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Phenyl 4-toluenesulfonate [640-60-8] M 248.2, m 94.5-95.5°. Crystd from MeOH or glacial acetic acid.

Phenyl 4-tolylcarbonate [13183-20-5] M 228.2, m 67°. Purified by preparative GLC with 20% Apiezon on Embacel, and sublimed *in vacuo*.

4-Phenyl-1,2,4-triazole-3,5-diol (4-phenylurazole) [15988-11-1] M 175.2, m 207-209°. Crystd from water.

4-Phenyl-1,2,4-triazoline-3,5-dione (PTAD) [4233-33-4] M 175.2, m 165-170°(dec), 170-177°(dec). Carmine red needles obtained by sublimation (ice cold finger) at 100°/0.1mm, and/or by recrystn from EtOH. IR: ν 1760, 1780 cm^{-1} . [Cookson et al. *Org Synth* 51 121 1971; Moore et al. *J Org Chem* 39 3700 1974.]

1-Phenyl-2,2,2-trifluoroethanol [*R*-(-)- 10531-50-7; *S*-(+)- 340-06-7] M 176.1, b 74-76°/10mm, 125-127°/760mm, d_4^{20} 1.301, n_D^{20} 1.4632, $[\alpha]_D^{20}$ (-) and (+) 31° (neat). Purified by fractional distn preferably in a vacuum. [Morrison and Ridgeway *Tetrahedron Lett* 573 1969; NMR: Pirkle and Beare *J Am Chem Soc* 90 6250 1968.] The racemate [340-05-6] has b 52-54°/2mm, 57-59°/2mm, 64-65°/5mm, d_4^{20} 1.293, n_D^{20} 1.457, and the 2-carbobenzoyl derivative has m 137-138° [Mosher et al. *J Am Chem Soc* 78 4374 1956].

Phenylurea [64-10-8] M 136.2, m 148°, pK^{25} -1.45 (aq H_2SO_4). Crystd from boiling water (10mL/g). Dried in a steam oven at 100°.

9-Phenyl-9-xanthenol (hydroxypixyl) [596-38-3] M 274.3, m 158-161°, 158.5-159°, 159°. Dissolve in AcOH and add H_2O whereby it separates as colourless prisms. It is slightly soluble in CHCl_3 , soluble in C_6H_6 but insoluble in pet ether. It sublimes on heating. UV in H_2SO_4 : λ_{max} 450nm (ϵ 5620) and 370nm (ϵ 24,900) and the HClO_4 salt in CHCl_3 has λ_{max} 450 (ϵ 404) and 375nm (ϵ 2420). [Sharp *J Chem Soc* 2558 1958; Bünzly and Decker *Chem Ber* 37 2983 1904; Chattopadhyaya and Reece *J Chem Soc, Chem Commun* 639 1978; Gomberg and Cone *Justus Liebigs Ann Chem* 370 142 1909.]

Phloretin [2',4',6'-trihydroxy-3-(*p*-hydroxyphenyl)propiophenone] [60-82-2] M 274.3, m 264-271°(dec), $\text{pK}_{\text{Est}(1)} \sim 7.5$, $\text{pK}_{\text{Est}(2)} \sim 8.0$, $\text{pK}_{\text{Est}(3)} \sim 10$, $\text{pK}_{\text{Est}(4)} \sim 12$ (phenolic OH's). Crystd from aqueous EtOH.

Phloridzin (2H₂O) [phloretin 2'-*O*- β -D-glucoside] [60-81-1] M 472.5, m 110°, $[\alpha]_{546}^{20}$ -62° (c 3.2, EtOH). Crystd as dihydrate from water.

Phloroacetophenone (2H₂O) (2',4',6'-trihydroxyacetophenone) [480-66-0] M 186.2, m 218-219°, $\text{pK}_{\text{Est}(1)} \sim 7.9$, $\text{pK}_{\text{Est}(2)} \sim 12.0$. Crystd from hot water (35mL/g).

Phloroglucinol (2H₂O) (benzene-1,3,5-triol) [6099-90-7 (2H₂O); 108-73-6 (anhydr)] M 126.1, m 217-219°, 117° (anhydrous), $\text{pK}_1^{25} \sim 7.74$ (HClO_4), pK_2^{20} 7.97, pK_3^{20} 9.23. Crystd from water, and stored in the dark under nitrogen.

Phorone (2,6-dimethylhepta-2,5-dien-4-one) [504-20-1] M 138.2, m 28°, b 197°/743mm. Crystd repeatedly from EtOH.

"Phosphine" [dye CI 793, Chrysaniline mononitrate, 3-amino-9-(4-aminophenyl)-acridinium mononitrate] [10181-37-0] M 348.4, m >250°(dec), $\text{pK}_{\text{Est}} \sim 8.0$. Crystd from *benzene/EtOH.

Phthalaldehyde [643-79-8] M 134.1, m 54-56°, 55.5-56°, 58°, b 83-84°/0.8mm. Purified by steam distillation better by using super heated steam (at 175-180°) and efficient cooling. The distillate is saturated with Na_2SO_4 extracted exhaustively with EtOAc, dried (Na_2SO_4), filtered and evaporated. The residue

is recrystd from pet ether (b 90-100°) [Beill and Tarbell *Org Synth Coll Vol IV* 808 1963]. It can be distd under vacuum. The bis-2,4-dinitrophenylhydrazone has m 278-280° [Hatt and Stephenson *J Chem Soc* 199 1952].

Phthalazine [253-52-1] M 130.2, m 90-91°, pK²⁰ 3.47. Crystd from diethyl ether or *benzene, and sublimed under vacuum.

Phthalazine-1,4-dione (phthalhydrazide) [1445-69-8] M 162.2, m 330-333°, 336°, 346°, pK₁²⁰ -3.29 pK₂²⁰ -0.99, pK₃²⁰ 5.67, pK₄²⁰ 13.0. Twice recrystd from 0.1M KOH [Merenyi et al. *J Am Chem Soc* 108 7716 1986], EtOH or dimethylformamide and sublimes >300°.

Phthalazone (1-hydroxyphthalazine) [119-39-1] M 146.2, m 183-184°, 186-188°, b 337°/760mm, pK₁²⁰ -2.2, pK₂²⁰ -1.4, pK₃²⁰ 11.99. Crystd from H₂O or EtOH and sublimed *in vacuo*.

o-Phthalic acid [88-99-3] M 166.1, m 211-211.5°, pK₁²⁵ 2.76 (3.05), pK₂²⁵ 4.92 (4.73). Crystd from water.

Phthalic anhydride [85-44-9] M 148.1, m 132°, b 295°. Distd under reduced pressure. Purified from the acid by extracting with hot CHCl₃, filtering and evaporating. The residue was crystd from CHCl₃, CCl₄ or *benzene, or sublimed. Fractionally crystd from its melt. Dried under vacuum at 100°. [Saltiel *J Am Chem Soc* 108 2674 1986.]

Phthalide [87-41-2] M 134.1, m 72-73°, pK -7.98 (aq H₂SO₄). Crystd from water (75mL/g) and dried in air on filter paper.

Phthalimide [85-41-6] M 147.1, m 235°, 238°, pK 8.30. Crystd from EtOH (20mL/g) (charcoal), or by sublimation. For potassium phthalimide see entry in Chapter 5.

Phthalimidoglycine [4702-13-0] M 205.2, m 192-193°, pK_{Est} ~3. Crystd from water or EtOH.

Phthalonitrile [91-15-6] M 128.1, m 141°. Crystd from EtOH, toluene or *benzene. Can also be distd under high vacuum.

Phthalylsulfacetamide [131-69-1] M 362.3, m 196°. Crystd from water.

Phthiocol (2-hydroxy-3-methylnaphthaquinone) [483-55-6] M 188.1, m 173-174°, pK_{Est} ~4.2. Crystd from diethyl ether/pet ether.

Physalien (all trans β-carotene-3,3'-diol dipalmitate) [144-67-2] M 1044, m 98.5-99.5°, A_{1m}^{1%} (λ_{max}) 1410 (449nm), 1255 (478nm) in hexane. Purified by chromatography on water-deactivated alumina, using hexane/diethyl ether (19:1) to develop the column. Crystd from *benzene/EtOH. Stored in the dark, in inert atmosphere, at 0°.

Physodic acid [4,4',6'-trihydroxy-6-(2-oxoheptyl)-2'-pentyl-2,3'-oxydibenzoic acid 1,5-lactone] [84-24-2] M 470.5, m 205°, pK_{Est(1)} ~3.0, pK_{Est(2)} ~10, pK_{Est(3)} ~13. Crystd from MeOH. The diacetate has m 155-156° (from Me₂CO/CS₂).

Phytoene (7,7',8,8',11,11',12,12'-octahydro-ψ,ψ-carotene) [540-04-5] M 544.9, A_{1m}^{1%} (λ_{max}) 850 (287nm) in hexane, λ_{max} 275, 287 and 297nm nm. Purified by chromatography on columns of magnesium oxide-Supercel (a diatomaceous filter aid) or alumina [Rabourn et al. *Arch Biochem Biophys* 48 267 1954]. Stored as a solution in pet ether under nitrogen at -20°.

Phytofluene [540-05-6] M 549.0, b 140-185°(bath temp)/0.0001m A_{1m}^{1%} (λ_{max}) 1350 (348nm) in pet ether, λ_{max} 331, 348, 267. Purified by chromatography on partially deactivated alumina [Kushwaha et al. *J Biol Chem* 245 4708 1970]. Stored as a soln in pet ether under nitrogen at -20°.

Picein (*p*-acetylphenyl- β -D-glucopyranoside) [530-14-3] M 298.3, m 195-196°, $[\alpha]_D^{20}$ -88° (c 1, H₂O). Crystd from MeOH or (as monohydrate) from water.

Picene [213-14-3] M 278.3, m 364°. Crystd from isopropylbenzene/xylene. Can also be sublimed.

2-Picoline-N-oxide (2-methylpyridine-1-oxide) [931-19-1] M 109.1, m 41-45°, b 89-90°/0.8-0.9mm, 90-100°/1mm, 110°/4mm, 135°/5mm, 123°/9mm, 123-124°/15mm, 259-261°/atm, n_D^{25} 1.5854 (supercooled), pK^{25} 1.10. Purified by fractional distillation and could be recrystd from *C₆H₆-hexane but is *hygroscopic*. [Bullitt and Maynard *J Am Chem Soc* 76 1370 1954; Ross et al. *J Am Chem Soc* 78 3625 1956; IR: Wiley and Slaym Aker *J Am Chem Soc* 79 2233 1957.] The *picrate* has m 125-126.5° (from EtOH) [Boekelheide and Linn *J Am Chem Soc* 76 1286 1954]. The *phthalate* has m 115-116° (from EtOH) [den Hertog et al. *Recl Trav Chim Pays-Bas* 70 591 1951.]

3-Picoline-N-oxide (3-methylpyridine-1-oxide) [1003-73-2] M 109.1, m 37-39°, 37-38° (evac capillary), 84-85°/0.3mm, 101-103°/0.7-0.8mm, 114-115°/1.5mm, 118°/2mm, pK^{25} 1.08. Purified by careful fractionation *in vacuo*. The distillate remains supercooled for several days before solidifying. It is a slightly *hygroscopic* solid which could melt in the hand. The *picrate* has m 149-151° (from EtOH). [Taylor and Corvetti *Org Synth Coll Vol IV* 654 1963; IR: Katritzky et al. *J Chem Soc* 3680 1959; Jaffé and Doak *J Am Chem Soc* 77 4441, 4481 1955; Boekelheide and Linn *J Am Chem Soc* 76 1286 1954].

4-Picoline-N-oxide (4-methylpyridine-1-oxide) [1003-67-4] M 109.1, m 182-184°, 185-186°, 186-188°, pK^{25} 1.29. Recryst from EtOH-EtOAc, Me₂CO-Et₂O or *C₆H₆. [Bullitt and Maynard *J Am Chem Soc* 76 1370 1954; Boekelheide and Linn *J Am Chem Soc* 76 1286 1954].

Picolinic acid (pyridine-2-carboxylic acid) [98-98-6] M 123.1, m 138°, pK_1^{25} 1.03 (1.36), pK_2^{25} 5.30 (5.80). Crystd from water or *benzene.

α -Picolinium chloride [14401-91-3] M 129.6, m 200°. 1:1 Mixture of α -picoline and HCl, distd at 275°. Then vacuum sublimed at 91-91.5°.

N-Picolinoylbenzimidazole [100312-29-6] M 173.3, m 105-107°. Recrystd three times from hexane [Fife and Przystas *J Am Chem Soc* 108 4631 1986].

Picric acid [88-89-1] M 229.1, m 122-123°, pK^{25} 0.33 (0.37). Crystd first from acetic acid then acetone, toluene, CHCl₃, aqueous 30% EtOH, 95% EtOH, MeOH or H₂O. Dried in a vacuum oven at 80° for 2h. Alternatively, dried over Mg(ClO₄)₂ or fused and allowed to freeze under vacuum three times. Because it is **EXPLOSIVE**, picric acid should be stored moistened with H₂O, and only small portions should be dried at any one time. The dried acid should **NOT** be heated.

Picrolonic acid [3-methyl-4-nitro-1-(4-nitrophenyl)-2-pyrazolin-5-one, picrolonic acid] [550-74-3] M 264.2, m 120°(dec), 116.5°(dec at 125°) 125°. Crystd from water or EtOH (Solubility is 0.123% at 15° and 1.203% at 100° in H₂O; and 1.107% at 0° and 11.68% at 81° in EtOH). It forms Ca, Cu Hg, Mg, Na, Sr, and Pb complexes [Maquestian et al *Bull Soc Chim Belg* 82 233 1973; Isaki et al. *Chem Ber* 74 1420 1941].

Picrotoxin [124-87-8] M 602.6, m 203°, $[\alpha]_{546}^{20}$ -40° (c 1, EtOH). Crystd from water.

Picryl chloride [88-88-0] M 226.3, m 83°. Crystd from CHCl₃ or EtOH.

Picryl iodide [4436-27-5] M 340.0, m 164-165°. Crystd from *benzene.

Pimelic acid (heptane-1,7-dioic acid) [111-16-0] M 160.2, m 105-106°, pK_1^{25} 4.46, pK_2^{25} 5.58. Crystd from water or from *benzene containing 5% diethyl ether.

Pinacol (hexahydrate) [6091-58-3 (6H₂O); 76-09-5 (anhydr)] M 194.3, m 46.5°, b 59°/4mm. Distd then crystd repeatedly from water.

Pinacol (anhydrous) [76-09-5] M 118.1, m 41.1°, b 172°. The hydrate is rendered anhydrous by azeotropic distn of water with *benzene. Recrystd from *benzene or toluene/pet ether, absolute EtOH or dry diethyl ether. Recrystn from water gives the hexahydrate.

Pinacolone oxime [2475-93-6] M 115.2, m 78°. Crystd from aqueous EtOH.

Pinacyanol chloride [2768-90-3] M 388.9, m 270°(dec). Crystd from EtOH/diethyl ether.

R- α -Pinene [7785-70-8] M 136.2, b 61°/30mm, 156.2°/760mm, d 0.858, n¹⁵ 1.4634, n 1.4658, $[\alpha]_D^{25}$ +47.3°. Isomerised by heat, acids and certain solvents. Should be distd under reduced pressure under nitrogen and stored in the dark. Purified *via* the nitrosochloride [Waterman et al. *Recl Trav Chim Pays-Bas* 48 1191 1929]. For purification of optically active forms see Lynn [*J Am Chem Soc* 91 361 1919]. Small quantities (0.5mL) have been purified by GLC using helium as carrier gas and a column at 90° packed with 20 wt% of polypropylene sebacate on a Chromosorb support. Larger quantities were fractionally distd under reduced pressure in a column packed with stainless steel gauze spirals. Material could be dried with CaH₂ or sodium, and stored in a refrigerator: CaSO₄ and silica gel were not satisfactory because they induced spontaneous isomerisation. [Bates, Best and Williams *J Chem Soc* 1521 1962.]

S- α -Pinene [7785-26-4] M 136.2, b 155-156°/760mm, d 0.858, n 1.4634, $[\alpha]_D^{20}$ -47.2°. Purification as for R- α -Pinene above.

dl-Pipecolic acid (piperidine-2-carboxylic acid) [4043-87-2] M 129.1, m 264°, pK₁²⁵ 2.29, pK₂²⁵ 10.77. Crystd from water.

Piperazine [110-85-0] M 86.1, m 110-112°, 44° (hexahydrate 142-63-2) b 125-130°/760mm, pK₁²⁵ 5.33, pK₂²⁵ 9.73. Crystd from EtOH or anhydrous *benzene, and dried at 0.01mm. It can be sublimed under vacuum and purified by zone melting.

§ Piperazine on polystyrene support is commercially available.

Piperazine-N,N'-bis(2-ethanesulfonic acid) (PIPES) [5625-37-6] M 302.4, pK₁²⁵ <3, pK₂²⁵ 6.82 (7.82). Crystd from boiling water (maximum solubility is about 1g/L) or as described for ADA [*N*-(2-acetamido)iminodiacetic acid, see above].

Piperazine dihydrochloride (H₂O) [142-64-3 (2HCl); 6094-40-2 (xHCl)] M 177.1, m 82.5-83.5°. Crystd from aqueous EtOH. Dried at 110°.

Piperazine phosphate (H₂O) [18534-18-4] M 197.6. Crystd twice from water, air-dried and stored for several days over Drierite. The salt dehydrates slowly if heated at 70°.

Piperic acid [*trans,trans*-5-(3,4-methylenedioxyphenyl)-2,4-pentadieneoic acid] [136-72-1] M 218.2, m 217°, pK_{Est} ~4.7. Crystd from EtOH. Turns yellow in light. Sublimes with partial dec.

Piperidine [110-89-4] M 85.2, f -9°, b 35.4°/40mm, 106°/760mm, d 0.862, n 1.4535, n²⁵ 1.4500, pK₂²⁵ 11.20. Dried with BaO, KOH, CaH₂, or sodium, and fractionally distd (optionally from sodium, CaH₂, or P₂O₅). Purified from pyridine by zone melting.

§ Piperidine on polystyrene support is commercially available.

Piperidinium chloride [6091-44-7] M 121.6, m 244-245°. Crystd from EtOH/diethyl ether in the presence of a small amount of HCl.

Piperidinium nitrate [6091-45-8] M 145.2, m 110°. Crystd from acetone/ethyl acetate.

Piperine (1-piperoylpiperidine) [94-62-2] M 285.4, m 129-129.5°, pK¹⁵ 1.98. Crystd from EtOH or *benzene/ligroin.

Piperonal [120-57-0] M 150.1, m 37°, b 140°/15mm, 263°/760mm. Crystd from aqueous 70% EtOH or EtOH/water.

Piperonylic acid [94-53-1] M 166.1, m 229°, pK²⁵ 4.50. Crystd from EtOH or water.

Pivalic acid (trimethylacetic acid) [75-98-9] M 102.1, m 35.4°, b 71-73°/0.1mm, pK²⁵ 5.03. Fractionally distd under reduced pressure, then fractionally crystd from its melt. Recrystd from *benzene.

Pivaloyl chloride (trimethylacetyl chloride) [3282-30-2] M 120.6, b 57.6°/150mm, 70.5-71/250mm, 104°/754mm, 104-105°/atm, 105-108°/atm, d₄²⁰ 1.003, n_D²⁰ 1.4142. First check the IR to see if OH bands are present. If absent, or present in small amounts, then redistil under moderate vac. If present in large amounts then treat with oxalyl chloride or thionyl chloride and reflux for 2-3h, evap and distil residue. **Strongly LACHRYMATORY - work in a fumecupboard.** Store in sealed ampoules under N₂. [Traynham and Battiste *J Org Chem* 22 1551 1957; Grignard reactns: Whitmore et al. *J Am Chem Soc* 63 647 1941.]

Plumbagin (5-hydroxy-2-methyl-1,4-naphthaquinone) [481-42-5] M 188.1, m 78-79°, pK_{Est(1)}~9.5, pK_{Est(2)}~11.0. Crystd from aqueous EtOH and sublimed in a vac. Steam distils.

Polyacrylonitrile [25014-41-9]. Ppted from dimethylformamide by addition of MeOH.

Poly(diallyldimethylammonium) chloride [26062-79-3]. Ppted from water in acetone, and dried in vacuum for 24h. [Hardy and Shriner *J Am Chem Soc* 107 3822 1985.]

Polyethylene [9002-88-4]. Crystd from thiophen-free *benzene and dried over P₂O₅ under vacuum.

Polymethyl acrylate [9003-21-8]. Ppted from a 2% soln in acetone by addition of water.

Polystyrene [9003-53-6]. Ppted repeatedly from CHCl₃ or toluene soln by addition of MeOH. Dried *in vacuo* [Miyasaka et al. *J Phys Chem* 92 249 1988].

Polyvinyl acetate [9003-20-7]. Ppted from acetone by addition of *n*-hexane.

Poly(*N*-vinylcarbazole) [25067-59-8]. Ppted seven times from tetrahydrofuran with MeOH, with a final freeze-drying from *benzene. Dried under vacuum.

Polyvinyl chloride [9002-81-2]. Ppted from cyclohexanone by addition of MeOH.

Poly(4-vinylpyridine) [25232-41-1] M (105.1)_n. Purified by repeated pptn from solns in EtOH and dioxane, and then EtOH and ethyl acetate. Finally, freeze-dried from *tert*-butanol.

Poly(*N*-vinylpyrrolidone) [9003-39-8] M (111.1)_n, crosslinked [25249-54-1] m >300°. Purified by dialysis, and freeze-dried. Also by pptn from CHCl₃ soln by pouring into ether. Dried in a vacuum over P₂O₅. For the crosslinked polymer purification is by boiling for 10min in 10% HCl and then washing with glass-distilled water until free from Cl ions. Final Cl ions were removed more readily by neutralising with KOH and continued washing.

Prednisone [53-03-2] M 358.5, m 238°(dec), [α]_D²⁰ +168° (c 1, dioxane), λ_{max} 238nm (log ε 4.18) in MeOH. Crystd from acetone/hexane.

Pregnane [481-26-5] M 300.5, m 83.5°, [α]_D²⁰ +21° (CHCl₃). Crystd from MeOH.

5 β -Pregnane-3 α ,20 α -diol [80-92-2] M 320.5, m 243-244 $^{\circ}$, $[\alpha]_{546}^{20}$ +31 $^{\circ}$ (c 1, EtOH). Crystd from acetone.

5 β -Pregnane-3 α ,20 β -diol [80-91-1] M 320.5, m 244-246 $^{\circ}$, $[\alpha]_{546}^{20}$ +22 $^{\circ}$ (c 1, EtOH). Crystd from EtOH.

Procaine [4-(2-diethylaminomethoxycarbonyl)aniline] [59-46-1] M 236.3, m 51 $^{\circ}$ (dihydrate), 61 $^{\circ}$ (anhydrous), pK_1^{15} 2.45, pK_2^{15} 8.91. Crystd as the dihydrate from aqueous EtOH and as anhydrous material from pet ether or diethyl ether. The latter is *hygroscopic*.

Proclavine (3,6-diaminoacridine) [92-62-6] M 209.2, m 284-286 $^{\circ}$, pK^{25} 9.60. Crystd from aqueous MeOH. For proflavin see 3,6-diaminoacridine hydrochloride

Progesterone [57-83-0] M 314.5, m 128.5 $^{\circ}$, $[\alpha]_{546}^{20}$ +220 $^{\circ}$ (c 2, dioxane). Crystd from EtOH. When crystd from pet ether m is 121 $^{\circ}$, λ_{\max} 240nm, log ϵ 4.25 (EtOH).

L-Proline [147-85-3] M 115.1, m 215-220 $^{\circ}$ (dec)(D-isomer), 220-222 $^{\circ}$ (dec) (L-form), 205 $^{\circ}$ (dec)(DL-isomer), $[\alpha]_D^{25}$ (H₂O, L-isomer), pK_1^{25} 1.95, pK_2^{25} 10.64. Likely impurity are hydroxyproline. Purified *via* its picrate which was crystd twice from water, then decomposed with 40% H₂SO₄. The picric acid was extracted with diethyl ether, the H₂SO₄ was pptd with Ba(OH)₂, and the filtrate evapd. The residue was crystd from hot absolute EtOH [Mellan and Hoover *J Am Chem Soc* 73 3879 1951] or EtOH/ether. *Hygroscopic*. Stored in a desiccator.

Polycopene (all Z- ψ , ψ -carotene) [2361-24-2] M 536.5, m 111 $^{\circ}$, λ_{\max} 443.5, 470nm in pet ether. Purified by chromatography on deactivated alumina [Kushwaha et al. *J Biol Chem* 245 4708 1970]. Crystd from pet ether. Stored in the dark, in an inert atmosphere at -20 $^{\circ}$.

L-Prolylglycine [2578-57-6] M 172.2, m 236 $^{\circ}$, $[\alpha]_D^{20}$ +21.1 $^{\circ}$ (c 4, H₂O), pK_1^{25} 3.19, pK_2^{25} 8.97. Crystd from water at 50-60 $^{\circ}$ by addition of EtOH.

Proneurosporene (3,4,7',8'-tetrahydrolycopene) [10467-46-6] M 538.9, λ_{\max} 408, 432, 461 nm, $\epsilon_{1\text{cm}}^{1\%}$ 2040 (432nm) in hexane. Purified by chromatography on deactivated alumina [Kushwaha et al. *J Biol Chem* 245 4708 1970]. Stored in the dark, in an inert atmosphere at 0 $^{\circ}$.

Propane [74-98-6] M 44.1, m -189.7, b -42.1 $^{\circ}$ /760mm, d 0.5005, n 1.2898. Purified by bromination of the olefinic contaminants. Propane was treated with bromine for 30min at 0 $^{\circ}$. Unreacted bromine was quenched, and the propane was distd through two -78 $^{\circ}$ traps and collected at -196 $^{\circ}$ [Skell et al. *J Am Chem Soc* 108 6300 1986].

Propane-1,2-diamine (propylenediamine) [78-90-0] M 74.1, b 120.5 $^{\circ}$, d 0.868, n 1.446, pK_1^{25} 6.61, pK_2^{25} 9.82. Purified by azeotropic distn with toluene. [Horton, Thomason and Kelly *Anal Chem* 27 269 1955.]

Propane-1,2-diol (propyleneglycol) [57-55-6] M 76.1, b 104 $^{\circ}$ /32mm, d 1.040, n 1.433. Dried with Na₂SO₄, decanted and distd under reduced pressure.

Propane-1,3-diol [504-63-2] M 76.1, b 110-122 $^{\circ}$ /12mm, d 1.053, $n^{18.5}$ 1.4398. Dried with K₂CO₃ and distd under reduced pressure. More extensive purification involved conversion with benzaldehyde to 2-phenyl-1,3-dioxane (m 47-48 $^{\circ}$) which was subsequently decomposed by shaking with 0.5M HCl (3mL/g) for 15min and standing overnight at room temperature. After neutralisation with K₂CO₃, the benzaldehyde was removed by distn and the diol was recovered from the remaining aqueous soln by continuous extraction with CHCl₃ for 1day. The extract was dried with K₂CO₃, the CHCl₃ was evaporated and the diol was distd. [Foster, Haines and Stacey *Tetrahedron* 6 177 1961.]

Propane-1-thiol [107-03-9] M 76.1, b 65.3°/702mm, d²⁵ 0.83598, n²⁵ 1.43511, pK²⁰ 10.82. Purified by soln in aqueous 20% NaOH, extraction with a small amount of *benzene and steam distn until clear. After cooling, the soln was acidified slightly with 15% H₂SO₄, and the thiol was distd out, dried with anhydrous CaSO₄ or CaCl₂, and fractionally distd under nitrogen. [Mathias and Filho *J Phys Chem* 62 1427 1958.] Also purified by liberation of the mercaptan by adding dilute HCl to the residue remaining after steam distn. After direct distn from the flask, and separation of the water, the mercaptan was dried (Na₂SO₄) and distd under nitrogen.

Propane-2-thiol (Isopropyl mercaptan) [75-33-2] M 76.1, b 49.8°/696mm, d²⁵ 0.80895, n²⁵ 1.42154, pK²⁵ 10.86. Purification as for propane-1-thiol above.

Propargyl alcohol (2-propyn-1-ol) [107-19-7] M 56.1, b 54°/57mm, 113.6°/760mm, d 0.947, n 1.432. Commercial material contains a stabiliser. An aqueous soln of propargyl alcohol can be concentrated by azeotropic distn with butanol or butyl acetate. Dried with K₂CO₃ and distd under reduced pressure, in the presence of about 1% succinic acid, through a glass helices-packed column.

Propargyl chloride (3-chloropropyne) [624-65-7] M 74.5, b 58°/760mm, 65°/760mm, d 1.03, n 1.435. Purified by fractional distn at atm press. Note that a possible impurity propargyl alcohol has b 114-115°/atm. [Henry *Chem Ber* 8 398 1875.] **HIGHLY TOXIC and FLAMMABLE.**

Propene [115-07-1] M 42.1, m -185.2°, b -47.8°/750mm, d 0.519, n⁷¹ 1.357. Purified by freeze-pump-thaw cycles and trap-to-trap distn.

p-(1-Propenyl)phenol [cis/trans 6380-21-8; 539-12-8] M 134.2, m 93-94°, pK_{Est} ~10.2. Crystd from water.

β-Propiolactone [57-57-8] M 72.1, b 83°/45mm, d 1.150, n²⁵ 1.4117. Fractionally distd under reduced pressure, from sodium. **CARCINOGEN.**

Propionaldehyde [123-38-6] M 58.1, b 48.5-48.7°, d 0.804, n 1.3733, n²⁵ 1.37115. Dried with CaSO₄ or CaCl₂, and fractionally distd under nitrogen or in the presence of a trace of hydroquinone (to retard oxidation). Blacet and Pitts [*J Am Chem Soc* 74 3382 1952] repeatedly vacuum distd the middle fraction until no longer gave a solid polymer when cooled to -80°. It was stored with CaSO₄.

Propionamide [79-05-0] M 73.1, m 79.8-80.8°, pK²⁴ -0.9 (H₀ scale, aq H₂SO₄). Crystd from acetone, *benzene, CHCl₃, water or acetone/water, then dried in a vacuum desiccator over P₂O₅ or conc H₂SO₄.

Propionic acid [79-09-4] M 74.1, b 141°, d 0.992, n 1.3865, n²⁵ 1.3843, pK₁²⁵ -6.8 (H₀ scale, aq H₂SO₄), pK₂²⁵ 4.88. Dried with Na₂SO₄ or by fractional distn, then redistd after refluxing with a few crystals of KMnO₄. An alternative purification uses the conversion to the ethyl ester, fractional distn and hydrolysis. [Bradbury *J Am Chem Soc* 74 2709 1952.] Propionic acid can also be heated for 0.5h with an amount of benzoic anhydride equivalent to the amount of water present (in the presence of CrO₃ as catalyst), followed by fractional distn. [Cham and Israel *J Chem Soc* 196 1960.]

Propionic anhydride [123-62-6] M 130.2, b 67°/18mm, 168°/780mm, d 1.407, n 1.012. Shaken with P₂O₅ for several minutes, then distd.

Propionitrile [107-12-0] M 55.1, b 97.2°, d 1.407, n¹⁵ 1.36812, n³⁰ 1.36132. Shaken with dil HCl (20%), or with conc HCl until the odour of isonitrile has gone, then washed with water, and aqueous K₂CO₃. After a preliminary drying with silica gel or Linde type 4A molecular sieves, it is stirred with CaH₂ until hydrogen evolution ceases, then decanted and distd from P₂O₅ (not more than 5g/L, to minimise gel formation). Finally, it is refluxed with, and slowly distd from CaH₂ (5g/L), taking precautions to exclude moisture.

***n*-Propyl acetate** [109-60-4] M 102.1, b 101.5°, d 0.887, n 1.38442, pK²⁵ -7.18 (H₀ scale, aq H₂SO₄). Washed with satd aqueous NaHCO₃ until neutral, then with satd aqueous NaCl. Dried with MgSO₄ and fractionally distd.

***n*-Propyl alcohol (1-propanol)** [71-23-8] M 60.1, b 97.2°, d²⁵ 0.79995, n 1.385, pK²⁵ 16.1. The main impurities in *n*-propyl alcohol are usually water and 2-propen-1-ol, reflecting the commercial production by hydration of propene. Water can be removed by azeotropic distn either directly (azeotrope contains 28% water) or by using a ternary system, e.g. by adding *benzene. Alternatively, for gross amounts of water, refluxing over CaO for several hours is suitable, followed by distn and a further drying. To obtain more nearly anhydrous alcohol, suitable drying agents are firstly NaOH, CaSO₄ or K₂CO₃, then CaH₂, aluminium amalgam, magnesium activated with iodine, or a small amount of sodium. Alternatively, the alcohol can be refluxed with *n*-propylsuccinate or phthalate in a method similar to the one described under EtOH. Allyl alcohol is removed by adding bromine (15mL/L) and then fractionally distilling from a small amount of K₂CO₃. Propionaldehyde, also formed in the bromination, is removed as the 2,4-dinitrophenylhydrazone. *n*-Propyl alcohol can be dried down to 20ppm of water by passage through a column of pre-dried molecular sieves (type 3 or 4A, heated for 3h at 300°) in a current of nitrogen. Distn from sulfanilic or tartaric acids removes impurities. Albrecht [*J Am Chem Soc* 82 3813 1960] obtained spectroscopically pure material by heating with charcoal to 50-60°, filtering and adding 2,4-dinitrophenylhydrazine and a few drops of conc H₂SO₄. After standing for several hours, the mixture was cooled to 0°, filtered and vac distd. Gold and Satchell [*J Chem Soc* 1938 1963] heated *n*-propyl alcohol with 3-nitrophthalic anhydride at 76-110° for 15h, then recrystd the resulting ester from H₂O, *benzene/pet ether (b 100-120°)(3:1), and *benzene. The ester was hydrolysed under reflux with aq 7.5M NaOH for 45min under nitrogen, followed by distn (also under nitrogen). The fraction (b 87-92°) was dried with K₂CO₃ and stirred under reduced pressure in the dark over 2,4-dinitrophenylhydrazine, then freshly distilled. Also purified by adding 2g NaBH₄ to 1.5L alcohol, gently bubbling with argon and refluxing for 1day at 50°. Then added 2g of freshly cut sodium (washed with propanol) and refluxed for one day. Distd, taking the middle fraction [Jou and Freeman *J Phys Chem* 81 909 1977].

***n*-Propylamine** [107-10-8] M 59.1, b 48.5°, d 0.716, n 1.38815, pK²⁵ 10.69. Distd from zinc dust, at reduced pressure, in an atmosphere of nitrogen.

***n*-Propyl bromide.** [106-94-5] M 123.0, b 71.0°, d 1.354., n¹⁵ 1.43695, n²⁵ 1.43123. Likely contaminants include *n*-propyl alcohol and isopropyl bromide. The simplest purification procedure uses drying with MgSO₄ or CaCl₂ (with or without a preliminary wash of the bromide with aq NaHCO₃, then water), followed by fractional distn away from bright light. Chien and Willard [*J Am Chem Soc* 79 4872 1957] bubbled a stream of oxygen containing 5% ozone through *n*-propyl bromide for 1h, then shook with 3% hydrogen peroxide soln, neutralised with aq Na₂CO₃, washed with distilled water and dried. Then followed vigorous stirring with 95% H₂SO₄ until fresh acid did not discolour within 12h. The propyl bromide was separated, neutralised, washed dried with MgSO₄ and fractionally distd. The centre cut was stored in the dark. Instead of ozone, Schuler and McCauley [*J Am Chem Soc* 79 821 1957] added bromine and stored for 4 weeks, the bromine then being extracted with aq NaHSO₃ before the sulfuric acid treatment was applied. Distd. Further purified by preparative gas chromatography on a column packed with 30% SE-30 (General Electric ethylsilicone rubber) on 42/60 Chromosorb P at 150° and 40psi, using helium. [Chu *J Phys Chem* 41 226 1964.]

***n*-Propyl chloride** [540-54-5] M 78.5, b 46.6°, d 0.890, n 1.3880. Dried with MgSO₄ and fractionally distd. More extensively purified using extraction with H₂SO₄ as for *n*-propyl bromide. Alternatively, Chien and Willard [*J Am Chem Soc* 75 6160 1953] passed a stream of oxygen containing about 5% ozone through the *n*-propyl chloride for three times as long as was needed to cause the first coloration of starch iodide paper by the exit gas. After washing with aqueous NaHCO₃ to hydrolyse ozonides and remove organic acids, the chloride was dried with MgSO₄ and fractionally distd.

1-Propyl-3-(*p*-chlorobenzenesulfonyl) urea [94-20-2] M 260.7, m 127-129°. Crystd from aqueous EtOH.

Propylene carbonate [108-32-7] M 102.1, b 110°/0.5-1mm, 238-239°/760mm, d 1.204, n 1.423. Manufactured by reaction of 1,2-propylene oxide with CO₂ in the presence of a catalyst (quaternary

ammonium halide). Contaminants include propylene oxide, carbon dioxide, 1,2- and 1,3-propanediols, allyl alcohol and ethylene carbonate. It can be purified by percolation through molecular sieves (Linde 5A, dried at 350° for 14h under a stream of argon), followed by distn under vac. [Jasinski and Kirkland *Anal Chem* **39** 163 1967.] It can be stored over molecular sieves under an inert gas atmosphere. When purified in this way it contains less than 2ppm water. Activated alumina and dried CaO have been also used as drying agents prior to fractional distn under reduced pressure. It has been dried with 3A molecular sieves and distd under nitrogen in the presence of *p*-toluenesulfonic acid. Then redistilled and the middle fraction collected.

***dl*-Propylene oxide** [75-56-9] M 58.1, b 34.5°, d 0.829, n 1.3664. Dried with Na₂SO₄ or CaH₂, and fractionally distilled through a packed column (glass helices), after refluxing with Na, CaH₂, or KOH pellets.

***n*-Propyl ether (dipropyl ether)** [111-43-3] M 102.2, b 90.1°, d 0.740, n¹⁵ 1.38296, n 1.3803, pK -4.40 (aq H₂SO₄). Purified by drying with CaSO₄, by passage through an alumina column (to remove peroxides), and by fractional distn.

Propyl formate [110-74-7] M 88.1, b 81.3°, d 0.9058, n 1.3779. Distd, then washed with satd aq NaCl, and with satd aq NaHCO₃ in the presence of solid NaCl, dried with MgSO₄ and fractionally distd.

***n*-Propyl gallate** [121-79-9] M 212.2, m 150°. Crystd from aqueous EtOH.

***n*-Propyl iodide (1-iodopropane)** [107-08-4] M 170.0, b 102.5°, d 1.745, n 1.5041. Should be distd at reduced pressure to avoid decomposition. Dried with MgSO₄ or silica gel and fractionally distd. Stored under nitrogen with mercury in a brown bottle. Prior to distn, free iodine can be removed by shaking with copper powder or by washing with aq Na₂S₂O₃ and drying. Alternatively, the *n*-propyl iodide can be treated with bromine, then washed with aq Na₂S₂O₃ and dried. See also *n*-butyl iodide.

***n*-Propyl propionate** [106-36-5] M 120.2, b 122°, d 0.881, n 1.393. Treated with anhydrous CuSO₄, then distd under nitrogen.

6-Propyl-2-thiouracil (propacil, propylail) [51-52-5] M 170.2, m 218-220°, 218-220°, pK₁²¹ -6.54 (aq H₂SO₄), pK₂²¹ -4.22 (aq H₂SO₄), pK₃²¹ 8.25 (4% aq EtOH). Purified by recrystn from H₂O (sol in 900 parts at 20°, and 100 parts at 100°). UV, MeOH: λ_{max} 277nm. [Anderson et al. *J Am Chem Soc* **67** 2197 1945; Vanderhaegue *Bull Soc Chim Belg* **59** 689 1950.]

Propyne [74-99-7] M 40.1, m -101.5°, b -23.2°/760mm, d⁵⁰ 0.7062, n⁴⁰ 1.3863. Purified by preparative gas chromatography.

Protocatechualdehyde [139-85-5] M 138.1, m 153°. Crystd from water or toluene and dried in a vacuum desiccator over KOH pellets or shredded wax respectively.

Protopine [fumarine, macleine, 4,6,7,14-tetrahydro-5-methyl-bis[1,3]-benzodioxolo[4,5-*c*:5',6'-*g*]azecine-13(5*H*)-one] [130-86-9] M 353.4, m 208°, pK 5.99. Crystd from EtOH/CHCl₃.

1*S*,2*S*-Pseudoephedrine (1-hydroxy-1-phenyl-2-methylaminopropane) [90-82-4] M 165.2, m 118-119°, [α]_D²⁰ +53.0° (EtOH), +40.0° (H₂O), pK²⁵ 9.71. Crystd from dry diethyl ether, or from water and dried in a vacuum desiccator.

1*S*,2*S*-Pseudoephedrine hydrochloride [345-78-8] M 210.7, m 181-182°, 185-188°, [α]_D²⁰ +61° (c 1 H₂O). Crystd from EtOH.

Pteridine [91-18-9] M 132.2, m 139.5-140°, pK₁²⁰ 4.05 (equilibrium, hydrate), pK₂²⁰ 11.90 (OH of hydrate). Crystd from EtOH, *benzene, *n*-hexane, *n*-heptane or pet ether. It sublimes at 120-130°/20mm. Stored at 0°, in the dark; turns green in the presence of light and on long standing in the dark.

2,4-(1*H*,3*H*)-Pteridinedione H₂O (lumazine) [487-21-8] M 182.1, m >350°, pK₁²⁰ <1.0, pK₂²⁰ 7.94. Crystd from water.

Pterin (2-aminopteridin-4(3*H*)-one) [2236-60-4] M 163.1, m >300°, pK₁²⁰ 2.27 (basic), pK₂²⁰ 7.96 (acidic). It was dissolved in hot 1% aqueous ammonia, filtered, and an equal volume of hot 1*M* aqueous formic acid was added. The soln was allowed to cool at 0-2° overnight. The solid was collected and washed with distilled water several times by centrifugation and dried *in vacuo* over P₂O₅ overnight, and then at 100° overnight.

Pterocarpin {(6*aR-cis*)-6*a*,12*a*-dihydro-3-methoxy-6*H*-[1,3]dioxolo[5,6]benzofuro[3,2*c*][1]-benzopyran} [524-97-0] M 298.3, m 165°, [α]₅₄₆²⁰ -215° (c 0.5, CHCl₃). Crystd from EtOH, or pet ether.

Pteronic acid (2-amino-6-*p*-carboxyanilinomethylpteridin-4(3*H*)-one) [119-24-4] M 312.3, m >300°(dec), pK_{Est(1)}~ 2.3 (basic, N1), pK_{Est(2)}~ 2.6 (basic, CH₂NH), pK_{Est(3)}~ 4.5 (COOH), pK_{Est(4)}~ 7.9 (acidic 4-OH). Crystd from dilute HCl. Hygroscopic IRRITANT

R(+)-Pulegone [89-82-7] M 152.2, b 69.5°/5mm, n 1.4849, d 0.935, [α]₅₄₆²⁰ +23.5°(neat). Purified *via* the semicarbazone. [Erskine and Waight *J Chem Soc* 3425 1960.]

Purine [120-73-0] M 120.1, m 216-217°, pK₁²⁰ 2.30, pK₂²⁰ 9.86. Crystd from toluene or EtOH.

Purpurin (1,2,4-trihydroxy-5,10-anthraquinone) [81-54-9] M 256.2, m 253-256°, pK_{Est(1)}~7.0 (2-OH), pK_{Est(2)}~9.0 (4-OH), pK_{Est(3)}~11.1 (1-OH). Cryst from aq EtOH, dry at 100°.

Purpurogallin (2,3,4,6-tetrahydroxy-5*H*-benzocyclohepten-5-one) [569-77-7] M 220.2, m 274° (rapid heating) (pK 7-10, phenolic OH). Crystd from acetic acid.

Pyocyanine (1-hydroxy-5-methylphenazinium zwitterion) [85-66-5] M 210.2, m 133° (sublimes and dec on further heating). Crystd from H₂O as dark blue needles. *Picrate* has m 190° dec.

Pyrazine [290-37-9] M 80.1, m 47°, b 115.5-115.8°, pK₁²⁰ -6.25 (aq H₂SO₄), pK₂²⁵ 1.1 (0.51 at 20°). Distd in steam and crystd from water. Purified by zone melting.

Pyrazinecarboxamide [98-96-4] M 123.1, m 189-191° (sublimes slowly at 159°), pK -0.5. Crystd from water or EtOH.

Pyrazinecarboxylic acid [98-97-5] M 124.1, m 225-229°(dec), pK²⁵ 2.92. Crystd from water.

Pyrazine-2,3-dicarboxylic acid [89-01-0] M 168.1, m 183-185°(dec), pK₁ <-2.0, pK₂ 0.9, pK₃ 2.77 (2.20). Crystd from water. Dried at 100°.

Pyrazole [288-13-1] M 68.1, m 70°, pK²⁵ 2.48. Crystd from pet ether, cyclohexane, or water. [Barszcz et al. *J Chem Soc, Dalton Trans* 2025 1986.]

Pyrazole-3,5-dicarboxylic acid [3112-31-0] M 174.1, m 287-289°(dec), pK_{Est(1)}~1.2 (CO₂H), pK_{Est(2)}~3.7 (CO₂H), pK_{Est(3)}~12 (NH). Crystd from water or EtOH.

Pyrene [129-00-0] M 202.3, m 149-150°. Crystd from EtOH, glacial acetic acid, *benzene or toluene. Purified by chromatography of CCl₄ solns on alumina, with *benzene or *n*-hexane as eluent. [Backer and Whitten *J Phys Chem* 91 865 1987.] Also zone refined, and purified by sublimation. Marvel and Anderson [*J Am Chem Soc* 76 5434 1954] refluxed pyrene (35g) in toluene (400mL) with maleic anhydride (5g) for 4days, then added 150mL of aqueous 5% KOH and refluxed for 5h with occasional shaking. The toluene layer was separated, washed thoroughly with H₂O, concentrated to about 100mL and allowed to cool. Crystalline pyrene was filtered off and recrystd three times from EtOH or acetonitrile. [Chu and Thomas *J Am Chem Soc* 108

6270 1986; Russell et al. *Anal Chem* **50** 2961 1986.] The material was free from anthracene derivatives. Another purification step involved passage of pyrene in cyclohexane through a column of silica gel. It can be sublimed in a vacuum and zone refined. [Kano et al. *J Phys Chem* **89** 3748 1985.]

Pyrene-1-aldehyde [3029-19-4] **M 230.3, m 125-126°**. Recrystd three times from aqueous EtOH.

1-Pyrenebutyric acid [3443-45-6] **M 288.4, m 184-186°, pK_{Est} ~4.1**. Crystd from *benzene, EtOH, EtOH/water (7:3 v/v) or *C₆H₆/AcOH. Dried over P₂O₅. [Chu and Thomas *J Am Chem Soc* **108** 6270 1986.]

1-Pyrenecarboxylic acid [19694-02-1] **M 230.3, m 126-127°, pK_{Est} ~3.2**. Crystd from *C₆H₆ or 95% EtOH.

1-Pyrenesulfonic acid [26651-23-0] **M 202.2, m >350°, pK_{Est} <0**. Crystd from EtOH/water. The tetra-Na salt cryst from H₂O and the sulfonyl chloride has **m 120°(dec)**. [Vollmann et al. *Justus Liebigs Ann Chem* **531** 32 1937 and *Justus Liebigs Ann Chem* **540** 189 1939.]

1,3,6,8-Pyrenetetrasulfonic acid [6528-53-6] **M 522.2, m >400°, pK_{Est} <0** Crystd from water [Tietz and Bayer *Justus Liebigs Ann Chem* **540** 189 1939.]

Pyridine [110-86-1] **M 79.1, f -41.8°, b 115.6°, d 0.9831, n 1.51021, pK²⁵ 5.23**. Likely impurities are H₂O and amines such as the picolines and lutidines. Pyridine is *hygroscopic* and is miscible with H₂O and organic solvents. It can be dried with solid KOH, NaOH, CaO, BaO or sodium, followed by fractional distn. Other methods of drying include standing with Linde type 4A molecular sieves, CaH₂ or LiAlH₄, azeotropic distn of the H₂O with toluene or *benzene, or treated with phenylmagnesium bromide in ether, followed by evaporation of the ether and distn of the pyridine. A recommended [Lindauer and Mukherjee *Pure Appl Chem* **27** 267 1971] method dries pyridine over solid KOH (20g/Kg) for 2weeks, and fractionally distils the supernatant over Linde type 5A molecular sieves and solid KOH. The product is stored under CO₂-free nitrogen. Pyridine can be stored in contact with BaO, CaH₂ or molecular sieves. Non-basic materials can be removed by steam distilling a soln containing 1.2 equivalents of 20% H₂SO₄ or 17% HCl until about 10% of the base has been carried over along with the non-basic impurities. The residue is then made alkaline, and the base is separated, dried with NaOH and fractionally distd.

Alternatively, pyridine can be treated with oxidising agents. Thus pyridine (800mL) has been stirred for 24h with a mixture of ceric sulfate (20g) and anhydrous K₂CO₃ (15g), then filtered and fractionally distd. Hurd and Simon [*J Am Chem Soc* **84** 4519 1962] stirred pyridine (135mL), water (2.5L) and KMnO₄ (90g) for 2h at 100°, then stood for 15h before filtering off the ppted manganese oxides. Addition of solid KOH (*ca* 500g) caused pyridine to separate. It was decanted, refluxed with CaO for 3h and distd.

Separation of pyridine from some of its homologues can be achieved by crstn of the oxalates. Pyridine is ppted as its oxalate by adding it to the stirred soln of oxalic acid in acetone. The ppte is filtered, washed with cold acetone, and pyridine is regenerated and isolated. Other methods are based on complex formation with ZnCl₂ or HgCl₂. Heap, Jones and Speakman [*J Am Chem Soc* **43** 1936 1921] added crude pyridine (1L) to a soln of ZnCl₂ (848g) in 730mL of water, 346mL of conc HCl and 690mL of 95% EtOH. The crystalline ppte of ZnCl₂.(pyridine)₂ was filtered off, recrystd twice from absolute EtOH, then treated with a conc NaOH soln, using 26.7g of solid NaOH to 100g of the complex. The ppte was filtered off, and the pyridine was dried with NaOH pellets and distd. Similarly, Kyte, Jeffery and Vogel [*J Chem Soc* 4454 1960] added pyridine (60mL) in 300mL of 10% (v/v) HCl to a soln of HgCl₂ (405g) in hot water (2.3L). On cooling, crystals of pyridine-HgCl₂ (1:1) complex separated and were filtered off, crystd from 1% HCl (to **m 178.5-179°**), washed with a little EtOH and dried at 110°. The free base was liberated by addition of excess aq NaOH and separated by steam distn. The distillate was saturated with solid KOH, and the upper layer was removed, dried further with KOH, then BaO and distd. Another possible purification step is fractional crstn by partial freezing.

Small amounts of pyridine have been purified by vapour-phase chromatography, using a 180-cm column of polyethyleneglycol-400 (Shell 5%) on Embacel (May and Baker) at 100°, with argon as carrier gas. The Karl Fischer titration can be used for determining water content. A colour test for pyrrole as a contaminant is described by Biddiscombe et al. [*J Chem Soc* 1957 1954].

§ Polystyrene supported pyridine is commercially available.

Pyridine-2-aldehyde [1121-60-4] M 107.1, b 81.5°/25mm, d 1.121, n 1.535, pK_1^{25} 3.84, pK_2^{25} 12.68. Sulfur dioxide was bubbled into a soln of 50g in 250mL of boiled water, under nitrogen, at 0°, until pptn was complete. The addition compound was filtered off rapidly and, after washing with a little water, it was refluxed in 17% HCl (200mL) under nitrogen until a clear soln was obtained. Neutralisation with $NaHCO_3$ and extraction with ether separated the aldehyde which was recovered by drying the extract, then distilling twice, under nitrogen. [Kyte, Jeffery and Vogel *J Chem Soc* 4454 1960.]

Pyridine-3-aldehyde [500-22-1] M 107.1, b 89.5°/14mm, d 1.141, n 1.549, pK_1^{20} 3.80, pK_2^{20} 13.10. Purification as for pyridine-2-aldehyde.

Pyridine-4-aldehyde [872-85-5] M 107.1, b 79.5°/12mm, d 1.137, n 1.544, pK_1^{20} 4.77, pK_2^{20} 12.20. . Purification as for pyridine-2-aldehyde.

Pyridine-2-aldoxime (pyridine-2-carboxaldoxime) [873-69-8] M 122.1, m 111-113°, 114°, pK_1^{25} 3.56, pK_2^{25} 10.17. Recrystd from Et_2O -pet ether or H_2O . The *picrate* has m 169-171° (from aqueous EtOH). It is used in peptide synthesis. [UV: Grammaticakis *Bull Chem Soc Fr* 109, 116 1956; Ginsberg and Wilson *J Am Chem Soc* 79 481 1957; Hanania and Irvine *Nature* 183 40 1959; Green and Saville *J Chem Soc* 3887 1956.]

Pyridine-3-aldoxime [1193-92-6] M 122.1, m 150°, pK_1^{20} 4.07, pK_2^{20} 10.39. Crystd from water.

Pyridine-4-aldoxime [696-54-8] M 122.1, m 129°, pK_1^{20} 4.73, pK_2^{20} 10.03. Crystd from water.

2,6-Pyridinedialdoxime [2851-68-5] M 165.1, m 212°, $pK_{Est(1)}$ ~3.0, $pK_{Est(2)}$ ~10. Crystd from water.

Pyridine-2,5-dicarboxylic acid [100-26-5] M 167.1, m 254°, pK_2^{25} 2.49, pK_3^{25} 5.12. Crystd from dil HCl.

Pyridine-3,4-dicarboxylic acid [490-11-9] M 167.1, m 256°, pK_1^{25} 2.43, pK_2^{25} 4.78. Crystd from dilute aqueous HCl.

Pyridine hydrobromide perbromide (pyridinium bromide perbromide) [39416-48-3] M 319.9, m 130° (dec), 132-134° (dec). It is a very good brominating agent - liberating one mol. of Br_2 . Purified by recrystn from glacial acetic acid (33g from 100mL of AcOH). [Fieser and Fieser *Reagents for Organic Chemistry* Vol 1 967 1967.]

Pyridine hydrochloride [628-13-7] M 115.6, m 144°, b 218°. Crystd from $CHCl_3$ /ethyl acetate and washed with diethyl ether.

Pyridine N-oxide [694-59-7] M 95.1, m 67°, pK^{24} 0.79. Purified by vacuum sublimation.

Pyridine 3-sulfonic acid [636-73-7] M 159.2, m 365-366° (dec), 357°, pK^{25} 2.89 (12% aq EtOH), 3.22 (H_2O)(protonation on N). Purified by recrystn from H_2O or aqueous EtOH as needles or plates. [pKa: Evans and Brown *J Org Chem* 27 3127 1962; IR: Arnett and Chawla *J Am Chem Soc* 100 214 1978.] UV in 50% aqueous EtOH: λ_{max} at 208 and 262nm. The *ammonium salt* has m 243° (from H_2O), the *sulfonyl chloride* has m 133-134° (from pet ether), the *amide* has m 110-111° (from H_2O), the *hydrochloride* has m > 300° (dec), and the *N-methyl betaine* has m 130° (from H_2O). [Gastel and Wibaut *Recl Trav Chim Pays Bas* 53 1031 1934; McIlvain and Goese *J Am Chem Soc* 65 2233 1943; Machek *Monatsh Chem* 72 77 1938.]

2-Pyridinethiol (2-mercaptopyridine) [2637-34-5] M 111.2, m 127.4°, 127-130°, 130-132°, pK_1^{20} -1.07, pK_2^{20} 9.97. If impure, dissolve in $CHCl_3$, wash with dil AcOH, H_2O , dry ($MgSO_4$), evaporate under reduced press and recryst residue from C_6H_6 or H_2O . *2-Methylmercaptopyridine* (b 100-104°/33mm) was

formed by treatment with MeI/NaOH. [Albert and Barlin *J Chem Soc* 2394 1959; Phillips and Shapiro *J Chem Soc* 584 1942.]

Pyridoxal hydrochloride, pyridoxamine hydrochloride and pyridoxine hydrochloride (vitamin B₆) see entries in Chapter 6.

1-(2-Pyridylazo)-2-naphthol (PAN) [85-85-8] M 249.3, m 140-142°, pK₁³⁰⁻³⁶ 2.9, pK₂³⁰⁻³⁶ 11.2. Purified by repeated crystn from MeOH. It can also be purified by sublimation under vacuum. Purity can be checked by TLC using a mixed solvent (pet ether, diethyl ether, EtOH; 10:10:1) on a silica gel plate.

4-(2-Pyridylazo)resorcinol (PAR) [1141-59-9] M 215.2, m >195°(dec), λ_{max} 415nm, ε 2.59 x 10⁴ (pH 6-12), pK₁²⁵ 2.69, pK₂²⁵ 5.50. Purified as the sodium salt by recrystn from 1:1 EtOH/water. Purity can be checked by TLC using a silica gel plate and a mixed solvent (*n*-BuOH:EtOH:2M NH₃; 6:2:2).

Pyridyldiphenyltriazine [1046-56-6] M 310.4, m 191-192°. Purified by repeated recrystn from EtOH/dimethylformamide.

1-(4-pyridyl)ethanol [*R*-(+)- 27854-88-2; *S*-(-)- 54656-96-1] M 123.2, m 63-65°, 67-69°, [α]_D²⁰ (+) and (-) 49.8° (c 0.5, EtOH), pK_{Est} ~5.4. Purified by recrystn from pet ether. The (-)-*di-O-benzoyl tartrate salt* has m 146-148° (from EtOH). [UV, ORD: Harelli and Samori *J Chem Soc Perkin Trans 2* 1462 1974.] The *racemate* recrystallises from Et₂O m 74-76°, b 90-94°/1mm [Ferles and Attia *Collect Czech Chem Commun* 38 611 1973; UV, NMR: Nielson et al. *J Org Chem* 29 2898 1964.]

Pyrogallol [87-66-1] M 126.1, m 136°, pK₁²⁰ 9.05, pK₂²⁰ 11.19, pK₃²⁰ 14. Crystd from EtOH*/benzene.

***R*-pyroglutamic acid (5-oxo-D-proline, *R*-2-Pyrrolidone-5-carboxylic acid)** [4042-36-8] M 129.1, m 156-158°, [α]₅₄₆²⁰ +11° (c 5, H₂O). Crystd from EtOH/pet ether.

***S*-Pyroglutamic acid (5-oxo-L-proline)** [98-79-3] M 129.1, m 156-158°, 162-164°, [α]₅₄₆²⁰ -11° (c 5, H₂O), pK 12.7 (by electron spin resonance). Crystd from EtOH by addition of pet ether. *NH₄* salt has m 184-186° (from EtOH).

Pyromellitic acid (benzene-1,2,4,5-tetracarboxylic acid) [89-05-4] M 254.2, m 276°, 281-284°, pK₁²⁵ 1.87, pK₂²⁵ 2.72, pK₃²⁵ 4.30, pK₄²⁵ 5.52. Dissolved in 5.7 parts of hot dimethylformamide, decolorised and filtered. The ppt obtained on cooling was separated and air dried, the solvent being removed by heating in an oven at 150-170° for several hours. Crystd from water.

Pyromellitic dianhydride [89-32-7] M 218.1, m 286°. Crystd from ethyl methyl ketone or dioxane. Dried, and sublimed *in vacuo*.

α-Pyrone (2*H*-pyran-2-one) [504-31-4] M 96.1, m 5°, 8-9°, b 103-111°/19-22mm, 110°/26mm, 104°/30mm, 115-118°/37mm, 206-207°/atm, d₄²⁰ 1.1972, n_D²⁰ 1.5298, pK -1.14 (aq H₂SO₄). Dissolve in Et₂O, wash with brine, dry (Na₂SO₄), filter, evaporate, distil residue under vacuum and redistil. It is a colourless liquid. [Zimmermann et al. *Org Synth Coll Vol V* 982 1973; Nakagawa and Saegusa *Org Synth* 56 49 1977; Elderfield *J Org Chem* 6 566 1941.] The *picrate* has m 106-107° (from EtOH).

γ-Pyrone (4*H*-pyran-4-one) [108-97-4] M 96.1, m 32.5-32.6°, 33°, 32-34°, b 88.5°/7mm, 91-91.5°/9mm, 95-97°/13mm, 105°/23mm, 215°/atm, pK₂₅ 0.10. Purified by vacuum distn, the distillate crystallises and is *hygroscopic*. It is non-steam volatile. The *hydrochloride* has m 139° (from EtOH), and the *picrate* has m 130.2-130.3° (from EtOH or H₂O). [Mayer *Chem Ber* 90 2362 1957; IR: Jones et al. *Can J Chem* 37 2007 1959; Neelakatan *J Org Chem* 22 1584 1957.]

Pyronin Y [3,6-bis(dimethylamino)xanthylium chloride] [92-32-0] M 302.8, m 250-260°, CI 45005, λ_{\max} 522nm, $pK_{\text{Est}} \sim 7.6$. Commercial material contained a large quantity of zinc. Purified by dissolving 1g in 50mL of hot water containing 5g NaEDTA. Cooled to 0°, filtered, evapd to dryness and the residue extracted with EtOH. The soln was evaporated to 5-10mL, filtered, and the dye pptd by addition of excess dry diethyl ether. It was centrifuged and the crystals were washed with dry ether. The procedure was repeated, then the product was dissolved in CHCl_3 , filtered and evapd. The dye was stored in a vacuum.

Pyrrole [109-97-7] M 67.1, m 23.4°, b 66°/80mm, 129-130°/atm, d 0.966, n 1.5097, $pK_1^{25} -4.4$ (Protonation on carbon), $pK_2^{25} 17.51$ (aq KOH, H. scale). Dried with NaOH, CaH_2 or CaSO_4 . Fractionally distd under reduced pressure from CaH_2 . Stored under nitrogen, turns brown in air. Redistd immediately before use.

Pyrrolidine [123-75-1] M 71.1, b 87.5-88.5°, d 0.860, n 1.443, $pK^{25} 11.31$. Dried with BaO or sodium, then fractionally distd, under N_2 , through a Todd column packed with glass helices (see p. 174).

Pyrrolidine-1-carbodithioic acid ammonium salt [5108-96-3] M 164.3, m 128-130°, $pK^{25} 3.25$ (free acid). Purified by recryst twice by dissolving in MeOH and adding Et_2O . Also by recrystn from EtOH. [Synth and Polarography: Kitagawa and Taku *Bull Chem Soc Jpn* 64 2151 1973; Malissa and Schöffmann *Mikrochim Acta* 187 1955.]

Pyruvic acid [127-17-3] M 88.1, m 13°, b 65°/10mm, $pK^{25} 2.39$ (2.60). Distd twice, then fractionally crystd by partial freezing.

***p*-Quaterphenyl** [135-70-6] M 306.4, m 312-314°. Recrystd from dimethyl sulfoxide at *ca* 50°.

Quercetin ($2\text{H}_2\text{O}$) (3,3',4',5,6-pentahydroxyflavone) [6151-25-3 ($2\text{H}_2\text{O}$); 117-39-3 (anhydr)] M 338.3, m *ca* 315°(dec), (phenolic pK_s 7-10). Crystd from aq EtOH and dried at 100°.

Quercitrin (quercetin glycoside) [522-12-3] M 302.2, m 168°, 176-178°. Crystd from aq EtOH and dried at 135° to give the higher melting form.

Quinaldic (quinoline-2-carboxylic) acid [93-10-7] M 173.2, m 156-157°, $pK_1^{25} 1.45$, $pK_2^{25} 2.49$ (2.97). Crystd from *benzene.

Quinalizarin (1,2,5,8-tetrahydroxy-9,10-anthraquinone) [81-61-8] M 272.2, m 275°, $pK_{\text{Est}(1)} \sim 7.1$ (1-OH), $pK_{\text{Est}(2)} \sim 9.9$ (8-OH), $pK_{\text{Est}(3)} \sim 11.1$ (5-OH), $pK_{\text{Est}(4)} \sim 11.8$ (2-OH). Crystd from acetic acid or nitrobenzene. It can be sublimed *in vacuo*.

Quinazoline [253-82-7] M 130.2, m 48.0-48.5°, b 120-121°/17-18mm, $pK_1^{20} -4.51$ (aq H_2SO_4 , anhydrous dication), $pK_2^{20} 2.01$ (anhydrous monocation), $pK_3^{20} 4.3$ (equilibrium with 3,4-hydrated species), $pK_4^{20} 12.1$ (hydrated anion). Purified by passage through an activated alumina column in *benzene or pet ether (b 40-60°). Distd under reduced pressure, sublimed under vacuum and crystd from pet ether. [Armarego *J Appl Chem* 11 70 1961.]

Quinhydrone [106-34-3] M 218.2, m 168°. Crystd from H_2O at 65°, then dried in a vac desiccator.

1R,3R,4R,5R-Quinic acid (1,3,4,5-tetrahydroxy-cyclohexane-carboxylic acid) [77-95-2] M 192.3, m 172°(dec), $[\alpha]_{546}^{20} -51^\circ$ (c 20, H_2O), $pK^{25} 3.58$. Crystd from water.

Quinidine [56-54-2] M 324.4, m 171°, $[\alpha]_{546}^{20} +301.1^\circ$ (CHCl_3 contg 2.5% (v/v) EtOH), $pK_1^{15} 4.13$, $pK_2^{15} 8.77$. Crystd from *benzene or dry CHCl_3 /pet ether (b 40-60°), discarding the initial, oily crop of crystals. Dried under vacuum at 100° over P_2O_5 .