaudhury and Awuwallia *Trans Faraday Soc* 77 3119 1981].

**Sodium 1-decanesulfonate** [13419-61-9] M 244.33. Recrystd from absolute EtOH and dried over silica gel.

**Sodium n-decylsulfate** [142-87-0] M 239.3. Rigorously purified by continuous  $\text{Et}_2\text{O}$  extraction of a 1% aqueous solution for two weeks.

**Sodium deoxycholate ( $\text{H}_2\text{O}$ )** [302-95-4] M 432.6,  $[\alpha]_{\text{D}}^{20} +48^\circ$  (c 1, EtOH). Crystd from EtOH and dried in an oven at  $100^\circ$ . The solution is freed from soluble components by repeated extraction with acid-washed charcoal.

**Sodium dibenzylthiocarbamate** [55310-46-8] M 295.4, m  $230^\circ(\text{dec})$ ,  $pK^{20} 3.13$  (for monobenzylthiocarbamic acid). The free acid when recrystd twice from dry  $\text{Et}_2\text{O}$  has m  $80\text{-}82^\circ$ . The Na salt is repped from aqueous EtOH or EtOH by addition of  $\text{Et}_2\text{O}$  or  $\text{Me}_2\text{CO}$  [*Anal Chem* 50 896 1978]. The  $\text{NH}_4$  salt has m  $130\text{-}133^\circ$ ; Cu salt (yellow crystals) has m  $284\text{-}286^\circ$  and the Ti salt has m  $64\text{-}70^\circ$ .

**Sodium 2,5-dichlorobenzenesulfonate** [5138-93-2] M 249.0,  $pK_{\text{Est}} < 0$  (for  $\text{SO}_3\text{H}$ ). Crystd from MeOH, and dried under vacuum.



**Sodium dichromate** [7789-12-0] M 298.0, m 84.6° (2H<sub>2</sub>O), 356° (anhydr); b 400°(dec), d<sub>4</sub><sup>25</sup> 2.348. Crystd from small volumes of H<sub>2</sub>O by evaporation to crystallisation. Solubility in H<sub>2</sub>O is 238% at 0° and 508% at boiling. Red dihydrate is slowly dehydrated by heating at 100° for long periods. It is deliquescent, a powerful oxidising agent-*do not place in contact with skin- wash immediately as it is caustic*. (Possible carcinogen).

**Sodium diethyldithiocarbamate (3H<sub>2</sub>O)** [20624-25-3] M 225.3, m 94-96°(anhydr), pK<sup>20</sup> 3.65 (diethyldithiocarbamic acid). Recrystd from water.

**Sodium di(ethylhexyl)sulfosuccinate (Aerosol-OT)** [577-11-7] M 444.6. Dissolved in MeOH and inorganic salts which ppted were filtered off. Water was added and the solution was extracted several times with hexane. The residue was evaporated to one fifth its original volume, \*benzene was added and azeotropic distillation was continued until no water remained. Solvent was then evaporated. The white solid was crushed and dried in vacuum over P<sub>2</sub>O<sub>5</sub> for 48h [El Seoud and Fendler *J Chem Soc, Faraday Trans 1* 71 452 1975].

**Sodium diethyloxaloacetate** [63277-17-8] M 210.2. Extracted several times with boiling Et<sub>2</sub>O (until the solvent remained colourless) and then the residue was dried in air.

**Sodium diformylamide** [18197-26-7] M 95.0. Grind under dry tetrahydrofuran (fumehood), filter and wash with this solvent then dry in vacuum. [IR and prepn: *Synthesis* 122 1990; *Chem Ber* 100 355 1967, 102 4089 1969.]

**Sodium dihydrogen orthophosphate (2H<sub>2</sub>O)** [13472-35-0 (2H<sub>2</sub>O); 10049-21-5 (H<sub>2</sub>O); 7558-80-7 (anhydr)] M 156.0, m 60°(dec), d 1.91. Crystd from warm water (0.5mL/g) by chilling.

**Sodium 2,2'-dihydroxy-1-naphthaleneazobenzene-5'-sulfonate** [2092-55-9] M 354.3. See Solochrome Violet R on p. 352 in Chapter 4.

**Sodium 2,4-dihydroxyphenylazobenzene-4'-sulfonate** [547-57-9] M 304.2. Crystd from absolute EtOH.

**Sodium *p*-(*p*-dimethylaminobenzeneazo)-benzenesulfonate** [23398-40-5] M 327.3. Crystd from water.

**Sodium *p*-dimethylaminoazobenzene-*o*'-carboxylate** [845-10-3] M 219.2. Ppted from aqueous soln as the free acid which was recrystallised from 95% EtOH, then reconverted to the sodium salt.

**Sodium *p*-dimethylaminoazobenzene-*p*'-carboxylate** [845-46-5] M 219.2. Ppted from aqueous soln as the free acid which was recrystallised from 95% EtOH, then reconverted to the sodium salt.

**Sodium 2,4-dimethylbenzenesulfonate** [827-21-4] M 208.2. Crystd from MeOH and dried under vacuum.

**Sodium 2,5-dimethylbenzenesulfonate** [827-19-0] M 208.2. Dissolved in distilled water, filtered, then evaporated to dryness. Crystd twice from absolute EtOH or MeOH and dried at 110° under vacuum.

**Sodium dimethyldithiocarbamate hydrate** [128-04-1] M 143.2, m 106-108°, 120-122°, pK<sup>25</sup> 3.36 (diethyldithiocarbamic acid). Crystallise from a small volume of H<sub>2</sub>O, or dissolve in minimum volume of H<sub>2</sub>O and add cold Me<sub>2</sub>CO and dry in air. The solution in Me<sub>2</sub>CO is ~50g/400mL. The dihydrate loses H<sub>2</sub>O on heating at 115° to give the hemi hydrate which decomposes on further heating [IR: *Can J Chem.* 34 1096 1956].

**Sodium *N,N*-dimethylsulfanilate** [2244-40-8] M 223.2, m >300°. Crystd from water.

**Sodium dithionite (2H<sub>2</sub>O)** [7631-94-9] M 242.1, m 110°(loses 2H<sub>2</sub>O), 267°(dec), d 2.19, pK<sub>Est(1)</sub> -3.4, pK<sub>2</sub><sup>25</sup> 0.49 (for dithionic acid). Crystd from hot water (1.1mL/g) by cooling.

**Sodium dodecanoate (sodium laurate)** [629-25-4] M 222.3, pK<sup>20</sup> 5.3 (-COOH). Neutralised by adding a slight excess of dodecanoic acid, removing it by ether extraction. The salt is recrystd from aq soln by adding pure Me<sub>2</sub>CO and repeating the process (see sodium decanoate on p. 468). Also recrystd from MeOH.

**Sodium 1-dodecanesulfonate** [2386-53-0] M 272.4. Twice recrystd from EtOH.

**Sodium dodecylbenzenesulfonate** [25155-30-0] M 348.5. Recrystd from propan-2-ol.

**Sodium dodecylsulfate (SDS, sodium laurylsulfate)** [151-21-3] M 288.4, m 204-207°. Purified by Soxhlet extraction with pet ether for 24h, followed by dissolution in acetone:MeOH:H<sub>2</sub>O 90:5:5(v/v) and recrystn [Politi et al. *J Phys Chem* **89** 2345 1985]. Also purified by two recrystns from absolute EtOH, aqueous 95% EtOH, MeOH, isopropanol or a 1:1 mixture of EtOH:isopropanol to remove dodecanol, and dried under vacuum [Ramesh and Labes *J Am Chem Soc* **109** 3228 1987]. Also purified by foaming [see Cockbain and McMullen *Trans Faraday Soc* **47** 322 1951] or by liquid-liquid extraction [see Harrold *J Colloid Sci* **15** 280 1960]. Dried over silica gel. For DNA work it should be dissolved in excess MeOH passed through an activated charcoal column and evaporated until it crystallises out. Also purified by dissolving in hot 95% EtOH (14mL/g), filtering and cooling, then drying in a vacuum desiccator. Alternatively, it was crystd from H<sub>2</sub>O, vacuum dried, washed with anhydrous Et<sub>2</sub>O, vacuum dried. These operations were repeated five times [Maritato *J Phys Chem* **89** 1341 1985; Lennox and McClelland *J Am Chem Soc* **108** 3771 1986; Dressik *J Am Chem Soc* **108** 7567 1986].

**Sodium ethoxide** [141-52-6] M 68.1. *Hygroscopic* powder which should be stored under N<sub>2</sub> in a cool place. Likely impurity is EtOH which can be removed by warming at 60-80° under high vacuum. Hydrolysed by H<sub>2</sub>O to yield NaOH and EtOH. Other impurities, if kept in air for long periods are NaOH and Na<sub>2</sub>CO<sub>3</sub>. In this case the powder cannot be used if these impurities affect the reactivity and a fresh sample should be acquired [IR: *J Org Chem* **21** 156 1956].

**Sodium ethylmercurithiosalicylate** [54-64-8] M 404.8. Crystd from ethanol-diethyl ether

**Sodium ethylsulfate** [546-74-7] M 166.1. Recrystd three times from MeOH-Et<sub>2</sub>O and vacuum dried.

**Sodium ferricyanide (H<sub>2</sub>O)** [14217-21-1; 13601-19-9 (anhydr)] M 298.9, pK<sup>25</sup> <1 (for ferricyanide). Crystd from hot water (1.5mL/g) or by precipitation from 95% EtOH.

**Sodium ferrocyanide (10H<sub>2</sub>O)** [13601-19-9] M 484.1, m 50-80° (loses 10H<sub>2</sub>O), 435°(dec), d 1.46, pK<sub>3</sub><sup>25</sup> 2.57, pK<sub>4</sub><sup>25</sup> 4.35 (for ferrocyanide). Crystd from hot water (0.7mL/g), until free of ferricyanide as shown by absence of Prussian Blue formation with ferrous sulfate soln.

**Sodium fluoride** [7681-49-4] M 42.0, m 996°, b 1695°, d 2.56. Crystd from water by partial evaporation in a vacuum desiccator, or dissolved in water, and ca half of it pptd by addition of EtOH. Ppte was dried in an air oven at 130° for one day, and then stored in a desiccator over KOH.

**Sodium fluoroacetate (mono)** [62-74-8] M 100.0, m 200-205°(dec). A free flowing white TOXIC powder which is purified by dissolving in ca 4 parts of H<sub>2</sub>O and the pH is checked. If it is alkaline, add a few drops of FCH<sub>2</sub>CO<sub>2</sub>H to make the solution just acidic. Evaporate (fumehood) on a steam bath until crystals start to separate, cool and filter the solid off. More solid can be obtained by adding EtOH to the filtrate. Dry at 100° in vacuum. [*J Chem Soc* 1778 1948.]

**Sodium fluoroborate** [13755-29-8] M 109.8, m 384°, d 2.47, pK -4.9 (for fluoroboric acid H<sub>3</sub>O<sup>+</sup>BF<sub>4</sub><sup>-</sup>). Crystd from hot water (50mL/g) by cooling to 0°. Alternatively, purified from insoluble material by dissolving in a minimum amount of water, then fluoride ion was removed by adding conc lanthanum nitrate in excess. After removing lanthanum fluoride by centrifugation, the supernatant was passed

through a cation-exchange column (Dowex 50, Na<sup>+</sup>-form) to remove any remaining lanthanum [Anbar and Guttman *J Phys Chem* **64** 1896 1960]. Also recrystd from anhydrous MeOH and dried in a vacuum at 70° for 16h. It is affected by moisture. [Delville et al. *J Am Chem Soc* **109** 7293 1987.]

**Sodium fluorosilicate** [16893-85-9] **M 188.1**. Crystd from hot water (40mL/g) by cooling.

**Sodium formaldehyde sulfoxylate dihydrate (sodium hydroxymethylsulfinate, Rongalite)** [149-44-0] **M 134.1, m 63-64° (dihydrate)**. Crystallises from H<sub>2</sub>O as the dihydrate, decomposes at higher temperatures. Store in a closed container in a cool place. It is insoluble in EtOH and Et<sub>2</sub>O and is a good reducing agent. [X-ray structure: *J Chem Soc* 3064 1955.] Note that this compound {HOCH<sub>2</sub>SO<sub>2</sub>Na} should not be confused with formaldehyde sodium bisulfite adduct {HOCH<sub>2</sub>SO<sub>3</sub>Na} from which it is prepared by reduction with Zn.

**Sodium formate (anhydrous)** [141-53-7] **M 68.0, m 253°, d 1.92**. A saturated aqueous solution at 90° (0.8mL water/g) was filtered and allowed to cool slowly. (The final temperature was above 30° to prevent formation of the hydrate.) After two such crystns the crystals were dried in an oven at 130°, then under high vacuum. [Westrum, Chang and Levitin *J Phys Chem* **64** 1553 1960; Roecker and Meyer *J Am Chem Soc* **108** 4066 1986.] The salt has also been recrystd twice from 1mM DTPA (diethylenetriaminepentaacetic acid which was recrystd 4x from MilliQ water and dried in a vac), then twice from water [Bielski and Thomas *J Am Chem Soc* **109** 7761 1987].

**Sodium D-gluconate** [527-07-1] **M 218.1, m 200-205°dec, [α]<sub>D</sub><sup>20</sup> +14°, [α]<sub>D</sub><sup>20</sup> +12 (c 20, H<sub>2</sub>O)**. Crystallise from a small volume of H<sub>2</sub>O (sol 59g/100mL at 25°), or dissolve in H<sub>2</sub>O and add EtOH since it is sparingly soluble in EtOH. Insoluble in Et<sub>2</sub>O. It forms a Cu complex in alkaline soln and a complex with Fe in neutral solution. [*J Am Chem Soc* **81** 5302 1959.]

**Sodium glycochenodeoxycholate** [16564-43-5] **M 472.6**. Dissolved in EtOH, filtered and concentrated to crystallisation, and recrystallised from a little EtOH.

**Sodium glycocholate** [863-57-0] **M 488.6**. Dissolved in EtOH, filtered and concentrated to crystallisation, and recrystallised from a little EtOH.

**Sodium glycolate (2H<sub>2</sub>O)** [2836-32-0] **M 98.0**. Ppted from aqueous solution by EtOH, and air dried.

**Sodium hexadecylsulfate** [1120-01-0] **M 344.5**. Recrystd from absolute EtOH or MeOH and dried in vac [Abu Hamdiyyah and Rahman *J Phys Chem* **91** 1531 1987].

**Sodium hexafluorophosphate** [21324-39-0] **M 167.9, pK<sub>1</sub><sup>2.5</sup>~ 0.5, pK<sub>2</sub><sup>2.5</sup> 5.12 (for fluorophosphoric acid H<sub>2</sub>PO<sub>3</sub>F)**. Recrystd from acetonitrile and vacuum dried for 2 days at room temperature. It is an irritant and is *hygroscopic*. [Delville et al. *J Am Chem Soc* **109** 7293 1987.]

**Sodium hexanitrocobaltate III (Na<sub>3</sub>[Co(NO)<sub>6</sub>])** [13600-98-1] **M 403.9**. Dissolve (ca 60g) in H<sub>2</sub>O (300mL), filter to obtain a clear solution, add 96% EtOH (250mL) with vigorous stirring. Allow the ppte to settle for 2h, filter, wash with EtOH (4 x 25mL), twice with Et<sub>2</sub>O and dry in air [*Handbook of Preparative Inorganic Chemistry (Ed. Brauer) Vol II* 1541 1965]. Yellow to brown yellow crystals which are very soluble in H<sub>2</sub>O, are decomposed by acid and form an insoluble K salt. Used for estimating K.

**Sodium hydrogen diglycollate** [50795-24-9] **M 156.1**. Crystd from hot water (7.5mL/g) by cooling to 0° with constant stirring, the crystals being filtered off on to a sintered-glass funnel and dried at 110° overnight.

**Sodium hydrogen oxalate (2H<sub>2</sub>O)** [1186-49-8] **M 130.0, m 100°(loses 2H<sub>2</sub>O), b 200°(dec)**. Crystd from hot water (5mL/g) by cooling.

**Sodium hydrogen succinate** [2922-54-5] **M 140.0**. Crystd from water and dried at 110°.

**Sodium hydrogen *d*-tartrate** [526-94-3] **M 190.1, m 100°(loses H<sub>2</sub>O), b 234°, [α]<sub>546</sub> +26° (c 1, H<sub>2</sub>O).** Crystd from warm water (10mL/g) by cooling to 0°.

**Sodium hydroxide (anhydrous)** [1310-73-2] **M 40.0, m 323°, b 1390°, d 2.13.** Common impurities are water and sodium carbonate. Sodium hydroxide can be purified by dissolving 100g in 1L of pure EtOH, filtering the solution under vacuum through a fine sintered-glass disc to remove insoluble carbonates and halides. (This and subsequent operations should be performed in a dry, CO<sub>2</sub>-free box.) The soln is concentrated under vacuum, using mild heating, to give a thick slurry of the mono-alcoholate which is transferred to a coarse sintered-glass disc and pumped free of mother liquor. After washing the crystals several times with purified alcohol to remove traces of water, they are vacuum dried, with mild heating, for about 30h to decompose the alcoholate, leaving a fine white crystalline powder [Kelly and Snyder *J Am Chem Soc* 73 4114 1951].

**Sodium hydroxide solutions (caustic), pK<sup>25</sup> 14.77.** Carbonate ion can be removed by passage through an anion-exchange column (such as Amberlite IRA-400; OH<sup>-</sup>-form). The column should be freshly prepared from the chloride form by slow prior passage of sodium hydroxide soln until the effluent gives no test for chloride ions. After use, the column can be regenerated by washing with dilute HCl, then water. Similarly, other metal ions are removed when a 1M (or more dilute) NaOH soln is passed through a column of Dowex ion-exchange A-1 resin in its Na<sup>+</sup>-form.

Alternatively, carbonate contamination can be reduced by rinsing sticks of NaOH (analytical reagent quality) rapidly with H<sub>2</sub>O, then dissolving in distilled H<sub>2</sub>O, or by preparing a concentrated aqueous soln of NaOH and drawing off the clear supernatant liquid. (Insoluble Na<sub>2</sub>CO<sub>3</sub> is left behind.) Carbonate contamination can be reduced by adding a slight excess of conc BaCl<sub>2</sub> or Ba(OH)<sub>2</sub> to a NaOH soln, shaking well and allowing the BaCO<sub>3</sub> ppte to settle. If the presence of Ba in the soln is unacceptable, an electrolytic purification can be used. For example, sodium amalgam is prepared by the electrolysis of 3L of 30% NaOH with 500mL of pure mercury for cathode, and a platinum anode, passing 15 Faradays at 4Amps, in a thick-walled polyethylene bottle. The bottle is then fitted with inlet and outlet tubes, the spent soln being flushed out by CO<sub>2</sub>-free N<sub>2</sub>. The amalgam is then washed thoroughly with a large volume of deionised water (with the electrolysis current switched on to minimize loss of Na). Finally, a clean steel rod is placed in contact in the solution with the amalgam (to facilitate hydrogen evolution), reaction being allowed to proceed until a suitable concentration is reached, before being transferred to a storage vessel and diluted as required [Marsh and Stokes *Aust J Chem* 17 740 1964].

**Sodium 2-hydroxy-4-methoxybenzophenone-5-sulfonate** [6628-37-1] **M 330.3.** Crystd from MeOH and dried under vacuum.

**Sodium *p*-hydroxyphenylazobenzene-*p*'-sulfonate** [2623-36-1] **M 288.2.** Crystd from 95% EtOH.

**Sodium hypophosphite monohydrate** [10039-56-2] **M 106.0 (see pK of hypophosphorous acid).** Dissolve in boiling EtOH, cool and add dry Et<sub>2</sub>O till all the salt separates. Collect and dry in vacuum. It is soluble in 1 part of H<sub>2</sub>O. It liberates PH<sub>3</sub> on heating and can *ignite* spontaneously when heated. The anhydrous salt is soluble in ethylene glycol (33% w/w) and propylene glycol (9.7%) at 25°.

**Sodium iodate** [7681-55-2] **M 197.9, m dec on heating, d 4.28.** Crystd from water (3mL/g) by cooling.

**Sodium iodide** [7681-82-5] **M 149.9, m 660°, b 1304°, d 3.67.** Crystd from water/ethanol soln and dried for 12h under vacuum, at 70°. Alternatively, dissolved in acetone, filtered and cooled to -20°, the resulting yellow crystals being filtered off and heated in a vacuum oven at 70° for 6h to remove acetone. The NaI was then crystd from very dilute NaOH, dried under vacuum, and stored in a vacuum desiccator [Verdin *Trans Faraday Soc* 57 484 1961].

**Sodium ionophore I (ETH 227) (*N,N,N*'-triheptyl-*N,N,N*'-trimethyl-4,4',4''-propylidynetris(3-oxabutynamide)** [61183-76-4] **M 642.0.** It is purified (*ca* 200mg) by TLC on Kieselgel F<sub>254</sub> with CHCl<sub>3</sub>/Me<sub>2</sub>CO (1:1) as solvent, followed by HPLC (50mg) with an octadecyltrimethylsilane modified column (Mercksorb SI 100, 10μm) [IR, NMR, MS: *Helv Chim Acta* 59 2417 1976].

**Sodium ionophore V (ETH 4120) [4-octadecanoyloxymethyl-*N,N,N',N'*-tetracyclohexyl-1,2-phenylenedioxydiacetamide] [129880-73-5] M 849.3.** Purified by recrystn from EtOAc. [Preparation and properties: *Anal Chim Acta* 233 295 1990].

**Sodium ionophore VI {bis[(12-crown-4)methyl]dodecyl methyl malonate} [80403-59-4] M 662.9.** Purified by gel permeation or column chromatography. [Preparation and NMR data: *J Electroanal Chem* 132 99 1982.]

**Sodium isopropylxanthate [140-93-2] M 158.2, pK 2.16 (for -S<sup>-</sup>).** Crystd from ligroin/ethanol.

**Sodium laurate [629-25-4] M 222.3.** See sodium dodecanoate on p. 470.

**Sodium *RS*-mandelate [114-21-6] M 174.1.** Crystd from 95% EtOH.

**Sodium 2-mercaptoethanesulfonate (MESNA) [19767-45-4] M 164.2, pK<sub>1</sub><sup>20</sup> < 0 (SO<sub>3</sub><sup>-</sup>), pK<sub>2</sub><sup>20</sup> 9.53 (SH).** It can be recrystd from H<sub>2</sub>O and does not melt below 250°. It can be purified further by converting to the free acid by passing a 2M soln through an ion exchange (Amberlite IR-120) column in the acid form, evaporating the eluate in a vacuum to give the acid as a viscous oil (readily dec) which can be checked by acid and SH titration. It is then dissolved in H<sub>2</sub>O, carefully neutralised with aqueous NaOH, evaporated and recrystd from H<sub>2</sub>O [*J Am Chem Soc* 77 6231 1955].

**Sodium metanilate [1126-34-7] M 195.2.** Crystd from hot water.

**Sodium metaperiodate (NaIO<sub>4</sub>) [7790-28-5] M 213.9, m ~300°(dec), d 4.17.** Crystd from hot water.

**Sodium metasilicate (5H<sub>2</sub>O) [6834-92-0] M 212.1, m 1088°, d 2.4.** Crystd from aqueous 5% NaOH solution.

**Sodium methanethiolate [sodium methylmercaptide] [5188-07-8] M 70.1, pK<sup>25</sup> 10.33 (MeS<sup>-</sup>).** Dissolve the salt (10g) in EtOH (10mL) and add Et<sub>2</sub>O (100mL). Cool and collect the ppte, wash it with Et<sub>2</sub>O and dry it in vacuum. It is a white powder which is very soluble in EtOH and H<sub>2</sub>O. [*Bull Soc Chim Fr* 3 2318 1936.]

**Sodium methoxide [124-41-4] M 54.0.** It behaves the same as sodium ethoxide. It is *hygroscopic* and is hydrolysed by moist air to NaOH and EtOH. Material that has been kept under N<sub>2</sub> should be used. If erratic results are obtained, even with recently purchased NaOMe it should be freshly prepared thus: Clean Na (37g) cut in 1-3g pieces is added in small portions to stirred MeOH (800mL) in a 2L three necked flask equipped with a stirrer and a condenser with a drying tube. After all the Na has dissolved the MeOH is removed by distillation under vacuum and the residual NaOMe is dried by heating at 150° under vacuum and kept under dry N<sub>2</sub> [*Org Synth* 39 51 1959].

**Sodium 3-methyl-1-butanesulfonate [5343-41-9] M 174.1.** Crystd from 90% MeOH.

**Sodium molybdate (2H<sub>2</sub>O) [10102-40-6] M 241.9, m 100°(loses 2H<sub>2</sub>O), 687°, d 3.28, pK<sup>25</sup> 4.08 (for H<sub>2</sub>MoO<sub>4</sub>).** Crystd from hot water (1mL/g) by cooling to 0°.

**Sodium monensin [22373-78-0] M 693.8.** Recrystd from EtOH-H<sub>2</sub>O [Cox et al. *J Am Chem Soc* 107 4297 1985].

**Sodium 1-naphthalenesulfonate [130-14-3] M 230.2.** Recrystd from water or aqueous acetone [Okadata et al. *J Am Chem Soc* 108 2863 1986].

**Sodium 2-naphthalenesulfonate [532-02-5] M 230.2.** Crystd from hot 10% aqueous NaOH or water, and dried in a steam oven.

**Sodium 2-naphthylamine-5,7-disulfonate** [79004-97-0] **M 235.4**. Crystd from water (charcoal) and dried in a steam oven.

**Sodium nitrate** [7631-99-4] **M 85.0, m 307°, b 380°, d 2.26**. Crystd from hot water (0.6mL/g) by cooling to 0°, or from concentrated aqueous solution by addition of MeOH. Dried under vacuum at 140°. After 2 recrystns tech grade had K, Mg, B, Fe Al, and Li at 100, 29, 0.6, 0.4, 0.2 and 0.2 ppm resp.

**Sodium nitrite** [7632-00-0] **M 69.0, m 271°, b 320°, d 2.17**. Crystd from hot water (0.7mL/g) by cooling to 0°, or from its own melt. Dried over P<sub>2</sub>O<sub>5</sub>.

**Sodium 1-octanesulfonate** [5324-84-5] **M 216.2**. Recrystd from absolute EtOH.

**Sodium oleate** [143-19-1] **M 304.4, m 233-235°**. Crystd from EtOH and dried in an oven at 100°.

**Sodium oxalate** [62-76-0] **M 134.0, m 250-270°(dec), d 2.34**. Crystd from hot water (16mL/g) by cooling to 0°. Before use as a volumetric standard, analytical grade quality sodium oxalate should be dried for 2h at 120° and allowed to cool in a desiccator.

**Sodium palmitate** [408-35-5] **M 278.4, m , 270°, 285-201°**. Crystd from EtOH, dried in an oven.

**Sodium perchlorate (anhydrous)** [7601-89-0] **M 122.4, m 130°(for monohydrate), d 2.02, pK<sup>25</sup> -2.4 to -3.1 (for HClO<sub>4</sub>)**. Because its solubility in water is high (2.1g/mL at 15°) and it has a rather low temperature coefficient of solubility, sodium perchlorate is usually crystd from acetone, MeOH, water-ethanol or dioxane-water (33g dissolved in 36mL of water and 200mL of dioxane). After filtering and crystallising, the solid is dried under vacuum at 140-150° to remove solvent of crystn. Basic impurities can be removed by crystn from hot acetic acid, followed by heating at 150°. If NaClO<sub>4</sub> is ppted from distilled water by adding HClO<sub>4</sub> to the chilled solution, the ppte contains some free acid. **EXPLOSIVE**

**Sodium phenol-4-sulfonate (2H<sub>2</sub>O) (4-hydroxybenzenesulfonic acid Na salt)** [825-90-1] **M 232.2**. Crystd from hot water (1mL/g) by cooling to 0°, or from MeOH, and dried in vacuum.

**Sodium phenoxide** [139-02-6] **M 116.1, m 61-64°**. Washed with Et<sub>2</sub>O, then heated under vacuum to 200° to remove any free phenol.

**Sodium phenylacetate** [114-70-5] **M 158.1**. Its aqueous solution was evaporated to crystallisation on a steam bath, the crystals being washed with absolute EtOH and dried under vacuum at 80°.

**Sodium o-phenylphenolate (4H<sub>2</sub>O)** [132-27-4] **M 264.3**. Crystd from acetone and dried under vacuum at room temperature.

**Sodium phosphoamidate** [3076-34-4] **M 119.0**. Dissolved in water below 10°, and acetic acid added dropwise to pH 4.0 so that the monosodium salt was ppted. The ppte was washed with water and Et<sub>2</sub>O, then air dried. Addition of one equivalent of NaOH to the solution gave the sodium salt, the solution being adjusted to pH 6.0 before use [Rose and Heald *Biochem J* **81** 339 1961].

**Sodium phytate (H<sub>2</sub>O) [*myo*-inositolhexakis(H<sub>2</sub>PO<sub>4</sub>) Na salt]** [14306-25-3] **M 857.9**. Crystd from water.

**Sodium piperazine-*N,N'*-bis(2-ethanesulfonate) H<sub>2</sub>O (PIPES-Na salt)** [76836-02-7] **M 364.3**. Crystd from water and EtOH.

**Sodium polyacrylate (NaPAA)** [9003-04-7]. Commercial polyacrylamide was neutralised with an aqueous solution of NaOH and the polymer ppted with acetone. The ppte was redissolved in a small amount of water and freeze-dried. The polymer was repeatedly washed with EtOH and water to remove traces of low

molecular weight material, and finally dried in vacuum at 60° [Vink *J Chem Soc, Faraday Trans 1* **75** 1207 1979]. Also dialysed overnight against distilled water, then freeze-dried.

**Sodium poly( $\alpha$ -L-glutamate).** It was washed with acetone, dried, dissolved in water and pptd with isopropanol at 5°. Impurities and low molecular weight fractions were removed by dialysis of the aqueous solution for 50h, followed by ultrafiltration through a filter impermeable to polymers of molecular weights greater the 10<sup>4</sup>. The polymer was recovered by freeze-drying. [Mori et al. *J Chem Soc, Faraday Trans 1* **2583** 1978.]

**Sodium propionate** [137-40-6] **M 96.1, m 287-289°.** Recrystd from H<sub>2</sub>O (solubility 10%), and dried by heating at 100° for 4h. Solubility of anhydrous salt in MeOH is 13% at 15° and 13.77% at 68°. It is insoluble in \*C<sub>6</sub>H<sub>6</sub> and Me<sub>2</sub>CO. [*J Chem Soc* 1341 1934.]

**Sodium pyrophosphate (10H<sub>2</sub>O)** [13472-36-1] **M 446.1, d 1.82, pK<sub>1</sub><sup>2.5</sup> 1.52, pK<sub>2</sub><sup>2.5</sup> 2.36, pK<sub>3</sub><sup>2.5</sup> 6.60, pK<sub>4</sub><sup>2.5</sup> 9.25 (for pyrophosphoric acid, H<sub>4</sub>P<sub>2</sub>O<sub>7</sub>).** Crystd from hot H<sub>2</sub>O and air dried at room temp.

**Sodium selenate** [13410-01-0] **M 188.9, pK<sub>1</sub><sup>2.5</sup> ~0, pK<sub>2</sub><sup>2.5</sup> 1.66 (for selenic acid, H<sub>2</sub>SeO<sub>4</sub>).** Crystd from water.

**Sodium selenite** [10102-18-8] **M 172.9, m >350°, pK<sub>1</sub><sup>2.5</sup> 2.62, pK<sub>2</sub><sup>2.5</sup> 8.32 (for H<sub>2</sub>SeO<sub>3</sub>).** Crystd from water.

**Sodium silicate solution** [1344-09-8] **pK<sub>1</sub><sup>2.5</sup> 9.51, pK<sub>2</sub><sup>2.5</sup> 11.77 (for silicic acid, H<sub>4</sub>SiO<sub>4</sub>)** Purified by contact filtration with activated charcoal.

**Sodium succinate** [150-90-3] **M 162.1.** See disodium succinate on p. 421.

**Sodium sulfanilate** [515-74-2] **M 195.2.** Crystd from water.

**Sodium sulfate (10H<sub>2</sub>O)** [7727-73-3 (10H<sub>2</sub>O); 7757-82-6 (anhydr)] **M 322.2, m 32°(dec), 884° (anhydr), d 2.68 (anhydr).** Crystd from water at 30° (1.1mL/g) by cooling to 0°. Sodium sulfate becomes anhydrous at 32°.

**Sodium sulfide (9H<sub>2</sub>O)** [1313-84-4 (9H<sub>2</sub>O); 1313-82-2 (anhydr)] **M 240.2, m ~50(loses H<sub>2</sub>O), 950(anhydr), d 1.43 (10H<sub>2</sub>O), 1.86 (anhydr).** Some purification of the hydrated salt can be achieved by selecting large crystals and removing the surface layer (contaminated with oxidation products) by washing with distilled water. Other metal ions can be removed from Na<sub>2</sub>S solutions by passage through a column of Dowex ion-exchange A-1 resin, Na<sup>+</sup>-form. The hydrated salt can be rendered anhydrous by heating in a stream of H<sub>2</sub> or N<sub>2</sub> until water is no longer evolved. (The resulting cake should not be heated to fusion because it is readily oxidised.) Recrystd from distilled water [Anderson and Azowlay *J Chem Soc, Dalton Trans* 469 1986].

**Sodium sulfite** [7757-83-7] **M 126.0, d 2.63.** Crystd from warm water (0.5mL/g) by cooling to 0°. Purified by repeated crystns from deoxygenated water inside a glove-box, finally drying under vacuum. [Rhee and Dasgupta *J Phys Chem* **89** 1799 1985.]

**Sodium R-tartrate (2H<sub>2</sub>O)** [6106-24-7] **M 230.1, m 120°(loses H<sub>2</sub>O), d 1.82.** Crystd from warm dilute aqueous NaOH by cooling.

**Sodium taurocholate** [145-42-6] **M 555.7.** Purified by recrystn and gel chromatography using Sephadex LH-20.

**Sodium tetradecylsulfate** [1191-50-0] **M 316.4.** Recrystd from absolute EtOH [Abu Hamdiyyah and Rahman *J Phys Chem* **91** 1531 1987].

**Sodium tetrafluoroborate** [13755-29-8] M 109.8, d 2.47,  $pK^{25}$  -4.9 (for  $\text{HBF}_4$ ). See Sodium fluoroborate on p. 470.

**Sodium tetrametaphosphate** [13396-41-3] M 429.9,  $pK_1^{25}$  2.60,  $pK_2^{25}$  6.4,  $pK_3^{25}$  8.22,  $pK_4^{25}$  11.4 (tetrametaphosphoric acid,  $\text{H}_4\text{P}_4\text{O}_{12}$ ). Crystd twice from water at room temperature by adding EtOH (300g of  $\text{Na}_4\text{P}_4\text{O}_{12}\cdot\text{H}_2\text{O}$ , 2L of water, and 1L of EtOH), washed first with 20% EtOH then with 50% EtOH and air dried [Quimby *J Phys Chem* 58 603 1954].

**Sodium tetraphenylborate [tetraphenyl boron Na]** [143-66-8] M 342.2. Dissolve in dry MeOH and add dry  $\text{Et}_2\text{O}$ . Collect the solid and dry in a vacuum at  $80^\circ/2\text{mm}$  for 4h. Also can be extracted (Soxhlet) using  $\text{CHCl}_3$  and crystallises from  $\text{CHCl}_3$  as snow white needles. It is freely sol in  $\text{H}_2\text{O}$ ,  $\text{Me}_2\text{CO}$  but insol in pet ether and  $\text{Et}_2\text{O}$ . An aqueous soln has pH ~ 5 and can be stored for days at  $25^\circ$  or lower, and for 5 days at  $45^\circ$  without deterioration. Its solubility in polar solvents increases with decrease in temp [Justus Liebigs Ann Chem 574 195 1950]. The salt can also be recrystd from acetone-hexane or  $\text{CHCl}_3$ , or from  $\text{Et}_2\text{O}$ -cyclohexane (3:2) by warming the soln to ppt the compound. Dried in a vacuum at  $80^\circ$ . Dissolved in  $\text{Me}_2\text{CO}$  and added to an excess of toluene. After a slight milkiness developed on standing, the mixture was filtered. The clear filtrate was evaporated at room temperature to a small bulk and again filtered. The filtrate was then warmed to  $50\text{-}60^\circ$ , giving clear dissolution of crystals. After standing at this temperature for 10min the mixture was filtered rapidly through a pre-heated Büchner funnel, and the crystals were collected and dried in a vacuum desiccator at room temperature for 3 days [Abraham et al. *J Chem Soc, Faraday Trans 1* 80 489 1984]. If the product gives a turbid aq solution, the turbidity can be removed by treating with freshly prepared alumina gel.

**Sodium thioantimonate ( $\text{Na}_3\text{SbS}_4\cdot 9\text{H}_2\text{O}$ , Schlippe's salt)** [13776-84-6] M 481.1, m  $87^\circ$ , b  $234^\circ$ , d 1.81. Crystd from warm water (2mL/g) by cooling to  $0^\circ$ .

**Sodium thiocyanate** [540-72-7] M 81.1, m  $300^\circ$ ,  $pK^{25}$  -1.85 (for HSCN). It is recrystd from EtOH or  $\text{Me}_2\text{CO}$  and the mother liquor is removed from the crystals by centrifugation. It is very deliquescent and should be kept in an oven at  $130^\circ$  before use. It can be dried in vacuum at  $120^\circ/\text{P}_2\text{O}_5$  [*Trans Faraday Soc* 30 1104 1934]. Its solubility in  $\text{H}_2\text{O}$  is 113% at  $10^\circ$ , 178% at  $46^\circ$ , 225.6% at  $101.4^\circ$ ; in MeOH 35% at  $15.8^\circ$ , 51% at  $48^\circ$ , 53.5% at  $52.3^\circ$ ; in EtOH 18.4% at  $18.8^\circ$ , 24.4% at  $70.9^\circ$ ; and in  $\text{Me}_2\text{CO}$  6.85% at  $18.8^\circ$  and 21.4% at  $56^\circ$  [*J Chem Soc* 2282 1929].

Sodium thiocyanate has also been recrystd from water, acetonitrile or from MeOH using  $\text{Et}_2\text{O}$  for washing, then dried at  $130^\circ$ , or dried under vacuum at  $60^\circ$  for 2 days. [Strasser et al. *J Am Chem Soc* 107 789 1985; Szezygiel et al. *J Am Chem Soc* 91 1252 1987.] (The latter purification removes material reacting with iodine.) Sodium thiocyanate solns can be freed from traces of iron by repeated batch extractions with  $\text{Et}_2\text{O}$ .

**Sodium thioglycolate** [367-51-1] M 114.1. Crystd from 60% EtOH (charcoal). Hygroscopic.

**Sodium thiosulfate ( $5\text{H}_2\text{O}$ )** [10102-17-7 (hydr); 7772-98-7 (anhydr)] M 248.2(anhydr), m 48(rapid heat), d 1.69,  $pK_1^{25}$  0.6,  $pK_2^{25}$  1.74 (for  $\text{H}_2\text{S}_2\text{O}_3$ ) Crystd from EtOH- $\text{H}_2\text{O}$  solns or from water (0.3mL/g) below  $60^\circ$  by cooling to  $0^\circ$ , and dried at  $35^\circ$  over  $\text{P}_2\text{O}_5$  under vacuum.

**Sodium *p*-toluenesulfinate** [824-79-3] M 178.2,  $pK^{25}$  2.80 (1.99)(for  $-\text{SO}_2^-$ ). Crystd from water (to constant UV spectrum), and dried under vacuum or extracted with hot \*benzene, then dissolved in EtOH- $\text{H}_2\text{O}$  and heated with decolorising charcoal. The solution was filtered and cooled to give crystals of the dihydrate.

**Sodium *p*-toluenesulfonate** [657-84-1] M 194.2,  $pK^{25}$  -1.34 (for  $-\text{SO}_3^-$ ). Dissolved in distilled water, filtered to remove insoluble impurities and evaporated to dryness. Then crystd from MeOH or EtOH, and dried at  $110^\circ$ . Its solubility in EtOH is not high (maximum 2.5%) so that Soxhlet extraction with EtOH may be preferable. Sodium *p*-toluenesulfonate has also been crystd from  $\text{Et}_2\text{O}$  and dried under vacuum at  $50^\circ$ .

**Sodium trifluoroacetate** [2923-18-4] M 136.0, m  $206\text{-}210^\circ(\text{dec})$ ,  $pK^{25}$  0.52 (for  $\text{CF}_3\text{CO}_2^-$ ). A possible contaminant is NaCl. The solid is treated with  $\text{CF}_3\text{CO}_2\text{H}$  and evaporated twice. Its solubility in  $\text{CF}_3\text{CO}_2\text{H}$  is 13.1% at  $29.8^\circ$ . The residue is crystd from dil EtOH and the solid dried in vacuum at  $100^\circ$ . [*J*



*Am Chem Soc* 76 4285 1954.] It can be pptd from EtOH by adding dioxane, then crystd several times from hot absolute EtOH. Dried at 120-130°/1mm.

**Sodium 2,2',4-trihydroxyazobenzene-5'-sulfonate** [3564-26-9] M 295.3. Purified by precipitating the free acid from aqueous solution using concentrated HCl, then washing and extracting with EtOH in a Soxhlet extractor. Evaporation of the EtOH left the purified acid.

**Sodium trimetaphosphate (6H<sub>2</sub>O)** [7785-84-4] M 320.2, m 53°, d 1.79, pK<sub>2</sub><sup>2.5</sup> 1.64, pK<sub>3</sub><sup>2.5</sup> 2.07 (for trimetaphosphoric acid, H<sub>3</sub>P<sub>3</sub>O<sub>9</sub>). Pptd from an aq soln at 40° by adding EtOH. Air dried.

**Sodium 2,4,6-trimethylbenzenesulfonate** [6148-75-0] M 222.1. Crystd twice from MeOH and dried under vacuum.

**Sodium trimethylsilanolate (sodium trimethylsilanol)** [18027-10-6] M 112.2, m 230°(dec). It is very soluble in Et<sub>2</sub>O and \*C<sub>6</sub>H<sub>6</sub> but moderately soluble in pet ether. It is purified by sublimation at 130-150° in a high vacuum. [IR: *J Am Chem Soc* 75 5615 1953; *J Org Chem* 17 1555 1952.]

**Sodium tripolyphosphate** [7758-29-4] M 367.9, pK<sub>1</sub><sup>2.5</sup> ~ 1, pK<sub>2</sub><sup>2.5</sup> 2.0, pK<sub>3</sub><sup>2.5</sup> 2.13, pK<sub>4</sub><sup>2.5</sup> 5.78, pK<sub>5</sub><sup>2.5</sup> 8.56 (for tripolyphosphoric acid, H<sub>5</sub>P<sub>3</sub>O<sub>10</sub>). Purified by repeated pptn from aqueous solution by slow addition of MeOH and air dried. Also a solution of anhydrous sodium tripolyphosphate (840g) in water (3.8L) was filtered, MeOH (1.4L) was added with vigorous stirring to ppte Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>·6H<sub>2</sub>O. The ppte was collected on a filter, air dried by suction, then left to dry in air overnight. It was crystd twice more in this way, using a 13% aqueous solution (w/w), and leaching the crystals with 200mL portions of water [Watters, Loughran and Lambert *J Am Chem Soc* 78 4855 1956]. Similarly, EtOH can be added to ppte the salt from a filtered 12-15% aqueous solution, the final solution containing ca 25% EtOH (v/v). Air drying should be at a relative humidity of 40-60%. Heat and vac drying should be avoided. [Quimby *J Phys Chem* 58 603 1954.]

**Sodium tungstate (2H<sub>2</sub>O)** [10213-10-2] M 329.9, m 698°, d 4.18, pK<sub>1</sub><sup>2.5</sup> 2.20, pK<sub>2</sub><sup>2.5</sup> 3.70 (for tungstic acid, H<sub>2</sub>WO<sub>4</sub>). Crystd from hot water (0.8mL/g) by cooling to 0°.

**Sodium m-xylenesulfonate** [30587-85-0] M 208.2. Dissolved in distilled water, filtered, then evaporated to dryness. Crystd twice from absolute EtOH and dried at 110°.

**Sodium p-xylenesulfonate** [827-19-0] M 208.2. See sodium 2,5-dimethylbenzenesulfonate on p. 469.

**Stannic chloride (tin IV chloride, stannic tetrachloride)** [7646-78-8] M 260.5, m -33°, -30.2°, b 114°/760mm, d 2.23, pK<sup>2.5</sup> 14.15 (for aquo Sn<sup>4+</sup> hydrolysis). Fumes in moist air due to hydrate formation. Fractionate in a ground glass still and store in the absence of air. Possible impurities are SO<sub>2</sub> and HCl [Baudler *Handbook of Preparative Inorganic Chemistry (Ed. Brauer)* Vol I p. 729 1963]. It forms a solid pentahydrate [10026-06-9] which smells of HCl and is formed when the anhydrous salt is dissolved in a small vol of H<sub>2</sub>O. Also refluxed with clean mercury or P<sub>2</sub>O<sub>5</sub> for several hours, then distd under (reduced) N<sub>2</sub> pressure into a receiver containing P<sub>2</sub>O<sub>5</sub>. Finally redistd. Alternatively, distd from Sn metal under vacuum in an all-glass system and sealed off in large ampoules. Fumes in moist air. SnCl<sub>4</sub> is available commercially as 1M solns in CH<sub>2</sub>Cl<sub>2</sub> or hexane. **HARMFUL VAPOURS.**

**Stannic iodide (SnI<sub>4</sub>)** [7790-47-8] M 626.3, m 144°, b 340, d 4.46. Crystd from anhydrous CHCl<sub>3</sub>, dried under vacuum and stored in a vacuum desiccator. Sublimes at 180°.

**Stannic oxide (SnO<sub>2</sub>)** [18282-10-5] M 150.7, m 1630°, d 6.95. Refluxed repeatedly with fresh HCl until the acid showed no tinge of yellow. The oxide was then dried at 110°.

**Stannous bis-cyclopentadienyl** [26078-96-6] M 248.9. Purified by vacuum sublimation. Handled and stored under dry N<sub>2</sub>. The related thallium and indium compounds are similarly prepared.

**Stannous chloride (anhydrous)** [7772-99-8] M 189.6, m 247°, b 606°, d 3.95,  $pK_1^{25}$  1.7,  $pK_2^{25}$  3.7 (for aquo  $\text{Sn}^{2+}$  hydrolysis). Analytical reagent grade stannous chloride dihydrate is dehydrated by adding slowly to vigorously stirred, redistilled acetic anhydride (120g salt per 100g of anhydride). (In a fume cupboard.) After *ca* an hour, the anhydrous  $\text{SnCl}_2$  is filtered on to a sintered-glass or Büchner funnel, washed free from acetic acid with dry  $\text{Et}_2\text{O}$  (2 x 30mL), and dried under vacuum. It is stored in a sealed container. [Stephen *J Chem Soc* 2786 1930].

**Strontium acetate** [543-94-2] M 205.7, d 2.1,  $pK^{25}$  13.0 (for aquo  $\text{Sr}^{2+}$  hydrolysis). Crystd from AcOH, then dried under vacuum for 24h at 100°.

**Strontium bromide** [10476-81-0] M 247.4, m 643°, d 4.22. Crystd from water (0.5mL/g).

**Strontium chloride (6H<sub>2</sub>O)** [1025-70-4] M 266.6, m 61°(rapid heating), 114-150°(loses 5H<sub>2</sub>O), 868°(anhydr). Crystd from warm water (0.5mL/g) by cooling to 0°.

**Strontium chromate** [7789-06-2] M 203.6, d 3.9,  $pK_1^{25}$  0.74,  $pK_2^{25}$  6.49 (for  $\text{H}_2\text{CrO}_4$ ). Crystd from water (40mL/g) by cooling.

**Strontium hydroxide (8H<sub>2</sub>O)** [1311-10-0 (8H<sub>2</sub>O); 18480-07-4 (anhydr)] M 265.8, m 100°(loses H<sub>2</sub>O), d 1.90, m 375(anhydr), d 3.63 (anhydr). Crystd from hot water (2.2mL/g) by cooling to 0°.

**Strontium lactate (3H<sub>2</sub>O)** [29870-99-3] M 319.8, m 120°(loses 3H<sub>2</sub>O). Crystd from aq EtOH.

**Strontium nitrate** [10042-76-9] M 211.6, m 570°, b 645°, d 2.99. Crystd from hot water (0.5mL/g) by cooling to 0°.

**Strontium oxalate (H<sub>2</sub>O)** [814-95-9] M 193.6, m 150°. Crystd from hot water (20mL/g) by cooling.

**Strontium salicylate** [526-26-1] M 224.7. Crystd from hot water (4mL/g) or EtOH.

**Strontium tartrate** [868-19-9] M 237.7. Crystd from hot water.

**Strontium thiosalicylate (5H<sub>2</sub>O)** [15123-90-7] M 289.8. Crystd from hot water (2mL/g) by cooling to 0°.

**Sulfamic acid** [5329-14-6] M 97.1, m 205°(dec),  $pK^{25}$  0.99 ( $\text{NH}_2\text{SO}_3\text{H}$ ). Crystd from water at 70° (300mL per 25g), after filtering, by cooling a little and discarding the first batch of crystals (about 25g) before standing in an ice-salt mixture for 20min. The crystals were filtered by suction, washed with a small quantity of ice water, then twice with cold EtOH and finally with  $\text{Et}_2\text{O}$ . Air dried for 1h, then stored in a desiccator over  $\text{Mg}(\text{ClO}_4)_2$  [Butler, Smith and Audrieth *Ind Eng Chem (Anal Ed)* 10 690 1938]. For preparation of primary standard material see *Pure Appl Chem* 25 459 1969.

**Sulfamide** [7803-58-9] M 96.1, m 91.5°. Crystd from absolute EtOH.

**Sulfur** [7704-34-9] M 32.1, m between 112.8° and 120°, depending on form. Murphy, Clabaugh and Gilchrist [*J Res Nat Bur Stand* 64A 355 1960] have obtained sulfur of about 99.999% purity by the following procedure: Roll sulfur was melted and filtered through a coarse-porosity glass filter funnel into a 2L round-bottomed Pyrex flask with two necks. Conc  $\text{H}_2\text{SO}_4$  (300mL) was added to the sulfur (2.5Kg), and the mixture was heated to 150°, stirring continuously for 2h. Over the next 6h, conc  $\text{HNO}_3$  was added in about 2mL portions at 10-15min intervals to the heated mixture. It was then allowed to cool to room temperature and the acid was poured off. The sulfur was rinsed several times with distilled water, then remelted, cooled, and rinsed several times with distd water again, this process being repeated four or five times to remove most of the acid entrapped in the sulfur. An air-cooled reflux tube (*ca* 40cm long) was attached to one of the necks of the flask, and a gas delivery tube (the lower end about 1in above the bottom of the flask) was inserted into the other. While the sulfur was boiled under reflux, a stream of helium or  $\text{N}_2$  was passed through to remove any

water,  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ , as vapour. After 4h, the sulfur was cooled so that the reflux tube could be replaced by a bent air-cooled condenser. The sulfur was then distilled, rejecting the first and the final 100mL portions, and transferred in 200mL portions to 400mL glass cylinder ampoules (which were placed on their sides during solidification). After adding about 80mL of water, displacing the air with  $\text{N}_2$ , and sealing the ampoule was cooled, and the water was titrated with 0.02M NaOH, the process being repeated until the acid content was negligible. Finally, entrapped water was removed by alternate evacuation to 10mm Hg and refilling with  $\text{N}_2$  while the sulfur was kept molten. Other purifications include crystn from  $\text{CS}_2$  (which is less satisfactory because the sulfur retains appreciable amounts of organic material), \*benzene or \*benzene/acetone, followed by melting and degassing. Has also been boiled with 1% MgO, then decanted, and dried under vacuum at  $40^\circ$  for 2 days over  $\text{P}_2\text{O}_5$ . [For purification of  $\text{S}_6$ , "recryst.  $\text{S}_8$ " and "Bacon-Fanelli sulfur" see Bartlett, Cox and Davis *J Am Chem Soc* **83** 103, 109 1961.]

**Sulfur dichloride** [10545-99-0] M 103.0, m  $-78^\circ$ , b  $59^\circ/760\text{mm}(\text{dec})$ , d 1.621. Twice distilled in the presence of a small amount of  $\text{PCl}_3$  through a 12in Vigreux column, the fraction boiling between  $55-61^\circ$  being redistd (in the presence of  $\text{PCl}_3$ ), and the fraction distilling between  $58-61^\circ$  retained. (The  $\text{PCl}_3$  is added to inhibit the decomposition of  $\text{SCl}_2$  into  $\text{S}_2\text{Cl}_2$  and  $\text{Cl}_2$ ). The  $\text{SCl}_2$  must be used as quickly as possible after distn, within 1h at room temperature, The sample contains 4%  $\text{S}_2\text{Cl}_2$ . On long standing this reaches 16-18%. **HARMFUL VAPOURS.**

**Sulfur dioxide** [7446-09-5] M 64.1, b  $-10^\circ$ . Dried by bubbling through concentrated  $\text{H}_2\text{SO}_4$  and by passage over  $\text{P}_2\text{O}_5$ , then passed through a glass-wool plug. Frozen with liquid air and pumped to a high vacuum to remove dissolved gases. **HARMFUL VAPOURS.**

**Sulfuric acid** [7664-93-9] M 98.1, d 1.83,  $\text{pK}_1^{25} \sim -8.3$ ,  $\text{pK}_2^{25} 1.99$ . Sulfuric acid, and also 30% fuming  $\text{H}_2\text{SO}_4$ , can be distilled in an all-Pyrex system, optionally from potassium persulfate. Also purified by fractional crystn of the monohydrate from the liquid. Dehydrates and attacks skin—wash immediately with  $\text{H}_2\text{O}$ .

**Sulfur monochloride (sulfur monochloride)** [10025-67-9] M 135.0, m  $-77^\circ$ ; b  $19.1^\circ$ ,  $29-30^\circ/12\text{mm}$ ,  $72^\circ/100\text{mm}$ ,  $138^\circ/760\text{mm}$ ,  $d^{20} 1.677$ ,  $n_D^{20} 1.67$ . Pungent, irritating golden yellow liquid. When impure its colour is orange to red due to  $\text{SCl}_2$  formed. It fumes in moist air and liberates HCl,  $\text{SO}_2$  and  $\text{H}_2\text{S}$  in the presence of  $\text{H}_2\text{O}$ . Distil and collect the fraction boiling above  $137^\circ$  at atmospheric pressure. Fractionate this fraction over sulfur at ca 12mm using ground glass apparatus (b  $29-30^\circ$ ). Alternatively purify by distn below  $60^\circ$  from a mixture containing sulfur (2%) and activated charcoal (1%), under reduced pressure (e.g. 50mm). It is soluble in EtOH, \* $\text{C}_6\text{H}_6$ ,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$  and  $\text{CCl}_4$ . Store in a closed container in the dark in a refrigerator. [*Handbook of Preparative Inorganic Chemistry (Ed. Brauer) Vol I* 371 1963.] **HARMFUL VAPOURS.**

**Sulfur trioxide pyridine complex** [26412-87-3] M 159.2, m  $155-165^\circ$ ,  $175^\circ$ . Wash the solid with a little  $\text{CCl}_4$ , then  $\text{H}_2\text{O}$  to remove traces of pyridine sulfate, and dry over  $\text{P}_2\text{O}_5$  [*Chem Ber* **59** 1166 1926; *Synthesis* **59** 1979].

**Sulfuryl chloride** [7791-25-2] M 135.0, m  $-54.1^\circ$ , b  $69.3^\circ/760\text{mm}$ ,  $d_4^{20} 1.67$ ,  $n_D^{30} 1.44$ . Pungent, irritating colourless liquid. It becomes yellow with time due to decomposition to  $\text{SO}_2$  and HCl. Distil and collect fraction boiling below  $75^\circ/\text{atm}$  which is mainly  $\text{SO}_2\text{Cl}_2$ . To remove  $\text{HSO}_3\text{Cl}$  and  $\text{H}_2\text{SO}_4$  impurities, the distillate is poured into a separating funnel filled with crushed ice and briefly shaken. The lower cloudy layer is removed, dried for some time in a desiccator over  $\text{P}_2\text{O}_5$  and finally fractionated at atmospheric pressure. The middle fraction boils at  $69-70^\circ$  and is pure  $\text{SO}_2\text{Cl}_2$ . It decomposes gradually in  $\text{H}_2\text{O}$  to  $\text{H}_2\text{SO}_4$  and HCl. Reacts violently with EtOH and MeOH and is soluble in \* $\text{C}_6\text{H}_6$ , toluene  $\text{Et}_2\text{O}$  and acetic acid. [*Handbook of Preparative Inorganic Chemistry (Ed Brauer) Vol I* 383 1963; *Inorg Synth* **1** 114 1939]. **HARMFUL VAPOURS.**

**Tantalum (V) chloride (tantalum pentachloride)** [7721-01-9] M 358.2, m  $216.2^\circ$ ,  $216.5-220^\circ$ ; b  $239^\circ/\text{atm.}$ , d 3.68. Purified by sublimation in a stream of  $\text{Cl}_2$ . Colourless