

Cytosine see entry in Chapter 6.

***cis*-Decahydroisoquinoline** [2744-08-3] **M 139.2, b 97-98°/15mm, 208-209°/730mm, pK²⁰ 11.32.** The free base is treated with satd aq picric acid, allowed to stand for 12h, fild, washed with MeOH to remove the more soluble *trans* isomer and recrystd from MeOH to give pure *cis-picrate* **m 149-150°.** The picrate (~5g) is shaken with 5M aq NaOH (50mL) and Et₂O (150mL) while H₂O is added to the aq phase to dissolve insoluble Na picrate. The Et₂O extract is dried over solid NaOH and then shaken with Al₂O₃ (Merck for chromatography) until the yellow color of traces of picric acid disappears (this color cannot be removed by repeated shaking with 5-10 M aq NaOH). The extract is concentrated to 50mL and dry HCl is bubbled through until separatrtn of the white crystals of the *cis-HCl* is complete. These are washed with Et₂O, dried at 100° and recrystd from EtOH + EtOAc to yield pure *cis-Hydrochloride* **m 182-183°** (dried in a vac desiccator over KOH) with IR (KBr) ν_{\max} 2920, 2820, 1582, 1470, 1445, 1410, 1395, 1313, 1135, 1080, 990, 870 cm⁻¹. The pure *free base* is prepared by dissolving the *hydrochloride* in 10 M aq NaOH, extracted with Et₂O, dried over solid KOH, fild and distd in vac. It has IR (film) ν_{\max} 2920, 2820, 2720, 2560, 1584, 1470, 1445, 1415, 1395, 1315, 1300, 1135, 1080, 1020, 990, 873 cm⁻¹. The ¹H NMR in CDCl₃ is characteristically different from that of the *trans*-isomer. [Armarego *J Chem Soc (C)* 377 1967; Gray and Heitmeier *J Am Chem Soc* 80 6274 1958; Witkop *J Am Chem Soc* 70 2617 1948; Skita *Chem Ber* 57 1982 1924; Helfer *Helv Chim Acta* 6 7991923.]

***trans*-Decahydroisoquinoline** [2744-09-4] **M 139.2, b 106°/15mm, pK²⁰ 11.32.** This is purified as the *cis*-isomer above. The *trans-picrate* has **m 175-176°**, and the *trans-hydrochloride* has **m 221-222°** and has IR (KBr) ν_{\max} 2930, 3800, 1589, 1450, 1400, 1070, 952, 837 cm⁻¹. The pure *free base* was prepared as above and had IR (film) ν_{\max} 2920, 2820, 2720, 2560, 1584, 1470, 1445, 1415, 1395, 1315, 1300, 1135, 1080, 1020, 990, 873 cm⁻¹. The ¹H NMR in CDCl₃ is characteristically different from that of the *cis*-isomer. (references as above and Helfer *Helv Chim Acta* 9 818 1926).

Decahydronaphthalene (decalin, mixed isomers) [91-17-8] **M 138.2, b 191.7°, d 0.886, n 1.476.** Stirred with conc H₂SO₄ for some hours. Then the organic phase was separated, washed with water, saturated aqueous Na₂CO₃, again with water, dried with CaSO₄ or CaH₂ (and perhaps dried further with Na), filtered and distd under reduced pressure (**b 63-70°/10mm**). Also purified by repeated passage through long columns of silica gel previously activated at 200-250°, followed by distn from LiAlH₄ and storage under N₂. Type 4A molecular sieves can be used as a drying agent. Storage over silica gel removes water and other polar substances.

***cis*-Decahydronaphthalene** [493-01-6] **M 138.2, f -43.2°, b 195.7°, d 0.897, n 1.48113.** Purification methods described for the mixed isomers are applicable. The individual isomers can be separated by very efficient fractional distn, followed by fractional crystn by partial freezing. The *cis*-isomer reacts preferentially with AlCl₃ and can be removed from the *trans*-isomer by stirring the mixture with a limited amount of AlCl₃ for 48h at room temperature, filtering and distilling.

***trans*-Decahydronaphthalene** [493-02-7] **M 138.2, f -30.6°, b 187.3°, d 0.870, n 1.46968.** See purification of *cis*-isomer above.

***cis*-Decahydroquinoline** [10343-99-4] **M 139.2, b 207-208°/708mm, pK²⁰ 11.29.** It is available as a *cis-trans*-mixture (**b 70-73°/10mm**, Aldrich, ~ 18% *cis*-isomer [2051-28-7]), but the isomers can be fractionated in a spinning band column (1~1.5 metre, type E) at atmospheric pressure and collecting 2mL fractions with a distillation rate of 1 drop in 8-10sec. The lower boiling fraction solidifies and contains the *trans*-isomer (see below, **m 48°**). The higher boiling fraction **b 207-208°/708mm**, remains liquid and is mostly the *cis*-isomer. This is reacted with PhCOCl and M aq NaOH to yield the *N-benzoyl derivative* **m 96°** after recrystn from pet ether (**b 80-100°**). It is hydrolysed with 20% aq HCl by refluxing overnight. PhCO₂H is fild off, the filtrate is basified with 5M aq NaOH and extracted with Et₂O. The dried extract (Na₂SO₄) is satd with dry HCl gas and the *cis-decahydroquinoline hydrochloride* which separates has **m 222-224°** after washing with Et₂O and drying at 100°; and has IR (KBr) ν_{\max} 2900, 2780, 2560, 1580, 1445, 1432, 1403, 1165, 1080,

1036, 990, 867 cm^{-1} . The *free base* is obtained by dissolving the *hydrochloride* salt in 5M aq NaOH, extracting with Et_2O and drying the extract (Na_2SO_4), evaporating and distilling the residue; it has IR (film) ν_{max} 2900, 2840, 2770, 1445, 1357, 1330, 1305, 1140, 1125, 1109, 1068, 844 cm^{-1} . The ^1H NMR in CDCl_3 is characteristically different from that of the *trans*-isomer. [Armarego *J Chem Soc (C)* 377 1967; Hüchel and Stepf *Justus Liebigs Ann Chem* 453 163 1927; Bailey and McElvain *J Am Chem Soc* 52 4013 1930.]

***trans*-Decahydroquinoline** [767-92-0] M 139.2, m 48°, b 205-206°/708mm, pK^{20} 11.29. The lower boiling fraction from the preceding spinning band column fractionation of the commercial *cis-trans*-mixture (~ 20:60; see the *cis*-isomer above) solidifies readily (m 48°) and the receiver has to be kept hot with warm water. It is further purified by conversion to the *Hydrochloride* m 285-286° after recrystn from EtOH/AcOEt . This has IR (KBr) ν_{max} 2920, 2760, 2578, 2520, 1580, 1455, 1070, 1050, 975, 950, 833 cm^{-1} . The *free base* is prepared as for the *cis*-isomer above and distd; and has IR (film, at ca 50°) ν_{max} 2905, 2840, 2780, 1447, 1335, 1305, 1240, 1177, 1125, 987, 900, 835 cm^{-1} . The ^1H NMR in CDCl_3 is characteristically different from that of the *cis*-isomer. [Armarego *J Chem Soc (C)* 377 1967; Hüchel and Stepf *Justus Liebigs Ann Chem* 453 163 1927; Bailey and McElvain *J Am Chem Soc* 52 4013 1930; Prelog and Szpilfogel *Helv Chim Acta* 28 1684 1945.]

***n*-Decane** [124-18-5] M 142.3, b 174.1°, d 0.770, n 1.41189, n^{25} 1.40967. It can be purified by shaking with conc H_2SO_4 , washing with water, aqueous NaHCO_3 , and more water, then drying with MgSO_4 , refluxing with Na and distilling. Passed through a column of silica gel or alumina. It can also be purified by azeotropic distn with 2-butoxyethanol, the alcohol being washed out of the distillate, using water; the decane is next dried and redistilled. It can be stored with NaH. Further purification can be achieved by preparative gas chromatography on a column packed with 30% SE-30 (General Electric methyl-silicone rubber) on 42/60 Chromosorb P at 150° and 40psig, using helium [Chu *J Chem Phys* 41 226 1964]. Also purified by zone refining.

Decan-1,10-diol [112-47-0] M 174.3, m 72.5-74°. Crystd from dry ethylene dichloride.

***n*-Decanol (*n*-decyl alcohol)** [112-30-1] M 158.3, f 6.0°, b 110-119°/0.1mm, d 0.823, n 1.434. Fractionally distd in an all-glass unit at 10mm pressure (b 110°), then fractionally crystd by partial freezing. Also purified by preparative GLC, and by passage through alumina before use.

***n*-Decyl bromide** [112-29-8] M 221.2, b 117-118°/15.5mm, d 1.066. Shaken with H_2SO_4 , washed with water, dried with K_2CO_3 , and fractionally distd.

Decyltrimethylammonium bromide [2082-84-0] M 280.3, m 239-242°. Crystd from 50% (v/v) $\text{EtOH}/\text{diethyl ether}$, or from acetone and washed with ether. Dried under vacuum at 60°. Also recrystd from EtOH and dried over silica gel. [McDonnell and Kraus *J Am Chem Soc* 73 2170 1952; Dearden and Wooley *J Phys Chem* 91 2404 1987.]

(+)-Dehydroabietylamine (abieta-8,11,13-triene-18-ylamine) [1446-61-3] M 285.5, m 41°, 42.5-45°, b 192-193°/1mm, 250°/12mm, n_D^{40} 1.546, $[\alpha]_{546}^{20} +51^\circ$ (c 1, EtOH), $\text{pK}_{\text{Est}} \sim 10.3$. The crude base is purified by converting 2g of base in toluene (3.3mL) into the acetate salt by heating at 65-70° with 0.46g of AcOH and the crystals are collected and dried (0.96g from two crops; m 141-143°). The acetate salt is dissolved in warm H_2O , basified with aqueous NaOH and extracted with $^*\text{C}_6\text{H}_6$. The dried extract (MgSO_4) is evaporated in vacuum leaving a viscous oil which crystallises and can be distd. [Gottstein and Cheney *J Org Chem* 30 2072 1965.] The *picrate* has m 234-236° (from aq MeOH), and the *formate* has m 147-148° (from heptane).

Dehydro-L(+)-ascorbic acid [490-83-5] M 174.1, m 196°(dec), $[\alpha]_{546}^{20} +42.5^\circ$ (c 1, H_2O), pK 3.90. Crystd from MeOH

7-Dehydrocholesterol [434-16-2] M 384.7, m 142-143°, $[\alpha]_D^{20} -122^\circ$ (c 1, CHCl_3). Crystd from MeOH .

Dehydrocholic acid [81-23-2] M 402.5, m 237°, $[\alpha]_{546}^{20}$ -159° (c 1, in CHCl₃), pK_{Est} ~4.9. Crystd from acetone.

Dehydroepiandrosterone [54-43-0] M 288.4, m 140-141° and 152-153° (dimorphic), $[\alpha]_{\text{D}}^{20}$ +13° (c 3, EtOH). Crystd from MeOH and sublimed in vacuum.

Delphinine [561-07-9] M 559.7, m 197-199°, $[\alpha]_{\text{D}}^{20}$ +26° (EtOH). Crystd from EtOH.

3-Deoxy-D-allose [6605-21-6] M 164.2, $[\alpha]_{\text{D}}^{20}$ +8° (c 0.25 in H₂O). Obtained from diethyl ether as a colourless syrup.

Deoxybenzoin [451-40-1] M 196.3, m 60°, b 177°/12mm, 320°/760mm. Crystd from EtOH.

Deoxycholic acid [83-44-3] M 392.6, m 171-174°, 176°, 176-178°, $[\alpha]_{546}^{20}$ +64° (c 1, EtOH), $[\alpha]_{\text{D}}^{20}$ +55° (c 2.5, EtOH), pK 6.58. Refluxed with CCl₄ (50mL/g), filtered, evaporated under vacuum at 25°, recrystd from acetone and dried under vacuum at 155° [Trenner et al. *J Am Chem Soc* 76 1196 1954]. A soln of (cholic acid-free) material (100mL) in 500mL of hot EtOH was filtered, evaporated to less than 500mL on a hot plate, and poured into 1500mL of cold diethyl ether. The ppte, filtered by suction, was crystd twice from 1-2 parts of absolute EtOH, to give an alcoholate, m 118-120°, which was dissolved in EtOH (100mL for 60g) and poured into boiling water. After boiling for several hours the ppte was filtered off, dried, ground and dried to constant weight [Sobotka and Goldberg *Biochem J* 26 555 1932]. Deoxycholic acid was also freed from fatty acids and cholic acid by silica gel chromatography by elution with 0.5% acetic acid in ethyl acetate [Tang et al. *J Am Chem Soc* 107 4058 1985]. It can also be recrystd from butanone. Its solubility in H₂O at 15° is 0.24g/L but in EtOH it is 22.07g/L.

11-Deoxycorticosterone (21-hydroxyprogesterone) [64-85-7] M 330.5, m 141-142°, $[\alpha]_{546}^{20}$ +178° and $[\alpha]_{546}$ +223° (c 1, EtOH). Crystd from diethyl ether.

2-Deoxy-β-D-galactose [1949-89-9] M 164.2, m 126-128°, $[\alpha]_{\text{D}}^{20}$ +60° (c 4, H₂O). Crystd from diethyl ether.

2-Deoxy-α-D-glucose [154-17-6] M 164.2, m 146°, $[\alpha]_{\text{D}}^{20}$ +46° (c 0.5, H₂O after 45h). Crystd from MeOH/acetone.

6-Deoxy-D-glucose (D-quinovose) [7658-08-4] M 164.2, m 146°, $[\alpha]_{\text{D}}^{20}$ +73° (after 5 min) and +30° (final, after 3h) (c 8.3, H₂O). It is purified by recrystn from EtOAc and is soluble in H₂O, EtOH but almost insoluble in Et₂O and Me₂CO. [Srivastava and Lerner *Carbohydr Res* 64 263 1978; NMR: Angyal and Pickles *Aust J Chem* 25 1711 1972.]

2-Deoxy-β-L-ribose [18546-37-7] M 134.1, m 77°, 80°, $[\alpha]_{\text{D}}^{25}$ +91.7° (c 7, pyridine, 40° final). Crystd from diethyl ether.

2-Deoxy-β-D-ribose [533-67-5] M 134.1, m 86-87°, 87-90°, $[\alpha]_{\text{D}}^{20}$ -56° (c 1, H₂O after 24h). Crystd from diethyl ether.

Desyl bromide (α-bromo-desoxybenzoin, ω-bromo-ω-phenyl acetophenone) [484-50-0] M 275.2, m 57.1-57.5°. Crystd from 95% EtOH.

Desyl chloride (α-chloro-desoxybenzoin, ω-chloro-ω-phenyl acetophenone) [447-31-4] M 230.7, m 62-64°, 66-67°, 67.5°, 68°. For the purification of small quantities recrystallise from pet ether (b 40-60°), but use MeOH or EtOH for larger quantities. For the latter solvent, dissolve 12.5g of chloride in 45mL of boiling EtOH (95%), filter and the filtrate yields colourless crystals (7.5g) on cooling. A further crop (0.9g) can be obtained by cooling in an ice-salt bath. It turns brown on exposure to sunlight but it is

stable in sealed dark containers. [Henley and Turner *J Chem Soc* 1182 1931; *Org Synth Coll Vol II* 159 1943.]

Dexamethasone (9- α -fluoro-16- α -methylprednisolone) [50-02-2] **M 392.5, m 262-264 $^{\circ}$, 268-271 $^{\circ}$, $[\alpha]_D^{25} +77.5^{\circ}$ (c 1, dioxane).** It has been recrystallised from Et₂O or small volumes of EtOAc. Its solubility in H₂O in 10 mg/100mL at 25 $^{\circ}$; and is freely soluble in Me₂CO, EtOH and CHCl₃. [Arth et al. *J Am Chem Soc* 80 3161 1958; for the β -methyl isomer see Taub et al. *J Am Chem Soc* 82 4025 1960.]

Dexamethasone 21-acetate (9- α -fluoro-16- α -methylprednisolone-21-acetate) [1177-87-3] **M 434.5, m 215-225 $^{\circ}$, 229-231 $^{\circ}$, $[\alpha]_D^{25} +77.6^{\circ}$ (c 1, dioxane), +73 $^{\circ}$ (c 1, CHCl₃).** Purified on neutral Al₂O₃ using CHCl₃ as eluent, fraction evaporated, and recrystd from CHCl₃. UV has λ_{\max} at 239nm. [Oliveto et al. *J Am Chem Soc* 80 4431 1958].

Diacetamide [625-77-4] **M 101.1, m 75.5-76.5 $^{\circ}$, b 222-223 $^{\circ}$.** Purified by crystn from MeOH [Arnett and Harrelson *J Am Chem Soc* 109 809 1987].

Diaceoxyiodobenzene (iodobenzenediacetate) [3240-34-4] **M 322.1, m 163-165 $^{\circ}$.** Purity can be checked by treatment with H₂SO₄ then KI and the liberated I₂ estimated with standard thiosulfate. It has been recrystd from 5M acetic acid and dried overnight in a vac desiccator over CaCl₂. The surface of the crystals may become slightly yellow but this does not affect its usefulness. [Sharefkin and Saltzman *Org Synth Coll Vol V* 600 1973.]

1,2-Diacetyl benzene [704-00-7] **M 162.2, m 39-41 $^{\circ}$, 41-42 $^{\circ}$, b 110 $^{\circ}$ /0.1mm, 148 $^{\circ}$ /20mm.** Purified by distn and by recrystn from pet ether. The *bis-2,4-dinitrophenyl hydrazone* has m 221 $^{\circ}$ dec. [Halford and Weissmann *J Org Chem* 17 1646 1952; Riemschneider and Kassahn *Chem Ber* 92 1705 1959.]

1,4-Diacetyl benzene [1009-61-6] **M 162.2, m 113-5-114.2 $^{\circ}$.** Crystd from *benzene and vacuum dried over CaCl₂. Also dissolved in acetone, treated with Norit, evapd and recrystd from MeOH [Wagner et al. *J Am Chem Soc* 108 7727 1986].

(+)-Di-O-acetyl-L-tartaric anhydride [(R,R)-2,3-diacetoxysuccinic anhydride] [6283-74-5] **M 216.2, m 129-132 $^{\circ}$, 133-134 $^{\circ}$, 135 $^{\circ}$, 137.5 $^{\circ}$, $[\alpha]_D^{20} +97.2^{\circ}$ (c 0.5, dry CHCl₃).** If the IR is good, i.e. no OH bands, then keep in a vacuum desiccator overnight (over P₂O₅/paraffin) before use. If OH bands are present then reflux 4g in Ac₂O (12.6mL) containing a few drops of conc H₂SO₄ for 10min (use a relatively large flask), pour onto ice, collect the crystals, wash with dry *C₆H₆ (2 x 2mL), stir with 17mL of cold Et₂O, filter and dry in a vacuum desiccator as above, and store in dark evacuated ampoules under N₂ in small aliquots. It is not very stable in air; the melting point of the crystals drop one degree in the first four days then remains constant (132-134 $^{\circ}$). If placed in a stoppered bottle it becomes gummy and the m falls 100 $^{\circ}$ in three days. Recrystn leads to decomposition. If good quality anhydride is required it should be prepared fresh from tartaric acid. It sublimes in a CO₂ atmosphere. [*Org Synth Coll Vol IV* 242 1963.]

Diallyl amine (N-2-propenyl-2-propen-1-amine) [124-02-7] **M 97.2, b 107-111 $^{\circ}$ /760mm, 112 $^{\circ}$ /760mm, d_4^{20} 0.789, n_D^{20} 1.440, pK^{20} 9.42.** Keep over KOH pellets overnight, decant and distil from a few pellets of KOH at atm pressure (b 108-111 $^{\circ}$), then fractionate through a Vigreux column. [Vliet *J Am Chem Soc* 46 1307 1924; *Org Synth Coll Vol I* 201 1941.] The *hydrochloride* has m 164-165 $^{\circ}$ (from Me₂CO + EtOH). [Butler and Angels *J Am Chem Soc* 79 3128 1957.]

(+)-N,N'-Diallyl tartramide (DATD) [58477-85-3] **M 228.3, m 184 $^{\circ}$, $[\alpha]_{546} +141^{\circ}$ (c 3, MeOH).** Wash with Et₂O containing 10% EtOH until the washings are clear and colourless, and dry *in vacuo*. [*FEBS Lett* 7 293 1970.]

Diamantane [2292-79-7] **M 188.3, m 234-235 $^{\circ}$.** Purified by repeated crystn from MeOH or pentane. Also dissolved in CH₂Cl₂, washed with 5% aq NaOH and water, and dried (MgSO₄). The soln was concentrated to a small volume, an equal weight of alumina was added, and the solvent evaporated. The residue was placed on

an activated alumina column (ca 4 x weight of diamantane) and eluted with pet ether (b 40-60°). Eight sublimations and twenty zone refining experiments gave material **m** 251° of 99.99% purity by differential analysis [*Tetrahedron Lett* 3877 1970; *J Chem Soc (C)* 2691 1972].

3,6-Diaminoacridine hydrochloride [952-23-8] **M** 245.7, **m** 270°(dec), ϵ_{456} 4.3×10^4 , pK_1^{20} 1.5, pK_2^{20} 9.60 (9.65 free base). First purified by pptn of the free base by adding aq NH_3 soln to an aq soln of the hydrochloride or hydrogen sulfate, drying the ppt and subliming at 0.01mm Hg [Müller and Crothers *Eur J Biochem*, 54 267 1975].

3,6-Diaminoacridine sulfate (proflavin sulfate) [1811-28-5] **M** 516.6, **m** >300°(dec), λ_{max} 456nm. An aqueous soln, after treatment with charcoal, was concentrated, chilled overnight, filtered and the ppt was rinsed with a little diethyl ether. The ppt was dried in air, then overnight in a vacuum oven at 70°.

1,3-Diaminoadamantane [10303-95-4] **M** 164.3, **m** 52°, $\text{pK}_{\text{Est}(1)}$ ~8.6, $\text{pK}_{\text{Est}(2)}$ ~10.6. Purified by zone refining.

1,4-Diaminoanthraquinone [128-95-0] **M** 238.3, **m** 268°. Purified by thin-layer chromatography on silica gel using toluene/acetone (9:1) as eluent. The main band was scraped off and extracted with MeOH. The solvent was evaporated and the quinone was dried in a drying pistol [Land, McAlpine, Sinclair and Truscott *J Chem Soc, Faraday Trans 1* 72 2091 1976]. Crystd from EtOH in dark violet crystals.

1,5-Diaminoanthraquinone [129-44-2] **M** 238.3, **m** 319°. Recrystd from EtOH or acetic acid [Flom and Barbara *J Phys Chem* 89 4481 1985].

2,6-Diaminoanthraquinone [131-14-6] **M** 238.3, **m** 310-320°. Crystd from pyridine. Column-chromatographed on Al_2O_3 /toluene to remove a fluorescent impurity, then recrystd from EtOH.

3,3'-Diaminobenzidine tetrahydrochloride (2H₂O) [7411-49-6] **M** 396.1, **m** >300°(dec), $\text{pK}_{\text{Est}(1)}$ ~3.3, $\text{pK}_{\text{Est}(2)}$ ~4.7 (free base). Dissolved in water and pted by adding conc HCl, then dried over solid NaOH.

3,4-Diaminobenzoic acid [619-05-6] **M** 152.2, **m** 213°(dec), 228-229°, pK_1^{25} 2.57 (4-NH₂), pK_2^{25} 3.39 (3-NH₂), $\text{pK}_{\text{Est}(3)}$ ~5.1 (CO₂H). Crystd from H₂O or toluene.

3,5-Diaminobenzoic acid [535-87-5] **M** 152.2, **m** 235-240°(dec), pK^{25} 5.13 (CO₂H). Crystd from water. *Dihydrochloride* has **m** 226-228°(dec).

4,4'-Diaminobenzophenone [611-98-3] **M** 212.3, **m** 242-244°, 243-245°, 246.5-247.5° (after sublimation at 0.0006 mm), pK_1^{25} 1.37, pK_2^{25} 2.92. Purified by recrystn from EtOH and by sublimation in high vacuum. The *dihydrochloride* has **m** 260° dec (from EtOH) and the *thiosemicarbazone* has **m** 207-207.5° dec (from aq EtOH). [Kuhn et al. *Chem Ber* 75 711 1942.]

1,4-Diaminobutane dihydrochloride (putrescine 2HCl) [333-93-7] **M** 161.1, **m** >290°, pK_1^{25} 9.63, pK_2^{25} 10.80. Crystd from EtOH/water.

1,2-Diamino-4,5-dichlorobenzene [5348-42-5] **M** 177.0, **m** 163°, $\text{pK}_{\text{Est}(1)}$ ~0, $\text{pK}_{\text{Est}(2)}$ ~2.9. Refluxed with activated charcoal in CH_2Cl_2 , followed by recrystn from diethyl ether/pet ether or pet ether [Koolar and Kochi *J Org Chem* 52 4545 1987].

2,2'-Diaminodiethylamine (diethylenetriamine) [111-40-0] **M** 103.2, **b** 208°, **d** 0.95, **n** 1.483, pK_1^{25} 4.34, pK_2^{25} 9.13, pK_3^{25} 9.94. Dried with Na and distd, preferably under reduced pressure, or in a stream of N₂. § Polymer-bound diethylenetriamine is commercially available.

4,5-Diamino-2,6-dihydroxypyrimidine (diamino uracil) sulfate [32014-70-3] **M** 382.3, **m** >300°, pK_1^{20} 1.7, pK_2^{20} 3.20, pK_3^{20} 4.56. The salt is quite insoluble in H₂O but can be converted to the

free base which is recrystd from H₂O and converted to the sulfate by addition of the required amount of H₂SO₄. The *hydrochloride* has *m* 300-305° dec and can be used to prepare the sulfate by addition of H₂SO₄; It is more soluble than the sulfate. The *perchlorate* has *m* 252-254°. The free base has λ_{\max} 260nm (log ϵ 4.24) in 0.1M HCl. [Bogert and Davidson *J Am Chem Soc* **86** 1668 1933; Bredereck et al. *Chem Ber* **86** 850 1953; *Org Synth Coll Vol IV* 247 1963; Barlin and Pfeleiderer *J Chem Soc (B)* 1425 1971.]

5,6-Diamino-1,3-dimethyluracil hydrate (5,6-diamino-1,3-dimethyl-2-pyrimidine-2,4-dione hydrate) [5440-00-6] *M* 188.2, *m* 205-208° dec, 209° dec, 210°dec, *pK*₁ 1.7, *pK*₂ 4.6. Recryst from EtOH. The *hydrochloride* has *m* 310° (from MeOH) and the *perchlorate* has *m* 246-248°. [UV: Bredereck et al. *Chem Ber* **92** 583 1959; Taylor et al. *J Am Chem Soc* **77** 2243 1955.]

4,4'-Diamino-3,3'-dinitrobiphenyl [6271-79-0] *M* 274.2, *m* 275°, *pK*_{Est} ~-0.2. Crystd from aqueous EtOH.

4,4'-Diaminodiphenylamine [537-65-5] *M* 199.3, *m* 158°, *pK*_{Est} ~5.0. Crystd from water.

4,4'-Diaminodiphenylmethane [101-77-9] *M* 198.3, *m* 91.6-92°, *pK*_{Est} ~4.9. Crystd from water, 95% EtOH or *benzene.

3,3'-Diaminodipropylamine [56-18-8] *M* 131.2, *b* 152°/50mm, *d* 0.938, *n* 1.481, *pK*₁²⁵ 7.72, *pK*₂²⁵ 9.57, *pK*₃²⁵ 10.65. Dried with Na and distd under vacuum.

6,9-Diamino-2-ethoxyacridine (Ethacridine) [442-16-0] *M* 257.3, *m* 226°, *pK*_{Est} ~11.5. Crystd from 50% EtOH.

2,7-Diaminofluorene [524-64-4] *M* 196.3, *m* 165°, *pK*_{Est} ~4.6. Recrystd from H₂O.

2,4-Diamino-6-hydroxypyrimidine [56-06-4] *M* 126.1, *m* 260-270°(dec), *pK*₁²⁵ 1.34, *pK*₂²⁵ 3.27, *pK*₃²⁵ 10.83. Recrystd from H₂O.

4,5-Diamino-6-hydroxypyrimidine hemisulfate [102783-18-6] *M* 350.3, *m* 268°, 270°, *pK*₁²⁵ 1.34, *pK*₂²⁵ 3.57, *pK*₃²⁵ 9.86. Recrystd from H₂O. The free base also recrystallises from H₂O (*m* 239°). [UV: Mason *J Chem Soc* 2071 1954; Elion et al. *J Am Chem Soc* **74** 411 1952.]

1,5-Diaminonaphthalene [2243-62-1] *M* 158.2, *m* 190°, *pK*²⁵ 4.12. Rerystd from boiling H₂O, but is wasteful due to poor solubility. Boil in chlorobenzene (charcoal), filter hot and cool the filtrate. This gives colourless crystals. Dry in a vac till free from chlorobenzene (odour), and store away from light.

1,8-Diaminonaphthalene [479-27-6] *M* 158.2, *m* 66.5°, *b* 205°/12mm, *pK* 4.44. Crystd from water or aqueous EtOH, and sublimed in a vacuum. *N,N'*-DiMe deriv [20734-56-9] has *m* 103-104° and *pK* 5.61; *N,N,N'*-TriMe deriv [20734-57-0] has *m* 29-30° and *pK* 6.43. [Hodgson et al. *J Chem Soc* 202 1945.]

2,3-Diaminonaphthalene [771-97-1] *M* 158.2, *m* 199°, *pK*²¹ 3.54 (in 50% aq EtOH). Crystd from water, or dissolved in 0.1M HCl, heated to 50°. After cooling, the soln was extracted with decalin to remove fluorescent impurities and centrifuged.

1,8-Diaminooctane [373-44-4] *M* 144.3, *m* 50-52°, 51-52°, 52-53°, *b* 121°/18mm, 120°/24mm, *pK*₁²⁰ 10.1, *pK*₂²⁰ 11.0. Distil under vacuum in an inert atmosphere(N₂, Ar), cool and store distillate in an inert atmosphere in the dark. The *dihydrochloride* has *m* 273-274°. [Nae and Le *Helv Chim Acta* **15** 55 1955.]

2,4-Diamino-5-phenylthiazole (Amiphenazole) [490-55-1] *M* 191.3, *m* 163-164°(dec). Crystd from aqueous EtOH or water. Stored in the dark under N₂.

1,5-Diaminopentane [462-94-2] M 102.2, m 14-16°, b 78-80°/12mm, 101-103°/35mm, 178-180°/750mm, d_4^{20} 0.869, n_D^{20} 1.458, pK_1^{20} 10.02, pK_2^{20} 10.96. Purified by distn, after standing over KOH pellets (at room temp; i.e. liquid form). It has pK_a^{20} values of 10.02 and 10.96 in H₂O. Its *dihydrochloride* has m 275° (sublimes in vac), and its *tetraphenyl boronate* has m 164°. [Schwarzenbach et al. *Helv Chim Acta* 35 2333 1952.]

***d,l*-2,6-Diaminopimelic acid** [583-93-7] M 190.2, m 313-315°(dec) $pK_{Est(1)}$ ~2.2, $pK_{Est(2)}$ ~9.7. Crystd from water.

1,3-Diaminopropane dihydrochloride [10517-44-9] M 147.1, m 243°, pK_1^{25} 8.29, pK_2^{25} 10.30. Crystd from EtOH/water.

1,3-Diaminopropan-2-ol [616-29-5] M 90.1, m 38-40°, pK_1^{25} 7.94, pK_2^{25} 9.57. Dissolved in an equal amount of water, shaken with charcoal and vacuum distd at 68°/0.1mm. It is too viscous to be distd through a packed column.

***L(S)*-2,3-Diaminopropionic acid monohydrochloride (3-amino-*L*-alanine hydrochloride)** [1482-97-9] M 140.6, m 132-133°dec, 237°dec, $[\alpha]_D^{25}$ +26.1° (c 5.8, M HCl), pK_1^{25} 1.30, pK_2^{25} 6.79, pK_3^{25} 9.51. Forms needles from H₂O and can be recrystd from aqueous EtOH. [Gmelin et al. *Z Physiol Chem.* 314 28 1959; IR: Koegel et al. *J Am Chem Soc* 77 5708 1977.]

2,3-Diaminopyridine [452-58-4] M 109.1, m 116°, pK_1^{25} -0.50, pK_2^{25} 6.92. Crystd from *benzene and sublimed *in vacuo*.

2,6-Diaminopyridine [141-86-6] M 109.1, m 121.5° $pK_{Est(1)}$ <-6.0, $pK_{Est(2)}$ ~7.3. Crystd from *benzene and sublimed *in vacuo*.

3,4-Diaminopyridine [54-96-6] M 109.1, m 218-219°, pK_1^{20} 0.49, pK_2^{20} 9.14. Crystd from *benzene and stored under H₂ because it is deliquescent and absorbs CO₂.

***meso*-2,3-Diaminosuccinic acid** [23220-52-2] M 148.1, m 305-306°(dec, and sublimes), $pK_{Est(1)}$ ~3.6, $pK_{Est(2)}$ ~9.8. Crystd from water.

Diaminotoluene see **toluenediamine**.

3,5-Diamino-1,2,4-triazole (Guanazole) [1455-77-2] M 99.1, m 206° pK_1^{20} 4.43, pK_2^{20} 12.12. Crystd from water or EtOH.

2,5-Di-*tert*-amylhydroquinone [79-74-3] M 250.4, m 185.8-186.5°. Crystd under N₂ from boiling glacial acetic acid (7mL/g) plus boiling water (2.5mL/g) [Stolow and Bonaventura *J Am Chem Soc* 85 3636 1963].

Di-*n*-amyl phthalate [131-18-0] M 306.4, b 204-206°/11mm, d_4^{25} 1.023, n 1.489. Washed with aqueous Na₂CO₃, then distilled water. Dried with CaCl₂ and distd under reduced pressure. Stored in a vacuum desiccator over P₂O₅.

1,3-Diazaazulene (cycloheptimidazole) [275-94-5] M 130.1, m 120°. Recrystd repeatedly from de-aerated cyclohexane in the dark.

1,5-Diazabicyclo[4.3.0]non-5-ene (DBN, 2,3,4,,6,7,8-hexahydropyrrolo[1,2-*a*]-pyrimidine) [3001-72-7] M 124.2, b 96-98°/11mm, 100-102°/12mm, 118-121°/32mm, d_4^{20} 1.040, n_D^{20} 1.520, pK >13.0. Distd from BaO. It forms a *hydroiodide* by addn of 47% HI, dry and dissolve in MeCN, evaporate and repeat, recrystallise from EtOH then dry at 25°/1mm for 5h, then at 80°/0.03mm for 12h and store and dispense in a dry box, m 154-156° [Jaeger et al. *J Am Chem Soc* 101 717 1979]. The *methiodide* is recrystd from CHCl₃ + Et₂O, m 248-250°, and *hydrogen fumarate* has m 159-160° and is crystd from *iso*-

PrOH [Rokach et al. *J Med Chem* **22** 237 1979; Oediger et al. *Chem Ber* **99** 2012 1966; Reppe et al. *Justus Liebig's Ann Chem* **596** 210 1955].

1,4-Diazabicyclo[2.2.2]octane (DABCO, triethylenediamine TED) [280-57-9] **M 112.2, m 156-157°** (sealed tube), **pK₁²⁵ 2.97, pK₂²⁵ 8.82** Crystd from 95% EtOH, pet ether or MeOH/diethyl ether (1:1). Dried under vacuum over CaCl₂ and BaO. It can be sublimed *in vacuo*, and readily at room temperature. Also purified by removal of water during azeotropic distn of a *benzene soln. It was then recrystd twice from anhydrous diethyl ether under argon, and stored under argon [Blackstock et al. *J Org Chem* **52** 1451 1987].

1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU, 2,3,4,6,7,8,9,10-octahydropyrimidino[1,2-a]-azepine) [6674-22-2] **M 152.2, b 115°/11mm, d 1.023, n 1.522, pK_{Est} ~ >13.** Fractionally dist under vac. Also purified by chromatography on Kieselgel and eluting with CHCl₃/EtOH/25% aq NH₃ (15:5:2) and checked by IR and MS. [Oediger et al. *Chem Ber* **99** 2012 1962; *Angew Chem, Int Ed Engl* **6** 76 1967; Guggisberg et al. *Helv Chim Acta* **61** 1057 1978.]

1,8-Diazabiphenylene [259-84-7] **M 154.2, pK_{Est} ~4.4.** Recrystd from cyclohexane, then sublimed in a vacuum.

2,7-Diazabiphenylene [31857-42-8] **M 154.2, pK_{Est} ~4.5.** Recrystd from cyclohexane, then sublimed in a vacuum.

Diazoaminobenzene (1,3-diphenyltriazeno) [136-36-6] **M 197.2, m 99°.** Crystd from pet ether (b 60-80°), 60% MeOH/water or 50% aqueous EtOH (charcoal) containing a small amount of KOH. Also purified by chromatography on alumina/toluene and toluene-pet ether. Stored in the dark.

6-Diazo-5-oxo-L-norleucine [157-03-9] **M 171.2, m 145-155°(dec), [α]_D²⁰ +21° (c 5, EtOH), pK₁ 2.1, pK₂ 8.95.** Crystd from EtOH, aq EtOH or MeOH.

Dibenzalacetone [538-58-9] **M 234.3, m 112°.** Crystd from hot ethyl acetate (2.5mL/g) or EtOH.

Dibenz[*a,h*]anthracene [53-70-3] **M 278.4, m 266-267°.** The yellow-green colour (due to other pentacyclic impurities) has been removed by crystn from *benzene or by selective oxidation with lead tetraacetate in acetic acid [Moriconi et al. *J Am Chem Soc* **82** 3441 1960].

Dibenzo-18-crown-6 [14187-32-7] **M 360.4, m 163-164°.** Crystd from *benzene, *n*-heptane or toluene and dried under vacuum at room temperature for several days. [Szezygiel *J Phys Chem* **91** 1252 1987.]

Dibenzo-18-crown-8 [14174-09-5] **M 448.5, m 103-106°.** Recrystd from EtOH, and vacuum dried at 60° over P₂O₅ for 16hours. [Delville et al. *J Am Chem Soc* **109** 7293 1987.]

Dibenzofuran [132-64-9] **M 168.2, m 82.4°.** Dissolved in diethyl ether, then shaken with two portions of aqueous NaOH (2M), washed with water, separated and dried (MgSO₄). After evaporating the ether, dibenzofuran was crystd from aq 80% EtOH and dried under vacuum. [Cass et al. *J Chem Soc* 1406 1958.] High purity material was obtained by zone refining.

Dibenzopyran (xanthene) [92-83-1] **M 182.2, m 100.5°, b 310-312°.** See entry on p. 386.

Dibenzothiophene [132-65-0] **M 184.3, m 99°.** Purified by chromatography on alumina with pet ether, in a darkened room. Crystd from water or EtOH.

***trans*-1,2-Dibenzoyl ethylene** [959-28-4] **M 236.3, m 109-112°, 111°.** Recrystd from MeOH or EtOH as yellow needles [Koller et al. *Helv Chim Acta* **29** 512 1946]. The *dioxime* has m 210-211°dec from AcOH. [IR: Kuhn et al. *J Am Chem Soc* **72** 5058 1950; Yates *J Am Chem Soc* **74** 5375 1952; Erickson et al. *J Am Chem Soc* **73** 5301 1951.]

Dibenzoylmethane (1,3-diphenyl-1,3-propanedione) [120-46-7] **M 224.3, m 80°**. Crystd from pet ether or MeOH.

Di-*O*-benzoyl-(*R* and *S*)-tartaric acid (H₂O) [*R*-(+)- 17026-42-5; *S*-(-)- 2743-38-6] **M 376.3, m 88-89° (hydrate), 173° (anhydrous), [α]_D²⁰ (+) and (-) 136° (c 2, EtOH), [α]_D²⁰ (+) and (-) 117° (c 5, EtOH), pK_{Est(1)} ~2.9, pK_{Est(2)} ~4.2**. Crystd from water (18g from 400 mL boiling H₂O) and stir vigorously while cooling in order to obtain crystals; otherwise an oil will separate which solidifies on cooling. Dry in a vacuum desiccator over KOH-H₂SO₄ - yield 16.4g) as monohydrate, **m 88-89°**. It crystallises from xylene as the anhydrous acid, **m 173° (150-153°)**. It does not cryst from *C₆H₆, toluene, *C₆H₆-pet ether (oil), or CHCl₃-pet ether. [Butler and Cretcher *J Am Chem Soc* 55 2605 1933; *Tetrahedron* 41 2465 1085.]

2,3,6,7-Dibenzphenanthrene [222-93-5] **M 276.3, m 257°**. Crystd from xylene.

Dibenzyl amine [103-49-1] **M 197.3, m -26°, b 113-114°/0.1mm, 174-175°/6mm, 270°/250mm, 300° (partial dec), d₄²⁰ 1.027, n_D²⁰ 1.576, pK²⁵ 8.52**. Purified by distn in a vacuum. It causes burns to the skin. The *dihydrochloride* has **m 265-266°** after recrystn from MeOH-HCl, and the *tetraphenyl boronate* has **m 129-133°**. [Bradley and Maisey *J Chem Soc* 247 1954; Hall *J Phys Chem* 60 63 1956; Donetti and Bellora *J Org Chem* 37 3352 1972.]

Dibenzyl disulfide [150-60-7] **M 246.4, m 71-72°**. Crystd from EtOH.

Dibenzylethylenediamine (benzathine, DBED) [140-28-3] **M 240.4, m 26°, b 195°/4mm, d 1.02, n 1.563, pK_{Est(1)} ~ 5.9, pK_{Est(2)} ~ 8.9**. Dissolve in acid, extract with toluene, basify, extract with Et₂O, dry over solid KOH, evap and fractionate *in vacuo*. The *diacetate* cryst from H₂O by addn of EtOH, has **m 111° (sol in H₂O is ~25%)**. [Frost et al., *J Am Chem Soc* 71 3842 1949.]

1,3,4,6-Di-*O*-benzylidene-D-mannitol [28224-73-9] **M 358.4, m 192-195°, 193°, [α]_D²⁰ -11.9° (c 0.7, Me₂CO)**. Recryst from Et₂O in long fine needles. λ_{max} 256nm (ε 435) in 95% EtOH, R_F 0.21 (1:1 CCl₄-EtOAc) on TLC Silica Gel G. [Sinclair *Carbohydr Res* 12 150 1970; ORD, CD, NMR, IR, MS: Brecknell et al. *Aust J Chem* 29 1749 1976.]

Dibenzyl ketone (1,3-diphenyl-2-propanone) [102-04-5] **M 210.3, m 34.0°**. Fractionally crystd from its melt, then crystd from pet ether. Stored in the dark.

Dibenzyl malonate [15014-25-2] **M 284.3, b 188-190°/0.2mm, 193-196°/1mm, d₄²⁰ 1.158, n_D²⁰ 1.5452**. Dissolve in toluene, wash with aqueous NaHCO₃, H₂O, dry over MgSO₄, filter, evaporate and distil. [Ginsburg and Pappo *J Am Chem Soc* 75 1094 1953; Baker et al. *J Org Chem* 17 77 1952.]

Dibenzyl sulfide [538-74-9] **M 214.3, m 48.5°, 50°**. Crystd from EtOH/water (10:1), or repeatedly from Et₂O. Also chromatographed on Al₂O₃ (pentane as eluent), then recrystd from EtOH [Kice and Bowers *J Am Chem Soc* 84 2390 1962]. Vacuum dried at 30° over P₂O₅, fused under nitrogen and re-dried.

2,4-Dibromoaniline [615-57-6] **M 250.9, m 79-80°, pK²⁵ 1.87**. Crystd from aqueous EtOH.

9,10-Dibromoanthracene [523-27-3] **M 336.0, m 226°**. Recrystd from xylene and vacuum sublimed [Johnston et al. *J Am Chem Soc* 109 1291 1987].

***p*-Dibromobenzene** [106-37-6] **M 235.9, m 87.8°**. Steam distd, crystd from EtOH or MeOH and dried in the dark under vacuum. Purified by zone melting.

2,5-Dibromobenzoic acid [610-71-9] **M 279.9, m 157°, pK_{Est} ~1.5**. Crystd from water or EtOH.

4,4'-Dibromobiphenyl [92-86-4] **M 312.0, m 164°, b 355-360°/760mm**. Crystd from MeOH.

***trans*-1,4-Dibromobut-2-ene** [821-06-7] **M 213.9, m 54°, b 85°/10mm**. Crystd from ligroin.

α,α -Dibromodeoxybenzoin [15023-99-1] M 354.0, m 111.8-112.7°. Crystd from acetic acid.

Dibromodichloromethane [594-18-3] M 242.7, m 22°. Crystd repeatedly from its melt, after washing with aqueous $\text{Na}_2\text{S}_2\text{O}_3$ and drying with BaO.

1,3-Dibromo-5,5-dimethylhydantoin [77-48-5] M 285.9, m 190-192°dec, 190-193°dec. Recrystd from H_2O . Solubility in CCl_4 is 0.003 mol/L at 25° and 0.024 mol/L at 76.5°.

1,2-Dibromoethane [106-93-4] M 187.9, f 10.0°, b 29.1°/10mm, 131.7°/760mm, d 2.179, n^{15} 1.54160. Washed with conc HCl or H_2SO_4 , then water, aqueous NaHCO_3 or Na_2CO_3 , more water, and dried with CaCl_2 . Fractionally distd. Alternatively, kept in daylight with excess bromine for 2 hours, then extracted with aqueous Na_2SO_3 , washed with water, dried with CaCl_2 , filtered and distd. It can also be purified by fractional crystn by partial freezing. Stored in the dark.

4',5'-Dibromofluorescein [596-03-2] M 490.1, m 285°. Crystd from aqueous 30% EtOH.

5,7-Dibromo-8-hydroxyquinoline [521-74-4] M 303.0, m 196°, pK_1^{25} 5.84, pK_2^{25} 9.56. Crystd from acetone/EtOH. It can be sublimed.

Dibromomaleic acid [608-37-7] M 273.9, m 123.5°, 125°dec, pK_1^{25} 1.45, pK_2^{25} 4.62. It has been recrystd from Et_2O or $\text{Et}_2\text{O}\text{-CHCl}_3$. It is slightly soluble in H_2O , soluble also in AcOH but insoluble in $^*\text{C}_6\text{H}_6$ and CHCl_3 . [Salmony and Simonis *Chem Ber* 38 2583 1905; Ruggli *Helv Chim Acta* 3 566 1929.]

2,5-Dibromonitrobenzene [3460-18-2] M 280.9, m 84°. Crystd from acetone.

2,6-Dibromo-4-nitrophenol [99-28-5] M 280.9, m 143-144°, pK^{25} 3.39. Crystd from aq EtOH.

2,4-Dibromophenol [615-58-7] M 251.9, m 37°, 41-42°, b 154°/10mm, 239°/atm, pK^{25} 7.79. Crystd from CHCl_3 at -40°.

2,6-Dibromophenol [608-33-3] M 251.9, m 56-57°, b 138°/10mm, 255-256°/740mm, pK^{25} 6.67. Vacuum distd (at 18mm), then crystd from cold CHCl_3 or from EtOH/water.

1,3-Dibromopropane [109-64-8] M 201.9, f -34.4°, b 63-63.5°/26mm, 76-77°/40mm, 90°/80mm, 165°/atm, d 1.977, n 1.522. Washed with dilute aqueous Na_2CO_3 , then water. Dried and fractionally distd under reduced pressure.

2,6-Dibromopyridine [626-05-1] M 236.9, m 117-119°, 118.5-119°, b 249°/757.5mm, $\text{pK}_{\text{Est}} < 0$. Purified by steam distn then twice recrystd from EtOH. Does not form an HgCl_2 salt. [den Hertog and Wibaut *Rec Trav Chim Pays Bas* 51 381 1932.]

meso-2,3-Dibromosuccinic acid [526-78-3] M 275.9, m 288-290° (sealed tube, dec), pK_1^{20} 1.56, pK_2^{20} 2.71. Crystd from distilled water, keeping the temperature below 70°.

1,2-Dibromotetrafluoroethane [124-73-2] M 259.8, b 47.3°/760mm. Washed with water, then with weak alkali. Dried with CaCl_2 or H_2SO_4 and distd. [Locke et al. *J Am Chem Soc* 56 1726 1934.] Also purified by gas chromatography on a silicone DC-200 column.

α,α' -Dibromo-*o*-xylene [91-13-4] M 264.0, m 95°, b 129-130°/4.5mm. Crystd from CHCl_3

α,α' -Dibromo-*m*-xylene [626-15-3] M 264.0, m 77°, b 156-160°/12mm. Crystd from acetone.

α,α' -Dibromo-*p*-xylene [623-24-5] M 264.0, m 145-147°, b 155-158°/12-15mm, 245°/760mm. Crystd from $^*\text{benzene}$ or chloroform.

Di-*n*-butylamine [111-92-2] **M 129.3, b 159°, n 1.41766, d 0.761, pK²⁵ 11.25.** Dried with LiAlH₄, CaH₂ or KOH pellets, filtered and distd from BaO or CaH₂.

α-Dibutylamino-α-(*p*-methoxyphenyl)acetamide (Ambucetamide) [519-88-0] **M 292.4, m 134°.** Crystd from EtOH containing 10% diethyl ether.

2,5-Di-*tert*-butyl aniline [21860-03-7] **M 205.4, m 103-104°, 103-106°, pK²⁵ 3.34 (50% aq MeOH), 3.58 (90% aq MeOH).** Recrystd from EtOH in fine needles after steam distn. It has a pK^{a25} of 3.58 (50% aq EtOH) and 3.34 (90% aq MeOH). The *tosylate* has **m 164°** (from AcOH). [Bell and Wilson *J Chem Soc* 2340 1956; Carpenter et al. *J Org Chem* 16 586 1951; Bartlett et al. *J Am Chem Soc* 76 2349 1954.]

Di-*tert*-butylazodicarboxylate [870-50-8] **M 230.3, m 90-92°.** Cryst from ligroin. Best purified by covering the dry solid (22g) with pet ether (b 30-60°, 35-40 mL) and adding ligroin (b 60-90°) to the boiling soln until the solid dissolves. On cooling, large lemon yellow crystals of the ester separate (~20g), **m 90.7-92°.** Evapn of the filtrate gives a further crop of crystals [Carpino and Crowley *Org Synth* 44 18 1964].

***p*-Di-*tert*-butylbenzene** [1012-72-2] **M 190.3, m 80°.** Crystd from diethyl ether, EtOH and dried under vacuum over P₂O₅ at 55°. [Tanner et al. *J Org Chem* 52 2142 1987.]

2,6-Di-*tert*-butyl-1,4-benzoquinone [719-22-2] **M 220.3, m 66-67°.** It can be recrystd from MeOH and sublimed in a vacuum.

3,5-Di-*tert*-butyl-*o*-benzoquinone [3383-21-9] **M 220.3, m 112-114°, 113-114°.** It can be recrystd from MeOH or pet ether, and forms fine red plates or rhombs. [Flaig et al. *Justus Liebigs Ann Chem* 597 196 1955; IR: Ley and Müller *Chem Ber* 89 1402 1956.]

3,5-Di-*tert*-butyl catechol [1020-31-1] **M 222.3, m 99°, 99-100°, pK_{Est(1)} ~11.0, pK_{Est(2)} ~13.1.** Recrystd from pet ether. [Ley and Müller *Chem Ber* 89 1402 1956; UV Flaig et al. *Z Naturforschung* 10b 668 1955.] Also crystd three times from pentane [Funabiki et al. *J Am Chem Soc* 108 2921 1986].

Dibutylcarbitol [di(ethyleneglycol)dibutyl ether] [112-73-2] **M 218.3, b 125-130°/0.1mm, d 0.883, n 1.424.** Freed from peroxides by slow passage through a column of activated alumina. The eluate was shaken with Na₂CO₃ (to remove any remaining acidic impurities), washed with water, and stored with CaCl₂ in a dark bottle [Tuck *J Chem Soc* 3202 1957].

2,6-Di-*tert*-butyl-*p*-cresol (2,6-di-*tert*-butyl-4-methylphenol, butylatedhydroxytoluene, BHT) [128-37-0] **M 230.4, m 71.5°, pK²⁵ 12.23.** Dissolved in *n*-hexane at room temperature, then cooled with rapid stirring, to -60°. The ppte was separated, redissolved in hexane, and the process was repeated until the mother liquor was no longer coloured. The final product was stored under N₂ at 0° [Blanchard *J Am Chem Soc* 82 2014 1960]. Also crystd from EtOH, MeOH, *benzene, *n*-hexane, methylcyclohexane or pet ether (b 60-80°), and dried under vacuum.

Di-*tert*-butyl dicarbonate (di-*tert*-butyl pyrocarbonate) [24424-99-5] **M 218.3, m 23° (21-22°), b 55-56°/0.15mm, 62-65°/0.4mm, d 0.950, n 1.409.** Melt by heating at ~35°, and distil in vac. If IR and NMR (ν 1810m 1765 cm⁻¹, δ in CCl₄ 1.50 singlet) suggest very impure then wash with equal vol of H₂O containing citric acid to make the aqueous layer slightly acidic, collect the organic layer and dry over anhyd MgSO₄ and distil in vac. [Pope et al. *Org Synth* 57 45 1977.] **FLAMMABLE.**

2,6-Di-*tert*-butyl-4-dimethylaminomethylphenol [88-27-7] **M 263.4, m 93-94°, b 172°/30mm, pK_{Est} ~12.0.** Crystd from *n*-hexane.

Di-*tert*-butyldiperphthalate [2155-71-7] **M 310.3, m dec explosively, CARE.** Crystd from diethyl ether. Dried over H₂SO₄.

2,6-Di-*tert*-butyl-4-ethylphenol [4130-42-1] M 234.4, m 42-44°, pK_{Est} ~12.3. Crystd from aqueous EtOH or *n*-hexane.

***N,N*-Dibutyl formamide** [761-65-9] M 157.3, b 63°/0.1mm, 118-120°/15mm, 244-246°/760mm, d₄²⁰ 0.878, n_D²⁰ 1.445. Purified by fractn distn [Mandel and Hill *J Am Chem Soc* 76 3981 1954].

2,5-Di-*tert*-butylhydroquinone [88-58-4] M 222.3, m 222-223°. Crystd from *C₆H₆ or AcOH.

2,4-Di-*tert*-butyl-4-isopropylphenol [5427-03-2] M 248.4, m 39-41°, pK_{Est} ~12.3. Crystd from *n*-hexane or aq EtOH.

2,6-Di-*tert*-butyl-4-methylpyridine [38222-83-2] M 205.4, m 31-32°, 33-36°, b 148-153°/95mm, 223°/760mm, n₄²⁰ 1.476, pK_{Est} ~5.7. Possible impurity is 2,6-di-*tert*-butyl-4-neopentylpyridine. Attempts to remove coloured impurities directly by distn, acid-base extraction or treatment with activated charcoal were unsuccessful. Pure material can be obtained by dissolving 0.3mole of the alkylpyridine in pentane (150mL) and introducing it at the top of a water jacketed chromatographic column (40 x 4.5cm) the cooling is necessary because the base in pentane reacts exothermically with alumina) containing activated and acidic alumina (300g). The column is eluted with pentane using a 1L constant pressure funnel fitted at the top of the column to provide slight press. All the pyridine is obtained in the first two litres of eluent (the progress of elution is monitored by spotting a fluorescent TLC plate and examining under short wave UV light - a dark blue spot is evidence for the presence of the alkylpyridine). Elution is complete in 1h. Pentane is removed on a rotovap with 90-93% recovery yielding a liquid which solidifies on cooling, m 31-32°, and the base can be distilled. The *HPtCl₆* salt has m 213-314° (dec), and the *CF₃SO₃H* salt has m 202.5-203.5° (from CH₂Cl₂). [*Org Synth* 60 34 1981.]

Di-*tert*-butyl peroxide (*tert*-butyl peroxide) [110-05-4] M 146.2, d 0.794, n 1.389. Washed with aqueous AgNO₃ to remove olefinic impurities, water and dried (MgSO₄). Freed from *tert*-butyl hydroperoxide by passage through an alumina column [Jackson et al. *J Am Chem Soc* 107 208 1985], and if necessary two high vacuum distns from room temp to a liquid-air trap [Offenbach and Tobolsky *J Am Chem Soc* 79 278 1957]. *The necessary protection from EXPLOSION should be used.*

2,6-Di-*tert*-butylphenol [128-39-2] M 206.3, m 37-38°, pK²⁵ 11.70. Crystd from aqueous EtOH or *n*-hexane.

Dibutyl phthalate [84-74-2] M 278.4, b 206°/20mm, 340°/760mm, d 1.4929, d⁵ 1.0426, n²⁵ 1.490. Washed with dilute NaOH (to remove any butyl hydrogen phthalate), aqueous NaHCO₃ (charcoal), then distd water. Dried with CaCl₂, distd under vacuum, and stored in a desiccator over P₂O₅. (See also p. 151.)

2,6-Di-*tert*-butylpyridine [585-48-8] M 191.3, b 100-101°/23mm, d 0.852, n 1.474, pK²⁵ 5.02. Redistd from KOH pellets.

§ Polystyrene supported version is commercially available.

Di-*n*-butyl sulfide [544-40-1] M 146.3, α-form b 182°, β-form 190-230°(dec). Washed with aq 5% NaOH, then water. Dried with CaCl₂ and distd from sodium.

Di-*n*-butyl sulfone [598-04-9] M 162.3, m 43.5°. Purified by zone melting.

***N,N'*-Di-*tert*-butylthiourea** [4041-95-6] M 188.3, m 174-175° (evac capillary). Recrystd from H₂O [Bortnick et al. *J Am Chem Soc* 78 4358 1956].

3,5-Dicarbethoxy-1,4-dihydrocollidine [632-93-9] M 267.3, m 131-132°. Crystd from hot EtOH/water.

Dichloramine-T (*N,N*-dichloro-*p*-toluenesulfonamide) [473-34-7] **M 240.1, m 83°**. Crystd from pet ether (b 60-80°) or CHCl₃/pet ether. Dried in air. (see also chloramine-T in Chapter 5).

Dichloroacetic acid [79-43-6] **M 128.9, m 13.5°, b 95.0-95.5°/17-18mm, d 1.563, n 1.466, pK²⁵ 1.35**. Crystd from *benzene or pet ether. Dried with MgSO₄ and fractionally distd. [Bernasconi et al. *J Am Chem Soc* **107** 3612 1985.]

sym-Dichloroacetone (1,3-dichloropropan-2-one) [534-07-6] **M 127.0, m 41-43°, 45°, b 86-88°/12mm, 75-77°/22mm, 172-172.5°/atm, 170-175° /atm, d 1.383**. Crystd from CCl₄, CHCl₃ and *benzene. Distd under vacuum. [Conant and Quayle *Org Synth Coll Vol* 211 1941; Hall and Sirel *J Am Chem Soc* **74** 836 1952]. It is dimorphic [Daasch and Kagarise *J Am Chem Soc* **77** 6156 1955]. The *oxime* has **m 130-131°, b 106°/25mm** [*Arzneimittel-Forsch* **8** 638 1958].

Dichloroacetonitrile [3018-12-0] **M 110.0, b 110-112°, d 1.369, n 1.440**. Purified by distn and by gas chromatography. **FLAMMABLE**.

2,4-Dichloroaniline [554-00-7] **M 162.0, m 63°, pK²⁵ 2.02**. Crystd from EtOH/water. Also crystd from EtOH and dried *in vacuo* for 6h at 40° [Moore et al. *J Am Chem Soc* **108** 2257 1986; Edidin et al. *J Am Chem Soc* **109** 3945 1987].

3,4-Dichloroaniline [95-76-1] **M 162.0, m 71.5°, pK²⁵ 2.97**. Crystd from MeOH.

9,10-Dichloroanthracene [605-49-1] **M 247.1, m 214-215°**. Purified by crystn from MeOH or EtOH, followed by sublimation under reduced pressure. [Masnori and Kochi *J Am Chem Soc* **107** 7880 1985.]

2,4-Dichlorobenzaldehyde [874-42-0] **M 175.0, m 72°**. Crystd from EtOH or ligroin.

2,6-Dichlorobenzaldehyde [83-38-5] **M 175.0, m 70.5-71.5°**. Crystd from EtOH/water or pet ether (b 30-60°).

***o*-Dichlorobenzene** [95-50-1] **M 147.0, b 81-82°/31-32mm, 180.5°/760mm, d 1.306, n 1.551, n²⁵ 1.549**. Contaminants may include the *p*-isomer and trichlorobenzene [Suslick et al. *J Am Chem Soc* **106** 4522 1984]. It was shaken with conc or fuming H₂SO₄, washed with water, dried with CaCl₂, and distd from CaH₂ or sodium in a glass-packed column. Low conductivity material (*ca* 10⁻¹⁰ mhos) has been obtained by refluxing with P₂O₅, fractionally distilled and passed through a column packed with silica gel or activated alumina: it was stored in a dry-box under N₂ or with activated alumina.

***m*-Dichlorobenzene** [541-73-1] **M 147.0, b 173.0°, d 1.289, n 1.54586, n²⁵ 1.54337**. Washed with aqueous 10% NaOH, then with water until neutral, dried and distd. Conductivity material (*ca* 10⁻¹⁰ mhos) has been prepared by refluxing over P₂O₅ for 8h, then fractionally distilling, and storing with activated alumina. *m*-Dichlorobenzene dissolves rubber stoppers.

***p*-Dichlorobenzene** [106-46-7] **M 147.0, m 53.0°, b 174.1°, d 1.241, n⁶⁰ 1.52849**. *o*-Dichlorobenzene is a common impurity. Has been purified by steam distn, crystn from EtOH or boiling MeOH, air-dried and dried in the dark under vacuum. Also purified by zone refining.

2,2'-Dichlorobenzidine [84-68-4] **M 253.1, m 165°, pK_{Est(1)} ~3.0, pK_{Est(2)} ~4.0**. Crystd from EtOH.

3,3'-Dichlorobenzidine [91-94-1] **M 253.1, m 132-133°, pK_{Est(1)} ~4.8, pK_{Est(2)} ~5.7**. Crystd from EtOH or *benzene. **CARCINOGEN**.

2,4-Dichlorobenzoic acid [50-84-0] **M 191.0, m 163-164°, pK²⁵ 2.68**. Crystd from aqueous EtOH (charcoal), then *benzene (charcoal). It can also be recrystd from water.

2,5-Dichlorobenzoic acid [50-79-3] M 191.0, m 154°, b 301°/760mm, pK^{25} 2.47. Crystd from water.

2,6-Dichlorobenzoic acid [50-30-6] M 191.0, m 141-142°, pK^{25} 1.59. Crystd from EtOH and sublimed *in vacuo*.

3,4-Dichlorobenzoic acid [51-44-5] M 191.0, m 206-207°, pK^{25} 3.64. Crystd from aqueous EtOH (charcoal) or acetic acid.

3,5-Dichlorobenzoic acid [51-36-5] M 191.0, m 188°, pK^{25} 3.54. Crystd from EtOH and sublimed in a vacuum.

2,6-Dichlorobenzonitrile [1194-65-6] M 172.0, m 145°. Crystd from acetone.

4,4'-Dichlorobenzophenone [90-98-2] M 251.1, m 145-146°. Recrystd from EtOH [Wagner et al. *J Am Chem Soc* 108 7727 1986].

2,5-Dichloro-1,4-benzoquinone [615-93-0] M 177.0, m 161-162°. Recrystd twice from 95% EtOH as yellow needles [Beck et al. *J Am Chem Soc* 108 4018 1986].

2,6-Dichloro-1,4-benzoquinone [697-91-6] M 177.0, m 122-124°. Recrystd from pet ether (b 60-70°) [Carlson and Miller *J Am Chem Soc* 107 479 1985].

2,6-Dichlorobenzoyl chloride [4659-45-4] M 209.5, m 15-17, b 122-124°/15mm, d 1.464. Reflux for 2h with excess of acetyl chloride (3 vols), distil off AcCl followed by the benzoyl chloride. Store away from moisture. It is an **IRRITANT**.

3,4-Dichlorobenzyl alcohol [1805-32-9] M 177.0, m 38-39°. Crystd from water.

2,3-Dichloro-1,3-butadiene [1653-19-6] M 123.0, b 41-43°/85mm, 98°/760mm. Crystd from pentane to constant melting point about -40°. A mixture of *meso* and *d,l* forms was separated by gas chromatography on an 8m stainless steel column (8mm i.d.) with 20% DEGS (diethyleneglycolsilyl chloride) on Chromosorb W (60-80 mesh) at 60° and 80mL He/min. [Su and Ache *J Phys Chem* 80 659 1976.]

(+) and (-) **(8,8-Dichlorocamphorylsulfonyl)oxaziridine** [127184-05-8] M 298.2, m 178-180°, 183-186°, $[\alpha]_D^{20}$ (+) and (-) 88.3° (c 1.3, $CHCl_3$), (+) and (-) 91° (c 5, $CHCl_3$). Recrystd from EtOH [Davis and Weismiller *J Org Chem* 55 3715 1990].

cis-3,4-Dichlorocyclobutene [2957-95-1] M 123.0, b 70-71°/55mm, 74-76°/55mm, d_4^{20} 1.297, n_D^{20} 1.499. Distd at 55mm through a 36-in platinum spinning band column, a fore-run b 58-62°/55mm is mainly 1,4-dichlorobutadiene. When the temperature reaches 70° the reflux ratio is reduced to 10:1 and the product is collected quickly. It is usually necessary to apply heat frequently with a sun lamp to prevent any dichlorobutadiene from clogging the exit in the early part of the distn [Pettit and Henery *Org Synth* 50 36 1970].

2,3-Dichloro-5,6-dicyano-p-benzoquinone (DDQ) [84-58-2] M 227.0, m 203° (dec). Crystd from $CHCl_3$, $CHCl_3$ /*benzene (4:1), or *benzene and stored at 0°. [Pataki and Harvey *J Org Chem* 52 2226 1987.]

β,β' -Dichlorodiethyl ether [111-44-4] M 143.0, b 79-80°/20mm, 176-177.0°/743mm, n 1.457, d 1.219. See bis-(β -dichloroethyl)ether on p. 134.

1,2-Dichloro-1,2-difluoroethane [431-06-1] M 134.9, b 59°, n 1.376. Purified by fract dist [Hazeldine *J Chem Soc* 4258 1952]. For purification of diastereoisomeric mixture, with resolution into *meso* and *rac* forms, see Machulla and Stocklin [*J Phys Chem* 78 658 1974].

Dichlorodifluoromethane (Freon 12) [75-71-8] M 120.9, m -158°, b -29.8°/atm, 42.5°/10atm. Passage through saturated aqueous KOH then conc H₂SO₄, and a tower packed with activated copper on Kiesselguhr at 200° removed CO₂ and O₂. A trap cooled to -29° removed a trace of high boiling material. It is a non-flammable propellant.

1,3-Dichloro-5,5'-dimethylhydantoin [118-52-5] M 197.0, m 132-134°, 136°. Purified by dissolving in conc H₂SO₄ and diluting with ice H₂O, dry and recrystd from CHCl₃. It sublimes at 100° in a vacuum. Exhibits time dependent hydrolysis at pH 9. [Pettersen and Grzeskowiak *J Org Chem* **24** 1414 1959.]

4,5-Dichloro-3H-1,2-dithiol-3-one [1192-52-5] M 187.1, m 52-56°, 61°, b 87°/0.5mm, 125°/11mm. Distd *in vacuo* and then recrystd from pet ether. IR: ν 1650 cm⁻¹ [Boberg *Justus Liebigs Ann Chem* **693** 212 1966].

1,1-Dichloroethane (ethylidene dichloride) [75-34-3] M 99.0, b 57.3°, d¹⁵ 1.18350, d 1.177, n¹⁵ 1.41975. Shaken with conc H₂SO₄ or aqueous KMnO₄, then washed with water, saturated aqueous NaHCO₃, again with water, dried with K₂CO₃ and distd from CaH₂ or CaSO₄. Stored over silica gel.

1,2-Dichloroethane [107-06-2] M 99.0, b 83.4°, d 1.256, n¹⁵ 1.44759. Usually prepared by chlorinating ethylene, so that likely impurities include higher chloro derivatives and other chloro compounds depending on the impurities originally present in the ethylene. It forms azeotropes with water, MeOH, EtOH, trichloroethylene, CCl₄ and isopropanol. Its azeotrope with water (containing 8.9% water, and b 77°) can be used to remove gross amounts of water prior to final drying. As a preliminary purification step, it can be steam distd, and the lower layer was treated as below.

Shaken with conc H₂SO₄ (to remove alcohol added as an oxidation inhibitor), washed with water, then dilute KOH or aqueous Na₂CO₃ and again with water. After an initial drying with CaCl₂, MgSO₄ or by distn, it is refluxed with P₂O₅, CaSO₄ or CaH₂ and fractionally distd. Carbonyl-containing impurities can be removed as described for chloroform.

1,2-Dichloroethylene [cis + trans 540-59-0] M 96.9, b 60° (cis), d 1.284, b 48° (trans), d 1.257. Shaken successively with conc H₂SO₄, water, aqueous NaHCO₃ and water. Dried with MgSO₄ and distn separated the *cis*- and *trans*-isomers.

***cis*-1,2-Dichloroethylene [156-59-2] M 96.9, b 60.4°, d 1.2830, n¹⁵ 1.44903, n 1.4495.** Purified by careful fractional distn, followed by passage through neutral activated alumina. Also by shaking with mercury, drying with K₂CO₃ and distn. from CaSO₄.

***trans*-1,2-Dichloroethylene [156-60-5] M 96.9, b 47.7°, n¹⁵ 1.45189, n 1.4462, d 1.2551.** Dried with MgSO₄, and fractionally distd under CO₂. Fractional crystn at low temperatures has also been used.

5,7-Dichloro-8-hydroxyquinoline [773-76-2] M 214.1, m 180-181°, pK₁ 1.89, pK₂ 7.62. Crystd from acetone/EtOH.

2,3-Dichloromaleic anhydride [1122-17-4] M 167.0, m 105-115°, 120°, 121-121.5°. Purified by sublimation *in vacuo* [Katakis et al. *J Chem Soc, Dalton Trans* 1491 1986]. It has also been purified by Soxhlet extraction with hexane, recrystd from CHCl₃ and sublimed [MS, Relles *J Org Chem* **37** 3630 1972].

Dichloromethane (methylene dichloride) [75-09-2] M 84.9, b 40.0°, d 1.325, n 1.42456, n²⁵ 1.4201. Shaken with portions of conc H₂SO₄ until the acid layer remained colourless, then washed with water, aqueous 5% Na₂CO₃, NaHCO₃ or NaOH, then water again. Pre-dried with CaCl₂, and distd from CaSO₄, CaH₂ or P₂O₅. Stored away from bright light in a brown bottle with Linde type 4A molecular sieves, in an atmosphere of dry N₂. Other purification steps include washing with aq Na₂S₂O₃, passage through a column of silica gel, and removal of carbonyl-containing impurities as described under **Chloroform**. It has also been purified by treatment with basic alumina, distd, and stored over molecular sieves under nitrogen [Puchot et al. *J Am Chem Soc* **108** 2353 1986].

Dichloromethane from Japanese sources contained MeOH as stabiliser which is not removed by distn. It can, however, be removed by standing over activated 3A Molecular Sieves (note that 4A Sieves cause the development of pressure in bottles), passed through activated Al₂O₃ and distd [Gao et al. *J Am Chem Soc* **109** 5771 1987]. It has been fractionated through a platinum spinning band column, degassed, and distd onto degassed molecular sieves, Linde 4A, heated under high vacuum at over 450° until the pressure readings reached the low values of 10⁻⁶ mm — ~1-2h [Mohammad and Kosower *J Am Chem Soc* **93** 2713 1971].

Rapid purification: Reflux over CaH₂ (5% w/v) and distil. Store over 4A molecular sieves.

3,9-Dichloro-7-methoxyacridine [86-38-4] M 278.1, m 160-161°. Crystd from *benzene.

5,7-Dichloro-2-methyl-8-hydroxyquinoline (5,7-dichloro-8-hydroxyquinaldine) [72-80-0] M 228.1, m 114-115°, pK_{Est(1)} ~2.0, pK_{Est(2)} ~8.4. Crystd from EtOH.

2,4-Dichloro-6-methylphenol [1570-65-6] M 177.0, m 55°, b 129-132°/40mm, pK²⁰ 8.14. Crystd from water.

2,4-Dichloro-1-naphthol [2050-76-2] M 213.1, m 106-107°, pK_{Est} ~7.7. Crystd from MeOH.

2,3-Dichloro-1,4-naphthoquinone [117-80-6] M 227.1, m 193°. Crystd from EtOH.

2,5-Dichloro-4-nitroaniline [6627-34-5] M 207.0, m 157-158°, pK²⁵ -1.74 (aq H₂SO₄). Crystd from EtOH, then sublimed.

2,6-Dichloro-4-nitroaniline [99-30-9] M 207.0, m 193°. Crystd from aq EtOH or *benzene/EtOH.

2,5-Dichloro-1-nitrobenzene [89-61-2] M 192.0, m 56°. Crystd from absolute EtOH.

3,4-Dichloro-1-nitrobenzene [99-54-7] M 192.0, m 43°. Crystd from absolute EtOH.

2,4-Dichloro-6-nitrophenol [609-89-2] M 208.0, m 122-123°, pK_{Est} ~5.0. Crystd from AcOH.

2,6-Dichloro-4-nitrophenol [618-00-4] M 208.0, m 125°, pK²⁵ 3.55. Crystd from EtOH and dried *in vacuo* over anhydrous MgSO₄.

4,6-Dichloro-5-nitropyrimidine [4316-93-2] M 194.0, m 100-103°, 101-102°, pK_{Est} <0. If too impure then dissolve in Et₂O, wash with H₂O, dry over MgSO₄, evaporate to dryness and recrystallise from pet ether (b 85-105°) as a light tan solid. It is sol in *ca* 8 parts of MeOH [Boon et al, *J Chem Soc* 96 1951; Montgomery et al. in *Synthetic Procedures in Nucleic Acid Chemistry* (Zorbach and Tipson eds) Wiley & Sons, NY, p76 1968].

Dichlorophen [2,2'-methylenebis(4-chlorophenol)] [97-23-4] M 269.1, b 177-178°, pK_{Est} ~9.7. Crystd from toluene.

2,3-Dichlorophenol [576-24-9] M 163.0, m 57°, pK²⁵ 7.70. Crystd from ether.

2,4-Dichlorophenol [120-83-2] M 163.0, m 42-43°, pK²⁵ 7.89. Crystd from pet ether (b 30-40°). Purified by repeated zone melting, using a P₂O₅ guard tube to exclude moisture. Very *hygroscopic* when dry.

2,5-Dichlorophenol [583-78-8] M 163.0, m 58°, b 211°/744mm, pK²⁵ 7.51. Crystd from ligroin and sublimed.

3,4-Dichlorophenol [95-77-2] M 163.0, m 68°, b 253.5°/767mm, pK²⁵ 8.58. Crystd from pet ether/*benzene mixture.

3,5-Dichlorophenol [591-35-5] M 163.0, m 68°, b 122-124°/8mm, 233-234°/760mm, pK²⁵ 8.81. Crystd from pet ether/*benzene mixture.

2,4-Dichlorophenoxyacetic acid (2,4-D) [94-75-7] M 221.0, m 146°, pK²⁵ 2.90. Crystd from MeOH. TOXIC.

α-(2,4-Dichlorophenoxy)propionic acid (2,4-DP, Dichloroprop) [120-36-5] M 235.1, m 117°, pK²⁰ 2.86, Crystd from MeOH. TOXIC.

2,4-Dichlorophenylacetic acid [19719-28-9] M 205.0, m 131°, 132-133°, pK_{Est} ~4.0. Crystd from aqueous EtOH.

2,6-Dichlorophenylacetic acid [6575-24-2] M 205.0, m 157-158°, pK_{Est} ~3.8. Crystd from aqueous EtOH.

3-(3,4-Dichlorophenyl)-1,1-dimethyl urea (Diuron) [330-54-1] M 233.1. Crystd four times from 95% EtOH [Beck et al. *J Am Chem Soc* 108 4018 1986].

4,5-Dichloro-*o*-phenylenediamine [5348-42-5] M 177.1, m 162°, 162-163°, pK_{Est(1)} ~-1.0, pK_{Est(2)} ~2.9. Recrystd from hexane, *C₆H₆, pet ether or H₂O (Na₂SO₄) and sublimed at 150°/15mm.

4,5-Dichlorophthalic acid [56962-08-4] M 235.0, m 200° (dec to anhydride), pK_{Est(1)} 2.2, pK_{Est(2)} ~4.7. Crystd from water. Can be purified by converting to the anhydride, reacting with boiling EtOH to form the *monoethyl ester* (m 133-134°) and hydrolysing back to the diacid

3,6-Dichlorophthalic anhydride [4466-59-5] M 189-191°, 191-191.5°, b 339°. Boil in xylene (allowing any vapours which would contain H₂O to be removed, e.g. Dean and Stark trap), which causes the acid to dehydrate to the anhydride and cool. Recryst from xylene [Villiger *Chem Ber* 42 3539 1909; Fedorow *Izv Akad Nauk SSSR Otd Khim Nauk* 397 1948, *Chem Abstr* 1585 1948].

1,2-Dichloropropane [78-87-5] M 113°, b 95.9-96.2°, d 1.158, n 1.439. Distd from CaH₂.

2,2-Dichloropropane [594-20-7] M 113.0, b 69.3°, d 1.090, n 1.415. Washed with aqueous Na₂CO₃ soln, then distilled water, dried over CaCl₂ and fractionally distd.

2,6-Dichloropurine [5451-40-1] M 189.0, m 180-181.5°, 181°, 185-195°(dec), 188-189°, pK₁²⁰ 1.16 (aq H₂SO₄), pK₂²⁰ 7.06. It can be recrystd from 150 parts of boiling H₂O and dried at 100° to constant weight. Soluble in EtOAc. The HgCl₂ salt separates from EtOH soln. UV: λ_{max} 275nm (ε 8.9K) at pH 1; and 280nm (ε 8.5K) at pH 11 [Elion and Hitchings *J Am Chem Soc* 78 3508 1956; Schaeffer and Thomas *J Am Chem Soc* 80 3738 1958; Beaman and Robins *J Appl Chem (London)* 12 432 1962; Montgomery *J Am Chem Soc* 78 1928 1956].

2,6-Dichloropyridine [2402-78-0] M 148.0, m 87-88°, pK -2.86 (aq H₂SO₄). Crystd from EtOH.

3,5-Dichloropyridine [2457-47-8] M 148.0, m 64-65°, pK²⁵ 0.67. Crystd from EtOH.

4,7-Dichloroquinoline [86-98-6] M 198.1, m 86.4-87.4°, b 148°/10mm, pK²⁵ 2.80. Crystd from MeOH or 95% EtOH.

2,3-Dichloroquinoxaline [2213-63-0] M 199.0, m 152-153°, 152-154°, pK_{Est} <0. Recrystd from *C₆H₆ and dried in a vacuum [Cheeseman *J Chem Soc* 1804 1955].

2,6-Dichlorostyrene [28469-92-3] M 173.0, b 72-73°/2mm, d 1.4045, n 1.5798. Purified by fractional crystn from the melt and by distn.

2,4-Dichlorotoluene [95-73-8] M 161.1, m -13.5°, b 61-62°/3mm, d 1.250, n 1.5513. Recrystd from EtOH at low temperature or fractionally distd.

2,6-Dichlorotoluene [118-69-4] M 161.1, b 199-200°/760mm, d 1.254, n 1.548. Fractionally distd and collecting the middle fraction.

3,4-Dichlorotoluene [95-75-0] M 161.1, m -16°, b 205°/760mm, d 1.2541, n 1.549. Recrystd from EtOH at very temperature or fractionally distd.

α,α'-Dichloro-*p*-xylene [623-25-6] M 175.1, m 100°. Crystd from *benzene and dried under vacuum.

Dicinnamalacetone (1,9-diphenyl-1,3,6,8-nonatetraen-5-one) [622-21-9] M 314.4, m 146°. Crystd from *benzene/isooctane (1:1).

Dicumyl peroxide [80-43-3] M 270.4, m 39-40°. Crystd from 95% EtOH (charcoal). Stored at 0°. **Potentially EXPLOSIVE.**

9,10-Dicyanoanthracene [1217-45-4] M 228.3, m 340°. Recrystd twice from pyridine [Mattes and Farid *J Am Chem Soc* 108 7356 1986].

1,2-Dicyanobenzene [91-15-6] M 128.1, m 141°. (See phthalonitrile on p. 334.)

1,4-Dicyanobenzene [623-26-7] M 128.1, m 222°. Crystd from EtOH.

1,4-Dicyanonaphthalene [3029-30-9] M 178.2, m 206°. Purified by crystn and sublimed *in vacuo*.

1,3-Dicyclohexylcarbodiimide (DCC) [538-75-0] M 206.3, m 34-35°, b 95-97°/0.2mm, 120-121°/0.6mm, 155°/11mm. It is sampled as a liquid after melting in warm H₂O. It is sensitive to air and *it is a potent skin irritant*. It can be distd in a vacuum and stored in a tightly stoppered flask in a freezer. It is very soluble in CH₂Cl₂ and pyridine where the reaction product with H₂O, after condensation, is dicyclohexyl urea which is insoluble and can be removed by filtration. Alternatively dissolve in CH₂Cl₂ add powdered anhyd MgSO₄ shake 4h, filter, evaporate and distil at 0.6 mm press and oil bath temperature 145°. [*Biochem Prep* 10, 122 1963; *Justus Liebigs Ann Chem* 571 83 1951; *Justus Liebigs Ann Chem* 612 11 1958.]

***cis*-Dicyclohexyl-18-crown-6** [16069-36-6] M 372.5, m 47-50°. Purified by chromatography on neutral alumina and eluting with an ether/hexane mixture [see *Inorg Chem* 14 3132 1975]. Dissolved in ether at *ca* 40°, and spectroscopic grade MeCN was added to the soln which was then chilled. The crown ether pptd and was filtered off. It was dried *in vacuo* at room temperature [Wallace *J Phys Chem* 89 1357 1985]. **SKIN IRRITANT.**

Di-*n*-decylamine [1120-49-6] M 297.6, m 34°. b 153°/1mm, 359°/760mm, pK_{Est} ~11.0. Dissolved in *benzene and pptd as its bisulfate by shaking with 4M H₂SO₄. Filtered, washed with *benzene, separating by centrifugation, then the free base was liberated by treating with aqueous NaOH [McDowell and Allen *J Phys Chem* 65 1358 1961].

Didodecylamine [3007-31-6] M 353.7, m 51.8°, pK²⁵ 11.00. Crystd from EtOH/*C₆H₆ under N₂.

Didodecyltrimethylammonium bromide [3282-73-3] M 463.6, m 157-162°. Recrystd from acetone, acetone/ether mixture, then from ethyl acetate, washed with ether and dried in a vacuum oven at 60° [Chen et al. *J Phys Chem* 88 1631 1984; Rupert et al. *J Am Chem Soc* 107 2628 1985; Halpern et al. *J Am Chem Soc* 108 3920 1986; Allen et al. *J Phys Chem* 91 2320 1987].

Dienestrol [4,4'-(diethylidene-ethylene)diphenol, Dienol] [84-17-3] M 266.3, m 227-228°, 231-233°, $pK_{\text{Est}} \sim 9.8$. Crystd from EtOH or dilute EtOH, sublimes at 130°/1mm. The *diacetate* has m 119-120° (from EtOH) [Hobday and Short *J Chem Soc* 609 1943].

Diethanolamine (2,2'-iminodiethanol) [111-42-2] M 105.1, m 28°, b 154-155°/10mm, 270°/760mm $pK^{25} 8.88$. Fractionally distd twice, then fractionally crystd from its melt.

3,4-Diethoxy-3-cyclobutene-1,2-dione (diethyl squarate) [5321-87-8] M 170.2, b 89-91°/0.4mm, 88-92°/0.4mm, $d_4^{20} 1.162$, $n_D^{25} 1.5000$. Dissolve in Et₂O, wash with Na₂CO₃, H₂O and dry (Na₂SO₄), filter, evaporate and distil using a Kügelrohr or purify by chromatography. Use a Kieselgel column and elute with 20% Et₂O-Pet ether (b 40-60°) then with Et₂O-pet ether (1:1), evaporate and distil *in vacuo*. [Dehmlow and Schell *Chem Ber* 113 1 1980; Perri and Moore *J Am Chem Soc* 112 1897 1990; IR: Cohen and Cohen *J Am Chem Soc* 88 1533 1966.] **It can cause severe dermatitis** [Foland et al. *J Am Chem Soc* 111 975 1989].

N,N-Diethylacetamide [685-91-6] M 157.2, b 86-88°, n 1.474, d 0.994. Dissolved in cyclohexane, shaken with anhydrous BaO and then filtered. The procedure was repeated three times, and the cyclohexane was distd off at 1 atmosphere pressure. The crude amide was also fractionally distd three times from anhydrous BaO.

Diethyl acetamidomalonate [1068-90-2] M 217.2, m 96°. Crystd from *benzene/pet ether.

Diethyl acetylenedicarboxylate [762-21-0] M 170.2, b 60-62°/0.3mm, 107-110°/11mm, 118-120°/20mm, $d_4^{20} 1.0735$, $n_D^{20} 1.4428$. Dissolve in *C₆H₆, wash with NaHCO₃, H₂O, dry over Na₂SO₄, filter, evaporate and distil in a vacuum [IR: Walton and Hughes *J Am Chem Soc* 79 3985 1957; Truce and Kruse *J Am Chem Soc* 81 5372 1959].

Diethylamine [109-89-7] M 73.1, b 55.5°, d 0.707, n 1.38637, $pK^{15} 11.38$. Dried with LiAlH₄ or KOH pellets. Refluxed with, and distd from, BaO or KOH. Converted to the *p*-toluenesulfonamide and crystd to constant melting point from dry pet ether (b 90-120°), then hydrolysed with HCl, excess NaOH was added, and the amine passed through a tower of activated alumina, redistd and dried with activated alumina before use [Swift *J Am Chem Soc* 64 115 1942].

§ A polystyrene diethylaminomethyl supported version is commercially available.

Diethylamine hydrochloride [660-68-4] M 109.6, m 223.5°. Crystd from absolute EtOH. Also crystd from dichloroethane/MeOH. *Hygroscopic*.

trans-4-(Diethylamino)azobenzene [3588-91-8] M 320.5, m 171° $pK_{\text{Est}(1)} \sim 5.4$, $pK_{\text{Est}(2)} \sim 3.0$. Purified by column chromatography [Flamigni and Monti *J Phys Chem* 89 3702 1985].

N,N-Diethylaniline [91-66-7] M 149.2, b 216.5°, d 0.938, n 1.5409 $pK^{25} 6.57$. Refluxed for 4h with half its weight of acetic anhydride, then fractionally distd under reduced pressure (b 92°/10mm).

Diethyl azodicarboxylate (DEAD) [1972-28-7] M 174.2, b 104.5°/12mm, 211-213°/atm, $d_4^{20} 1.110$, $n_D^{20} 1.420$. Dissolve in toluene, wash with 10% NaHCO₃ till neutral (may require several washes if too much hydrolysis had occurred (check IR for OH bands), then wash with H₂O (2 x), dry over Na₂SO₄, filter, evaporate the toluene and distil through a short Vigreux column. Main portion boils at 107-111°/15mm [*Org Synth Coll Vol III* 376 1955].

§ A polystyrene supported DEAD version is commercially available.

5,5-Diethylbarbituric acid (Barbital) [57-44-3] M 184.2, m 188-192°, $pK_1^{25} 8.02$, $pK_2^{25} 12.7$. Crystd from water or EtOH. Dried in a vacuum over P₂O₅.

Diethyl bromomalonate [685-87-0] M 239.1, b 116-118°/10mm, 122-123°/20mm, d_4^{20} 1.420, n_D^{20} 1.4507. Purified by fractional distn in a vacuum. IR: 1800 and 1700 cm^{-1} [Abramovitch *Can J Chem* 37 1146 1959; Bretschneider and Karpitschka *Monatsh Chem* 84 1091 1953].

Diethyl tert-butylmalonate [759-24-0] M 216.3, b 40-42°/0.03, 102-104°/11mm, 109.5-110.5°/17mm, 205-210°/760mm, d_4^{20} 0.980, n_D^{20} 1.425. Dissolve in Et_2O , wash with aqueous NaHCO_3 , H_2O , dry (MgSO_4), filter, evaporate and distil residue. Identified by hydrolysis to the acid and determining the neutralisation equiv (theor: 80.0). The acid has m 155-157° efferv [Hauser, Abramovitch and Adams *J Am Chem Soc* 64 2715 1942; Bush and Beauchamp *J Am Chem Soc* 75 2949 1953].

***N,N'*-Diethylcarbanilide (sym-Diethyldiphenylurea)** [85-98-3] M 268.4, m 79°. Crystd from EtOH.

Diethyl carbonate [105-58-8] M 118.1, b 126.8°, d 0.975, n_D^{25} 1.38287. It was washed (100mL) with an aqueous 10% Na_2CO_3 (20mL) solution, saturated CaCl_2 (20mL), then water (30mL). After drying by standing over solid CaCl_2 for 1h (note that prolonged contact should be avoided because slow combination with CaCl_2 occurs), it should be fractionally distd. Also dried over MgSO_4 and distd.

1,1'-Diethyl-2,2'-cyanine iodide [977-96-8] M 454.4, m 274°(dec). Crystd from EtOH and dried in a vacuum oven at 80° for 4h.

***N,N*-Diethylcyclohexylamine** [91-65-6] M 155.3, b 193°/760mm, d 0.850, n 1.4562, pK^{25} 10.72. Dried with BaO and fractionally distd.

Diethylene glycol [111-46-6] M 106.1, f -10.5°, b 244.3°, d 1.118, n_D^{15} 1.4490, n 1.4475. Fractionally distd under reduced pressure (b 133°/14mm), then fractionally crystd by partial freezing.

Diethylene glycol diethyl ether [112-36-7] M 162.2, b 85-86°/10mm, 188.2-188.3°/751mm, d 0.909, n 1.412. Dried with MgSO_4 , then CaH_2 or LiAlH_4 , under N_2 . If sodium is used the ether should be redistd alone to remove any products which may be formed by the action of sodium on the ether. As a preliminary purification, the crude ether (2L) can be refluxed for 12h with 25mL of conc HCl in 200mL of water, under reduced pressure, with slow passage of N_2 to remove aldehydes and other volatile substances. After cooling, addn of sufficient solid KOH pellets (slowly and with shaking until no more dissolves) gives two liquid phases. The upper of these is decanted, dried with fresh KOH pellets, decanted, then refluxed over, and distd from sodium. Can be passed through (alkaline) alumina prior to purification.

Diethylene glycol ditosylate [7460-82-4] M 414.5, m 86-87°, 87-88°, 88-89°. Purified by recrystn from Me_2CO and dried in a vacuum.

Diethylene glycol mono-*n*-butyl ether (butyl carbitol) [112-34-5] M 162.2, b 69-70°/0.3mm, 230.5°/760mm, d 0.967, n 1.4286. Dried with anhydrous K_2CO_3 or CaSO_4 , filtered and fractionally distd. Peroxides can be removed by refluxing with stannous chloride or a mixture of FeSO_4 and KHSO_4 (or, less completely, by filtration under slight pressure through a column of activated alumina).

Diethylene glycol monoethyl ether [111-90-0] M 134.2, b 201.9°, d 0.999, n 1.4273, n_D^{25} 1.4254. Ethylene glycol can be removed by extracting 250g in 750mL of *benzene with 5mL portions of water, allowing for phase separation, until successive aqueous portions show the same volume increase. Dried, and freed from peroxides, as described for diethylene glycol mono-*n*-butyl ether.

Diethylene glycol monomethyl ether [111-77-3] M 120.2, b 194°, d 1.010, n 1.423. Purified as for diethylene glycol mono-*n*-butyl ether.

Diethylenetriaminepenta-acetic acid (DTPA) [67-43-6] M 393.4, m 219-220°, pK_1^{25} 1.79, pK_2^{25} 2.56, pK_3^{25} 4.42, pK_4^{25} 8.76, pK_5^{25} 10.42. Crystd from water. Dried under vacuum or at 110°. [Bielski and Thomas *J Am Chem Soc* 109 7761 1987].

Diethyl ether (ethyl ether) [60-29-7] **M 74.1, b 34.6°/760mm, d 0.714, n¹⁵ 1.3555, n 1.35272.** Usual impurities are water, EtOH, diethyl peroxide (which is explosive when concentrated), and aldehydes. Peroxides [detected by liberation of iodine from weakly acid (HCl) solutions of KI, or by the blue colour in the ether layer when 1mg of Na₂Cr₂O₇ and 1 drop of dil H₂SO₄ in 1mL of water is shaken with 10mL of ether] can be removed in several different ways. The simplest method is to pass dry ether through a column of activated alumina (80g Al₂O₃/700mL of ether). More commonly, 1L of ether is shaken repeatedly with 5-10mL of a soln comprising 6.0g of ferrous sulfate and 6mL of conc H₂SO₄ in 110mL of water. Aqueous 10% Na₂SO₃ or stannous chloride can also be used. The ether is then washed with water, dried for 24h with CaCl₂, filtered and dried further by adding sodium wire until it remains bright. The ether is stored in a dark cool place, until distd from sodium before use. Peroxides can also be removed by wetting the ether with a little water, then adding excess LiAlH₄ or CaH₂ and leaving to stand for several hours. (This also dried the ether.)

Werner [*Analyst* 58 335 1933] removed peroxides and aldehydes by adding 8g AgNO₃ in 60mL of water to 1L of ether, then 100mL of 4% NaOH and shaking for 6min. Fierz-David [*Chimia* 1 246 1947] shook 1L of ether with 10g of a zinc-copper couple. (This reagent was prepared by suspending zinc dust in 50mL of hot water, adding 5mL of 2M HCl and decanting after 20sec, washing twice with water, covering with 50mL of water and 5mL of 5% cuprous sulfate with swirling. The liquid was decanted and discarded, and the residue was washed three times with 20mL of ethanol and twice with 20mL of diethyl ether).

Aldehydes can be removed from diethyl ether by distn from hydrazine hydrogen sulfate, phenyl hydrazine or thiosemicarbazide. Peroxides and oxidisable impurities have also been removed by shaking with strongly alkaline satd KMnO₄ (with which the ether was left to stand in contact for 24h), followed by washing with water, conc H₂SO₄, water again, then drying (CaCl₂) and distn from sodium, or sodium containing benzophenone to form the ketyl. Other purification procedures include distn from sodium triphenylmethide or butyl magnesium bromide, and drying with solid NaOH or P₂O₅.

Rapid purification: Same as for 1,4-dioxane.

Diethyl ethoxymethylene malonate [87-13-8] **M 216.2, b 014°/0.2mm, 109°/0.5mm, 279-283°/atm, d₄²⁰ 1.079, n_D²⁰ 1.4623.** Likely impurity is diethyl diethoxymethylene malonate which is difficult to separate from diethyl ethoxymethylene malonate by distn and it is necessary to follow the course of the distn by the change in refractive index instead of boiling point. After a low boiling fraction is collected, there is obtained an intermediate fraction (n_D²⁰ 1.414—1.458) the size of which depends on the amount of diethoxymethylene compound. This fraction is fractionated through a 5-inch Vigreux column at low pressure avoiding interruption in heating. Fraction **b** 108-110°/0.25mm was *ca* 10° lower than the submitters' (**b** 97.2°/0.25mm (n_D²⁰ 1.4612—1.4623) [*Org Synth Coll Vol III* 395 1955; Fuson et al. *J Org Chem* 11 197 1946; Duff and Kendal *J Chem Soc* 893 1948].

N,N'-Diethylformamide [617-84-5] **M 101.2, b 29°/0.5mm, 61-63°/10mm, 178.3-178.5°/760mm, d₄²⁰ 0.906, n_D²⁵ 1.4313.** Distd under reduced pressure then at atmospheric pressure [Wintcler et al. *Helv Chim Acta* 37 2370 1954; NMR: Hoffmann *Z Anal Chem* 170 177 1959].

Diethyl fumarate [623-91-6] **M 172.2, b 218°, d 1.052, n 1.441.** Washed with aqueous 5% Na₂CO₃, then with saturated CaCl₂ soln, dried with CaCl₂ and distd.

Di-(2-ethylhexyl)phthalate ('di-iso-octyl' phthalate) [117-81-7] **M 390.6, b 384°, 256-257°/1mm, d 0.9803, n 1.4863.** Washed with Na₂CO₃ soln, then shaken with water. After the resulting emulsion had been broken by adding ether, the ethereal soln was washed twice with water, dried (CaCl₂), and evaporated. The residual liquid was distd several times under reduced pressure, then stored in a vacuum desiccator over P₂O₅ [French and Singer *J Chem Soc* 1424 1956]

Diethyl ketone (3-pentanone) [96-22-0] **M 86.1, b 102.1°, d 0.8099, n 1.392.** Dried with anhydrous CaSO₄ or CuSO₄, and distd from P₂O₅ under N₂ or under reduced pressure. Further purification by conversion to the semicarbazone (recrystd to constant **m** 139°, from EtOH) which, after drying under vacuum over CaCl₂ and paraffin wax, was refluxed for 30min with excess oxalic acid, then steam distd and salted out with K₂CO₃. Dried with Na₂SO₄ and distd [Cowan, Jeffrey and Vogel *J Chem Soc* 171 1940].