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under reduced pressure through a column packed with stainless steel helices. Again dried with molecular sieves and distd. [Cram et al. *J Am Chem Soc* **83** 3678 1961; Coetzee *Pure Appl Chem* **49** 211 1977.]

Also, it was stirred at 50° and small portions of solid KMnO₄ were added until the colour persisted during 1h. Dropwise addition of MeOH then destroyed the excess KMnO₄, the soln was filtered, freed from potassium ions by passage through an ion-exchange column and dried under vacuum. It has also been vacuum distd from KOH pellets. It is *hygroscopic*. [See Sacco et al. *J Phys Chem* **80** 749 1976; *J Chem Soc, Faraday Trans 1* **73** 1936 1977; **74** 2070 1978; *Trans Faraday Soc* **62** 2738 1966.] Coetzee has reviewed the methods of purification of sulfolane, and also the removal of impurities. [Coetzee in *Recommended Methods of Purification of Solvents and Tests for Impurities*, Coetzee Ed. Pergamon Press, 1982.]

5-Sulfosalicylic acid [5965-83-3] M 254.2, m 108-110°, pK₁²⁵ <0, pK₂²⁵ 2.67, pK₃²⁵ 11.67. Crystd from water. Alternatively, it was converted to the monosodium salt which was crystd from water and washed with a little water, EtOH and then diethyl ether. The free acid was recovered by acidification.

Syringaldehyde (3,5-dimethoxy-4-hydroxybenzaldehyde) [134-96-3] M 182.2, m 113°, pK_{Est} ~8. Crystd from pet ether.

Syringic acid (3,5-dimethoxy-4-hydroxybenzoic acid) [530-57-4] M 198.2, m 204-205°, 206.5°, 206-209°, 209-210°, pK₁²⁵ 4.34, pK₂²⁵ 9.49. Recrystd from H₂O using charcoal [Bogert and Coyne *J Am Chem Soc* **51** 571 1929; Anderson and Nabenhauer *J Am Chem Soc* **48** 3001 1926.] The *methyl ester* has m 107° (from MeOH), the *4-acetyl* derivative has m 190° and the *4-benzoyl* derivative has m 229-232°. [Hahn and Wassmuth *Chem Ber* **67** 2050 1934; UV: Lemon *J Am Chem Soc* **69** 2998 1947 and Pearl and Beyer *J Am Chem Soc* **72** 1743 1950.]

D(-)-Tagatose [87-81-0] M 180.2, m 134-135°, [α]₅₄₆ -6.5° (c 1, H₂O). Crystd from aqueous EtOH.

d- Tartaric acid [147-71-7] M 150.1, m 169.5-170° (2*S*,3*S*-form, natural) [α]₅₄₆²⁰ -15° (c 10, H₂O); m 208° (2*R**S*,3*R**S*-form), pK₁²⁵ 3.03, pK₂²⁵ 4.46, pK₃²⁵ 14.4. Crystd from distilled H₂O or *benzene/diethyl ether containing 5% of pet ether (b 60-80°) (1:1). Soxhlet extraction with diethyl ether has been used to remove an impurity absorbing at 265nm. It has also been crystd from absolute EtOH/hexane, and dried in a vacuum for 18h [Kornblum and Wade *J Org Chem* **52** 5301 1987].

meso-Tartaric acid [147-73-9] M 150.1, m 139-141°, pK₁²⁵ 3.17, pK₂²⁵ 4.91. Crystd from water, washed with cold MeOH and dried at 60° under vacuum.

Taurocholic acid [81-24-3] M 515.6, m 125°(dec), [α]_D +38.8 (c 2, EtOH), pK 1.4. Crystd from EtOH/diethyl ether.

Terephthalaldehyde [623-27-8] M 134.1, m 116°, b 245-248°/771mm. Crystd from water.

Terephthalic acid (benzene-1,4-dicarboxylic acid) [100-21-0] M 166.1, m sublimes >300° without melting, pK₁²⁰ 3.4, pK₂²⁰ 4.34. Purified *via* the sodium salt which, after crystn from water, was reconverted to the acid by acidification with mineral acid.

Terephthaloyl chloride [100-20-9] M 203.0, m 80-82°. Crystd from dry hexane.

o-Terphenyl [84-15-1] M 230.3, m 57-58°. Crystd from EtOH. Purified by chromatography of CCl₄ solns on alumina, with pet ether as eluent, followed by crystn from pet ether (b 40-60°) or pet ether/*benzene. They can also be distd under vacuum.

m-Terphenyl [92-06-8] M 230.3, m 88-89°. Purification as for *o*-terphenyl above.

***p*-Terphenyl** [92-94-4] M 230.3, m 212.7°. Crystd from nitrobenzene or trichlorobenzene. It was purified by chromatography on alumina in a darkened room, using pet ether, and then crystallizing from pet ether (b 40-60°) or pet ether/*benzene.

Terpin hydrate [2451-01-6] M 190.3, m 105.5° (*cis*), 156-158° (*trans*). Crystd from H₂O or EtOH.

2,2':6',2''-Terpyridyl (2,2':6',2''-terpyridyl) [1148-79-4] M 233.3, m 91-92°, pK₁²³ 2.64, pK₂²³ 4.33. Crystd from diethyl ether, toluene or from pet ether, then aqueous MeOH, followed by vacuum sublimation at 90°.

Terreic acid (2-hydroxy-3-methyl-1,4-benzoquinone-5,6-epoxide) [121-40-4] M 154.1, m 127-127.5°, [α]_D²² +74° (pH 4, phosphate buffer), -17° (CHCl₃), pK 4.5. Crystd from *benzene or hexane. Sublimed *in vacuo*.

Terthiophene (2,5-di[thienyl]thiophene; α-terthienyl) [1081-34-1] M 248.4, m 94-95.5°, 94-96°. Possible impurities are bithienyl and polythienyls. Suspend in H₂O and steam distil to remove bithienyl. The residue is cooled and extracted with CHCl₃, dried (MgSO₄), filtered, evaporated and the residue chromatographed on Al₂O₃ using pet ether-3% Me₂CO as eluant. The terphenyl zone is then eluted from the Al₂O₃ with Et₂O, the extract is evaporated and the residue is recrystd from MeOH (40mL per g). The platelets are washed with cold MeOH and dried in air. [UV: Sease and Zechmeister *J Am Chem Soc* 69 270 1947; Uhlenbroek and Bijloo *Recl Trav Chim Pays-Bas* 79 1181 1960.] See also entry on p. 568 in Chapter 6.

Testosterone [58-22-0] M 288.4, m 155°, [α]₅₄₆²⁰ +130° (c 1, dioxane). Crystd from aq acetone.

Testosterone propionate [57-85-2] M 344.5, m 118-122°, [α]₅₄₆²⁰ +100° (c 1, dioxane). Crystd from aqueous EtOH.

2,4,5,6-Tetraaminopyrimidine sulfate [5392-28-9] M 238.2, m 255° (dec), >300°, >350° (dec), pK²⁰ 6.82. Purified by recrystn from H₂O, 2N H₂SO₄ (20 parts, 67% recovery) or 0.1N H₂SO₄ (40 parts, 62% recovery), and dried in air. [UV: Konrad and Pfeleiderer *Chem Ber* 103 722 1970; Malletta et al. *J Am Chem Soc* 69 1814 1947; Cavalieri et al. *J Am Chem Soc* 70 3875 1948.]

Tetra-*n*-amylammonium bromide [866-97-7] M 378.5, m 100-101°. Crystd from pet ether, *benzene or acetone/ether mixtures and dried in vacuum at 40-50° for 2 days.

Tetra-*n*-amylammonium iodide [2498-20-6] M 425.5, m 135-137°. Crystd from EtOH and dried at 35° under vac. Also purified by dissolving in acetone and pptd by adding diethyl ether; and dried at 50° for 2 days.

1,4,8,11-Tetraazacyclotetradecane [295-37-4] M 200.33, m 173° (closed capillary and sublimes at 125°), 183-185°, 185°, pK_{Est(1)}~3.8, pK_{Est(2)}~6.0, pK_{Est(3)}~9.0, pK_{Est(4)}~9.6. Purified by recrystn from dioxane (white needles) and sublimes above 120°. It has been distilled, b 132-140°/4-8mm. It forms complexes with metals and gives a sparingly soluble nitrate salt, m 205° (dec), which crystallises from H₂O and is dried at 150°. [UV: Bosnich et al. *Inorg Chem* 4 1102 1963, van Alphen *Recl Trav Chim Pays-Bas* 56 343 1937.]

Tetrabenazine (2-oxo-3-isobutyl-9,10-dimethoxy-1,2,3,4,6,7,-hexahydro-11bH-benzo[a]-quinolizine) [58-46-8] M 317.4, m 127-128°, pK_{Est} ~ 8. Crystd from MeOH. The *hydrochloride* has m 208-210° and the *oxime* has m 158° (from EtOH).

1,1,2,2,-Tetrabromoethane [79-27-6] M 345.7, f 0.0°, b 243.5°, d 2.965, n 1.63533. Washed successively with conc H₂SO₄ (three times) and H₂O (three times), dried with K₂CO₃ and CaSO₄ and distd.

3',3'',5',5''-Tetrabromophenolphthalein ethyl ester [1176-74-5] M 662.0, m 212-214°. Crystd from *benzene, dried at 120° and kept under vacuum.

Tetra-*n*-butylammonium bromide [1643-19-2] M 322.4, m 119.6°. Crystd from *benzene (5mL/g) at 80° by adding hot *n*-hexane (three volumes) and allowing to cool. Dried over P₂O₅ or Mg(ClO₄)₂, under vacuum. The salt is *very hygroscopic*. It can also be crystd from ethyl acetate or dry acetone by adding diethyl ether and dried *in vacuo* at 60° for 2 days. It has been crystd from acetone by addition of diethyl ether. So *hygroscopic* that all manipulations should be carried out in a dry-box. Purified by precipitation of a saturated solution in dry CCl₄ by addition of cyclohexane or by recrystallisation from ethyl acetate, then heating in vacuum to 75° in the presence of P₂O₅. [Symons et al. *J Chem Soc, Faraday Trans 1* 76 2251 1908.] Also recrystallised from CH₂Cl₂/diethyl ether and dried in a vacuum desiccator over P₂O₅. [Blau and Espenson *J Am Chem Soc* 108 1962 1986.]

Tetra-*n*-butylammonium chloride [1112-67-0] M 277.9, m 15.7°. Crystd from acetone by addition of diethyl ether. Very *hygroscopic* and forms crystals with 34H₂O.

Tetra-*n*-butylammonium fluoroborate [429-42-5] M 329.3, m 161-163°. See tetrabutylammonium fluoroborate on p. 480 in Chapter 5.

Tetra-*n*-butylammonium hexafluorophosphate [3109-63-5] M 387.5, m 239-241°. Recrystd from satd EtOH/water and dried for 10h in vac at 70°. It was also recrystd three times from abs EtOH and dried for 2 days in a drying pistol under vac at boiling toluene temperature [Bedard and Dahl *J Am Chem Soc* 108 5933 1986].

Tetra-*n*-butylammonium hydrogen sulfate [32503-27-8] M 339.5, m 171-172°. Crystd from acetone.

Tetra-*n*-butylammonium iodide [311-28-4] M 369.4, m 146°. Crystd from toluene/pet ether (see entry for the corresponding bromide), acetone, ethyl acetate, EtOH/diethyl ether, nitromethane, aq EtOH or water. Dried at room temperature under vac. It has also been dissolved in MeOH/acetone (1:3, 10mL/g), filtered and allowed to stand at room temperature to evaporate to *ca* half its original volume. Distilled water (1mL/g) was then added, and the ppte was filtered off and dried. It was also dissolved in acetone, ppted by adding ether and dried in vac at 90° for 2 days. It has also been recrystallised from CH₂Cl₂/pet ether or hexane, or anhydrous methanol and stored in a vacuum desiccator over H₂SO₄. [Chau and Espenson *J Am Chem Soc* 108 1962 1986.]

Tetra-*n*-butylammonium nitrate [1941-27-1] M 304.5, m 119°. Crystd from *benzene (7mL/g) or EtOH, dried in a vacuum over P₂O₅ at 60° for 2 days.

Tetra-*n*-butylammonium perchlorate [1923-70-2] M 341.9°, m 210°(dec). Crystd from EtOH, ethyl acetate, from *n*-hexane or diethyl ether/acetone mixture, ethyl acetate or hot CH₂Cl₂. Dried in vacuum at room temperature over P₂O₅ for 24h. [Anson et al. *J Am Chem Soc* 106 4460 1984; Ohst and Kochi *J Am Chem Soc* 108 2877 1986; Collman et al. *J Am Chem Soc* 108 2916 1986; Blau and Espenson *J Am Chem Soc* 108 1962 1986; Gustowski et al. *J Am Chem Soc* 108 1986; Ikezawa and Kutal *J Org Chem* 52 3299 1987.]

Tetra-*n*-butylammonium picrate [914-45-4] M 490.6, m 89°. Crystd from EtOH. Dried in a vacuum desiccator over P₂O₅.

Tetra-*n*-butylammonium tetrabutylborate (Bu₄N⁺ Bu₄B⁻) [23231-91-6] M 481.7, m 109.5°. Dissolved in MeOH or acetone, and crystd by adding distd water. Dried in vacuum at 70°. It has also been successively recrystd from isopropyl ether, isopropyl ether/acetone (50:1) and isopropyl ether/EtOH (50:1) for 10h, then isopropyl ether/acetone for 1h, and dried at 65° under reduced pressure for 1 week. [Kondo et al. *J Chem Soc, Faraday Trans 1* 76 812 1980.]

- 2,3,4,5-Tetrachloroaniline** [634-83-3] M 230.9, m 119-120°, pK_{Est} ~-0.26. Crystd from EtOH.
- 2,3,5,6-Tetrachloroaniline** [3481-20-7] M 230.9, m 107-108°, pK_{Est} ~-1.8. Crystd from EtOH.
- 1,2,3,4-Tetrachlorobenzene** [634-66-2] M 215.9, m 45-46°, b 254°/760mm. Crystd from EtOH.
- 1,2,3,5-Tetrachlorobenzene** [634-90-2] M 215.9, m 51°, b 246°/760mm. Crystd from EtOH.
- 1,2,4,5-Tetrachlorobenzene** [95-94-3] M 215.9, m 139.5-140.5°, b 240°/760mm. Crystd from EtOH, ether, *benzene, *benzene/EtOH or carbon disulfide.
- 3,4,5,6-Tetrachloro-1,2-benzoquinone** [2435-53-2] M 245.9, m 130°. Crystd from AcOH. Dry in vacuum desiccator over KOH.
- 1,1,2,2-Tetrachloro-1,2-difluoroethane** [72-12-0] M 203.8, f 26.0°, b 92.8°/760 mm. Purified as for trichlorotrifluoroethane.
- sym-Tetrachloroethane** [79-34-5] M 167.9, b 62°/100mm, 146.2°/atm, d 1.588, n¹⁵ 1.49678. Stirred, on a steam-bath, with conc H₂SO₄ until a fresh portion of acid remained colourless. The organic phase was then separated, distd in steam, dried (CaCl₂ or K₂CO₃), and fractionally distd in a vac.
- Tetrachloroethylene** [127-18-4] M 165.8, b 62°/80mm, 121.2°, d¹⁵ 1.63109, d 1.623, n¹⁵ 1.50759, n 1.50566 It decomposes under similar conditions to CHCl₃, to give phosgene and trichloroacetic acid. Inhibitors of this reaction include EtOH, diethyl ether and thymol (effective at 2-5ppm). Tetrachloroethylene should be distd under a vac (to avoid phosgene formation), and stored in the dark out of contact with air. It can be purified by washing with 2M HCl until the aq phase no longer becomes coloured, then with water, drying with Na₂CO₃, Na₂SO₄, CaCl₂ or P₂O₅, and fractionally distilling just before use. 1,1,2-Trichloroethane and 1,1,1,2-tetrachloroethane can be removed by counter-current extraction with EtOH/water.
- Tetrachloro-N-methylphthalimide** [14737-80-5] M 298.9, m 209.7°. Crystd from absolute EtOH.
- 2,3,4,6-Tetrachloronitrobenzene** [879-39-0] M 260.9, m 42°. Crystd from aqueous EtOH.
- 2,3,5,6-Tetrachloronitrobenzene** [117-18-0] M 260.9, m 99-100°. Crystd from aqueous EtOH.
- 2,3,4,5-Tetrachlorophenol** [4901-51-3] M 231.9, m 116-117°, pK_{Est} ~6.2. Crystd from pet ether.
- 2,3,4,6-Tetrachlorophenol** [58-90-2] M 231.9, m 70°, b 150°/15mm, pK_{Est} ~5.4. Crystd from pet ether.
- 2,3,5,6-Tetrachlorophenol** [935-95-5] M 231.9, m 115°, pK_{Est} ~5.0. Crystd from pet ethers.
- Tetrachlorophthalic anhydride** [117-08-8] M 285.9, m 255-257°. Crystd from chloroform or *benzene, then sublimed.
- 2,3,4,6-Tetrachloropyridine** [14121-36-9] M 216.9, m 74-75°, b 130-135°/16-20mm, pK_{Est} ~-5.7. Crystd from 50% EtOH.
- Tetracosane** [646-31-1] M 338.7, m 54°, b 243-244°/15mm. Crystd from diethyl ether.
- Tetracosanoic (lignoceric) acid** [557-59-5] M 368.7, m 84°, 87.5-88°, pK_{Est} ~5.0. Crystd from acetic acid, Me₂CO, toluene, pet ether/Me₂CO, *C₆H₆/Me₂CO.

1,2,4,5-Tetracyanobenzene [712-74-3] M 178.1, m 270-272° (280°). Crystd from EtOH and sublimed *in vacuo*. [Lawton and McRitchie *J Org Chem* 24 26 1959; Bailey et al. *Tetrahedron* 19 161 1963.]

Tetracyanoethylene [670-54-2] M 128.1, m 199-200° (sealed tube). Crystd from chlorobenzene, dichloroethane, or dichloromethane [Hall et al. *J Org Chem* 52 5528 1987]. Stored at 0° in a desiccator over NaOH pellets. (It slowly evolves HCN on exposure to moist air.) It can also be sublimed at 120° under vacuum. Also purified by repeated sublimation at 120-130°/0.5mm. [Frey et al. *J Am Chem Soc* 107 748 1985; Traylor and Mikszal *J Am Chem Soc* 109 2778 1987.]

7,7,8,8-Tetracyanoquinodimethane [1518-16-7] M 204.2, m 287-290°(dec). Recrystd from distd, dried, acetonitrile.

Tetradecane [629-59-4] M 198.4, m 6°, b 122°/10mm, 252-254°, d 0.763, n 1.429. Washed successively with 4M H₂SO₄ and water. Dried over MgSO₄ and distd several times under reduced pressure [Poë et al. *J Am Chem Soc* 108 5459 1986].

1-Tetradecanol [112-72-1] M 214.4, m 39-39.5°, b 160°/10mm, 170-173°/20mm. Crystd from aq EtOH. Purified by zone melting.

Tetradecyl ether (di-tetradecyl ether) [5412-98-6] M 410.7. Distd under vac and then crystd repeatedly from MeOH/*benzene.

Tetradecyltrimethylammonium bromide [1119-97-7] M 336.4, m 244-249°. Crystd from acetone or a mixture of acetone and >5% MeOH. Washed with diethyl ether and dried in a vacuum oven at 60°. [Dearden and Wooley *J Phys Chem* 91 2404 1987.]

Tetraethoxymethane [78-09-1] M 192.3, b 159°. See tetraethyl orthocarbonate on p. 360.

Tetraethylammonium bromide [71-91-0] M 210.2, m 269°(dec), 284°(dec). Recrystd from EtOH, CHCl₃ or diethyl ether, or, recrystd from acetonitrile, and dried over P₂O₅ under reduced pressure for several days. Also recrystd from EtOH/diethyl ether (1:2), EtOAc, water or boiling MeOH/acetone (1:3) or by adding equal volume of acetone and allowing to cool. Dried at 100° *in vacuo* for 12 days, and stored over P₂O₅.

Tetraethylammonium chloride hydrate [56-34-8] M 165.7, m dec>200°. Crystd from EtOH by adding diethyl ether, from warm water by adding EtOH and diethyl ether, from dimethylacetamide or from CH₂Cl₂ by addition of diethyl ether. Dried over P₂O₅ in vacuum for several days. Also crystd from acetone/CH₂Cl₂/hexane (2:2:1) [Blau and Espenson *J Am Chem Soc* 108 1962 1986; White and Murray *J Am Chem Soc* 109 2576 1987].

Tetraethylammonium iodide [68-05-3] M 257.2, m 302°, >300°(dec). Crystd from acetone/MeOH, EtOH/water, dimethylacetamide or ethyl acetate/EtOH (19:1). Dried under vacuum at 50° and stored over P₂O₅.

Tetraethylammonium perchlorate [2567-83-1] M 229.7, m 345°(dec). Crystd repeatedly from water, aqueous MeOH, acetonitrile or acetone, and dried at 70° under vacuum for 24h. [Cox et al. *J Am Chem Soc* 106 5965 1984; Liu et al. *J Am Chem Soc* 108 1740 1986; White and Murray *J Am Chem Soc* 109 2576 1987.] Also twice crystd from ethyl acetate/95% EtOH (2:1) [Lexa et al. *J Am Chem Soc* 109 6464 1987].

Tetraethylammonium picrate [741-03-7] M 342.1, m >300°(dec). Purified by successive crystns from water or 95% EtOH followed by drying in vacuum at 70°.

Tetraethylammonium tetrafluoroborate [429-06-1] M 217.1, m 235°, 275-277°, 289-291°. Recrystd three times from a mixture of ethyl acetate/hexane (5:1) or MeOH/pet ether, then stored at 95° for 48h

under vacuum [Henry and Faulkner *J Am Chem Soc* **107** 3436 1985; Huang et al. *Anal Chem* **58** 2889 1986]. See entry on p. 481 in Chapter 5.

Tetraethylammonium tetraphenylborate [12099-10-4] M 449.4. Recrystd from aqueous acetone. Dried in a vacuum oven at 60° for several days. *Similarly for the propyl and butyl homologues.*

Tetraethyl 1,1,2,2-ethanetetracarboxylate [632-56-4] M 318.3, m 73-74°. Twice recrystd from EtOH by cooling to 0°.

Tetraethylene glycol dimethyl ether [143-24-8] M 222.3, b 105°/1mm, d 1.010, n 1.435. Stood with CaH₂, LiAlH₄ or sodium, and distd when required.

Tetraethylenepentamine [112-57-2] M 189.3, b 169-171°/0.05mm, d 0.999, n 1.506, pK₁²⁵ 2.98, pK₂²⁵ 4.72, pK₃²⁵ 8.08, pK₄²⁵ 9.10, pK₅²⁵ 9.68. Distd under vacuum. Purified *via* its pentachloride, nitrate or sulfate. Jonassen, Frey and Schaafsma [*J Phys Chem* **61** 504 1957] cooled a soln of 150g of the base in 300mL of 95% EtOH, and added dropwise 180mL of conc HCl, keeping the temperature below 20°. The white ppt was filtered, crystd three times from EtOH/water, then washed with diethyl ether and dried by suction. Reilley and Holloway [*J Am Chem Soc* **80** 2917 1958], starting with a similar soln cooled to 0°, added slowly (keeping the temperature below 10°) a soln of 4.5g-moles of HNO₃ in 600mL of aqueous 50% EtOH (also cooled to 0°). The ppt was filtered by suction, recrystd five times from aqueous 5% HNO₃, then washed with acetone and absolute EtOH and dried at 50°. [For purification *via* the sulfate see Reilley and Vavoulis (*Anal Chem* **31** 243 1959), and for an additional purification step using the Schiff base with benzaldehyde see Jonassen et al. *J Am Chem Soc* **79** 4279 1957].

Tetraethyl orthocarbonate (ethyl orthocarbonate, tetraethoxy ethane) [78-09-1] M 192.3, b 59.6-60°/14mm, 158°/atm, 159°/atm, 160-161°/atm, d₄²⁰ 0.9186, n_D²⁰ 1.3932. Likely impurities are hydrolysis products. Shake with brine (satd NaCl; dilute with a little Et₂O if amount of material is small) and dry (MgSO₄). The organic layer is filtered off and evaporated, and the residue is distd through a helices packed fractionating column with a total reflux partial take-off head. All distns can be done at atmospheric pressure in an inert atmosphere (e.g. N₂). [Roberts and McMahon *Org Synth Coll Vol IV* 457 1963; Connolly and Dyson *J Chem Soc* 828 1937; Tieckelmann and Post *J Org Chem* **13** 266 1948.]

1,1,2,2-Tetrafluorocyclobutane [374-12-9] M 128.1, b 50-50.7°, d 1.275, n 1.3046. Purified distn or by preparative gas chromatography using a 2m x 6mm(i.d.) column packed with β,β'-oxydipropionitrile on Chromosorb P at 33°. [Conlin and Fey *J Chem Soc, Faraday Trans 1* **76** 322 1980; Coffmann et al. *J Am Chem Soc* **71** 490 1949.]

Tetrafluoro-1,3-dithietane [1717-50-6] M 164.1, m -6°, b 47-48°/760mm, d²⁵ 1.6036, n²⁵ 1.3908. Purified by preparative gas chromatography or by distn through an 18in spinning band column. Also purified by shaking vigorously *ca* 40mL with 25mL of 10% NaOH, 5mL of 30% H₂O₂ until the yellow colour disappeared. The larger layer was separated, dried over silica gel to give a colourless liquid boiling at 48°. It had a singlet at -1.77ppm in the NMR spectrum. [Middleton, Howard and Sharkey, *J Org Chem* **30** 1375 1965.]

2,2,3,3-Tetrafluoropropanol [76-37-9] M 132.1, b 106-106.5°, pK²⁵ 12.74. Tetrafluoropropanol (450mL) was added to a soln of 2.25g of NaHSO₃ in 90mL of water, shaken vigorously and stood for 24h. The fraction distilling at or above 99° was refluxed for 4h with 5-6g of KOH and rapidly distd, followed by a final fractional distn. [Kosower and Wu *J Am Chem Soc* **83** 3142 1961.] Alternatively, shaken with alumina for 24h, dried overnight with anhydrous K₂CO₃ and distd, taking the middle fraction (b 107-108°).

Tetra-*n*-heptylammonium bromide [4368-51-8] M 490.7, m 89-91°. Crystd from *n*-hexane, then dried in a vacuum oven at 70°.

Tetra-*n*-heptylammonium iodide [3535-83-9] M 537.7. Crystd from EtOH.

Tetra-*n*-hexylammonium bromide [4328-13-6] M 434.6, m 99-100°. Washed with ether, and dried in a vacuum at room temperature for 3 days.

Tetra-*n*-hexylammonium chloride [5922-92-9] M 390.1. Crystd from EtOH.

Tetra-*n*-hexylammonium iodide [2138-24-1] M 481.6, m 99-101°. Washed with diethyl ether and dried at room temperature *in vacuo* for 3 days.

Tetrahexylammonium perchlorate [4656-81-9] M 454.1, m 104-106°. Crystd from acetone and dried *in vacuo* at 80° for 24h.

Tetrahydrofuran [109-99-9] M 72.1, b 25°/176mm, 66°/760mm, d_4^{20} 0.889, n_D^{20} 1.4070, pK -2.48 (aq H₂SO₄). It is obtained commercially by catalytic hydrogenation of furan from pentosan-containing agricultural residues. It was purified by refluxing with, and distilling from LiAlH₄ which removes water, peroxides, inhibitors and other impurities [Jaeger et al. *J Am Chem Soc* 101 717 1979]. Peroxides can also be removed by passage through a column of activated alumina, or by treatment with aq ferrous sulfate and sodium bisulfate, followed by solid KOH. In both cases, the solvent is then dried and fractionally distd from sodium. Lithium wire or vigorously stirred molten potassium have also been used for this purpose. CaH₂ has also been used as a drying agent.

Several methods are available for obtaining the solvent almost anhydrous. Ware [*J Am Chem Soc* 83 1296 1961] dried vigorously with sodium-potassium alloy until a characteristic blue colour was evident in the solvent at Dry-ice/cellosolve temperatures. The solvent was kept in contact with the alloy until distd for use. Worsfold and Bywater [*J Chem Soc* 5234 1960], after refluxing and distilling from P₂O₅ and KOH, in turn, refluxed the solvent with sodium-potassium alloy and fluorenone until the green colour of the disodium salt of fluorenone was well established. [Alternatively, instead of fluorenone, benzophenone, which forms a blue ketyl, can be used.] The tetrahydrofuran was then fractionally distd, degassed and stored above CaH₂. *p*-Cresol or hydroquinone inhibit peroxide formation. The method described by Coetzee and Chang [*Pure Appl Chem* 57 633 1985] for 1,4-dioxane also applies here. Distns should always be done in the presence of a reducing agent, e.g. FeSO₄. **It irritates the skin, eyes and mucous membranes and the vapour should never be inhaled. It is HIGHLY FLAMMABLE and the necessary precautions should be taken.**

Rapid purification: Purification as for diethyl ether.

***l*-Tetrahydropalmitine (2,3,9,10-tetramethoxy-6*H*-dibenzo[*a,g*]quinolizidine)** [10097-84-4] M 355.4, m 148-149°, $[\alpha]_D^{20}$ -291° (EtOH). Crystd from MeOH by addition of water [see *J Chem Soc (C)* 530 1967].

Tetrahydropyran [142-68-7] M 86.1, b 88.0°, n 1.4202, d 0.885, pK -2.79 (aq H₂SO₄). Dried with CaH₂, then passed through a column of silica gel to remove olefinic impurities and fractionally distd. Freed from peroxides and moisture by refluxing with sodium, then distilling from LiAlH₄. Alternatively, peroxides can be removed by treatment with aqueous ferrous sulfate and sodium bisulfate, followed by solid KOH, and fractional distn from sodium.

Tetrahydro-4*H*-pyran-4-one [29943-42-8] M 100.1, b 57-59°/11mm, 65-66°/15mm, 67-68°/18mm, 73°/20mm, 164.7°/atm, 166-166.5°/atm, d_4^{20} 1.0844, n_D^{20} 1.4551. Purified by repeated distn preferably in a vacuum. [Baker *J Chem Soc* 296 1944; IR: Olsen and Bredoch *Chem Ber* 91 1589 1958.] The *oxime* has m 87-88° and b 110-111°/13mm [Cornubert et al. *Bull Soc Chim Fr* 36 1950]. The 4-nitrophenylhydrazone forms orange-brown needles from EtOH, m 186° [Cawley and Plant *J Chem Soc* 1214 1938].

Tetrahydrothiophene [110-01-0] M 88.2, m -96°, b 14.5°/10mm, 120.9°/760mm, d 0.997, n 1.5289. Crude material was purified by crystn of the mercuric chloride complex to a constant melting point. It was then regenerated, washed, dried, and fractionally distd. [Whitehead et al. *J Am Chem Soc* 73 3632 1951.] It has been dried over Na₂SO₄ and distd in a vacuum [Roberts and Friend *J Am Chem Soc* 108 7204 1986].

Tetrahydro-4H-thiopyran-4-one [1072-72-6] *M* 116.2, *m* 60-62°, 61-62°, 64-65°, 65-67°. Purified by recrystn from diisopropyl ether or pet ether and dried in air. If too impure then dissolve in Et₂O, wash with aq NaHCO₃, then H₂O, dried (MgSO₄), fild, evapd and the residue recrystd as before. [Cardwell *J Chem Soc* 715 1949.] The *oxime* can be recrystd from CHCl₃-pet ether (at -20°) and has *m* 84-85° [Barkenbus et al. *J Org Chem* 20 871 1955]. The *2,4-dinitrophenylhydrazone* has *m* 186° (from EtOAc) [Barkenbus et al. *J Org Chem* 16 232 1951]. The *S-dioxide* is recrystd from AcOH, *m* 173-174° [Fehnel and Carmack *J Am Chem Soc* 70 1813 1948].

Tetrahydroxy-*p*-benzoquinone (2H₂O) [5676-48-2] *M* 208.1, *pK*₁³⁰ 4.80, *pK*₂³⁰ 6.8. Crystd from water.

Tetrakis(dimethylamino)ethylene [996-70-3] *M* 300.2, *b* 60°/1mm, *d* 0.861, *n* 1.4817, *pK*_{Est(1)}<0, *pK*_{Est(2)}<0, *pK*_{Est(3)}~1.5, *pK*_{Est(4)} 5.1. Impurities include tetramethylurea, dimethylamine, tetramethylethanediamine and tetramethyloxamide. It was washed with water while being flushed with nitrogen to remove dimethylamine, dried over molecular sieves, then passed through a silica gel column (previously activated at 400°) under nitrogen. Degassed on a vacuum line by distn from a trap at 50° to one at -70°. Finally, it was stirred over sodium-potassium alloy for several days. [Holroyd et al. *J Phys Chem* 89 4244 1985; Wiberg *Angew Chem Int Ed Engl* 7 766 1968.]

Tetralin (1,2,3,4-tetrahydronaphthalene) [119-64-2] *M* 132.2, *b* 65-66°/5mm, 207.6°/760 mm, *d* 0.968, *n* 1.5413. It was washed with successive portions of conc H₂SO₄ until the acid layer no longer became coloured, then washed with aq 10% Na₂CO₃, and then distd water. Dried (CaSO₄ or Na₂SO₄), filtered, refluxed and fractionally distd at under reduced pressure from sodium or BaO. It can also be purified by repeated fractional freezing.

Bass [*J Chem Soc* 3498 1964] freed tetralin, purified as above, from naphthalene and other impurities by conversion to ammonium tetralin-6-sulfonate. Conc H₂SO₄ (150mL) was added slowly to stirred tetralin (272mL) which was then heated on a water bath for about 2h to give complete soln. The warm mixture, when poured into aq NH₄Cl soln (120g in 400mL water), gave a white ppte which, after filtering off, was crystd from boiling water, washed with 50% aq EtOH and dried at 100°. Evapn of its boiling aq soln on a steam bath removed traces of naphthalene. The pure salt (229g) was mixed with conc H₂SO₄ (266mL) and steam distd from an oil bath at 165-170°. An ether extract of the distillate was washed with aq Na₂SO₄, and the ether was evapd, prior to distilling the tetralin from sodium. Tetralin has also been purified *via* barium tetralin-6-sulfonate, conversion to the sodium salt and decomposition in 60% H₂SO₄ using superheated steam.

Tetralin hydroperoxide [771-29-9] *M* 164.2, *m* 56°. Crystd from hexane.

α-Tetralone (1,2,3,4-tetrahydro-1-oxonaphthalene) [529-34-0] *M* 146.2, *m* 2-7°, 7.8-8.0°, *b* 75-85°/0.3mm, 89°/0.5mm, 94-95°/2mm, 132-134°/15mm, 143-145°/20mm, *d*₄²⁰ 1.0695, *n*_D²⁰ 1.5665. Check the IR first. Purify by dissolving 20mL in Et₂O (200mL), washing with H₂O (100mL), 5% aq NaOH (100mL), H₂O (100mL), 3% aq AcOH (100mL), 5% NaHCO₃ (100mL) then H₂O (100mL) and dry the ethereal layer over MgSO₄. Filter, evap and fractionate the residue through a 6in Vigreux column under reduced pres to give a colourless oil (~17g) with *b* 90-91°/0.5-0.7mm. [Snyder and Werber *Org Synth Coll Vol III* 798 1955.] It has also been fractionated through a 0.5metre packed column with a heated jacket under reflux using a partial take-off head. [Olson and Bader *Org Synth Coll Vol IV* 898 1963.]

β-Tetralone (1,2,3,4-tetrahydro-2-oxonaphthalene) [530-93-8] *M* 146.2, *m* 17-18°, ~18°, *b* 93-95°/2mm, 104-105°/4mm, 114-115°/4-5mm, 140°/18mm, *d*₄²⁰ 1.1000, *n*_D²⁰ 1.5598. If reasonably pure then fractionate through an efficient column. Otherwise purify *via* the *bisulfite adduct*. To a soln of NaHSO₃ (32.5g, 0.31mol) in H₂O (57mL) is added 95% EtOH (18mL) and set aside overnight. Any bisulfite-sulfate that separated is removed by filtration and the filtrate is added to the tetralone (14.6g, 0.1mol) and shaken vigorously. The adduct separates in a few minutes as a white ppte and kept on ice for ~3.5h with occasional shaking. The ppte is collected, washed with 95% EtOH (13mL), then with Et₂O (4 x 15mL, by stirring the suspension in the solvent, filtering and repeating the process). The colourless product is dried in air and stored in air tight containers in which it is stable for extended periods (yield is ~17g). This bisulfite (5g) is suspended in H₂O (25mL) and Na₂CO₃.H₂O (7.5g) is added (pH of soln is ~10). The mixture is then extracted

with Et₂O (5 x 10mL, i.e. until the aqueous phase does not test for tetralone — see below). Wash the combined extracts with 10% aqueous HCl (10mL), H₂O (10mL, i.e. until the washings are neutral), dry (MgSO₄), filter, evaporate and distil the residual oil using Claisen flask under reduced pressure and in a N₂ atm. The pure tetralone is a colourless liquid **b** 70-71°/0.25mm (see also above). The yield is ~2g. **Tetralone test:** Dissolve a few drops of the tetralone soln (ethereal or aqueous) in 95% EtOH in a test tube and add 10 drops of 25% NaOH down the side of the tube. A deep blue colour develops at the interface with air. [Soffer *Org Synth Coll Vol IV* 903 1963; Cornforth et al. *J Chem Soc* 689 1942; UV: Soffer et al. *J Am Chem Soc* 1556 1952.] The *phenylhydrazone* has **m** 108° [Crawley and Robinson *J Chem Soc* 2001 1938].

Tetramethylammonium bromide [64-20-0] **M 154.1, sublimes with dec >230°**. Crystd from EtOH, EtOH/diethyl ether, MeOH/acetone, water or from acetone/MeOH (4:1) by adding an equal volume of acetone. It was dried at 110° under reduced pressure or at 140° for 24h.

Tetramethylammonium chloride [75-57-0] **M 109.6, m >230°(dec)**. Crystd from EtOH, EtOH/CHCl₃, EtOH/diethyl ether, acetone/EtOH (1:1), isopropanol or water. Traces of the free amine can be removed by washing with CHCl₃.

Tetramethylammonium hydroxide (5H₂O) [10424-65-4 (5H₂O); 75-59-2 (aq soln)] **M 181.2, m 63°, 65-68°**. Freed from chloride ions by passage through an ion-exchange column (Amberlite IRA-400, prepared in its OH⁻ form by passing 2M NaOH until the effluent was free from chloride ions, then washed with distilled H₂O until neutral). A modification, to obtain carbonate-free hydroxide, uses the method of Davies and Nancollas [*Nature* 165 237 1950].

Tetramethylammonium iodide [75-58-1] **M 201.1, m >230°(dec)**. Crystd from water or 50% EtOH, EtOH/diethyl ether, ethyl acetate, or from acetone/MeOH (4:1) by adding an equal volume of acetone. Dried in a vacuum desiccator.

Tetramethylammonium perchlorate [2537-36-2] **M 173.6, m >300 °(dec)**. Crystd from acetone and dried *in vacuo* at 60° for several days.

Tetramethylammonium tetraphenylborate [15525-13-0] **M 393.3**. Recrystd from acetone, acetone/CCl₄ and from acetone/1,2-dichloroethane. Dried over P₂O₅ in vacuum, or in a vacuum oven at 60° for several days.

1,2,3,4-Tetramethylbenzene (prehnitine) [488-23-3] **M 134.2, m -6.3°, b 79.4°/10mm, 204-205°/760mm, d 0.905, n 1.5203**. Dried over sodium and distd under reduced pressure.

1,2,3,5-Tetramethylbenzene (isodurene) [527-53-7] **M 134.2, m -23.7°, b 74.4°/10mm, 198°/760mm, d 0.890, n 1.5130**. Refluxed over sodium and distd under reduced pressure.

1,2,4,5-tetramethylbenzene (durene) [95-93-2] **M 134.2, m 79.5-80.5°**. Chromatographed on alumina, and recrystd from aqueous EtOH or *benzene. Zone-refining removes duroaldehydes. Dried under vacuum. [Yamauchi et al. *J Phys Chem* 89 4804 1985.] It has also been sublimed *in vacuo* [Johnston et al. *J Am Chem Soc* 109 1291 1987].

N,N,N',N'-Tetramethylbenzidine [366-29-0] **M 240.4, m 195.4-195.6°, pK_{Est(1)}~3.4, pK_{Est(2)}~4.5**. Crystd from EtOH or pet ether, then from pet ether/*benzene, and sublimed in a vacuum. [Guarr et al. *J Am Chem Soc* 107 5104 1985.] Dried under vac in an Abderhalden pistol, or carefully on a vacuum line.

2,2,4,4-Tetramethylcyclobutan-1,3-dione [933-52-8] **M 140.2, m 114.5-114.9°**. Crystd from *benzene and dried under vacuum over P₂O₅ in an Abderhalden pistol.

3,3,5,5-Tetramethylcyclohexanone [14376-79-5] **M 154.3, m 11-12°, 13.2°, b 59-61°, 80-82°/13mm, 196°/760mm, 203.8-204.8°/760mm, d_D²⁰ 0.8954, n_D²⁰ 1.4515**. Purified first through a

24in column packed with Raschig rings then a 40cm Vigreux colum under reduced pressure (b 69-69.3°/7mm, see above). The *oxime* has m 144-145° (from 60% EtOH) and the *semicarbazone* has m 196-197°, 197-198° (214.5°, 217-218°) [Karasch and Tawney *J Am Chem Soc* **63** 2308 1941; UV: Sandris and Ourisson *Bull Soc Chim Fr* 958 1956].

***p,p'*-Tetramethyldiaminodiphenylmethane [bis(*p*-dimethylaminophenyl)methane, Michler's base]** [101-61-1] M 254.4, m 89-90°, b 155-157°/0.1mm, pK_{Est(1)}~5.8, pK_{Est(2)}~5.1. Crystd from EtOH (2mL/g) or 95% EtOH (ca 12mL/g). It sublimes on heating.

Tetramethylene sulfoxide (tetrahydrothiophen 1-oxide) [1600-44-8] M 104.2, b 235-237°, d 1.175, n 1.525. Shaken with BaO for 4 days, then distd from CaH₂ under reduced pressure.

***N,N,N',N'*-Tetramethylethylenediamine (TMEDA, TEMED)** [110-18-9] M 116.2, b 122°, d 1.175, n²⁵ 1.4153, pK₁²⁵ 5.90, pK₂²⁵ 9.14. Partially dried with molecular sieves (Linde type 4A), and distd in vacuum from butyl lithium. This treatment removes all traces of primary and secondary amines and water. [Hay, McCabe and Robb *J Chem Soc, Faraday Trans 1* **68** 1 1972.] Or, dried with KOH pellets. Refluxed for 2h with one-sixth its weight of *n*-butyric anhydride (to remove primary and secondary amines) and fractionally distd. Refluxed with fresh KOH, and distd under nitrogen. [Cram and Wilson *J Am Chem Soc* **85** 1245 1963.] Also distd from sodium.

Tetramethylethylenediamine dihydrochloride [7677-21-8] M 198.2, m ~300°. Crystd from 98% EtOH/conc HCl. Hygroscopic. [Knorr *Chem Ber* **37** 3510 1904.]

1,1,3,3-Tetramethylguanidine [80-70-6] M 115.2, b 159-160°, d 0.917 n 1.470, pK²⁵ 13.6. Refluxed over granulated BaO, then fractionally distd.

***N,N,N',N'*-Tetramethyl-1,8-naphthalenediamine** [20734-58-1] M 214.3, m 45-48°, 47-48°, b 144-145°/4mm, pK₁ -10.5 (aq H₂SO₄, diprotonation), pK₂ 12.34 (monoprotonation). It is prepared by methylating 1,8-diaminonaphthalene and likely impurities are methylated products. The tetramethyl compound is a stronger base than the unmethylated, di and trimethylated derivatives. The pK_a values are: 1,8-(NH₂)₂ = 4.61, 1,8-(NHMe)₂ = 5.61, 1-NHMe-8-NHMe₂ = 6.43 and 1,8-(NMe₂)₂ = 12.34. The mixture is then treated H₂O at pH 8 (where all but the required base are protonated) and extracted with Et₂O or CHCl₃. The dried extract (K₂CO₃) yields the tetramethyldiamine on evapn which can be distd. It is a strong base with weak nucleophilic properties, e.g. it could not be alkylated by refluxing with EtI in MeCN for 4 days and on treatment with methyl fluorosulfonate only the fluorosulfonate salt of the base is obtained. [NMR: Adler et al. *J Chem Soc, Chem Commun* 723 1968; *J Am Chem Soc* **63** 358 1941.] See Proton sponge p. 134.

Tetramethyl orthocarbonate (methyl orthocarbonate, tetramethoxy methane) [1850-14-2] M 136.2, m -5.6°, -5°, -2°, b 113.5°/760mm, 113.5-114°/755mm, 112-114°/atm, d₄²⁰ 1.0202, n_D²⁰ 1.3860. Purified in the same way as for tetraethyl orthocarbonate. [Smith *Acta Chem Scand* **10** 1006 1956; Tiekemann and Post *J Org Chem* **13** 266 1948.]

2,6,10,14-Tetramethylpentadecane (pristane, norphytane) [1921-70-6] M 268.5, b 68° (bath temp)/0.004mm, 158°/10mm, 296°/atm, d₄²⁰ 0.7827, n_D²⁰ 1.4385. Purified by shaking with conc H₂SO₄ (care with this acid, if amount of pristane is too small then it should be diluted with pet ether *not* Et₂O which is quite sol in the acid), the H₂O (care as it may heat up if in contact with conc H₂SO₄), dried (MgSO₄) evaporated and distd over Na metal. [Sørensen and Sørensen *Acta Chem Scand* **3** 939 1949.]

***N,N,N',N'*-Tetramethyl-1,4-phenylenediamine** [100-22-1] M 164.3, m 51°, b 260°/760mm, pK₁²⁰ 2.29, pK₂²⁰ 6.35. Crystd from pet ether or water. It can be sublimed or dried carefully in a vacuum line, and stored in the dark under nitrogen. Also recrystd from its melt.

***N,N,N',N'*-Tetramethyl-1,4-phenylenediamine dihydrochloride** [637-01-4] M 237.2, m 222-224°. Crystd from isopropyl or *n*-butyl alcohols, satd with HCl. Treated with aq NaOH to give the free base which was filtered, dried and sublimed in a vacuum. [Guarr et al. *J Am Chem Soc* **107** 5104 1985.]

2,2,6,6-Tetramethylpiperidinyl-1-oxy (TEMPO) [2564-83-2] M 156.3, m 36-38°. Purified by sublimation (33°, water aspirator) [Hay and Fincke *J Am Chem Soc* 109 8012 1987].

2,2,6,6-Tetramethyl-4-piperidone hydrochloride (triacetoneamine) [33973-59-0] M 191.7, m 190° (dec), 198-199° (dec), $pK^{2.5}$ 7.90. Purified by recrystn from EtOH/Et₂O, MeCN or Me₂CO/MeOH. The *free base* has m 37-39° (after sublimation), b 102-105°/18mm, and *hydrate* m 56-58° (wet Et₂O); the *hydrobromide* has m 203° (from EtOH-Et₂O) and the *picrate* has m 196° (from aq EtOH). [Sandris and Ourisson *Bull Soc Chim Fr* 345 1958.]

Tetramethylthiuram disulfide [bis-(dimethylthiocarbamyl)-disulfide] [137-26-8] M 240.4, m 146-148°, 155-156°. Crystd (three times) from boiling CHCl₃, then recrystd from boiling CHCl₃ by adding EtOH dropwise to initiate pptn, and allowed to cool. Finally it was pptd from cold CHCl₃ by adding EtOH (which retained the monosulfide in soln). [Ferington and Tobolsky *J Am Chem Soc* 77 4510 1955.]

1,1,3,3-Tetramethyl urea [632-22-4] M 116.2, f -1.2°, b 175.2°/760mm, d 0.969, n 1.453. Dried over BaO and distd under nitrogen.

Tetramethyl uric acid [2309-49-1] M 224.2, m 225°, 228°, $pK_{Est} < 0$. Crystd from H₂O or MeOH.

1,3,5,5-Tetranitrohexahydropyrimidine [81360-42-1] M 270.1, m 153-154°. Crystd from EtOH (5x), and sublimed (~65°/0.05mm) [*J Org Chem* 47 2474 1982; *J Labelled Comp Radiopharm* 29 1197 1991].

Tetranitromethane [509-14-8] M 196.0, m 14.2°, b 21-23°/23mm, 126°/760mm, d 1.640, n 1.438. Shaken with dilute NaOH, washed, steam distd, dried with Na₂SO₄ and fractionally crystd by partial freezing. The melted crystals were dried with MgSO₄ and fractionally distd under reduced pressure. Shaken with a large volume of dilute NaOH until no absorption attributable to the *aci*-nitro anion (from mono- di- and trinitromethanes) is observable in the water. Then washed with distilled water, and distilled at room temperature by passing a stream of air or nitrogen through the liquid and condensing in a trap at -80°. It can be dried with MgSO₄ or Na₂SO₄, fractionally crystd from the melt, and fractionally distd under reduced pressure.

Tetra(*p*-nitrophenyl)ethylene [47797-98-8] M 512.4, m 306-307°. Crystd from dioxane or AcOH and dried at 150°/0.1mm. [Gorvin *J Chem Soc* 678 1959.]

4,7,13,18-Tetraoxa-1,10-diazabicyclo[8.5.5]eicosane (Cryptand 211) [31250-06-3] M 288.1, $pK_{Est} \sim 7.9$. Redistd, dried under high vacuum over 24h, and stored under nitrogen.

1,7,10,16-Tetraoxa-4,13-diazacyclooctadecane (4,13-diaza-18-crown-6) [23978-55-4] M 262.3, m 118-116°, $pK_{Est} \sim 8.8$. Twice recrystd from *benzene/*n*-heptane, and dried for 24h under high vacuum [E.Weber and F.Vögtle *Top Curr Chem* (Springer Verlag, Berlin) 98 1 1981; D'Aprano and Sesta *J Phys Chem* 91 2415 1987].

Tetrapentylammonium bromide [866-97-7] M 378.5, m 100-101°. See tetra-*n*-amylammonium bromide on p. 356.

Tetraphenylethylene [632-51-9] M 332.4, m 223-224°, b 415-425°/760mm. Crystd from dioxane or from EtOH/*benzene. Sublimed under high vacuum.

Tetraphenylhydrazine [632-52-0] M 336.4, m 147°, $pK_{Est} \sim 0$. Crystd from 1:1 CHCl₃/toluene or CHCl₃/EtOH. Stored in a refrigerator, in the dark.

***trans*-1,1,4,4-Tetraphenyl-2-methylbutadiene** [20411-57-8] M 372.5. Crystd from EtOH.

1,2,3,4-Tetraphenylnaphthalene [751-38-2] M 432.6, m 199-201°, 204-204.5°. Crystd from MeOH or as EtOH. [Fieser and Haddadin *Org Synth* 46 107 1966.]

5,6,11,12-Tetraphenylnaphthacene (Rubrene) [517-51-1] M 532.7, m >315°, 322°, d 1.255. Orange crystals by sublimation at 250-260°/3-4mm [UV Badger and Pearce *Spectrochim Acta* 4 280 1950]. Also recrystd from *benzene under red light because it is chemiluminescent and light sensitive.

5,10,15,20-Tetraphenylporphyrin (TPP) [917-23-7] M 614.7, λ_{\max} 482nm. Purified by chromatography on neutral (Grade I) alumina, and recrystd from CH₂Cl₂/MeOH [Yamashita et al. *J Phys Chem* 91 3055 1987].

Tetra-*n*-propylammonium bromide [1941-30-6] M 266.3, m >280°(dec). Crystd from ethyl acetate/EtOH (9:1), acetone or MeOH. Dried at 110° under reduced pressure.

Tetra-*n*-propylammonium iodide [631-40-3] M 313.3, m >280°(dec). Purified by crystallization from EtOH, EtOH/diethyl ether (1:1), EtOH/water or aqueous acetone. Dried at 50° under vacuum. Stored over P₂O₅ in a vacuum desiccator.

Tetra-*n*-propylammonium perchlorate [15780-02-6] M 285.8, m 239-241°. See tetrapropylammonium perchlorate on p. 483 in Chapter 5.

5,10,15,20-Tetra-4'-pyridinylporphyrin [16834-13-2] M 618.7, m >300°(dec). Purified by chromatography on alumina (neutral, Grade I), followed by crystallization from CH₂Cl₂/MeOH [Yamashita et al. *J Phys Chem* 91 3055 1987].

Tetrathiafulvalene [31366-25-3] M 204.4, m 122-124°. Recrystd from cyclohexane/hexane under an argon atmosphere [Kauzlarich et al. *J Am Chem Soc* 109 4561 1987].

1,2,3,4-Tetrazole [288-94-8] M 70.1, m 156°, pK 4.89 (acidic). Crystd from EtOH, sublimed under high vacuum at ca 120° (care should be taken due to possible EXPLOSION).

Thebaine [115-37-7] M 311.4, m 193°, $[\alpha]_D^{25}$ -219° (EtOH), pK¹⁵ 8.15. Sublimed at 170-180°.

2-Thenoyltrifluoroacetone [326-91-0] M 222.2, m 42-44°, b 96-98°/9mm. Crystd from hexane or *benzene. (Aqueous solutions slowly decompose).

2-Thenylamine [27757-85-3] M 113.1, b 78.5°/15mm, pK³⁰ 8.92. Distd under reduced pressure (nitrogen), from BaO, through a column packed with glass helices.

Theobromine [83-67-0] M 180.2, m 337°, pK₁⁴⁰ -0.16, pK₂²⁵ 9.96. Crystd from water.

Theophylline [58-55-9] M 180.2, m 270-274°, pK₁⁴⁰ -0.24, pK₂ 2.5, pK₃⁴⁰ 8.79. Crystd from H₂O.

Thevetin [11018-93-2] M 858.9, m softens at 194°, m 210°. Crystd (as trihydrate) from isopropanol. Dried at 100°/10mm to give the hemihydrate (very hygroscopic).

Thianthrene [92-85-3] M 216.3, m 158°. Crystd from Me₂CO (charcoal), AcOH or EtOH. Sublimes in a vacuum.

ϵ -[2-(4-Thiazolidone)]hexanoic acid [539-35-5] M 215.3, m 140°, pK_{Est} ~4.7. Crystd from H₂O, Me₂CO or MeOH.

Thiazoline-2-thiol [96-53-7] M 119.2, m 106-107°, 106-108°, pK_{Est} ~13.0. Purified by dissolution in alkali, pptn by addition of HCl and then recrystd from H₂O as needles. [IR: Flett *J Chem Soc* 347 1953 and Mecke et al. *Chem Ber* 90 975; Gabriel and Stelzner *Chem Ber* 28 2931 1895.]

4-(2-Thiazolylazo)resorcinol [2246-46-0] M 221.2, m 200-202°(dec), λ_{\max} 500 nm, $\text{pK}_1^{2.5}$ 1.25, $\text{pK}_2^{2.5}$ 6.53, $\text{pK}_3^{2.5}$ 10.76. Dissolved in alkali, extracted with diethyl ether, and re-ppted with dil HCl. The purity was checked by TLC on silica gel using pet ether/diethyl ether/EtOH (10:10:1) as the mobile phase.

Thietane (trimethylene sulfide) [287-27-4] M 74.1, m -64°, -73.2°, b 93.8-94.2°/752mm, 95°/atm, d_4^{20} 1.0200, n_D^{20} 1.5020. Purified by preparative gas chromatography on a dinonyl phthalate column. It has also been purified by drying over anhydrous K_2CO_3 , and distd through a 25 cm glass helices packed column (for 14g of thietane), then dried over CaSO_4 before sealing in a vac. [Haines et al. *J Phys Chem* 58 270 1954.] It is characterised as the *dimethylsulfonium iodide* m 97-98° [Bennett and Hock *J Chem Soc* 2496 1927]. The *S-oxide* has b 102°/25mm, n_D^{21} 1.5075 [Tamres and Searles *J Am Chem Soc* 81 2100 1959].

Thioacetamide [62-55-5] M 75.1, m 112-113°, $\text{pK}^{2.5}$ 13.4. Crystd from absolute diethyl ether or *benzene. Dried at 70° in vacuum and stored over P_2O_5 at 0° under nitrogen. (*Develops an obnoxious odour on storage*, and absorption at 269nm decreases, hence it should be freshly crystd before use).

Thioacetanilide [677-53-6] M 151.2, m 75-76°, pK_{Est} ~13.1. Crystd from H_2O and dried *in vacuo*.

Thiobarbituric acid [504-17-6] M 144.2, m 235°(dec), $\text{pK}_1^{2.5}$ 2.25, $\text{pK}_2^{2.5}$ 10.72 (2% aq ETOH). Crystd from water.

Thiobenzanilide [636-04-4] M 213.2, m 101.5-102°, pK_{Est} ~12.6. Crystd from MeOH at Dry-ice temperature.

(1R)-(-)-Thiocamphor (1R-bornane-2-thione, 1R-(-)-1,7,7-trimethylbicyclo[2.2.1]heptane-2-thione) [53402-10-1] M 168.3, m 136-138°, 146°, $[\alpha]_D^{22}$ -22° (c 3, EtOAc). Forms red prisms from EtOH and sublimes under vacuum. It possesses a sulfurous odour and is volatile as camphor. [Sen *J Indian Chem Soc* 12 647 1935; 18 76 1941.] The *racemate* crystallises from * C_6H_6 and has m 145° [138.6-139°, White and Bishop *J Am Chem Soc* 62 10 1940].

1,1'-Thiocarbonyldiimidazole [6160-65-2] M 178.1, m 100-102°, 105-106°. It forms yellow crystals by recrystn from tetrahydrofuran or by sublimation at 10^{-3} Torr (bath temp 70-80°). Hydrolysed by H_2O , store dry. [Staab and Walther *Justus Liebigs Ann Chem* 657 98 1962; Pullukat et al. *Tetrahedron Lett* 1953 1967.]

Thiochrome {2,7-dimethyl-5H-thiachromine-8-ethanol; 3,8-dimethyl-2-hydroxyethyl-5H-thiazolo[2,3:1',2']pyrimido[4',5'-d]pyrimidine} [92-35-3] M 262.3, m 227-228°, pK_{Est} ~5.8 (thiazol-N protonation). Crystd from chloroform.

Thiodiglycollic acid [123-93-3] M 150.2, m 129°, $\text{pK}_1^{2.5}$ 3.15 (3.24), $\text{pK}_2^{2.5}$ 4.13 (4.56). Crystd from water.

3,3'-Thiodipropionic acid [111-17-1] M 178.2, $\text{pK}_1^{2.5}$ 3.84, $\text{pK}_2^{2.5}$ 4.66. Crystd from water.

Thioflavine T [2390-54-7] M 318.9, $\text{pK}^{2.5}$ 2.7. Crystd from *benzene/EtOH (1:1).

Thioformamide [115-08-2] M 61.0, m 29°, pK_{Est} ~12.4. Crystd from ethyl acetate or ether/pet ether.

Thioglycollic acid [68-11-1] M 92.1, b 95-96°/8mm, d 1.326, n 1.505, $\text{pK}_1^{2.5}$ 3.42, $\text{pK}_2^{2.5}$ 10.20. Mixed with an equal volume of *benzene, the *benzene is then distd to dehydrate the acid. After heating to 100° to remove most of the *benzene, the residue was distd under vacuum and stored in sealed ampoules at 3°. [Eshelman et al. *Anal Chem* 22 844 1960.]

Thioguanosine (2-amino-6-mercapto-9- β -D-ribofuranosylpurine) [85-31-4] M 299.3, m 230-231°(dec), $[\alpha]_D^{20}$ -64° (c 1.3, 0.1N NaOH), pK 8.33. Crystd (as hemihydrate) from water.