Previous Page

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, H_2O), pK_1^{25} -6.23 (aq H_2SO_4) , 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 P_2O_5.

Tryptophol [3-(2-hydroxyethyl)indole] *[526-55-6]* **M 161.2, m 59°, b 174°/2mm.** Crystd from diethyl ether/pet ether, *C_6H_6, *C_6H_6/pet ether. The *picrate* has **m** 98-100° (from *C_6H_6).

(+)-Tubocurarine chloride ($5H_2O$) *[57-94-3]* **M 771.7, m 274-275°(dec) (anhydrous), $[\alpha]_{546}^{20}$ +235° (c 0.5, H_2O), $pK_{Est(1)}$~8.5, $pK_{Est(2)}$~8.8.** Crystd from water and forms various hydrates.

D(+)-Turanose *[547-25-1]* **M 342.3, m 168-170°, $[\alpha]_D^{20}$ +88° (c 4, H_2O).** 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 (NH_2).** 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 (CO_2H), pK_2^{25} 9.21 (OH), pK_3^{20} 10.47 (NH_2).** 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 P_2O_5.

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 ppte 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

Amberlite MB-1 cation- and anion-exchange resin, and allowing to crystallise. [Benesch, Lardy and Benesch *J Biol Chem* **216** 663 *1955*.] Also crystd from MeOH or EtOH, and dried under vacuum at room temperature.

Urea nitrate *[124-47-0]* **M 123.1, m 152°(dec).** Crystd from dilute HNO_3.

Uric acid *[69-93-2]* **M 168.1, m >300°, pK_1 5.75, pK_2 10.3.** Crystd from hot distilled water.

Uridine *[58-96-8]* **M 244.2, m 165°, $[\alpha]_D^{20}$ +4.0° (H_2O), pK^{25} 9.51 (9.25).** Crystd from aqueous 75% MeOH.

Urocanic acid (4-imidazolylacrylic acid) *[104-98-3]* **M 138.1, m 225°, 226-228°, $pK_{Est(1)}$~2.5, $pK_{Est(2)}$~6, $pK_{Est(3)}$~11.** Crystd from water and dried at 100°.

Ursodeoxycholic acid *[128-13-2]* **M 392.5, m 203°, $[\alpha]_D^{20}$ +60° (c 0.2, EtOH), pK_{Est} ~4.8.** Crystd from EtOH.

(+)-Usnic acid [2,6-diacetyl-3,7,9-trihydroxy-8,9b-dimethyldibenzofuran-1(2*H*)-one] *[7562-61-0]* **M 344.3, m 204°.** See (+)-usnic acid on p. 573 in Chapter 6.

trans-Vaccenic acid

(octadec-11-enoic acid) *[693-72-1]* **M 282.5, m 43-44°, pK_{Est} ~ 4.9.** Crystd from acetone. The *methyl ester* has **b** 174-175°/5mm.

***n*-Valeraldehyde** *[110-62-3]* **M 86.1, b 103°, d 0.811, n^{25} 1.40233.** Purified *via* the bisulfite derivative. [Birrell and Trotman-Dickinson *J Chem Soc* 2059 *1960*.]

***n*-Valeramide (pentanamide)** *[626-97-1]* **M 101.1, m 115-116°.** Crystd from EtOH.

Valeric acid (*n*-pentanoic acid) *[109-52-4]* **M 102.1, b 95°/22mm, 186.4°/atm, d 0.938, n 1.4080, pK^{25} 4.81.** Water was removed from the acid by distn using a Vigreux column, until the boiling point reached 183°. A few crystals of $KMnO_4$ were added, and after refluxing, the distn was continued, [Andrews and Keefer *J Am Chem Soc* **83** 3708 *1961*.]

δ-Valerolactam (2-piperidone) *[675-20-7]* **M 99.1, m 38.5-39.5°, 39-40°, 40°, b 81-82°/0.1mm, 136-137°/15mm, pK 0.75 (in AcOH).** Purified by repeated fractional distn. [Cowley *J Org Chem* **23** 1330 *1958*; Reppe et al. *Justus Liebigs Ann Chem* **596** 198 *1955*; IR: Huisgen et al. *Chem Ber* **90** 1437 *1957*.] The *hydrochloride* has **m** 183-184° (from isoPrOH or EtOH-Et_2O) [Hurd et al. *J Org Chem* **17** 865 *1952*], and the *oxime* has **m** 122.5° (from pet ether) [Behringer and Meier *Justus Liebigs Ann Chem* **607** 67 *1957*]. *Picrate* has **m** 92-93°.

γ-Valerolactone (± 4,5-dihydro-5-methyl-2(3*H*)-furanone) *[108-29-2]* **M 100.1, m -37°, 36°, b 82-85°/10mm, 102-103°/28mm, 125.3°/68mm, 136°/100mm, 205.75-206.25°/754mm, d_4^{20} 1.072, n_D^{20} 1.4322.** Purified by repeated fractional distillation [Boorman and Linstead *J Chem Soc* 577, 580 *1933*]. IR ν: 1790 (CS_2), 1775 ($CHCl_3$) cm^{-1} [Jones et al. *Can J Chem* **37** 2007 *1959*]. The *BF_3-complex* distils at 110-111°/20mm [Reppe et al. *Justus Liebigs Ann Chem* **596** 179 *1955*]. It is characterised by conversion to γ-hydroxy-*n*-valeramide by treatment with NH_3, **m** 51.5-52° (by slow evapn of a $CHCl_3$ soln).

δ-Valerolactone (tetrahydro-2*H*-pyran-2-one) *[542-28-9]* **M 100.1, m -13°, -12°, b 88°/4mm, 97°/10mm, 124°/24mm, 145-146°/40mm, 229-229.5°/atm, d_4^{20} 1.1081, n_D^{20} 1.4568.** Purified by repeated fractional distn. IR ν: 1750 (in CS_2), 1732 (in $CHCl_3$), 1748 (in CCl_4) and 1733 (in MeOH) cm^{-1}. [Huisgen and Ott *Tetrahedron* **6** 253 *1959*; Linstead and Rydon *J Chem Soc* 580 *1933*; Jones et al. *Can J Chem* **37** 2007 *1959*.]

Valeronitrile *[110-59-8]* **M 83.1, b 142.3°, d 0.799, n^{15} 1.39913, n^{30} 1.39037.** Washed with half its volume of conc HCl (twice), then with saturated aqueous $NaHCO_3$, dried with $MgSO_4$ and fractionally distd from P_2O_5.

L-Valine *[72-18-4]* **M 117.2, m 315°, $[\alpha]_D^{20}$ +266.7° (6M HCl), pK_1 2.26, pK_2 9.68.** Crystd from water by addition of EtOH.

Vanillin (4-hydroxy-3-methoxybenzaldehyde) *[121-33-5]* **M 152.2, m 83°, b 170°/15mm, pK^{25} 7.40.** Crystd from water or aqueous EtOH, or by distn *in vacuo*.

Veratraldehyde *[120-14-9]* **M 166.2, m 42-43°.** Crystd from diethyl ether, pet ether, CCl_4 or toluene.

Variamine Blue RT [4-(phenylamino)benzenediazonium sulfate (1:1)] *[4477-28-5]* **M 293.3, CI 37240, λ_{max} 377 nm.** Dissolved 10g in 100mL of hot water. Sodium dithionite (0.4g) was added, followed by active carbon (1.5g) and filtered hot. To the colourless or slightly yellow filtrate a soln of saturated NaCl was added and the mixture cooled. The needles were filtered off, washed with cold water, dried at room temperature, and stored in a dark bottle (light sensitive). [Erdey *Chem Analyst* **48** 106 *1959*.]

Vicine (2,4-diamino-5-β-D-glucopyranosidoxy-6-hydroxypyrimidine) *[152-93-2]* **M 304.3, m 243-244°, $[\alpha]_D^{20}$ -12° (c 4, 0.2N NaOH).** Crystd from water or aqueous 85% EtOH, and dried at 135°.

Vinyl acetate *[108-05-4]* **M 86.1, b 72.3°, d 0.938, n 1.396.** Inhibitors such as hydroquinone, and other impurities are removed by drying with $CaCl_2$ and fractionally distilling under nitrogen, then refluxing briefly with a small amount of benzoyl peroxide and redistilling under nitrogen. Stored in the dark at 0°.

9-Vinylanthracene *[2444-68-0]* **M 204.3, m 65-67°, b 61-66°/10mm.** Purified by vacuum sublimation. Also by chromatography on silica gel with cyclohexane as eluent, and recrystd from EtOH [Werst et al. *J Am Chem Soc* **109** 32 *1987*].

Vinyl butoxyethyl ether *[4223-11-4]* **M 144.2.** Washed with aqueous 1% NaOH, dried with CaH_2, then refluxed with and distd from, sodium.

***N*-Vinylcarbazole** *[1484-13-5]* **M 193.3, m 66°.** Crystd repeatedly from MeOH in amber glassware. Vacuum sublimed.

Vinylene carbonate *[872-36-6]* **M 86.1, m 22°.** Purified by zone melting.

Vinyl chloroformate *[5130-24-5]* **M 106.5, b 46.5°/80mm, 67-69°/atm, 109-110°/760mm, d_4^{20} 1.136, n_D^{23} 1.420.** It has been fractionated through a Todd column (Model A with ~60 plates, see p. 174) under atmospheric pressure and purity can be checked by gas chromatography. It has IR with ν at 3100 + 2870 (CH_2), 1780 (C=O), 1640 (C=C) and 940 (CH_2 out-of-plane) and 910 (CH_2 wagging) cm^{-1}. [IR: Lee *J Org Chem* **30** 3943 *1965*; Levaillant *Ann Chim (Paris)* **6** 504 *1936*.] Used for protecting NH_2 groups in peptide synthesis [Olofson et al. *Tetrahedron Lett* 1563 *1977*].

1-Vinylnaphthalene *[826-74-4]* **M 154.2, b 124-125°/15mm.** Fractionally distd under reduced pressure on a spinning-band column, dried with CaH_2 and again distd under vacuum. Stored in sealed ampoules in a freezer.

2-Vinylpyridine monomer *[100-69-6]* **M 105.1, b 79-82°/29mm, d 0.974, n 1.550, pK^{25} 4.92.** Steam distd, then dried with $MgSO_4$ and distd under vacuum.

4-Vinylpyridine monomer *[100-43-6]* **M 105.1, b 40-41°/1.4mm, 54°/5mm, 58-61°/12mm, 68°/18mm, 79°/33mm, d_4^{20} 0.9836, n_D^{20} 1.5486, pK^{25} 5.62.** Purified by fractional distillation under a good vacuum and in a N_2 atmosphere and stored in sealed ampoules under N_2, and kept in the dark at -20°.

The *picrate* has **m** 175-176°. [UV: Coleman and Fuoss *J Am Chem Soc* **77** 5472 *1955*; Overberger et al. *J Polymer Sci* **27** 381 *1958*; Petro and Smyth *J Am Chem Soc* **79** 6142 *1957*.] Used for alkylating SH groups in peptides [Anderson and Friedman *Can J Biochem* **49** 1042 *1971* ; Cawins and Friedman *Anal Biochem* **35** 489 *1970*].

Vinyl stearate *[111-63-7]* **M 310.5, m 35°, b 166°/1.5mm.** Vacuum distd under nitrogen, then crystd from acetone (3mL/g) or ethyl acetate at 0°.

Violanthrene (dibenzanthrene, 5,10-dihydroviolanthrene A) *[81-31-2]* **M 428.5.** Purified by vacuum sublimation over Cu in a muffle furnace at 450°/25mm in a CO_2 atmosphere [Scholl and Meyer *Chem Ber* **67** 1229 *1934*]. *Violanthrene A* (anthro[9,1,2-cde]benzo[rst]pentaphene *[188-87-4]* **M** 426.5 has **m** 506°. [Clar *Chem Ber* **76** 458 *1943*.]

Viologen (4,4'-dipyridyl dihydrochloride) *[27926-72-3]* **M 229.1, m >300°.** Purified by pptn on adding excess of acetone to a concentrated solution in aqueous MeOH. It has also been recrystd several times from MeOH and dried at 70° under vacuum for 24h [Prasad et al. *J Am Chem Soc* **108** 5135 *1986*], and recrystd three times from MeOH/isopropanol [Stramel and Thomas *J Chem Soc, Faraday Trans* **82** 799 *1986*].

Visnagin (4-methoxy-7-methyl-5*H*-furo[3,2-g][1]benzopyran-5-one) *[82-57-5]* **M 230.2, m 142-145°.** Crystd from water.

dl-Warfarin (4-hydroxy-3-(3-oxo-1-phenylbutyl)-2*H*-1-benzopyran-2-one) *[81-81-2]* **M 308.3, m 161°.** Crystd from MeOH. The *acetate* has **m** 182-183° and *2,4-dinitrophenylhydrazone* has **m** 215-216°. Effective anticoagulant and rodenticide.

Xanthatin (3-methylene-7-methyl-6-[3-oxo-1-buten-1-yl]cyclohept-5-ene-[10,11-*b*]furan-2-one) *[26791-73-1]* **M 246.3, m 114.5-115°, $[\alpha]_D$ -20° (EtOH).** Crystd from MeOH or EtOH. UV: λ_{max} 213 and 275nm (ε 22800 and 7300).

Xanthene *[92-83-1]* **M 182.2, m 100.5°, b 310-312°/760mm.** Crystd from *benzene or EtOH.

9-Xanthenone (xanthone) *[90-47-1]* **M 196.2, m 175.6-175.4°.** Crystd from EtOH (25mL/g) and dried at 100°. It has also been recrystd from *n*-hexane three times and sublimed *in vacuo*. [Saltiel *J Am Chem Soc* **108** 2674 *1986*].

Xanthopterin (H_2O) see entry on p. 576 in Chapter 6.

Xanthorhamnin *[1324-63-6]* **M 770.7, m 195°, $[\alpha]_D^{20}$ +3.75° (EtOH).** Crystd from a mixture of ethyl and isopropyl alcohols, air dried, then dried for several hours at 110°.

Xanthosine ($2H_2O$) [9-(β-D-ribosyl)purin-2,6(1*H*,3*H*)-dione] *[5968-90-1]* **M 320.3, $[\alpha]_D^{20}$ -53° (c 8, 0.3M NaOH), pK_1^{25} <2.5, pK_2^{25} 5.67, pK_3^{25} 12.85.** Crystd from EtOH or water (as dihydrate).

Xanthurenic acid (5,8-dihydroxyquiniline-2-carboxylic acid) *[59-00-7]* **M 205.2, m 286°, 290-295°(dec), $pK_{Est(1)}$~ 1.5, $pK_{Est(2)}$~ 4.9, $pK_{Est(3)}$~ 9.8.** Ppted by the addition of 2N formic acid to its soln in hot 2M ammonia (charcoal). Filter solid off, dry in a vac at ~80° in the dark. UV (H_2O) has λmax nm (ε $M^{-1}cm^{-1}$): 243 (30,000) and 342 (6,500). The *methyl ester* has **m** 262° (from MeOH).

Xanthydrol *[90-46-0]* **M 198.2, m 123-124°.** Crystd from EtOH and dried at 40-50°.

Xylene *[1330-20-7]* **M 106.1 (mixed isomers).** Usual impurites are ethylbenzene, paraffins, traces of sulfur compounds and water. It is not practicable to separate the *m*-, and *p*-isomers of xylene by fractional distn, although, with a sufficiently efficient still, *o*-xylene can be fractionally distd from a mixture of isomers. Purified (and dried) by fractional distn from $LiAlH_4$, P_2O_5, CaH_2 or sodium. This treatment can be preceded by shaking successively with conc H_2SO_4, water, aqueous 10% NaOH, water and mercury, and drying with $CaCl_2$ for several days. Xylene can be purified by azeotropic distn with 2-ethoxyethanol or 2-methoxyethanol, the distillate being washed with water to remove the alcohol, then dried and fractionally distilled.

***o*-Xylene** *[95-47-6]* **M 106.2, f -25.2°, b 84°/14mm, 144.4°/760mm, d 0.88020, d^{25} 0.87596, n 1.50543, n^{25} 1.50292.** The general purification methods listed under xylene are applicable [Clarke and Taylor *J Am Chem Soc* **45** 831 *1923*]. *o*-Xylene (4.4Kg) is sulfonated by stirring for 4h with 2.5L of conc H_2SO_4 at 95°. After cooling, and separating the unsulfonated material, the product was diluted with 3L of water and neutralised with 40% NaOH. On cooling, sodium *o*-xylene sulfonate separated and was recrystd from half its weight of water. [A further crop of crystals was obtained by concentrating the mother liquor to one-third of its volume]. The salt was dissolved in the minimum amount of cold water, then mixed with the same amount of cold water, and with the same volume of conc H_2SO_4 and heated to 110°. *o*-Xylene was regenerated and steam distd. It was then dried and redistd.

***m*-Xylene** *[108-38-3]* **M 106, f -47.9°, b 139.1°, d 0.86417, d^{25} 0.85990, n 1.49721, n^{25} 1.49464.** The general purification methods listed under *xylene* are applicable. The *o*- and *p*-isomers can be removed by their selective oxidation when a *m*-xylene sample containing them is boiled with dilute HNO_3 (one part conc acid to three parts water). After washing with water and alkali, the product can be steam distd, then distd and purified by sulfonation. [Clarke and Taylor *J Am Chem Soc* **45** 831 *1923*.] *m*-Xylene is selectively sulfonated when a mixture of xylenes is refluxed with the theoretical amount of 50-70% H_2SO_4 at 85-95° under reduced pressure. By using a still resembling a Dean and Stark apparatus, water in the condensate can be progressively withdrawn while the xylene is returned to the reaction vessel. Subsequently, after cooling, then adding water, unreacted xylenes are distd off under reduced pressure. The *m*-xylene sulfonic acid is subsequently hydrolysed by steam distn up to 140°, the free *m*-xylene being washed, dried with silica gel and again distd. Stored over molecular sieves Linde type 4A.

***p*-Xylene** *[106-42-3]* **M 106.2, f 13.3, b 138.3°, d 0.86105, d^{25} 0.85669, n 1.49581, n^{25} 1.49325.** The general purification methods listed for *xylene* are applicable. *p*-Xylene can readily be separated from its isomers by crystn from such solvents as MeOH, EtOH, isopropanol, acetone, butanone, toluene, pentane or pentene. It can be further purified by fractional crystn by partial freezing, and stored over sodium wire or molecular sieves Linde type 4A. [Stokes and French *J Chem Soc, Faraday Trans 1* **76** 537 *1980*.]

Xylenol Orange {3*H*-2,1-benzoxathiol-3-ylidene-bis-[(6-hydroxy-5-methyl-*m*-phenylene)-methylnitrilo]tetraacetic acid, S,S-dioxide} *[1611-35-4]* **M 672.6, m 210°(dec), ε_{578} 6.09 x 10^4 (pH 14), ε_{435} 2.62 x 10^4 (pH 3.1), pK_1 -1.74, pK_2 -1.09 (aq H_2SO_4-HNO_3), pK_3 2.58, pK_4 3.23, pK_5 6.46, pK_6 10.46, pK_7 12.28.** Generally contaminated with starting material (cresol red) and semixylenol orange. Purified by ion-exchange chromatography using DEAE-cellulose, eluting with 0.1M NaCl soln which will give the sodium salt. Cresol Red, semixylenol orange and iminodiacetic acid bands elute first. This procedure will give the sodium salt of the dye. To obtain the free acid dissolve the salt in H_2O and acidify with AcOH. Filter off, wash with H_2O and dry first in air and then in a vac deisiccator over P_2O_5 in the dark [Sato, Yokoyama and Momoki *Anal Chim Acta* **94** 317 *1977*].

α-D-Xylose *[58-86-6]* **M 150.1, m 146-147°, $[\alpha]_D^{20}$ -18.8° (c 4, H_2O).** Purified by slow crystn from aq 80% EtOH or EtOH, then dried at 60° under vac over P_2O_5. Stored in a vacuum desiccator over $CaSO_4$.

***m*-Xylylene diisocyanate** *[3634-83-1]* **M 188.2, b 88-89°/0.02mm, 130°/2mm, d_4^{20} 1.204, n_D^{20} 1.4531.** Purified by repeated distn through a 2 plate column. [Ferstundig and Scherrer *J Am Chem Soc* **81** 4838 *1959*.]

α-Yohimbine *[146-48-5]* **M 354.5, m 278°(dec), $[\alpha]_D^{20}$ +55.6° (c 2, EtOH), pK_1^{22} 3.0, pK_2^{22} 7.45.** Crystd from EtOH, and dried to remove EtOH of crystn. For γ-Yohimbine see ajmalicine on p. 98.

Zeaxanthin **[all *trans*-β-carotene-3,3'(*R,R'*)-diol]** *[144-68-3]* **M 568.9, m 207°, 215.5°, λ_{max} 275 (log ε 4.34), 453 (log ε 5.12), 480 (log ε 5.07) in EtOH.** Yellow plates (with a blue lustre) from MeOH or EtOH.