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Thioindigo [522-75-8] M 296.2, m >280°. Adsorbed on silica gel from CCl₄/*benzene (3:1), eluted with *benzene, crystd from CHCl₃ and dried at 60-65°. [Wyman and Brode *J Am Chem Soc* 73 1487 1951.] This paper also gives details of purification of other thioindigo dyes.

Thiomalic (mercaptosuccinic) acid [70-49-5] M 150.2, m 153-154°, pK₁²⁵ 3.64 (3.17), pK₂²⁵ 4.64 (4.67), pK₃²⁵ 10.37 (10.52). Extracted from aqueous soln several times with diethyl ether, and the aqueous soln freeze-dried.

Thio-Michler's Ketone [1226-46-6] M 284.6, λ_{max} 457 nm (ε 2.92 x 10⁴ in 30% aq *n*-propanol). Purified by recrystn from hot EtOH or by triturating with a small volume of CHCl₃, followed by filtration and washing with hot EtOH [Terbell and Wystrade *J Phys Chem* 68 2110 1964].

Thionanthone (thioxanthone) [492-22-8] M 212.3, m 212-213°, b 371-373°/712mm. See 9*H*-thioxanthene-9-one on p. 369.

2-Thionaphthol [91-60-1] M 160.2, m 81°, 82°, b 153.5°/15mm, 286°/760mm, pK_{Est} ~6.1. Crystd from EtOH.

Thionine (3,7-diaminophenothiazine) [135-59-1; 581-64-6 (HCl)] M 263.7, ε₅₉₀ 6.2 x 10⁴ M⁻¹ cm⁻¹, pK¹⁵ 6.9. The standard biological stain is highly pure. It can be crystd from water or 50% EtOH, then chromatographed on alumina using CHCl₃ as eluent [Shepp, Chaberek and McNeil *J Phys Chem* 66 2563 1962]. Dried overnight at 100° and stored in a vacuum. The *hydrochloride* can be crystd from 50% EtOH or dilute HCl and aqueous *n*-butanol. Purified also by column chromatography and washed with CHCl₃ and acetone. Dried *in vacuo* at room temperature.

Thioxine hydrochloride (8-mercaptoquinoline hydrochloride) [34006-16-1] M 197.7, m 170-175° (dec), pK₁²⁵ 2.16, pK₂²⁵ 8.38. Crystallises from EtOH and the crystals are yellow in colour. It has pK_a²⁰ values of 2.05 and 8.29 in H₂O. [UV: Albert and Barlin *J Chem Soc* 2384 1959.]

Thiophane (tetrahydrothiophene) [110-01-0] M 88.2, b 40.3°/39.7mm. See tetrahydrothiophene on p. 361.

Thiophene [110-02-1] M 84.1, f -38.5°, b 84.2°, d 1.525, n 1.52890, n³⁰ 1.5223. The simplest purification procedure is to dry with solid KOH, or reflux with sodium, and fractionally distd through a glass-helices packed column. More extensive treatments include an initial wash with aq HCl, then water, drying with CaSO₄ or KOH, and passage through columns of activated silica gel or alumina. Fawcett and Rasmussen [*J Am Chem Soc* 67 1705 1945] washed thiophene successively with 7M HCl, 4M NaOH, and distd water, dried with CaCl₂ and fractionally distd. *Benzene was removed by fractional crystn by partial freezing, and the thiophene was degassed and sealed in Pyrex flasks. [Also a method is described for recovering the thiophene from the *benzene-enriched portion.]

Thiophene-2-acetic acid [1918-77-0] M 142.2, m 76°, pK²⁵ 3.89. Crystd from ligroin.

Thiophene-3-acetic acid [6964-21-2] M 142.2, m 79-80°, pK_{Est} ~3.1. Crystd from ligroin.

2-Thiophenecarboxaldehyde [98-03-3] M 112.2, b 106°/30mm, d 1.593, n 1.222. Washed with 50% HCl and distd under reduced pressure just before use.

Thiophene-2-carboxylic acid [527-72-0] M 128.2, m 129-130°, pK²⁵ 3.89. Crystd from water.

Thiophene-3-carboxylic acid [88-31-1] M 128.1, m 137-138°, pK²⁵ 6.23. Crystd from water.

Thiophenol (benzenethiol) [108-98-5] M 110.2, f -14.9°, b 46.4°/10mm, 168.0°/760mm, d 1.073, n 1.5897, pK²⁵ 6.62. Dried with CaCl₂ or CaSO₄, and distd at 10mm pressure or at 100mm (b 103.5°) in a stream of nitrogen.

Thiopyronine (2,7-dimethylaminothioxanthane) [2412-14-8] M 318.9, λ_{\max} 564nm (ϵ 78,500) H₂O, pK_{Est} ~ 7. Purified as the hydrochloride by recrystn from hydrochloric acid. [Fanghanel et al. *J Phys Chem* 91 3700 1987.]

Thiosalicylic (2-mercaptobenzoic) acid [147-93-3] M 154.2, m 164-165°, pK₁²⁵ 3.54, pK₂²⁵ 8.80. Crystd from hot EtOH (4mL/g), after adding hot distd water (8mL/g) and boiling with charcoal. The hot soln was filtered, cooled, the solid collected and dried *in vacuo* (P₂O₅). Cryst from AcOH and sublimes *in vacuo*.

Thiosemicarbazide [79-19-6] M 91.1, m 181-183°, pK₁²⁵ 1.88, pK₂²⁵ 12.81. Crystd from water.

Thiothienoyltrifluoroacetone [4552-64-1] M 228.2, m 61-62°. Easily oxidised and has to be purified before use. This may be by recrystd from *benzene or by dissolution in pet ether, extraction into 1M NaOH soln, acidification of the aqueous phase with 1-6M HCl soln, back extraction into pet ether and final evapn of the solvent. The purity can be checked by TLC. It was stored in ampoules under nitrogen at 0° in the dark. [Muller and Rother *Anal Chim Acta* 66 49 1973.]

Thiouracil [141-90-2] M 128.2, m 240°(dec), pK²⁵ 7.52. Crystd from water or EtOH.

Thiourea [62-56-6] M 76.1, m 179°, pK²⁰ -1.19 (aq H₂SO₄). Crystd from absolute EtOH, MeOH, acetonitrile or water. Dried under vacuum over H₂SO₄ at room temperature.

9H-Thioxanthene-9-one (thioxanthone) [492-22-8] M 212.3, m 200-202°, 209°, 212-214°, b 371-373°/712mm. Yellow needles from CHCl₃ or EtOH and sublimes *in vacuo*. Sol in CS₂, hot AcOH and soln in concn H₂SO₄ to give a yellow color with green fluorescence in VIS light. The sulfone has m 187° (from EtOH), and the hydrazone has m 115° (yellow leaflets from EtOH/*C₆H₆). [Szmant et al. *J Org Chem* 18 745 1953; Ullmann et al. *Chem Ber* 49 2509 1916; NMR: Sharpless et al. *Org Magn Res* 6 115 1974.]

L-Threonine [72-19-5] M 119.1, m 251-253°, [α]_D²⁶ -28.4° (H₂O), pK₁²⁵ 2.17, pK₂²⁵ 9.00. Likely impurities are *allo*-threonine and glycine. Crystd from water by adding 4 volumes of EtOH. Dried and stored in a desiccator.

Thymidine [50-89-5] M 242.2, m 185°, pK₂²⁵ 9.65. Crystd from ethyl acetate.

Thymine [65-71-4] M 126.1, m 326°, pK²⁵ 9.82. Crystd from ethyl acetate or water. Purified by preparative (2mm thick) TLC plates of silica gel, eluting with ethyl acetate/isopropanol/water (75:16:9, v/v; R_F 0.75). Spot localised by uv lamp, cut from plate, placed in MeOH, shaken and filtered through a millipore filter, then rotary evapd. [Infante et al. *J Chem Soc, Faraday Trans 1* 68 1586 1973.]

Thymolphthalein complexone [1913-93-5] M 720.8, m 190°(dec), pK₁^{18.2} 7.35, pK₂^{18.2} 12.25. Purification as for phthalein complexone except that it was synthesised from thymolphthalein instead of cresolphthalein.

Tiglic acid [80-59-1] M 100.1, m 63.5-64°, b 198.5°, pK¹⁸ 4.96. Crystd from water.

Tinuvin P (2-[2H-benzotriazol-2-yl]-p-cresol) [50936-05-5] M 225.3, m 131-133°, pK_{Est(1)} ~ 1.6 (N protonation), pK_{Est(2)} ~ 8 (phenolic OH). Recrystd from *n*-heptane or Me₂CO/pentane. [Woessner et al. *J Phys Chem* 81 3629 1985.]

***o*-Tolidine (3,3'-dimethylbenzidine)** [119-93-7] M 212.3, m 131-132°, pK²⁵ 4.45. Dissolved in *benzene, percolated through a column of activated alumina and crystd from *benzene/pet ether.

***p*-Tolualdehyde** [104-87-0] M 120.2, b 83-85°/0.1mm, 199-200°/760mm, d 1.018, n 1.548. Steam distd, dried with CaSO₄ and fractionally distd.

***o*-Toluamide** [527-85-5] M 135.2, m 141°. Crystd from hot water (10mL/g) and dried in air.

Toluene [108-88-3] M 92.1, b 110.6°, d¹⁰ 0.87615, d²⁵ 0.86231, n 1.49693, n²⁵ 1.49413. Dried with CaCl₂, CaH₂ or CaSO₄, and dried further by standing with sodium, P₂O₅ or CaH₂. It can be fractionally distd from sodium or P₂O₅. Unless specially purified, toluene is likely to be contaminated with methylthiophenes and other sulfur containing impurities. These can be removed by shaking with conc H₂SO₄, but the temperature must be kept below 30° if sulfonation of toluene is to be avoided. A typical procedure consists of shaking toluene twice with cold conc H₂SO₄ (100mL of acid per L), once with water, once with aqueous 5% NaHCO₃ or NaOH, again with H₂O, then drying successively with CaSO₄ and P₂O₅, with final distn from P₂O₅ or over LiAlH₄ after refluxing for 30min. Alternatively, the treatment with NaHCO₃ can be replaced by boiling under reflux with 1% sodium amalgam. Sulfur compounds can also be removed by prolonged shaking of the toluene with mercury, or by two distns from AlCl₃, the distillate then being washed with water, dried with K₂CO₃ and stored with sodium wire. Other purification procedures include refluxing and distn of sodium dried toluene from diphenylpicrylhydrazyl, and from SnCl₂ (to ensure freedom from peroxides). It has also been co-distd with 10% by volume of ethyl methyl ketone, and again fractionally distd. [Brown and Pearsall *J Am Chem Soc* 74 191 1952.] For removal of carbonyl impurities see **benzene*. Toluene has been purified by distn under nitrogen in the presence of sodium benzophenone ketyl. Toluene has also been dried with MgSO₄, after the sulfur impurities have been removed, and then fractionally distd from P₂O₅ and stored in the dark [Tabushi et al. *J Am Chem Soc* 107 4465 1985]. Toluene can be purified by passage through a tightly packed column of Fuller's earth.

Rapid purification: Alumina, CaH₂ and 4A molecular sieves (3% w/v) may be used to dry toluene (6h stirring and standing). Then the toluene is distd, discarding the first 5% of distillate, and is stored over molecular sieves (3A, 4A) or Na wire.

Toluene-2,4-diamine [95-80-7] M 122.2, m 99°, b 148-150°/8mm, 292°/760mm, pK_{Est(1)}~2.5, pK_{Est(2)}~4.4. Recrystd from water containing a very small amount of sodium dithionite (to prevent air oxidation), and dried under vacuum. Also cryst from **benzene*.

***o*-Toluenesulfonamide** [88-19-7] M 171.2, m 155.5°. Crystd from hot water, then from EtOH or Et₂O-pet ether.

***p*-Toluenesulfonamide** [70-55-3] M 171.2, m 137-137.5°, 138°. Crystd from hot water, then from EtOH or Et₂O-pet ether.

***p*-Toluenesulfonic acid** [6192-52-5] M 190.2, m 38° (anhydrous), m 105-107° (monohydrate), pK 1.55. Purified by pptn from a satd soln at 0° by introducing HCl gas. Also crystd from conc HCl, then crystd from dilute HCl (charcoal) to remove benzenesulfonic acid. It has been crystd from EtOH/water. Dried in a vacuum desiccator over solid KOH and CaCl₂. *p*-Toluenesulfonic acid can be dehydrated by azeotropic distn with **benzene* or by heating at 100° for 4h under water-pump vacuum. The anhydrous acid can be crystd from **benzene*, CHCl₃, ethyl acetate, anhydrous MeOH, or from acetone by adding a large excess of **benzene*. It can be dried under vacuum at 50°.

Toluenesulfonic acid hydrazide (tosylhydrazide) [1576-35-8] M 186.2, m 108-110°, 109-110°. Dissolve in hot MeOH (~1g/4mL), filter through Celite and ppte material by adding 2-2.5 vols of distd H₂O. Air or vac dry. [Fiedman et al. *Org Synth Coll Vol V* 1055 1973.]

***p*-Toluenesulfonyl chloride (tosyl chloride)** [98-59-9] M 190.7, m 66-69°, 67.5-68.5°, 69°, b 138-139°/9mm, 146°/15mm, 167°/36mm. Material that has been standing for a long time contains tosic acid and HCl and has m ca 65-68°. It is purified by dissolving (10g) in the minimum volume of CHCl₃ (ca 25mL) filtered, and diluted with five volumes (i.e. 125mL) of pet ether (b 30-60°) to precipitate impurities. The soln is filtered, clarified with charcoal and concentrated to 40mL by evaporation. Further evaporation to a very small volume gave 7g of white crystals which were analytically pure, m 67.5-68.5°. (The insoluble material was largely tosic acid and had m 101-104°). [Pelletier *Chem Ind (London)* 1034 1953.] Also crystd from toluene/pet ether in the cold, from pet ether (b 40-60°) or **benzene*. Its soln in diethyl ether has been washed with aqueous 10% NaOH until colourless, then dried (Na₂SO₄) and crystd by cooling in

powdered Dry-ice. It has also been purified by dissolving in *benzene, washing with aqueous 5% NaOH, then dried with K_2CO_3 or $MgSO_4$, and distd under reduced pressure and can be sublimed at high vacuum [Ebel *Chem Ber* **60** 2086/1927].

p-Toluenethiol [106-45-6] M 124.2, m 43.5-44°, pK^{25} 6.82. Crystd from pet ether (b 40-70°).

Tolhydroquinone (2-methylbenzene-1,4-diol) [95-71-6] M 124.1, m 128-129°, pK_1^{20} 10.15, pK_2^{20} 11.75. Crystd from EtOH.

o-Toluic acid [118-90-1] M 136.2, m 102-103°, pK^{25} 3.91. Crystd from *benzene (2.5mL/g) and dried in air.

m-Toluic acid [99-04-7] M 136.2, m 111-113°, pK^{25} 4.27. Crystd from water.

p-Toluic acid [99-94-5] M 136.2, m 178.5-179.5°, pK^{25} 4.37. Crystd from water, water/EtOH (1:1), MeOH/water or *benzene.

o-Toluidine (2-methylaniline) [95-53-4] M 107.2, f -16.3°, b 80.1°/10mm, 200.3°/760mm, d 0.999, n 1.57246, n^{25} 1.56987, pK^{25} 4.45. In general, methods similar to those for purifying aniline can be used, e.g. distn from zinc dust, at reduced pressure, under nitrogen. Berliner and May [*J Am Chem Soc* **49** 1007 1927] purified *via* the oxalate. Twice-distd *o*-toluidine was dissolved in four times its volume of diethyl ether and the equivalent amount of oxalic acid needed to form the dioxalate was added as its soln in diethyl ether. (If *p*-toluidine is present, its oxalate pptes and can be removed by filtration.) Evapn of the ether soln gave crystals of *o*-toluidine dioxalate. They were filtered off, recrystd five times from water containing a small amount of oxalic acid (to prevent hydrolysis), then treated with dilute aqueous Na_2CO_3 to liberate the amine which was separated, dried ($CaCl_2$) and distd under reduced pressure.

m-Toluidine (3-methylaniline) [108-44-1] M 107.2, f -30.4°, b 82.3°/10mm, 203.4°/760mm, d 0.989, n 1.56811, n^{25} 1.56570, pK^{25} 4.71. It can be purified as for aniline. Twice-distd, *m*-toluidine was converted to the hydrochloride using a slight excess of HCl, and the salt was fractionally crystd from 25% EtOH (five times), and from distd water (twice), rejecting, in each case, the first material that crystd. The amine was regenerated and distd as for *o*-toluidine. [Berliner and May *J Am Chem Soc* **49** 1007 1927.]

p-Toluidine (4-methylaniline) [106-49-0] M 107.2, m 44.8°, b 79.6°/10mm, 200.5°/760mm, d 0.962, n 1.5636, $n^{59.1}$ 1.5534, pK^{25} 5.08. In general, methods similar to those for purifying aniline can be used. It can be separated from the *o*- and *m*-isomers by fractional crystn from its melt. *p*-Toluidine has been crystd from hot water (charcoal), EtOH, *benzene, pet ether or EtOH/water (1:4), and dried in a vacuum desiccator. It can also be sublimed at 30° under vacuum. For further purification, use has been made of the oxalate, the sulfate and acetylation. The oxalate, formed as described for *o*-toluidine, was filtered, washed and recrystd three times from hot distd water. The base was regenerated with aq Na_2CO_3 and recrystd three times from distd water. [Berliner and May *J Am Chem Soc* **49** 1007 1927.] Alternatively, *p*-toluidine was converted to its acetyl derivative which, after repeated crystn from EtOH, was hydrolysed by refluxing (50g) in a mixture of 500mL of water and 115mL of conc H_2SO_4 until a clear soln was obtained. The amine sulfate was isolated, suspended in water, and NaOH was added. The free base was distd twice from zinc dust under vacuum. The *p*-toluidine was then recrystd from pet ether and dried in a vacuum desiccator or in a vacuum for 6h at 40°. [Berliner and Berliner *J Am Chem Soc* **76** 6179 1954; Moore et al. *J Am Chem Soc* **108** 2257 1986.]

Toluidine Blue O [93-31-9] M 305.8, CI 52040, λ_{max} 626nm, pK^{25} 7.5. Crystd from hot water (18mL/g) by adding one and a half volumes of alcohol and chilling on ice. Dried at 100° in an oven for 8-10h.

p-Toluidine hydrochloride [540-23-8] M 143.6, m 245.9-246.1°. Crystd from MeOH containing a few drops of conc HCl. Dried under vacuum over paraffin chips.

2-*p*-Toluidinylnaphthalene-6-sulfonic acid [7724-15-4] M 313.9, $pK_{Est} \sim 0$. Crystd twice from 2% aqueous KOH and dried under high vacuum for 4h at room temperature. Crystd from water. Tested for purity by TLC on silica gel with isopropanol as solvent. The free acid was obtained by acidifying a satd aqueous soln.

***o*-Tolunitrile** [529-19-1] M 117.2, b 205.2°, d 0.992, n 1.5279. Fractionally distd, washed with conc HCl or 50% H₂SO₄ at 60° until the smell of isonitrile had gone (this also removed any amines), then washed with saturated NaHCO₃ and dilute NaCl solns, then dried with K₂CO₃ and redistd.

***m*-Tolunitrile** [620-22-4] M 117.2, b 209.5-210°/773mm, d 0.986, n 1.5250. Dried with MgSO₄, fractionally distd, then washed with aqueous acid to remove possible traces of amines, dried and redistd.

***p*-Tolunitrile** [104-85-8] M 117.2, m 29.5°, b 104-106°/20mm. Melted, dried with MgSO₄, fractionally crystd from its melt, then fractionally distd under reduced pressure in a 6-in spinning band column. [Brown *J Am Chem Soc* 81 3232 1959.] It can also be crystd from *benzene/pet ether (b 40-60°).

4-Tolyl-2-benzoic acid (4'-methylbiphenyl-2-carboxylic acid) [7148-03-0] M 196.2, m 138-139°, $pK^{25} 3.64$. Crystd from toluene.

***p*-Tolylacetic acid** [622-47-9] M 150.2, m 94°. See 4-methylphenylacetic acid on p. 297.

***p*-Tolyl carbinol (4-methylbenzyl alcohol)** [589-18-4] M 122.2, m 61°, b 116-118°/20mm, 217°/760mm. Crystd from pet ether (b 80-100°, 1g/mL). It can also be distd under reduced pressure.

***p*-Tolyl disulfide** [103-19-5] M 246.4, m 45-46°. Purified by chromatography on alumina using hexane as eluent, then crystd from MeOH. [Kice and Bowers *J Am Chem Soc* 84 2384 1962.]

***p*-Tolylsulfonylmethyl isocyanide (tosylmethyl isocyanide, TOSMIC)** [36635-61-7] M 195.2, m 114-115°(dec), 116-117°(dec). Use an efficient fume cupboard. Purify by dissolving TOSMIC (50g) in CH₂Cl₂ (150mL) and pass through a column (40x3cm) containing neutral alumina (100g) in CH₂Cl₂ and eluting with CH₂Cl₂. A nearly colorless soln (700mL) is collected, evaporated *in vacuo* and the residue (42-47g) of TOSMIC (m 113-114° dec) is recrystd once from MeOH (m 116-117° dec). [Hoogenboom et al. *Org Synth* 57 102 1977; Lensen *Tetrahedron Lett* 2367 1972..] Also recrystd from EtOH (charcoal) [Saito and Itano, *J Chem Soc, Perkin Trans 1* 1 1986].

***p*-Tolyl urea** [622-51-5] M 150.2, m 181°. Crystd from EtOH/water (1:1).

***trans*-Traumatic acid (2-dodecene-1,12-dioic acid)** [6402-36-4] M 228.3, m 165-166°, 150-160°/0.001mm, $pK_{Est(1)} \sim 4.2$, $pK_{Est(2)} \sim 4.6$. Crystd from EtOH, acetone or glyme.

α, α' -Trehalose (2H₂O) [6138-23-4] M 378.3, m 96.5-97.5°, 203° (anhydrous). Crystd (as the dihydrate) from aqueous EtOH. Dried at 13°.

1,2,3-Triaminopropane trihydrochloride [free base 21291-99-6] M 198.7, m 250°, $pK_1^{20} 3.72$, $pK_2^{20} 7.95$, $pK_3^{20} 9.59$. Crystd from EtOH.

1,5,7-Triazabicyclo[4.4.0]dec-5-ene (TBD, 1,3,4,6,7,8-hexahydro-2*h*-pyrimido[1,2-*a*]-pyrimidine) [5807-14-7] M 139.2, m 125-130°, $pK \sim 16$ Cryst from Et₂O but readily forms white crystals of the carbonate. It is a strong base (see pK , i.e. about 100 times more basic than tetramethylguanidine. The *picrate* has m 220.5-222° (from EtOH). Forms the *5-nitro derivative* m 14.5-160° that gives a *5-nitro nitrate* salt m 100-101° (from EtOH-Et₂O) and a *5-nitro picrate* m 144-145° (from H₂O). [McKay and Kreling *Can J Chem* 35 1438 1957; Schwesinger *Chimia* 39 369 1985; Hilpert et al. *J Chem Soc, Chem Commun* 1401 1983; Kamfen and Eschenmoser *Helv Chim Acta* 72 185 1989].

1,2,4-Triazole [288-88-0] M 69.1, m 121°, 260°, $pK_1^{25} 2.27$ (basic), $pK_2^{25} 10.26$ (acidic). Crystd from EtOH or water [Barszcz et al. *J Chem Soc, Dalton Trans* 2025 1986].

Tribenzylamine [620-40-6] M 287.4, m 93-94°, 230°/13mm, $pK_{Est} < 0$. Crystd from abs EtOH or pet ether. Dried in a vacuum over P_2O_5 at room temperature. *HCl* has m 226-228° (from EtOH) and *picrate* has m 191° (from H_2O or aq EtOH).

2,4,6-Tribromoacetanilide [607-93-2] M 451.8, m 232°. Crystd from EtOH.

2,4,6-Tribromoaniline [147-82-0] M 329.8, m 120°, $pK_{Est} \sim -0.5$ (aq H_2SO_4). Crystd from MeOH.

sym-Tribromobenzene [626-39-1] M 314.8, m 122°. Crystd from glacial acetic acid/water (4:1), then washed with chilled EtOH and dried in air.

Tribromochloromethane [594-15-0] M 287.2, m 55°. Melted, washed with aqueous $Na_2S_2O_3$, dried with BaO and fractionally crystd from its melt.

2,4,6-Tribromophenol [118-79-6] M 330.8, m 94°, pK^{25} 6.00. Crystd from EtOH or pet ether. Dried under vacuum over P_2O_5 at room temperature.

Tri-*n*-butylamine [102-82-9] M 185.4, b 68°/3mm, 120°/44mm, d 0.7788, n 1.4294, pK^{25} 9.93. Purified by fractional distn from sodium under reduced pressure. Pegolotti and Young [*J Am Chem Soc* 83 3251 1961] heated the amine overnight with an equal volume of acetic anhydride, in a steam bath. The amine layer was separated and heated with water for 2h on the steam bath (to hydrolyse any remaining acetic anhydride). The soln was cooled, solid K_2CO_3 was added to neutralize any acetic acid that had been formed, and the amine was separated, dried (K_2CO_3) and distd at 44mm pressure. Davis and Nakshbendi [*J Am Chem Soc* 84 2085 1926] treated the amine with one-eighth of its weight of benzenesulfonyl chloride in aqueous 15% NaOH at 0-5°. The mixture was shaken intermittently and allowed to warm to room temperature. After a day, the amine layer was washed with aq NaOH, then water and dried with KOH. (This treatment removes primary and secondary amines.) It was further dried with CaH_2 and distd under vacuum.

Tri-*n*-butylammonium hydrobromide [37026-85-0] M 308.3, m 75.2-75.9°. Crystd from ethyl acetate.

Tri-*n*-butylammonium nitrate [33850-87-2] M 304.5. Crystd from mixtures of *n*-hexane and acetone (95:5). Dried over P_2O_5 .

Tri-*n*-butylammonium perchlorate [14999-66-7] M 285.5. Recrystd from *n*-hexane.

sym-Tri-*tert*-butylbenzene [1460-02-2] M 246.4, m 73.4-73.9°. Crystd from EtOH.

2,4,6-Tri-*tert*-butylphenol [732-26-3] M 262.4, m 129-132°, 131°/1mm, 147°/10mm, 278°/760mm, pK^{25} 12.19. Crystd from *n*-hexane or several times from 95% EtOH until the EtOH soln was colourless [Balasubramanian and Bruice *J Am Chem Soc* 108 5495 1986]. It has also been purified by sublimation [Yuan and Bruice *J Am Chem Soc* 108 1643 1986; Wong et al. *J Am Chem Soc* 109 3428 1987]. Purification has been achieved by passage through a silica gel column followed by recrystn from *n*-hexane [Kajii et al. *J Phys Chem* 91 2791 1987].

Tricarballic acid (propane-1,2,3-tricarboxylic acid) [99-14-9] M 176.1, m 166°, pK_1^{25} 3.47, pK_2^{25} 4.54, pK_3^{25} 5.89. Crystd from diethyl ether.

Trichloroacetamide [594-65-0] M 162.4, m 139-141°, b 238-240°. Its xylene soln was dried with P_2O_5 , then fractionally distd.

Trichloroacetanilide [2563-97-5] M 238.5, m 95°. Crystd from *benzene.

Trichloroacetic acid [76-03-9] M 163.4, m 59.4-59.8°, pK²⁵ 0.51. Purified by fractional crystn from its melt, then crystd repeatedly from dry *benzene and stored over conc H₂SO₄ in a vac desiccator. It can also be crystd from CHCl₃ or cyclohexane, and dried over P₂O₅ or Mg(ClO₄)₂ in a vac desiccator. Trichloroacetic acid can be fractionally distd under reduced pressure from MgSO₄. Layne, Jaffé and Zimmer [*J Am Chem Soc* 85 435 1963] dried trichloroacetic acid in *benzene by distilling off the *benzene-water azeotrope, then crystd the acid from the remaining *benzene soln. Manipulations were carried out under nitrogen. [Use a well ventilated fume cupboard].

2,3,4-Trichloroaniline [634-67-3] M 196.5, m 67.5°, b 292°/774mm, pK_{Est} ~1.3. Crystd from ligroin.

2,4,5-Trichloroaniline [636-30-6] M 196.5, m 96.5°, b 270°/760mm, pK 1.09. Crystd from ligroin.

2,4,6-Trichloroaniline [634-93-5] M 196.5, m 78.5°, b 127°/14mm, 262°/746mm, pK 0.03. Crystd from ligroin.

1,2,3-Trichlorobenzene [87-61-6] M 181.5, m 52.6°. Crystd from EtOH.

1,2,4-Trichlorobenzene [120-82-1] M 181.5, m 17°, b 210°. Separated from a mixture of isomers by washing with fuming H₂SO₄, then water, drying with CaSO₄ and slowly fractionally distilling. [Jensen, Marino and Brown *J Am Chem Soc* 81 3303 1959.]

1,3,5-Trichlorobenzene [108-70-3] M 181.5, m 64-65°. Recrystd from dry *benzene or toluene.

3,4,5-Trichloro-*o*-cresol (3,4,5-trichloro-2-methylphenol) [608-92-4] M 211.5, m 77°, pK_{Est} ~7.6. Crystd from pet ether.

2,3,5-Trichloro-*p*-cresol [608-91-3] M 211.5, m 66-67°, pK_{Est} ~6.9. Crystd from pet ether.

1,1,1-Trichloroethane [71-55-6] M 133.4, f -32.7°, b 74.0°, d 1.337, n 1.4385. Washed successively with conc HCl (or conc H₂SO₄), aq 10% K₂CO₃ (Na₂CO₃), aq 10% NaCl, dried with CaCl₂ or Na₂SO₄, and fractionally distd. It can contain up to 3% dioxane as preservative. This is removed by washing successively with 10% aq HCl, 10% aq NaHCO₃ and 10% aq NaCl; and distd over CaCl₂ before use.

1,1,2-Trichloroethane [79-00-5] M 133.4, f -36.3°, b 113.6°, d 1.435, n 1.472. Purification as for 1,1,1-trichloroethane above.

Trichloroethylene [79-01-6] M 131.4, f -88°, b 87.2°, d 1.463, n²¹ 1.4767. Undergoes decomposition in a similar way to CHCl₃, giving HCl, CO, COCl₂ and organic products. It reacts with KOH, NaOH and 90% H₂SO₄, and forms azeotropes with water, MeOH, EtOH, and acetic acid. It is purified by washing successively with 2M HCl, water and 2M K₂CO₃, then dried with K₂CO₃ and CaCl₂, and fractionally distd immediately before use. It has also been steam distd from 10% Ca(OH)₂ slurry, most of the water being removed from the distillate by cooling to -30° to -50° and filtering off the ice through chamois skin: the trichloroethylene was then fractionally distd at 250mm pressure and collected in a blackened container. [Carlisle and Levine *Ind Eng Chem (Anal Ed)* 24 1164 1932.]

2,4,5-Trichloro-1-nitrobenzene [89-69-0] M 226.5, m 57°. Crystd from EtOH.

3,4,6-Trichloro-2-nitrophenol [82-62-2] M 242.4, m 92-93°, pK_{Est} ~4.1. Crystd from pet ether or EtOH.

2,4,5-Trichlorophenol [95-95-4] M 197.5, m 67°, b 72°/1mm, pK²⁵ 7.0. Crystd from EtOH or pet ether.

2,4,6-Trichlorophenol [88-06-2] M 197.5, m 67-68°, pK²⁵ 6.23. Crystd from *benzene, EtOH or EtOH/water.

3,4,5-Trichlorophenol [609-19-8] M 197.5, m 100°, pK²⁵ 7.84. Crystd from pet ether/*benzene mixture.

2,4,5-Trichlorophenoxyacetic acid (2,4,5-T) [93-76-5] M 255.5, m 153°, 155-158°, pK²⁵ 2.83. Crystd from *benzene. (CANCER SUSPECT)

1,1,2-Trichlorotrifluoroethane [76-13-1] M 187.4, b 47.6°/760mm, d 1.576, n 1.360. Washed with water, then with weak alkali. Dried with CaCl₂ or H₂SO₄ and distd. [Locke et al. *J Am Chem Soc* 56 1726 1934.]

Tricycloquinazoline [195-84-6] M 230.3, m 322-323°. Crystd repeatedly from toluene, followed by vac sublimation at 210° at a pressure of 0.15-0.3 Torr in subdued light.

Tridecanoic acid [638-53-9] M 214.4, m 41.8°, 44.5-45.5° (several forms), b 199-200°/24mm, pK_{Est} ~5.0. Crystd from acetone.

7-Tridecanone [462-18-0] M 198.4, m 33°, b 255°/766mm. Crystd from EtOH.

Tri-*n*-dodecylammonium nitrate [2305-34-2] M 585.0. Crystd from *n*-hexane/acetone (95:5) and kept in a desiccator over P₂O₅.

Tri-*n*-dodecylammonium perchlorate [5838-82-4] M 622.4. Recrystd from *n*-hexane or acetone and kept in a desiccator over P₂O₅.

Triethanolamine hydrochloride [637-39-8] M 185.7, m 177°, pK²⁵ 7.92 (free base). Crystd from EtOH. Dried at 80°.

1,1,2-Triethoxyethane [4819-77-6] M 162.2, b 164°, d 0.897, n 1.401. Dried with Na₂SO₄, and distd.

Triethylamine [121-44-8] M 101.2, b 89.4°, d 0.7280, n 1.4005, pK²⁵ 10.82. Dried with CaSO₄, LiAlH₄, Linde type 4A molecular sieves, CaH₂, KOH, or K₂CO₃, then distd, either alone or from BaO, sodium, P₂O₅ or CaH₂. It has also been distd from zinc dust, under nitrogen. To remove traces of primary and secondary amines, triethylamine has been refluxed with acetic anhydride, benzoic anhydride, phthalic anhydride, then distd, refluxed with CaH₂ (ammonia-free) or KOH (or dried with activated alumina), and again distd. Another purification involved refluxing for 2h with *p*-toluenesulfonyl chloride, then distd. Grovenstein and Williams [*J Am Chem Soc* 83 412 1961] treated triethylamine (500mL) with benzoyl chloride (30mL), filtered off the ppte, and refluxed the liquid for 1h with a further 30mL of benzoyl chloride. After cooling, the liquid was filtered, distd, and allowed to stand for several hours with KOH pellets. It was then refluxed with, and distd from, stirred molten potassium. Triethylamine has been converted to its hydrochloride, crystd from EtOH (to m 254°), then liberated with aq NaOH, dried with solid KOH and distd from sodium under nitrogen.

Triethylammonium hydrobromide [636-70-4] M 229.1, m 248°. Equimolar portions of triethylamine and aqueous solutions of HBr in acetone were mixed. The ppted salt was washed with anhydrous acetone and dried in vacuum for 1-2h. [Odinekov et al. *J Chem Soc, Faraday Trans 2* 80 899 1984.] Recrystd from CHCl₃ or EtOH.

Triethylammonium hydrochloride [554-68-7] M 137.7, m 257-260°(dec). Purified like the bromide above.

Triethylammonium hydroiodide [4636-73-1] M 229.1, m 181°. Purified as for triethylammonium bromide, except the soln for pptn was precooled acetone at -10° and the ppte was twice recrystd from a cooled acetone/hexane mixture at -10°.

Triethylammonium trichloroacetate [4113-06-8] M 263.6. Equimolar solns of triethylamine and trichloroacetic acid in *n*-hexane were mixed at 10°. The solid so obtained was recrystd from CHCl₃/*benzene.

Triethylammonium trifluoroacetate [454-49-9] M 196.2. Purified as for the corresponding trichloroacetate. The salt was a colourless liquid at ambient temperature.

1,2,4-Triethylbenzene [877-44-1] M 162.3, b 96.8-97.1°/12.8mm, d 0.8738, n 1.5015. For separation from a commercial mixture see Dillingham and Reid [*J Am Chem Soc* 60 2606 1938].

1,3,5-Triethylbenzene [102-25-0] M 162.3, b 102-102.5°, d 0.8631, n 1.4951. For separation from a commercial mixture see Dillingham and Reid [*J Am Chem Soc* 60 2606 1938].

Triethylene glycol [112-27-6] M 150.2, b 115-117°/0.1mm, 278°/760mm, n¹⁵ 1.4578, d¹⁵ 1.1274. Dried with CaSO₄ for 1 week, then repeatedly and very slowly fractionally distd under vacuum. Stored in a vacuum desiccator over P₂O₅. It is very *hygroscopic*.

Triethylene glycol dimethyl ether (triglyme) [112-49-2] M 178.2, b 225°, d 0.987, n 1.425. Refluxed with, and distd from sodium hydride or LiAlH₄.

Triethylenetetramine (TRIEN, TETA, trientine) [112-24-3] M 146.2, m 12°, b 157°/20mm, d 0.971, n 1.497, pK₁^{2.5} 3.32, pK₂^{2.5} 6.67, pK₃^{2.5} 9.20, pK₄^{2.5} 9.92. Dried with sodium, then distd under vac. Further purification has been *via* the nitrate or the chloride. For example, Jonassen and Strickland [*J Am Chem Soc* 80 312 1958] separated TRIEN from admixture with TREN (38%) by soln in EtOH, cooling to approximately 5° in an ice-bath and adding conc HCl dropwise from a burette, keeping the temperature below 10°, until all of the white crystalline ppte of TREN.HCl had formed and been removed. Further addition of HCl then ppted thick creamy white TRIEN.HCl which was crystd several times from hot water by adding an excess of cold EtOH. The crystals were finally washed with Me₂CO, then Et₂O and dried in a vacuum desiccator.

Triethylenetetramine tetrahydrochloride (TRIEN.HCl) [4961-10-4] M 292.1, m 266-270°. Crystd repeatedly from hot water by pptn with cold EtOH or EtOH/HCl. Washed with acetone and abs EtOH and dried in a vacuum oven at 80° (see TRIEN above).

Triethyl orthoformate (1,1,1-triethoxymethane) [122-51-0] M 148.2, m 30°, b 60°/30mm, 144-146°, d 0.891, n 1.392. Fractionate first at atm press, then in a vac. If impure, then wash with H₂O, dry over anhyd K₂CO₃, filter and fractionate through a Widmer column. [Sah and Ma *J Am Chem Soc* 54 2964 1932; Ohme and Schmitz *Justus Liebigs Ann Chem* 716 207 1968.] **IRRITANT** and **FLAMMABLE**.

Triethyloxonium fluoroborate [368-39-8] M 190.0, m 92-93°(dec). Crystd from diethyl ether. *Very hygroscopic*, and should be handled in a dry box and stored at 0°. [*Org Synth* 46 113 1966.] Pure material should give a clear and colourless soln in dichloromethane (1 in 50, w/v).

Trifluoroacetic acid [76-05-1] M 114.0, f -15.5°, b 72.4°, d 1.494, n 1.2850, pK^{2.5} 0.52. The purification of trifluoroacetic acid, reported in earlier editions of this work, by refluxing over KMnO₄ for 24h and slowly distilling has resulted in very **SERIOUS EXPLOSIONS** on various occasions, but not always. This apparently depends on the source and/or age of the acid. The method is **NOT RECOMMENDED**. Water can be removed by adding trifluoroacetic anhydride (0.05%, to diminish water content) and distd. [Conway and Novak *J Phys Chem* 81 1459 1977]. It can be refluxed and distd from P₂O₅. It is further purified by fractional crystn by partial freezing and again distd. **Highly TOXIC vapour**.

Trifluoroacetic anhydride [407-25-0] M 210.0, b 38-40°/760mm, d 1.508. Purification by distilling over KMnO_4 , as for the acid above is **EXTREMELY DANGEROUS** due to the possibility of **EXPLOSION**. It is best purified by distilling from P_2O_5 slowly, and collecting the fraction boiling at 39.5°. Store in a dry atmosphere. **Highly TOXIC vapour**.

1,1,1-Trifluoro-2-bromoethane [421-06-7] M 163.0. See 1-bromo-3,3,3-trifluoroethane on p. 142.

2,2,2-Trifluoroethanol [75-89-8] M 100.0, b 72.4°/738mm, d 1.400, pK^{25} 12.8. Dried with CaSO_4 and a little NaHCO_3 (to remove traces of acid). **Highly TOXIC vapour**.

Trifluoromethanesulfonic anhydride (triflic anhydride) [358-23-6] M 282.1, b 82-85°, 84°, d 1.71, n 1.322. Distil through a short Vigreux column. Could be freshly prepd from the anhydrous acid (11.5g) and P_2O_5 (11.5g, or half this weight) by setting aside at room temp for 1h, distilling off volatile products then through a short Vigreux column. Readily hydrolysed by H_2O and decomposes appreciably after a few days to liberate SO_2 and produce a viscous liquid. Store dry at low low temp. [Burdon et al. *J Chem Soc* 2574 1957; Beard et al. *J Org Chem* 38 373 1973.] **Highly TOXIC vapour**.

4-(Trifluoromethyl)acetophenone [709-63-7] M 188.2, m 31-33°, b 79-81°/9mm. Purified by distillation or sublimation *in vacuo*.

3-Trifluoromethyl-4-nitrophenol [88-30-2] M 162.1, m 81°, $\text{pK}_{\text{Est}} \sim 6.1$. Crystd from *benzene or from pet ether/*benzene mixture.

α,α,α -Trifluorotoluene (benzotrifluoride) [98-08-8] M 144.1, b 102.5°, d 1.190, n^{30} 1.4100. Purified by repeated treatment with boiling aqueous Na_2CO_3 (until no test for chloride ion was obtained), dried with K_2CO_3 , then with P_2O_5 , and fractionally distd.

Triglycyl glycine (tetraglycine) [637-84-3] M 246.2, m 270-275°(dec). Crystd from distilled water (optionally, by the addition of EtOH).

Trigonelline (1-methylnicotinic acid zwitterion) [535-83-1] M 137.1, m 218°(dec). Crystd (as monohydrate) from aqueous EtOH, then dried at 100°.

2,3,4-Trihydroxybenzoic acid [610-02-6] M 170.1, m 207-208°, $\text{pK}_{\text{Est}(1)} \sim 3.4$, $\text{pK}_{\text{Est}(2)} \sim 7.8$, $\text{pK}_{\text{Est}(3)} > 12$. Crystd from water.

2,4,6-Trihydroxybenzoic acid [83-30-7] M 170.1, m 205-212°(dec), $\text{pK}_{\text{Est}(1)} \sim 1.5$, $\text{pK}_{\text{Est}(2)} \sim 8.0$, $\text{pK}_{\text{Est}(3)} > 12$. Crystd from water.

4',5,7-Trihydroxyflavone (apigenin) [520-36-5] M 270.2, m 296-298°, 300-305°, 345-350° (pK 's 7—10, for phenolic OH). Crystd from aq pyridine or aq EtOH. Dyes wool yellow when with added Cr.

3,4,5-Triiodobenzoic acid [2338-20-7] M 499.8, m 289-290°, 293°, pK^{25} 0.65. Crystd from aqueous EtOH or water.

3,4,5-Triiodobenzyl chloride [52273-54-8] M 504.3, m 138°. Crystd from CCl_4 /pet ether (charcoal).

3,3',5-Triiodo-S-thyronine [6893-02-3] M 651.0, m 236-237°(dec), $[\alpha]_{\text{D}}^{29.5} + 21.5^\circ$ (EtOH/1M aq HCl, 2:1), pK_1^{25} 6.48, pK_2^{25} 7.62, pK_3^{25} 7.82. Likely impurities are as in *thyroxine*. Purified by dissolving in dilute NH_3 at room temperature, then crystd by addition of dilute acetic acid to pH 6.

Trimellitic (benzene-1,2,4-tricarboxylic) acid [528-44-9] M 210.1, m 218-220°, pK_1^{25} 2.42, pK_2^{25} 3.71, pK_3^{25} 5.01. Crystd from acetic acid or aqueous EtOH.

1,2,3-Trimethoxybenzene [634-36-6] M 168.2, m 45-46°. Sublimed under vacuum.

1,3,5-Trimethoxybenzene [621-23-8] M 168.2, m 53°. Sublimed under vacuum.

Trimethylamine [75-50-3] M 59.1, b 3.5°, pK²⁵ 9.80. Dried by passage of the gas through a tower filled with solid KOH. Water and impurities containing labile hydrogen were removed by treatment with freshly sublimed, ground, P₂O₅. Has been refluxed with acetic anhydride, and then distd through a tube packed with HgO and BaO. [Comyns *J Chem Soc* 1557 1955.] For more extensive purification, trimethylamine has been converted to the hydrochloride, crystd (see below), and regenerated by treating the hydrochloride with excess aq 50% KOH, the gas passing through a CaSO₄ column into a steel cylinder containing sodium ribbon. After 1-2 days, the cylinder was cooled at -78° and hydrogen and air were removed by pumping. [Day and Felsing *J Am Chem Soc* 72 1698 1950.] Trimethylamine has also been trap-to-trap distd and then freeze-pump-thaw degassed [Halpern et al. *J Am Chem Soc* 108 3907 1986].

Trimethylamine hydrochloride [593-81-7] M 95.7, m >280°(dec). Crystd from CHCl₃, EtOH or *n*-propanol, and dried under vacuum. It has also been crystd from *benzene/MeOH, MeOH/diethyl ether and dried under vacuum over paraffin wax and H₂SO₄. Stood over P₂O₅. It is *hygroscopic*.

Trimethylamine hydroiodide [20230-89-1] M 186.0, m 263°. Crystd from MeOH.

1,2,4-Trimethylbenzene (pseudocumene) [95-63-6] M 120.2, m -43.8°, b 51.6°/10mm, 167-168°/760mm, d 0.889, n 1.5048. Refluxed over sodium and distd under reduced pressure.

2,4,6-Trimethylbenzoic acid (mesitoic acid) [480-63-7] M 164.2, m 155°, pK²⁵ 3.45. Crystd from water, ligroin or carbon tetrachloride [Ohwada et al. *J Am Chem Soc* 108 3029 1986].

Trimethyl-1,4-benzoquinone [935-92-2] M 150.1, m 29-30°, 36°, b 98°/10mm, 108°/18mm. Distd in a vac or sublimed *in vacuo* before use. [Smith et al. *J Am Chem Soc* 60 318 1939.]

R-(-)-2,2,6-Trimethyl-1,4-cyclohexanedione [60046-49-3] M 154.2, m 88-90°, 91-92°, [α]_D²⁰ -270° (c 0.4%, MeOH), [α]_D²⁰ -275° (c 1, CHCl₃). Obtained from fermentation and purified by recrystn from diisopropyl ether. [ORD: Leuenberger et al. *Helv Chim Acta* 59 1832 1976.] The *racemate* has m 65-67° and the 4-(4-phenyl)semicarbazone has m 218-220° (from CH₂Cl₂-MeOH) [Isler et al. *Helv Chim Acta* 39 2041 1956.]

2,2,5-Trimethylhexane [3522-94-9] M 128.3, m -105.8°, b 124.1°, d 0.716, n 1.39971, n²⁵ 1.39727. Extracted with conc H₂SO₄, washed with H₂O, dried (type 4A molecular sieves), and fractionally distd.

Trimethyl-1,4-hydroquinone (2,3,5-trimethylbenzene-1,4-diol) [700-13-0] M 152.2, m 173-174°, pK_{Est(1)}~ 11.1, pK_{Est(2)}~ 12.7. Recrystd from water, under anaerobic conditions.

1',3',3'-Trimethyl-6-nitrospiro[2H-benzopyran-2,2'-indoline] [1498-88-0] M 322.4, m 180°. Recrystd from absolute EtOH [Hinnen et al. *Bull Soc Chim Fr* 2066 1968; Ramesh and Labes *J Am Chem Soc* 109 3228 1987].

Trimethylolpropane [77-99-6] M 134.2, m 57-59°. Crystd from acetone and ether.

2,2,3-Trimethylpentane [564-02-3] M 114.2, b 109.8°, d 0.7161, n 1.40295, n²⁵ 1.40064. Purified by azeotropic distn with 2-methoxyethanol, which was subsequently washed out with water. The trimethylpentane was then dried and fractionally distd. [Forziati et al. *J Res Nat Bur Stand* 36 129 1946.]

2,2,4-Trimethylpentane (isooctane) [540-84-1] M 114.2, b 99.2°, d 0.693, n 1.39145, n²⁵ 1.38898. Distd from sodium, passed through a column of silica gel or activated alumina (to remove traces of

olefins), and again distd from sodium. Extracted repeatedly with conc H_2SO_4 , then agitated with aqueous KMnO_4 , washed with water, dried (CaSO_4) and distd. Purified by azeotropic distn with EtOH, which was subsequently washed out with water, and the trimethylpentane was dried and fractionally distd. [Forziati et al. *J Res Nat Bur Stand* **36** 126 1946.] Also purified by fractional crystn.

2,3,5-Trimethylphenol [697-82-5] M 136.2, m 95-96°, b 233°/760mm, pK^{25} 10.67. Crystd from water or pet ether.

2,4,5-Trimethylphenol [496-78-6] M 136.2, m 70.5-71.5°, pK^{25} 10.57. Crystd from water.

2,4,6-Trimethylphenol [527-60-6] M 136.2, m 69°, b 220°/760mm, pK^{25} 10.86. Crystd from water and sublimed *in vacuo*.

3,4,5-Trimethylphenol [527-54-8] M 136.2, m 107°, b 248-249°/760mm, pK^{25} 10.25. Crystd from pet ether.

Trimethylphenylammonium benzenesulfonate [16093-66-6] M 293.3. Crystd repeatedly from MeOH (charcoal).

2,2,4-Trimethyl-6-phenyl-1,2-dihydroquinoline [3562-69-4] M 249.3, m 102°. Vacuum distd, then crystd from absolute EtOH.

2,4,6-Trimethylpyridine (sym-collidine) [108-75-8] M 121.2, m -46°, b 10°/2.7mm, 36-37°/2mm, 60.7°/13mm, 65°/31mm, 170.4°/760mm, 175-178°/atm, d^{25} 0.9100, n_D^{20} 1.4939, 1.4981, n^{25} 1.4959, pK^{25} 6.69. Commercial samples may be grossly impure. Likely contaminants include 3,5-dimethylpyridine, 2,3,6-trimethylpyridine and water. Brown, Johnson and Podall [*J Am Chem Soc* **76** 5556 1954] fractionally distd 2,4,6-trimethylpyridine under reduced pressure through a 40-cm Vigreux column and added to 430mL of the distillate slowly, with cooling to 0°, 45g of BF_3 -diethyl etherate. The mixture was again distd, and an equal volume of dry *benzene was added to the distillate. Dry HCl was passed into the soln, which was kept cold in an ice-bath, and the hydrochloride was filtered off. It was recrystd from abs EtOH (1.5mL/g) to m 286-287°(sealed tube). The free base was regenerated by treatment with aq NaOH, then extracted with *benzene, dried (MgSO_4) and distd under reduced pressure. Sisler et al. [*J Am Chem Soc* **75** 446 1953] pted trimethylpyridine as its phosphate from a soln of the base in MeOH by adding 85% H_3PO_4 , shaking and cooling. The free base was regenerated as above. Garrett and Smythe [*J Chem Soc* 763 1903] purified the trimethylpyridine via the HgCl_2 complex. It is more soluble in cold than hot H_2O [sol 20.8% at 6°, 3.5% at 20°, 1.8% at 100°].

Also purified by dissolving in CHCl_3 , adding solid K_2CO_3 and Drierite, filtering and fractionally distilling through an 8in helix packed column. The *sulfate* has m 205°, and the *picrate* (from hot H_2O) has m 155-156°. [Frank and Meikle *J Am Chem Soc* **72** 4184 1950.]

Trimethylsulfonium iodide [2181-42-2] M 204.1, m 215-220°(dec). Crystd from EtOH.

1,3,7-Trimethyluric acid [5415-44-1] M 210.2, m 345°(dec), pK 6.0. Crystd from water.

1,3,9-Trimethyluric acid [7464-93-9] M 210.2, m 340°, 347°, pK^{20} 9.39. Crystd from water.

1,7,9-Trimethyluric acid [55441-82-2] M 210.2, m 316-318 (dec)°, 345°, pK_{Est} ~9.0. Crystd from water or EtOH, and sublimed *in vacuo*.

Trimyristin [555-45-3] M 723.2, m 56.5°. Crystd from diethyl ether.

2,4,6-Trinitroanisole [606-35-9] M 243.1, m 68°. Crystd from EtOH or MeOH. Dried under vac.

1,3,6-Trinitrobenzene [99-35-4] M 213.1, m 122-123°. Crystd from glacial acetic acid, CHCl₃, CCl₄, EtOH aq EtOH or EtOH/*benzene, after (optionally) heating with dil HNO₃. Air dried. Fused, and crystd under vacuum.

2,4,6-Trinitrobenzenesulfonic acid hydrate (TNBS, picrylsulfonic acid) [2508-19-2] M 293.2, m 180°, λ_{max} 240nm (ε 650 M⁻¹cm⁻¹), pK_{Est} ~ <0. It is also available as 0.1M and 5%w/v solns in H₂O. Recrystd from 1M HCl and dried at 100° or a mixt of EtOH (50mL), H₂O (30mL) and conc HCl (70mL) for 65g of acid. The *diethanolamine salt* had m 182-183° [Golumbic *J Org Chem* 11 518 1946].

2,4,6-Trinitrobenzoic acid [129-66-8] M 225.1, m 227-228°, pK²⁵ 0.65. Crystd from distilled water. Dried in a vacuum desiccator.

2,4,6-Trinitro-*m*-cresol [602-99-3] M 243.1, m 107.0-107.5°, pK 2.8. Crystd successively from H₂O, aq EtOH and *benzene/cyclohexane, then dried at 80° for 2h. [Davis and Paabo *J Res Nat Bur Stand* 64A 533 1960.]

2,4,7-Trinitro-9-fluorenone [129-79-3] M 315.2, m 176°. Crystd from nitric acid/water (3:1), washed with water and dried under vacuum over P₂O₅, or recrystd from dry *benzene.

2,4,6-Trinitrotoluene (TNT) [118-96-7] M 227.1, m 81.0-81.5°. Crystd from *benzene and EtOH. Then fused and allowed to cryst under vacuum. Gey, Dalbey and Van Dolah [*J Am Chem Soc* 78 1803 1956] dissolved TNT in acetone and added cold water (1:2:15), the ppte was filtered, washed free from solvent and stirred with five parts of aq 8% Na₂SO₃ at 50-60° for 10min. It was filtered, washed with cold water until the effluent was colourless, and air dried. The product was dissolved in five parts of hot CCl₄, washed with warm water until the washings were colourless and TNT was recovered by cooling and filtering. It was recrystd from 95% EtOH and carefully dried over H₂SO₄. The dry solid should not be heated without taking precautions for a possible **EXPLOSION**.

2,4,6-Trinitro-*m*-xylene [632-92-8] M 241.2, m 182.2°. Crystd from ethyl methyl ketone.

Tri-*n*-octylamine [1116-76-3] M 353.7, b 164-168°/0.7mm, 365-367°/760mm, d 0.813, n 1.450, pK²⁵ 10.65. It was converted to the amine hydrochloride etherate which was recrystd four times from diethyl ether at -30° (see below). Neutralisation of this salt regenerated the free amine. [Wilson and Wogman *J Phys Chem* 66 1552 1962.] Distd at 1-2mm pressure.

Tri-*n*-octylammonium chloride [1188-95-0] M 384.2, m 78-79°, pK 8.35 (in 70% aq EtOH). Crystd from Et₂O, then *n*-hexane (see above). [Burrows et al. *J Chem Soc* 200 1947.]

Tri-*n*-octylammonium perchlorate [2861-99-6] M 454.2, m >300°(dec). Crystd from *n*-hexane.

1,3,5-Trioxane [110-88-3] M 90.1, m 64°, b 114.5°/759mm. Crystd from sodium-dried diethyl ether or water, and dried over CaCl₂. Purified by zone refining.

Trioxsalen (2,5,9-trimethyl-7H-furo[3,2-*g*]benzopyran-7-one) [3902-71-4] M 228.3, m 233-235°, 234.5-235°. Purified by recrystn from CHCl₃. If too impure it is fractionally crystd from CHCl₃-pet ether (b 30-60°) using Norit and finally crystd from CHCl₃ alone to give colourless prisms, m 234.5-235°. It is a photosensitiser so it should be stored in the dark. [UV: Kaufmann *J Org Chem* 26 117 1961; Baeme et al. *J Chem Soc* 2976 1949.]

Tripalmitin [555-44-2] M 807.4, m 66.4°. Crystd from acetone, diethyl ether or EtOH.

Triphenylamine [603-34-9] M 245.3, m 127.3-127.9°, pK -5.0 (in fluorosulfuric acid). Crystd from EtOH or from *benzene/abs EtOH, diethyl ether and pet ether. It was sublimed under vacuum and carefully dried in a vacuum line. Stored in the dark under nitrogen.

1,3,5-Triphenylbenzene [612-71-5] M 306.4, m 173-175°. Purified by chromatography on alumina using *benzene or pet ether as eluents.

Triphenylene [217-59-4] M 228.3, m 198°, b 425°. Purified by zone refining or crystn from EtOH or CHCl₃, and sublimed.

1,2,3-Triphenylguanidine [101-01-9] M 287.3, m 144°, pK 9.10. Crystd from EtOH or EtOH/water, and dried under vacuum.

Triphenylmethane [519-73-3] M 244.3, m 92-93°. Crystd from EtOH or *benzene (with one molecule of *benzene of crystallisation which is lost on exposure to air or by heating on a water bath). It can also be sublimed under vacuum. It can also be given a preliminary purification by refluxing with tin and glacial acetic acid, then filtered hot through a glass sinter disc, and pptd by addition of cold water.

Triphenylmethanol (triphenylcarbinol) [76-84-6] M 260.3, m 164°, b 360-380° (without dec), pK²⁵ -6.63 (aq H₂SO₄). Crystd from EtOH, MeOH, CCl₄ (4mL/g), *benzene, hexane or pet ether (b 60-70°). Dried at 90°. [Ohwada et al. *J Am Chem Soc* 108 3029 1986.]

Triphenylmethyl chloride (trityl chloride) [76-83-5] M 278.9, m 111-112°. Crystd from iso-octane. Also crystd from 5 parts of pet ether (b 90-100°) and 1 part of acetyl chloride using 1.8g of solvent per g of chloride. Dried in a desiccator over soda lime and paraffin wax. [*Org Synth Coll Vol III* 841 1955; Thomas and Rochow *J Am Chem Soc* 79 1843 1957; Moisel et al. *J Am Chem Soc* 108 4706 1986.]

2,3,5-Triphenyltetrazolium chloride (TTC) [298-96-4] M 334.8, m 243°(dec). Crystd from EtOH or CHCl₃, and dried at 105°.

Tri-*n*-propylamine [102-69-2] M 143.3, b 156.5°, d 0.757, n 1.419, pK²⁵ 10.66. Dried with KOH and fractionally distd. Also refluxed with toluene-*p*-sulfonyl chloride and with KOH, then fractionally distd. The distillate, after addn of 2% phenyl isocyanate, was redistd and the residue fractionally distd from sodium. [Takahashi et al. *J Org Chem* 52 2666 1987.]

Tripyridyl triazine [3682-35-7] M 312.3, m 245-248°. Purified by repeated crystn from aq EtOH.

Tris-(2-aminoethyl)amine (TREN) [4097-89-6] M 146.2, b 114°/15mm, 263°/744mm, d 0.977, n 1.498, pK₁²⁵ 8.42, pK₂²⁵ 9.44, pK₃²⁵ 10.13. For a separation from a mixture containing 62% TRIEN, see entry under triethylenetetramine. Also purified by conversion to the hydrochloride (see below), recrystn and regeneration of the free base [Xie and Hendrickson *J Am Chem Soc* 109 6981 1987].

Tris-(2-aminoethyl)amine trihydrochloride [14350-52-8] M 255.7, m 300°(dec). Crystd several times by dissolving in a minimum of hot water and precipitating with excess cold EtOH. The ppte was washed with acetone, then diethyl ether and dried in a vacuum desiccator.

Tris(d,d-dicamopholylmethanato)europium (III) [52351-64-1] M 108.5, m 220-227.5°, 229-232°, [α]_D²⁵ +28.6° (c 5.4, CCl₄; and varies markedly with concentration). Dissolve in pentane, filter from any insol material, evaporate to dryness and dry the residue (white powder) at 100°/0.1mm for 36h. The IR has ν 1540cm⁻¹. [McCreary et al. *J Am Chem Soc* 96 1038 1974.]

Tris-(dimethylamino)methane (N,N,N',N',N'',N''-hexamethylmethanetriamine) [5762-56-1] M 145.3, b 42-43°/12mm, n 1.4349, pK_{Est} ~ 10. Dry over KOH and dist through a Vigreux column at waterpump vacuum. Store in absence of CO₂. [Bredereck et al. *Chem Ber* 101 1885 1968 and *Angew Chem, Int Ed Engl* 5 132 1966.]

Tris-(hydroxymethyl)methylamine (TRIS) [77-86-1] M 121.1, m 172°, pK²⁵ 8.07. Tris can ordinarily be obtained in highly pure form suitable for use as an acidimetric standard. If only impure material is available, it should be crystd from 20% EtOH. Dry in a vacuum desiccator over P₂O₅ or CaCl₂.

Alternatively, it is dissolved in twice its weight of water at 55-60°, filtered, concd to half its volume and poured slowly, with stirring, into about twice the volume of EtOH. The crystals which separate on cooling to 3-4° are filtered off, washed with a little MeOH, air dried by suction, then finally ground and dried in a vacuum desiccator over P₂O₅. It has also been crystd from water, MeOH or aq MeOH, and vacuum dried at 80° for 2 days.

Tris-(hydroxymethyl)methylamonium hydrochloride (TRIS-HCl) [1185-53-1] M 157.6, m 149-150°(dec). Crystd from 50% EtOH, then from 70% EtOH. Tris-hydrochloride is also available commercially in a highly pure state. Otherwise, crystd from 50% EtOH, then 70% EtOH, and dried below 40° to avoid risk of decomposition.

1,1,1-Tris-(hydroxymethyl)ethane (2-hydroxymethyl-2-methyl-1,3-propanediol) [77-85-0] M 120.2, m 200°. Dissolved in hot tetrahydrofuran, filtered and ppted with hexane. It has also been crystd from acetone/water (1:1). Dried in vacuum.

N-Tris-(hydroxymethyl)methyl-2-aminomethanesulfonic acid (TES) [7365-44-8] M 229.3, m 224-226°(dec), pK²⁰ 7.50. Crystd from hot EtOH containing a little water.

N-Tris-(hydroxymethyl)methylglycine (TRICINE) [5704-04-1] M 179.2, m 186-188°(dec), pK₁²⁰ ~2.3, pK₂²⁰ 8.15. Crystd from EtOH and water.

Tris-(hydroxymethyl)nitromethane [2-(hydroxymethyl)-2-nitro-1,3-propanediol] [126-11-4] M 151.1, m 174-175°(dec, tech grade), 214°(pure). Crystd from CHCl₃/ethyl acetate or ethyl acetate/*benzene. It is an acid and a 0.1M sol in H₂O has pH 4.5. **IRRITANT.**

Tris-[(3-trifluoromethylhydroxymethylene)-d-camphorato] europium (III) [Eu(tfc)₃] [34830-11-0] M 893.6, m 195-299° (dec), ~220°, [α]_D²⁴ +152° (c 2, CCl₄; and varies markedly with concentration). Purified by extraction with pentane, filtered and filtrate evapd and the residual bright yellow amorphous powder is dried at 100°/0.1mm for 36h. A sample purified by fractional molecular distn at 180-200°/0.004mm gave a liquid which solidified and softened at ~130° and melted at ~180° and was analytically pure. IR (CCl₄) v: 1630-1680cm⁻¹ and NMR (CCl₄) δ broad: -1.3 to 0.5, -0.08 (s), 0.41 (s), 1.6-2.3 and 3.39 (s). [McCreary et al. *J Am Chem Soc* 96 1038 1974; ; Goering et al. *J Am Chem Soc* 93 5913 1971.]

1,3,5-Trithiane [291-21-4] M 138.3, m 220°(dec). Crystd from acetic acid.

Triuret (1,3-dicarbamoylurea) [556-99-0] M 146.1, m 233°(dec). Crystd from aq ammonia. Gives mono and dipotassium salts.

Tropaeolin 00, [554-73-4] M 316.3, pK_{Est(2)} ~ 5.8, pK_{Est(3)} ~ 10.3. Recrystd twice from water [Kolthoff and Gus *J Am Chem Soc* 60 2516 1938].

Tropaeolin 000 (see Orange II p. 477 in Chapter 5). Purified by salting out from hot distilled water using sodium acetate, then three times from distilled water and twice from EtOH.

3-Tropanol (Tropine) [120-29-6] M 141.2, m 63°, b 229°/760mm, pK¹⁵ 3.80. Distd in steam and crystd from diethyl ether. *Hygroscopic.*

dl-Tropic (3-hydroxy-2-phenylpropionic) acid [529-64-6] M 166.2, m 118°, pK²⁵ 4.12. Crystd from water or *benzene.

Tropolone [533-75-5] M 122.1, m 49-50°, b 81-84°/0.1mm, pK₁ -0.53 (protonation of CO, aq H₂SO₄), pK₂ 6.67 (acidic OH). Crystd from hexane or pet ether and sublimed at 40°/4mm.

Tryptamine [(3-2-aminoethyl)indole] [61-54-1] M 160.1, m 116°, pK₁²⁵ -6.31 (aq H₂SO₄, diprotonation), pK_{Est(2)} ~4.9, pK₃²⁵ 16.60 (acidic indole NH). Crystd from *benzene.

Tryptamine hydrochloride [343-94-2] M 196.7, m 252-253°. Crystd from EtOH/water.

L-Tryptophan [73-22-3] M 204.3, m 278°, $[\alpha]_D^{20}$ -33.4° (EtOH), $[\alpha]_{546}^{20}$ -36° (c 1, H₂O), pK_1^{25} -6.23 (aq H₂SO₄), pK_2^{25} 2.46, pK_3^{25} 9.41, pK_4^{25} 14.82 (acidic NH, in aq NaOH). Crystd from water/EtOH, washed with anhydrous diethyl ether and dried at room temperature under vac over P₂O₅.

Tryptophol [3-(2-hydroxyethyl)indole] [526-55-6] M 161.2, m 59°, b 174°/2mm. Crystd from diethyl ether/pet ether, *C₆H₆, *C₆H₆/pet ether. The *picrate* has m 98-100° (from *C₆H₆).

(+)-Tubocurarine chloride (5H₂O) [57-94-3] M 771.7, m 274-275°(dec) (anhydrous), $[\alpha]_{546}^{20}$ +235° (c 0.5, H₂O), $pK_{Est(1)}$ ~8.5, $pK_{Est(2)}$ ~8.8. Crystd from water and forms various hydrates.

D(+)-Turannose [547-25-1] M 342.3, m 168-170°, $[\alpha]_D^{20}$ +88° (c 4, H₂O). Crystd from water by addition of EtOH.

Tyramine (4-hydroxybenzylamine) [51-67-2] M 137.2, m 164-165°, pK_1^{25} 9.74 (OH), pK_2^{25} 10.52 (NH₂). Crystd from *benzene or EtOH.

Tyramine hydrochloride [60-19-5] M 173.6, m 274-276°. Crystd from EtOH by addition of diethyl ether, or from conc HCl.

Tyrocidine A (cyclic decapeptide antibiotic with two D-Phe amino acids) [1481-70-5] M 1268.8, m 240°(dec), $[\alpha]_D^{25}$ -115° (c 0.91, MeOH). Crystd as hydrochloride from MeOH or EtOH and HCl. [Paladin and Craig *J Am Chem Soc* 76 688 1954; King and Craig *J Am Chem Soc* 77 6624 1955; Okamoto et al. *Bull Chem Soc Jpn* 50 231 1977.]

L-Tyrosine [60-18-4] M 181.2, m 290-295°(dec), $[\alpha]_D^{25}$ -10.0° (5M HCl), pK_1^{25} 2.18 (CO₂H), pK_2^{25} 9.21 (OH), pK_3^{20} 10.47 (NH₂). Likely impurities are L-cysteine and the ammonium salt. Dissolved in dilute ammonia, then crystd by adding dilute acetic acid to pH 5. Also crystd from water or EtOH/water, and dried at room temperature under vacuum over P₂O₅.

Umbelliferone (7-hydroxycoumarin) [93-35-6] M 162.2, m 225-228°, pK_{Est} ~8.0. Crystd from water.

Undecan-1-ol [112-42-5] M 172.3, m 16.5°. Purified by repeated fractional crystn from its melt or by distn in a vacuum.

Undec-10-enoic acid [112-38-9] M 184.3, m 25-25.5°, b 131°/1mm, 168°/15mm, pK_{Est} ~5.0. Purified by repeated fractional crystn from its melt or by distn in a vacuum.

Uracil [66-22-8] M 122.1, m 335°(dec), pK_1^{25} 9.43, pK_2^{25} 13.3-14.2. Crystd from water.

Uramil (5-aminobarbituric acid) [118-78-5] M 143.1, m 310-312°, 320°, >400°(dec), $pK_{Est(1)}$ ~3.9, $pK_{Est(2)}$ ~8.0, $pK_{Est(3)}$ ~12.5. Crystd from water.

Urea [57-13-6] M 60.1, m 132.7-132.9°, pK^{25} 0.12. Crystd twice from conductivity water using centrifugal drainage and keeping the temperature below 60°. The crystals were dried under vacuum at 55° for 6h. Levy and Margouls [*J Am Chem Soc* 84 1345 1962] prepared a 9M soln in conductivity water (keeping the temperature below 25°) and, after filtering through a medium-porosity glass sinter, added an equal volume of absolute EtOH. The mixture was set aside at -27° for 2-3 days and filtered cold. The ppt was washed with a small amount of EtOH and dried in air. Crystn from 70% EtOH between 40° and -9° has also been used. Ionic impurities such as ammonium isocyanate have been removed by treating the conc aqueous soln at 50° with