

Diethyl malonate [105-53-3] M 160.2, b 92°/22mm, 198-199°/760mm, d 1.056, d_4^{25} 1.0507, n 1.413. If too impure (IR, NMR) the ester (250g) has been heated on a steam bath for 36h with absolute EtOH (125mL) and conc H₂SO₄ (75mL), then fractionally distd under reduced pressure. Otherwise fractionally distil under reduced pressure and collect the steady boiling middle fraction.

Diethyl phenyl orthoformate (diethoxy phenoxy ethane) [14444-77-0] M 196.3, b 111°/11mm, 122°/13mm, d_4^{20} 1.0099, n_D^{20} 1.4799. Fractionated through an efficient column under vacuum [Smith *Acta Chem Scand* 10 1006 1956].

Diethyl phthalate [84-66-2] M 222.2, b 172°/12mm, b 295°/760mm, d^{25} 1.1160, n 1.5022. Washed with aqueous Na₂CO₃, then distilled water, dried (CaCl₂), and distd under reduced pressure. Stored in a vacuum desiccator over P₂O₅.

Diethyl phthalimidomalonate [56680-61-5] M 305.3, m 72-74°, 73-74°, pK 9.17. Dissolve in xylene and when the temperature is 30° add pet ether (b 40-60°) and cool to 20° whereby the malonate separates as a pale brown powder [Booth et al. *J Chem Soc* 666 1944]. Alternatively, dissolve in *C₆H₆, dry over CaCl₂, filter, evaporate and the residual oil solidifies. This is ground with Et₂O, filter and wash with Et₂O until white in colour, and dry in a vacuum. The anion has λ_{max} 254nm (ϵ 18.5K) [Clark and Murray *Org Synth Coll Vol I* 271 1941; UV of Na salt: Nnadi and Wang *J Am Chem Soc* 92 4421 1970].

2,2-Diethyl-1,3-propanediol [115-76-4] M 132.2, m 61.4-61.8°. Crystd from pet ether (b 65-70°).

Diethyl pyrocarbonate (DEP) [1609-47-8] M 162.1, b 38-40°/12mm, 160-163°/atm, d_4^{20} 1.119, n_D^{20} 1.398. Dissolve in Et₂O, wash with dilute HCl, H₂O, dry over Na₂SO₄, filter, evaporate and distil the residue first *in vacuo* then at atmospheric pressure. It is soluble in alcohols, esters, ketones and hydrocarbon solvents. A 50% w/w soln is usually prepared for general use. **Treat with great CAUTION as DEP irritates the eyes, mucous membranes and skin.** [Boehm and Mehta *Chem Ber* 71 1797 1938; Thoma and Rinke *Justus Liebigs Ann Chem* 624 30 1959.]

Diethylstilboesterol [56-23-1] M 268.4, m 169-172°. Crystd from *benzene.

Diethyl succinate [123-25-1] M 174.2, b 105°/15mm, d 1.047, n 1.4199. Dried with MgSO₄, and distd at 15mm pressure.

Diethyl sulfate [64-67-5] M 154.2, b 96°/15mm, 118°/40mm, d 1.177, n 1.399. Washed with aqueous 3% Na₂CO₃ (to remove acidic material), then distilled water, dried (CaCl₂), filtered and distd. *Causes blisters to the skin.*

Diethyl disulfide [110-81-6] M 122.3, b 154-155°, d 0.993, n 1.506. Dried with silica gel or MgSO₄ and distd under reduced pressure (optionally from CaCl₂).

Diethyl sulfide [352-93-2] M 90.2, m 0°/15mm, 90.1°/760mm, d 0.837, n 1.443. Washed with aq 5% NaOH, then water, dried with CaCl₂ and distd from sodium. Can also be dried with MgSO₄ or silica gel. Alternative purification is *via* the Hg(II) chloride complex [(Et)₂S.2HgCl₂] (see dimethyl sulfide).

Diethyl (-)-D- (from the non-natural) [13811-71-7] and (+)-L- (from the natural acid) [89-91-2] tartrate M 206.2, m 17°, b 80°/0.5mm, 162°/19mm, 278-282°/atm, d_4^{20} 1.204, n_D^{20} 1.4476, $[\alpha]_D^{20}$ (-) and (+) 26.5° (c 1, H₂O) and (-) and (+) 8.5° (neat), $[\alpha]_{546}^{20}$ (-) and (+) 30° (c 1, H₂O). Distd under high vacuum and stored under vacuum or in an inert atm in a desiccator in round bottomed flasks equipped with a vac stopcock. Have also been dist by Kügelrohr distn and/or by 'wiped-film' molecular distn. Slightly sol in H₂O but miscible in EtOH and Et₂O. [Gao et al. *J Am Chem Soc* 109 5770 (5771) 1987; IR: Pristera *Anal Chem* 25 844 1953.]

Diethyl terephthalate [636-09-0] M 222.2, m 44°, 142°/2mm, 302°/760mm. Crystd from toluene and distd under reduced pressure.

sym-Diethylthiourea [105-55-5] M 132.2, m 76-77°. Crystd from *benzene.

Difluoroacetic acid [381-73-7] M 96.0, m -0.35°, b 67-70°/20mm, 134°/760mm, d_4^{20} 1.530, n_D^{20} 1.3428, pK^{25} 1.28. Purified by distilling over P_2O_5 . The *acid chloride* is a fuming liquid b 25°/atm, and the *amide* has b 108.6°/35mm, m 52° (from * C_6H_6), and the *anilide* has b 90°/1mm, 114°/5mm, m 58° [Henne and Pelley *J Am Chem Soc* 74 1426 1952, Coffman et al. *J Org Chem* 14 749 1949; NMR: Meyer et al. *J Am Chem Soc* 75 4567 1953; pK: Wegscheider *Z Phys Chem* 69 614 1909].

Diglycolic acid (2-oxapentane-1,5-dioic acid) [110-99-6] M 134.1, m 148° (monohydrate), pK_1^{25} 2.97, pK_2^{25} 4.37. Crystd from water.

Diglycyl glycine [556-33-2] M 189.2, m 246°(dec), pK_1^{25} 3.30, pK_2^{25} 7.96. Crystd from H_2O or $H_2O/EtOH$ and dried at 110°.

Diglyme [bis-(2-methoxyethyl) ether, diethylene glycol dimethyl ether] [111-96-6] M 134.2, b 62°/17mm, 75°/35mm, 160°/760mm, d 0.917, n 1.4087. Dried with NaOH pellets or CaH_2 , then refluxed with, and distd (under reduced pressure) from Na, CaH_2 , $LiAlH_4$, $NaBH_4$ or NaH. These operations were carried out under N_2 . The amine-like odour of diglyme has been removed by shaking with a weakly acidic ion-exchange resin (Amberlite IR-120) before drying and distn. Addn of 0.01% $NaBH_4$ to the distillate inhibits peroxidation. Purification as for dioxane. Also passed through a 12-in column of molecular sieves to remove water and peroxides.

Digoxin [20830-75-5] M 781.0, m 265°(dec), $[\alpha]_{546}^{20}$ +14.0° (c 10, pyridine). Crystd from aqueous EtOH or aqueous pyridine.

4,4'-Di-n-heptyloxyazoxybenzene [2635-26-9] M 426.6, m 75°, 95° (smectic → nematic) and 127° (nematic → liquid), pK_{Est} --5. Purified by chromatography on Al_2O_3 (*benzene), recrystd from hexane or 95% EtOH and dried by heating under vacuum. The liquid crystals can be sublimed *in vacuo*. [Mellifiori et al. *Spectrochim Acta Part A* 37(A) 605 1981; Dewar and Schroeder *J Am Chem Soc* 86 5235 1964; Weygand and Glaber *J Prakt Chem* 155 332 1940].

9,10-Dihydroanthracene [613-31-0] M 180.3, m 110-110.5°. Crystd from EtOH [Rabideau et al. *J Am Chem Soc* 108 8130 1986].

2,3-Dihydrobenzofuran (coumaran) [496-16-2] M 120.2, m -21.5°, 72-73°/12mm, 84°/17mm, 188°/atm, d_4^{20} 1.065, n_D^{20} 1.5524. Suspend in aqueous NaOH and steam distil. Saturate the distillate with NaCl and extract with Et_2O , dry extract ($MgSO_4$), filter, evap and distil the residue. It gives a strong violet colour with $FeCl_3 + H_2SO_4$ and forms a yellow *picrate*, m 76°, from EtOH or * C_6H_6 which loses coumaran in a desiccator [Bennett and Hafez *J Chem Soc* 287 1941; Baddeley et al. *J Chem Soc* 2455 1956].

Dihydrochloranil (tetrachloro-1,4-hydroquinone) [87-87-6] M 247.9, m 240.5°. Crystd from EtOH or AcOH+EtOH. Sublimes at 77°/0.6x10⁻³mm. The *dibenzoyl derivative* has m 233°. [Conant and Fieser *J Am Chem Soc* 45 2207 1923 ; Rabideau et al. *J Am Chem Soc* 108 8130 1986.]

Dihydrocodeine [125-28-0] M 301.4, m 112-113°, b 248/14mm°. Crystd from aqueous methanol.

1,4-Dihydro-1,4-epoxynaphthalene [573-57-9] M 144.2, m 53-54.5°, 53-56°, 55-56°. Dissolve in Et_2O , wash with H_2O , dry over K_2CO_3 , filter, evaporate and dry the residue at 15mm, then recrystallise from pet ether (b 40-60°), dry at 25°/0.005mm and sublime (sublimes slowly at room temp)[Wittig and Pohmer *Chem Ber* 89 1334 1956; Gilman and Gorsich *J Am Chem Soc* 79 2625 1957].

Dihydropyran (3,4-dihydro-2H-pyran) [110-87-2] M 84.1, b 84.4°/742mm, 85.4-85.6°/760mm, d_4^{20} 0.9261, n_D^{20} 1.4423, pK_{Est} ~ 4.2. Partially dried with Na_2CO_3 , then fractionally distd. The fraction b 84-85°, was refluxed with Na until hydrogen was no longer evolved when fresh Na was

added. It was then dried, and distd again through a 60 x 1.2cm column packed with glass rings [Brandon et al. *J Am Chem Soc* 72 2120 1950; UV: Elington et al. *J Chem Soc* 2873 1952, NMR: Bushweller and O'Neil *Tetrahedron Lett* 4713 1969]. It has been characterised as the 2,3,5-dinitrobenzoyloxy-tetrahydrofuran derivative, **m** 103° which forms pale yellow crystals from dihydropyran-Et₂O [Woods and Kramer *J Am Chem Soc* 69 2246 1947].

3,4-Dihydro-2H-pyrido[1,2a]-pyrimidin-2-one [5439-14-5] **M** 148.2, **m** 185-187°, 187-188°, 191-191.5°. Dissolve in CHCl₃, filter, evaporate then recrystallise the residue from EtOH-Me₂CO (needles) which can be washed with Et₂O and dried. It can also be recrystd from CHCl₃-pet ether or CHCl₃-hexane. The *hydrochloride* has **m** 295-295° (dec, from EtOH or MeOH-Et₂O), the *hydrobromide* has **m** 299-300°(dec, from MeOH-Et₂O) and the *picrate* has **m** 224-226°(corr), **m** 219-220° from EtOH. [Adams and Pachter *J Am Chem Soc* 74 4906 1952; Lappin *J Org Chem* 23 1358 1958; Hurd and Hayao *J Am Chem Soc* 77 115 1955.]

Dihydrotachysterol [67-96-9] **M** 398.7, **m** 125-127°, $[\alpha]_D^{20} +97^\circ$ (CHCl₃). Crystd from 90% MeOH.

1,8-Dihydroxyanthraquinone [117-10-2] **M** 240.1, **m** 193-197°, **pK₁²⁵ 8.30**, **pK₂²⁵ 12.46**. Crystd from EtOH and sublimed in a vacuum.

2,4-Dihydroxyazobenzene (Sudan orange G) [2051-85-6] **M** 214.2, **m** 228°, **pK_S(.Est(1)) <0**, **pK_{Est(2)} ~7.3**, **pK_{Est(3)} ~9.3**. Crystd from hot EtOH (charcoal).

2,3-Dihydroxybenzaldehyde [24677-78-9] **M** 138.1, **m** 135-136°, **pK₁²⁰ 7.73**, **pK₂²⁰ 10.91**. Crystd from water.

2,4-Dihydroxybenzoic acid [89-86-1] **M** 154.1, **m** 226-227°(dec), **pK₁²⁵ 3.30**, **pK₂²⁵ 9.12**, **pK₃²⁵ 15.6**. Crystd from water.

2,5-Dihydroxybenzoic acid [490-79-9] **M** 154.1, **m** 204.5-205°, **pK²⁵ 2.95**. Crystd from hot water or *benzene/acetone. Dried in a vacuum desiccator over silica gel.

2,6-Dihydroxybenzoic acid [303-07-1] **M** 154.1, **m** 167°(dec), **pK²⁵ 1.05**. Dissolved in aqueous NaHCO₃ and the soln was washed with ether to remove non-acidic material. The acid was pptd by adding H₂SO₄, and recrystd from water. Dried under vacuum and stored in the dark [Lowe and Smith *J Chem Soc, Faraday Trans 1* 69 1934 1973].

2,4-Dihydroxybenzophenone [131-56-6] **M** 214.2, **m** 145.5-147° **pK_{Est(1)} ~7.0**, **pK_{Est(2)} ~12.0**. Recrystd from MeOH.

2,5-Dihydroxybenzyl alcohol (Gentisyl alcohol) [495-08-9] **M** 140.1, **m** 100° **pK_{Est(1)} ~9.3**, **pK_{Est(2)} ~11.3**. Crystd from CHCl₃. Sublimes at ~70° under high vacuum.

2,2'-Dihydroxybiphenyl [1806-29-7] **M** 186.2, **m** 108.5-109.5°, **pK₁²⁵ 7.56**, **pK₂²⁵ 11.80**. Repeatedly crystd from toluene, then sublimed at 60°/10⁻⁴mm.

3 α ,7 α -Dihydroxycholanolic acid (Chenodeoxycholic acid) [474-25-9] **M** 239.6, **m** 143°, $[\alpha]_{546}^{20} +14^\circ$ (c 2, EtOH), **pK_{Est} ~4.9**. Crystd from ethyl acetate.

7,8-Dihydroxycoumarin (Daphnetin) [486-35-1] **M** 178.2, **m** 256°(dec), **pK_{Est(1)} ~8.5**, **pK_{Est(2)} ~12.3**. Crystd from aqueous EtOH. Sublimed.

2,2'-Dihydroxy-6,6'-dinaphthyl disulfide [6088-51-3] **M** 350.5, **m** 220-223°. See 6-hydroxy-2-naphthyl disulfide on p. 264.

trans-2,3-Dihydroxy-1,4-dioxane [4845-50-5] M 120.1, m 91-95°, 100°. Recryst from Me₂CO. With phenylhydrazine it gives *glyoxal phenylhydrazone* m 175° (from Me₂CO-pet ether). The *diacetyl* derivative has m 105-106° [Head *J Chem Soc* 1036 1955, Raudnitz *Chem Ind (London)* 166 1956].

2,5-Dihydroxy-1,4-dithiane [40018-26-6] M 152.2, m (142-147° ?) 150-152°, 151°. Recrystd from EtOH. The *2,5-diethoxy-dithiane* has m 91° (92-93°) crystallises from pet ether and can be sublimed at 60°/0.001mm [Hormatka and Haber *Monatsh Chem* 85 1088 1954; Thiel et al. *Justus Liebigs Ann Chem* 611 121 1958; Hesse and Jøeder *Chem Ber* 85 924 1952].

(N,N-Dihydroxyethyl)glycine (BICINE) [150-25-4] M 163.2, m 193°(dec), pK₁²⁵ 1.81, pK₂²⁵ 8.27. Dissolved in a small volume of hot water and ppted with EtOH, twice. Repeated once more but with charcoal treatment of the aqueous soln, and filtered before addition of EtOH.

Dihydroxyfumaric (1,2-dihydroxybut-1-ene-1,2-dioic) acid dihydrate [133-38-0] M 184.1, m 155°(dec), pK₁²⁵ 1.57, pK₂²⁵ 3.36. Crystd from water.

3,4'-Dihydroxyisoflavone [578-86-9] M 256.3, m 234-236°. Crystd from aqueous 50% EtOH.

5,7-Dihydroxy-4'-methoxyflavone [480-44-4] M 284.3, m 261°. Crystd from 95% EtOH.

1,8-Dihydroxy-3-methylantraquinone (chrysophanic acid) [481-74-3] M 245.3, m 196°, pK_{Est(1)} ~8.2, pK_{Est(2)} ~12.4. Crystd from EtOH or *benzene and sublimed in a vacuum.

1,5-Dihydroxynaphthalene [83-56-7] M 160.2, m 260°, 250-261°, pK_{Est} ~9.6. Crystd from nitromethane.

1,6-Dihydroxynaphthalene [575-44-0] M 160.2, m 138-139° (with previous softening), pK_{Est} ~9.4. Crystd from *benzene or *benzene/EtOH after treatment with charcoal.

2,5-Dihydroxyphenylacetic acid (homogentisic acid) [451-13-8] M 168.2, m 152°, 154-152°, pK²⁰ 4.14 (COOH). Crystd from EtOH/CHCl₃ or H₂O (sol 85% at 25°).

3,4-Dihydroxytoluene [452-86-8] M 124.1, m 65-66°, 68°, b 112°/3mm, 241°/760mm, pK₁²⁵ 9.44 (9.7), pK₂²⁵ 10.90 (11.9). Crystd from *C₆H₆. Purity checked by TLC. Crystd from high-boiling pet ether and distd in a vacuum.

1,3-Diiminoisoindoline [3468-11-9] M 145.2, m 193-194° (dec), 196° (dec), pK 8.27. It crystallises from H₂O, MeOH or MeOH-Et₂O (charcoal) in colourless prisms that become green on heating. [Elvidge and Linstead *J Chem Soc* 5000 1952]. IR (nujol): 3150 and 690 cm⁻¹, and UV: λ_{max} 251nm (ε 12.5K), 256nm (ε 12.5K) and 303nm (ε 4.6K) [Elvidge and Golden *J Chem Soc* 700 1957; Clark et al. *J Chem Soc* 3593 1953]. The *thiocyanate* has m 250-255° (dec), the *monohydrochloride* has m 300-301° (turns green) and the *dihydrochloride* has m 326-328° (turns green) and the *picrate* cryst from EtOH has m 299° (dec).

1,4-Diiodobenzene [624-38-4] M 329.9, m 132-133°. Crystd from EtOH or boiling MeOH, then air dried.

1,2-Diiodoethane [624-73-7] M 281.9, m 81-84°, d 2.134. Dissolved in ether, washed with satd aq Na₂S₂O₃, drying it over MgSO₄ and evap the ether *in vacuo* [Molander et al. *J Am Chem Soc* 109 453 1987].

5,7-Diiodo-8-hydroxyquinoline [83-73-8] M 397.0, m 214-215°(dec) pK_{Est(1)} ~3.2, pK_{Est(2)} ~8.2. Crystd from xylene and dried at 70° in a vacuum.

Diiodomethane (methylene diiodide) [75-11-6] M 267.8, m 6.1°, b 66-70°/11-12mm, d 3.325. Fractionally distd under reduced pressure, then fractionally crystd by partial freezing, and stabilized

with silver wool if necessary. It has also been purified by drying over CaCl_2 and fractionally distd from Cu powder.

S-3,5-Diiodotyrosine (iodogorgoic acid) [300-39-0] M 469.0, m 204°(dec), $[\alpha]_{\text{D}} +1.5^\circ$ (in 1M HCl) pK_1 2.12, pK_2 6.48, pK_3 7.82. See 3,5-diiodo-L-tyrosine dihydrate on p. 530 in Chapter 6.

Diisopropanolamine [110-97-4] M 133.2, m 41-44°, d 1.004, $\text{pK}_{\text{Est}} \sim 10.7$. Repeatedly crystd from dry diethyl ether.

Diisopropylamine [108-18-9] M 101.2, b 83.5°/760mm, n 1.39236, d 0.720, pK^{25} 11.20. Distd from NaOH, or refluxed over Na wire or NaH for three minutes and distd into a dry receiver under N_2 .
§ A polystyrene supported version of diisopropylamine is commercially available.

Diisopropylethylamine [7087-68-5] M 129.3, b 127°, $\text{pK}_{\text{Est}} \sim 10.9$. Distd from ninhydrin, then from KOH [Dryland and Sheppard, *J Chem Soc, Faraday Trans 1* 125 1986]. It is a strong base and should be stored in the absence of carbon dioxide.

(-)-2,3:4,6-Di-O-isopropylidene-2-keto-L-gulonic acid monohydrate (- DAG) [18467-77-1] M 292.3, m 100-101°, 103°, $[\alpha]_{\text{D}}^{25} -21.6^\circ$ (c 2.3, MeOH). Dissolve in Et_2O , filter, dry (MgSO_4), filter, evaporate to give a yellow oil. Addition of one drop of H_2O induces crystn to the monohydrate, which also forms rhombic crystals by recrystn from 95% $\text{EtOH-H}_2\text{O}$ at room temperature. [Flatt et al. *Synthesis* 815 1979; Reichstein and Grussner *Helv Chim Acta* 17 311 1934; Takagi and Jeffrey *Acta Crystallogr Sect B* 34 2932 1978; cf *Org Synth* 55 80 1976.]

1,2:5,6-Di-O-isopropylidene-D-mannitol [1707-77-3] M 262.3, m 121-125°, 122°, $[\alpha]_{\text{D}}^{25} +1.2^\circ$ (c 3, H_2O). Although quite soluble in H_2O it gives a purer product from this solvent, forming needles [Baer *J Am Chem Soc* 67 338 1945; NMR: Curtis et al. *J Chem Soc, Perkin Trans 1* 1756 1977].

Diisopropyl ketone (2,4-dimethyl-3-pentanone) [565-80-0] M 114.2, b 124°, d 0.801, n 1.400. Dried with CaSO_4 , shaken with chromatographic alumina and fractionally distd from P_2O_5 under N_2 .

Diketene [674-82-8] M 84.1, m -7°, b 66-68°/90mm, d 1.440, n 1.4376, n^{25} 1.4348. Diketene polymerizes violently in the presence of alkali. Distd at reduced pressure, then fractionally crystd by partial freezing (using as a cooling bath a 1:1 soln of $\text{Na}_2\text{S}_2\text{O}_3$ in water, cooled with Dry-ice until slushy, and stored in a Dewar flask). Freezing proceeds slowly, and takes about a day for half completion. The crystals are separated and stored in a refrigerator under N_2 . See ketene on p. 276.

2,2'-Diketospirilloxanthin [24009-17-4] M 624.9, m 225-227°, $\epsilon_{1\text{cm}}^{1\%}(\lambda_{\text{max}})$ 550(349nm), 820(422nm), 2125(488nm), 2725(516nm), 2130(551nm) in hexane. Purified by chromatography on a column of partially deactivated alumina. Crystd from acetone/pet ether. Stored in the dark, in an inert atmosphere at 0°.

Dilauroyl peroxide [105-74-8] M 398.6, m 53-54°. See lauryl peroxide (dodecyl peroxide) on p. 278.

Dimedone (5,5-dimethylcyclohexane-1,3-dione) [126-81-8] M 140.2, m 148-149°, pK^{25} 5.27. Crystd from acetone (ca 8mL/g), water or aqueous EtOH . Dried in air.

2,3-Dimercapto-1-propanol (BAL, British Anti-Lewisite) [59-52-9] M 124.2, b 82-84°/0.8mm, d 1.239, n 1.5732, pK_1^{25} 8.62, pK_2^{25} 10.75. Ppted as the Hg mercaptide [see Bjöberg *Chem Ber* 75 13 1942], regenerated with H_2S , and distd at 2.7mm [Rosenblatt and Jean *Anal Chem* 951 1955].

1,3-Dimercapto-2-propanol [584-04-3] M 124.2, b 82°/1.5mm. Purified as for 2,3-dimercapto-1-propanol above.

meso-2,3-Dimercaptosuccinic acid [304-55-2] M 182.2, m 191-192° (dec), 210° (dec), 210-211° (dec), pK_1^{25} 2.71, pK_2^{25} 3.48, pK_3^{25} 8.89, pK_4^{25} 10.75. Purified by dissolving in NaOH and precipitating with dilute HCl, dry and recrystallise from MeOH. IR has ν at 2544 (SH) and 1689 (CO₂H) cm^{-1} . The *bis-S-acetyl* deriv has m 183-185° (from EtOAc or Me₂CO) and its *Me ester* has m 119-120° (from pet ether) [Gerecke et al. *Helv Chim Acta* 44 957 1961; Owen and Sultanbawa *J Chem Soc* 3112 1949].

4,4'-Dimethoxyazobenzene [501-58-6] M 242.3, m 162.7-164.7°, pK_{Est} ~0. Chromatographed on basic alumina, eluted with *benzene. Crystd from 2:2:1 (v/v) methanol/ethanol/*benzene.

4,4'-Dimethoxyazoxybenzene [1562-94-3] M 258.3, transition temp 118-121°. See *p,p'*-azoxyanisole on p. 118.

1,2-Dimethoxybenzene (veratrole) [91-16-7] M 137.2, m 23°, b 208.5-208.7, d 1.085, n^{25} 1.53232. Steam distd. Fractionally distd from BaO, CaH₂ or Na. Crystd from *benzene or low-boiling pet ether at 0°. Fractionally crystd from its melt. Stored over anhydrous Na₂SO₄.

1,3-Dimethoxybenzene [151-10-0] M 137.2, b 212-213°, d 1.056, n 1.5215. Extracted with aqueous NaOH, and water, then dried. Fractionally distd from BaO or Na.

1,4-Dimethoxybenzene [150-78-7] M 137.2, m 57.2-57.8°. Steam distd. Crystd from hexane or *benzene, and from MeOH or EtOH but these are wasteful due to high solubilities. Dried under vacuum. Also sublimes under vacuum.

2,4-Dimethoxybenzoic acid [91-52-1] M 182.2, m 109°, pK^{25} 4.36. Crystd from water and dried in a vacuum desiccator over H₂SO₄.

2,6-Dimethoxybenzoic acid [1466-76-8] M 182.2, m 186-187°, pK^{25} 3.44. Crystd from water.

3,4-Dimethoxybenzoic acid (veratric acid) [93-07-2] M 182.2, m 181-182°, pK^{25} 4.43. Crystd from water or aq acetic acid.

3,5-Dimethoxybenzoic acid [1132-21-4] M 182.2, m 185-186°, pK^{25} 3.97. Crystd from water, EtOH or aq acetic acid.

***p,p'*-Dimethoxybenzophenone** [90-96-0] M 242.3, m 144.5°. Crystd from absolute EtOH.

2,6-Dimethoxy-1,4-benzoquinone [530-55-2] M 168.1, m 256°. Crystd from acetic acid. Sublimes in a vacuum.

1,1-Dimethoxyethane (acetaldehyde dimethyl acetal) [534-15-6] M 90.1, b 212°/760mm, d 0.828, n 1.4140. Purification as for acetal on p. 81. Also purified by GLC.

1,2-Dimethoxyethane (glycol dimethyl ether, glyme) [110-71-4] M 90.1, b 84°, d 0.867, n 1.380. Traces of water and acidic materials have been removed by refluxing with Na, K or CaH₂, decanting and distilling from Na, K, CaH₂ or LiAlH₄. Reaction has been speeded up by using vigorous high-speed stirring and molten potassium. For virtually complete elimination of water, 1,2-dimethoxyethane has been dried with Na-K alloy until a characteristic blue colour was formed in the solvent at Dry-ice/cellosolve temperatures: the solvent was kept with the alloy until distd for use [Ward *J Am Chem Soc* 83 1296 1961]. Alternatively, glyme, refluxed with benzophenone and Na-K, was dry enough if, on distn, it gave a blue colour of the ketyl immediately on addition to benzophenone and sodium [Ayscough and Wilson *J Chem Soc* 5412 1963]. Also purified by distn under N₂ from sodium benzophenone ketyl (see above).

5,6-Dimethoxy-1-indanone [2107-69-9] M 192.2, m 118-120°. Crystd from MeOH, then sublimed in a vacuum.

Dimethoxymethane (methylal) [109-87-5] M 76.1, b 42-46°/atm, d_4^{20} 0.8608, n_D^{20} 1.35335. See formaldehyde dimethyl acetal on p. 245.

1,4-Dimethoxynaphthalene [10075-62-4] M 188.2, m 87-88°. Crystd from EtOH.

1,5-Dimethoxynaphthalene [10075-63-5] M 188.2, m 183-184°. Crystd from EtOH.

2,6-Dimethoxyphenol [91-10-1] M 154.2, m 54-56°, pK_{Est} ~9.6. Purified by zone melting or sublimation in a vacuum.

3,5-dimethoxyphenol (phloroglucinol dimethylether) [500-99-2] M 154.2, m 42-43°, b 115°/0.04mm, pK^{25} 9.35. Purified by distn followed by sublimation in a vacuum.

3,4-Dimethoxyphenyl acetic acid (homoveratric acid) [93-40-3] M 196.2, m 97-99°, pK^{25} 4.33. Crystd from water or *benzene/ligroin.

3,5-Dimethoxyphenylacetonitrile [13388-75-5] M 177.1, m 53°. Crystd from MeOH or pet ether (b 90-110°). [Adams et al. *J Am Chem Soc* 70 664 1948; Sankaraman et al. *J Am Chem Soc* 109 5235 1987.]

4,4'-Dimethoxythiobenzophenone [958-80-5] M 258.3, m 120°. Recrystd from a mixture of cyclohexane/dichloromethane (4:1).

2,6-Dimethoxytoluene [5673-07-4] M 152.2, m 39-41°. Sublimed *in vacuo* [Sankaraman et al. *J Am Chem Soc* 109 5235 1987].

4,4'-Dimethoxytrityl chloride (DMT) [40615-36-9] M 338.8, m 114°. Crysts from cyclohexane-acetyl chloride as the hydrochloride and dry over KOH pellets in a desiccator. When dissolved in *C₆H₆ and air is blown through, HCl is removed. It crystallises from Et₂O. [*Justus Liebigs Ann Chem* 370 142 1909; *Chem Ber* 36 2774 1903; Smith et al. *J Am Chem Soc* 84 430 1962; Smith et al. *J Am Chem Soc* 85 3821 1963.] If it had hydrolysed considerably (see OH in IR) then repeat the crystallisation from cyclohexane-acetyl chloride — excess of AcCl is removed in vac over KOH.

N,N-Dimethylacetamide [127-19-5] M 87.1, b 58.0-58.5°/11.4mm, d 0.940, n 1.437. Shaken with BaO for several days, refluxed with BaO for 1h, then fractionally distd under reduced pressure, and stored over molecular sieves.

β,β -Dimethylacrylic acid (senecioic acid) [541-47-9] M 100.1, m 68°, pK^{25} -5.4 (aq H₂SO₄). Crystd from hot water or pet ether (b 60-80°).

Dimethyl adipate [627-93-0] M 174.2, m 9-11°, b 109°/10mm, 121-123°/20mm, 235°/760mm, d_4^{20} 1.0642, n_D^{20} 1.4292. Dissolve in Et₂O, wash with NaHCO₃, H₂O, dry over MgSO₄, filter, evaporate and distil several times until the IR and NMR are consistent with the structure [Lorette and Brown *J Org Chem* 24 261 1959; Hoffmann and Weiss *J Am Chem Soc* 79 4759 1957].

Dimethyl adipimidate dihydrochloride [14620-72-5] M 245.1, m 218-220°, 222-224°. If the salt smells of HCl then wash with MeOH and dry Et₂O (1:3) under N₂ until the HCl is completely removed. Recryst from MeOH-Et₂O (it is very important that the solvents are super dry) [Hartman and Wold *Biochemistry* 6 2439 1967; McElvain and Shroeder *J Am Chem Soc* 71 40 1949].

Dimethylamine [124-40-3] M 45.1, f -92.2°, b 0°/563mm, 6.9°/760mm, pK^{25} 10.73. Dried by passage through a KOH-filled tower, or by standing with sodium pellets at 0° during 18h.
§ A dimethylaminomethyl polystyrene supported version is commercially available.

Dimethylamine hydrochloride [506-59-2] M 81.6, m 171°. Crystd from hot CHCl_3 or abs EtOH. Also recrystd from MeOH/ether soln. Dried in a vacuum desiccator over H_2SO_4 , then P_2O_5 . *Hygroscopic*.

4-*N,N'*-Dimethylaminoazo-benzene-4'-isothiocyanate {DABITC, 4-[(4-isocyanatophenyl)-azo]-*N,N'*-dimethylaniline} [7612-98-8] M 282.4, m 170-171°, $\text{pK}_{\text{Est}} \sim 2.5$. Crystd by dissolving 1g in 150mL of boiling Me_2CO , filtering hot and allowing to cool at -20° overnight collecting the solid and drying in vac. Solns in pyridine should be used immediately otherwise it dec. Moisture sensitive. [Chang *Methods Enzymol* 91 79, 455 1983.]

***p*-Dimethylaminoazobenzene** (Methyl Yellow) [60-11-7] M 225.3, m 118-119°(dec), $\text{pK}_1^{25} -5.34$ (aq H_2SO_4), $\text{pK}_2^{25} 2.96$. Crystd from acetic acid or isooctane, or from 95% EtOH by adding hot water and cooling. Dried over KOH under vacuum at 50° . **CARCINOGEN**.

***p*-Dimethylaminobenzaldehyde** (Ehrlich's Reagent) [100-10-7] M 149.2, m 74-75°, $\text{pK}_{\text{Est}} \sim 2.6$. Crystd from water, hexane, or from EtOH (2mL/g), after charcoal treatment, by adding excess of water. Also dissolved in aqueous acetic acid, filtered, and ppted with ammonia. Finally recrystd from EtOH.

***p*-Dimethylaminobenzoic acid** [619-84-1] M 165.2, m 242.5-243.5°(dec), $\text{pK}_1 2.51$, $\text{pK}_2 6.03$. Crystd from EtOH/water.

***p*-Dimethylaminobenzophenone** [530-44-9] M 225.3, m 92-93°, $\text{pK}_{\text{Est}} \sim 2.7$. Crystd from EtOH.

***N,N*-Dimethylamino-*p*-chlorobenzene** (*p*-chloro-*N,N*-dimethylaniline) [698-69-1] M 155.6, m 32-33.5°, 35.5°, b 231°/atm. Purified by vacuum sublimation [Guarr et al. *J Am Chem Soc* 107 5104 1985]. The *picrate* has m 126-128° (from methanol).

2*S*,3*R*-(+)-4-Dimethylamino-1,2-diphenyl-3-methyl-2-butanol [38345-66-3] M 283.4, m 55-57°, $[\alpha]_{546}^{20} +9.3^\circ$ (c 9.6, EtOH), $[\alpha]_{\text{D}}^{20} +7.7^\circ$ (c 9.6, EtOH), $\text{pK}_{\text{Est}} \sim 10.0$. Purification of the *hydrochloride* by dissolving 1.5g in 13.5 mL of 5N HCl heating to boiling and evaporate in a vacuum. Recrystn of the *hydrochloride* three times from MeOH-EtOAc gives m 189-190°, $[\alpha]_{\text{D}} -33.7^\circ$ (c 1, H_2O) {enantiomer has $+34.2^\circ$ }. The *hydrochloride* in the minimum volume of water is basified with aqueous 5N NaOH and extracted with Et_2O . The extract is dried (K_2CO_3) and evap leaving a residue which is stored in a desiccator over solid KOH as a low melting solid. It can be recovered with these procedures from asymmetric reductions with LAH, and reused. [*J Am Chem Soc* 77 3400 1955; *J Org Chem* 28 2381 2483 1963.]

***dl*-4-Dimethylamino-2,2-diphenylvaleramide** [60-46-8] M 296.4, m 183-184°, $\text{pK}_{\text{Est}} \sim 9.8$. Crystd from aqueous EtOH.

(-)-*L*-4-Dimethylamino-2,2-diphenylvaleramide [6078-64-4] M 296.4, m 136.5-137.5°. Crystd from pet ether or EtOH.

2-Dimethylaminoethanol [108-01-0] M 89.1, b 134.5-135.5°, d 1.430, n 1.4362, $\text{pK}^{25} 9.23$. Dried with anhydrous K_2CO_3 or KOH, and fractionally distd.

1-(3-Dimethylamino-propyl)-3-ethylcarbodiimide hydrochloride (EDCI, DEC, 1-ethyl-3-(3-dimethyl-aminopropyl) carbodiimide hydrochloride) [25952-53-8] M 191.7, m 113.5-114.5°, 114-116°, $\text{pK}_{\text{Est}} \sim 10.3$. An excellent H_2O -soluble peptide coupling reagent. It is purified by dissolving (ca 1g) in CH_2Cl_2 (10mL) at room temperature and then add dry Et_2O (~110mL) dropwise and the crystals that separate are collected, washed with dry Et_2O and recrystd from CH_2Cl_2 - Et_2O and dried in a vacuum over P_2O_5 . It is important to work in a dry atmosphere or work rapidly and then dry the solid as soon as possible. Material is moderately *hygroscopic* but once it becomes wet it reacts slowly with H_2O . Store away from moisture and at -20° to slow down the hydrolysis process. The *free base* has b 47-48°/0.27mm, 53-54°/0.6mm, $n_{\text{D}}^{25} 1.4582$. The *methiodide* is recrystallised from CHCl_3 -EtOAc, the crystals are filtered off, washed with dry Et_2O and recrystd from CHCl_3 - Et_2O , and dried *in vacuo* over P_2O_5 , m 93-95°, 94-95°. [Sheehan et al. *J Am Chem Soc* 87 2492 1965; Sheehan and Cruickshank *Org Synth Coll Vol V* 555 1973.]

§ A polymer bound version is commercially available.

6-Dimethylaminopurine [938-55-6] M 163.1, m 257.5-258.5°, 259-262°, 263-264°, pK₁²⁵ 3.87, pK₂²⁵ 10.5. It is purified by recrystn from H₂O, EtOH (0.32g in 10mL) or CHCl₃. [Albert and Brown *J Chem Soc* 2060 1954; UV: Mason *J Chem Soc* 2071 1954.] The *monohydrochloride* crystallises from EtOH-Et₂O, m 253° (dec) [Elion et al. *J Am Chem Soc* 74 411 1952], the *dihydrochloride* has m 225° (dec) and the *picrate* has m 245° (235-236.5°) [Fryth et al. *J Am Chem Soc* 80 2736 1958].

4-Dimethylaminopyridine (DMAP) [1122-58-3] M 122.2, m 108-109°, b 191°, pK²⁵ 9.61. Recrystd from toluene [Sadownik et al. *J Am Chem Soc* 108 7789 1986].

§ A polystyrene supported version (PS-DMAP) is commercially available.

N,N-Dimethylaniline [121-69-7] M 121.2, f 2°, b 84°/15mm, 193°/760mm, d 0.956, n²⁵ 1.5556, pK²⁵ 5.07. Primary and secondary amines (including aniline and monomethylaniline) can be removed by refluxing for some hours with excess acetic anhydride, and then fractionally distilling. Crocker and Jones (*J Chem Soc* 1808 1959) used four volumes of acetic anhydride, then distd off the greater part of it, and took up the residue in ice-cold dil HCl. Non-basic materials were removed by ether extraction, then the dimethylaniline was liberated with ammonia, extracted with ether, dried, and distd under reduced pressure. Metzler and Tobolsky (*J Am Chem Soc* 76 5178 1954) refluxed with only 10% (w/w) of acetic anhydride, then cooled and poured into excess 20% HCl, which, after cooling, was extracted with diethyl ether. (The amine hydrochloride, remains in the aqueous phase.) The HCl soln was cautiously made alkaline to phenolphthalein, and the amine layer was drawn off, dried over KOH and fractionally distd under reduced pressure, under nitrogen. Suitable drying agents for dimethylaniline include NaOH, BaO, CaSO₄, and CaH₂.

Other purification procedures include the formation of the picrate, prepared in *benzene soln and crystd to constant melting point, then decomposed with warm 10% NaOH and extracted into ether: the extract was washed with water, and distd under reduced pressure. The oxalate has also been used. The base has been fractionally crystd by partial freezing and also from aq 80% EtOH then from absolute EtOH. It has been distd from zinc dust, under nitrogen.

2,6-Dimethylaniline [87-62-7] M 121.2, m 11°, b 210-211°/736mm, d 0.974, n 1.5604, pK²⁵ 3.95. Converted to its hydrochloride which, after recrystn, was decomposed with alkali to give the free base. Dried over KOH and fractionally distd.

3,4-Dimethylaniline [95-64-7] M 121.2, m 51°, b 116-118°/25mm, b 226°/760mm, pK²⁵ 5.17. Crystd from ligroin and distilled under vacuum.

9,10-Dimethylanthracene [781-43-1] M 206.3, m 180-181°. Crystd from EtOH, and by recrystn from the melt.

1,3-Dimethylbarbituric acid [769-42-6] M 156.1, m 123°, pK²⁵ 4.56. Crystd from water and sublimed in a vacuum. Also purified by dissolving 10g in 100mL of boiling CCl₄/CHCl₃ (8:2) (1g charcoal), filtered and cooled to 25°. Dried *in vacuo* [Kohn et al. *Anal Chem* 58 3184 1986].

7,12-Dimethylbenz[a]anthracene [57-97-6] M 256.4, m 122-123°. Purified by chromatography on alumina/toluene or *benzene. Crystd from acetone/EtOH.

5,6-Dimethylbenzimidazole [582-60-5] M 146.2, m 205-206°, pK₁²⁵ 5.96, pK₂²⁵ 12.52. Crystd from diethyl ether. Sublimed at 140°/3mm.

2,3-Dimethylbenzoic acid [603-79-2] M 150.2, m 146°, pK²⁵ 3.72. Crystd from EtOH and is volatile in steam.

2,4-Dimethylbenzoic acid [611-01-8] M 150.2, m 126-127°, b 267°/727mm, pK²⁵ 4.22. Crystd from EtOH, and sublimed in a vacuum.

2,5-Dimethylbenzoic acid [610-72-0] M 150.2, m 134°, b 268°/760mm, pK²⁵ 4.00. Steam distd, and crystd from EtOH.

2,6-Dimethylbenzoic acid [632-46-2] M 150.2, m 117°, pK²⁵ 3.35. Steam distd, and crystd from EtOH.

3,4-Dimethylbenzoic acid [619-04-5] M 150.2, m 166°, pK²⁵ 4.50. Crystd from EtOH and sublimed *in vacuo*.

3,5-Dimethylbenzoic acid [499-06-9] M 150.2, m 170°, pK²⁵ 4.30. Distd in steam, crystd from water or EtOH and sublimed in a vacuum.

4,4'-Dimethylbenzophenone [611-97-2] M 210.3, m 95°, b 333-334°/725mm. Purified by zone refining.

2,5-Dimethyl-1,4-benzoquinone [137-18-8] M 136.1, m 124-125°. Crystd from EtOH.

2,6-Dimethyl-1,4-benzoquinone [527-61-7] M 136.1, m 72° (sealed tube). Crystd from water/EtOH (8:1).

2,3-Dimethylbenzothiophene [31317-17-6] M 212.3, b 123-124°/10mm, n¹⁹ 1.6171. Fractionated through a 90cm Monel spiral column, or other efficient fractionating or spinning band column and collecting the middle fraction.

N,N-Dimethylbenzylamine [103-83-3] M 135.2, b 66-67°/15mm, 181°/760mm, d₄²⁰ 0.898, n_D²⁰ 1.516, pK²⁵ 8.91. See *N*-benzyl dimethylamine on p. 128.

4,4'-Dimethyl-2,2'-bipyridine [1134-35-6] M 184.2, m 175-176°, pK_{Est(1)} ~0.2, pK_{Est(2)} ~4.9. Crystd from ethyl acetate. [Elliott et al. *J Am Chem Soc* 107 4647 1985.]

1,1'-Dimethyl-4,4'-bipyridylum dichloride (3H₂O; Methyl Viologen Dichloride, paraquat dichloride) [1910-42-5] M 311.2, m >300°(dec). Recrystd from MeOH/acetone mixture. Also crystd three times from absolute EtOH [Bancroft et al. *Anal Chem* 53 1390 1981]. Dried at 80° in a vacuum.

N,N-Dimethylbiuret [7710-35-2] M 131.1, m 178°. Purified by repeated crystn from the melt, or H₂O. [Bredereck and Richter *Chem Ber* 99 2461 1968; Dunning and Close *J Am Chem Soc* 75 3615 1953.]

2,3-Dimethyl-1,3-butadiene [513-81-5] M 82.2, m -69-70°, b 68-69°/760mm, d 0.727, n 1.4385. Distd from NaBH₄, and purified by zone melting.

1,3-Dimethylbutadiene sulfone (1,3-dimethylsulfolene) [10033-92-8] M 145.2, m 40.4-41.0°. Crystd from diethyl ether.

2,2-Dimethylbutane [75-83-2] M 86.2, b 49.7°, d 0.649, n²⁵ 1.36595. Distd azeotropically with MeOH, then washed with water, dried and distd.

2,3-Dimethylbutane [79-29-8] M 86.2, b 58.0°, d 1.375, n²⁵ 1.37231. Distd from sodium, passed through a column of silica gel (activated by heating in nitrogen to 350° before use) to remove unsaturated impurities, and again distd from sodium. Also distilled azeotropically with MeOH, then washed with water, dried and redistd.

2,3-Dimethylbut-2-ene [563-79-1] M 84.2, b 72-73°/760mm, d 0.708, n 1.41153. Purified by GLC on a column of 20% squalene on chromosorb P at 50° [Flowers and Rabinovitch *J Phys Chem* 89 563 1985]. Also washed with 1M NaOH soln followed by H₂O. Dried over Na₂SO₄, distd over powdered KOH

under nitrogen and passed through activated alumina before use. [Woon et al. *J Am Chem Soc* **108** 7990 1986; Wong et al. *J Am Chem Soc* **109** 3428 1987.]

Dimethylcarbamoyl chloride [79-44-7] **M 107.5, m -33°, b 34°/0.1mm, d 1.172, n 1.4511.** Must distil under high vacuum to avoid decomposition.

3,3'-Dimethylcarbanilide [620-50-8] **M 240.3, m 225°.** Crystd from ethyl acetate.

Dimethyl carbonate [616-38-5] **M 90.1, m 4.65°, b 90-91°, d 1.070, n 1.369.** Contains small amounts of water and alcohol which form azeotropes. Stood for several days in contact with Linde type 4A molecular sieves, then fractionally distd. The middle fraction was frozen slowly at 2°, several times, retaining 80% of the solvent at each cycle.

cis- and trans-1,4-Dimethylcyclohexane [589-90-2] **M 112.2, b 120°, d 0.788, n 1.427.** Freed from olefins by shaking with conc H₂SO₄, washing with water, drying and fractionally distilling.

1,2-Dimethylcyclohexene [1674-10-8] **M 110.2, b 135-136°/760mm, d 0.826, n 1.4591.** Passed through a column of basic alumina and distd.

1,5-Dimethyl-1,5-diazaundecamethylene polymethobromide (Hexadimethrene, Polybrene) [28728-55-4] **M 5000—10,000 polymer** Purified by chromatography on Dowex 50 and/or by filtration through alumina before use [Frank Hoppe-Seyler's *Z Physiol Chemie* **360** 997 1979]. Hygroscopic, sol in H₂O is 10%.

2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline [4733-39-5] **M 360.5, m >280°, pK_{Est} ~5.6.** Purified by recrystn from *benzene.

Dimethyl disulfide [624-92-0] **M 94.2, f -98°, b 40°/12mm, 110°/760mm, d 1.0605, n 1.5260.** Passed through neutral alumina before use.

2,2-Dimethylethyleneimine [2658-24-4] **M 71.1, b 70.5-71.0°, pK²⁵ 8.64.** Freshly distd from sodium before use.

N,N-Dimethylformamide (DMF) [68-12-2] **M 73.1, b 40°/10mm, 61°/30mm, 88°/100mm, 153°/760mm, d 0.948, n²⁵ 1.4269, pK -0.3.** Decomposes slightly at its normal boiling point to give small amounts of dimethylamine and carbon monoxide. The decomposition is catalysed by acidic or basic materials, so that even at room temperature DMF is appreciably decomposed if allowed to stand for several hours with solid KOH, NaOH or CaH₂. If these reagents are used as dehydrating agents, therefore, they should not be refluxed with the DMF. Use of CaSO₄, MgSO₄, silica gel or Linde type 4A molecular sieves is preferable, followed by distn under reduced pressure. This procedure is adequate for most laboratory purposes. Larger amounts of water can be removed by azeotropic distn with *benzene (10% v/v, previously dried over CaH₂), at atmospheric pressure: water and *benzene distil below 80°. The liquid remaining in the distn flask is further dried by adding MgSO₄ (previously ignited overnight at 300-400°) to give 25g/L. After shaking for one day, a further quantity of MgSO₄ is added, and the DMF distd at 15-20mm pressure through a 3-ft vacuum-jacketed column packed with steel helices. However, MgSO₄ is an inefficient drying agent, leaving about 0.01M water in the final DMF. More efficient drying (to around 0.001-0.007M water) is achieved by standing with powdered BaO, followed by decanting before distn, with alumina powder (50g/L; previously heated overnight to 500-600°), and distilling from more of the alumina; or by refluxing at 120-140° for 24h with triphenylchlorosilane (5-10g/L), then distilling at ca 5mm pressure [Thomas and Rochow *J Am Chem Soc* **79** 1843 1957]. Free amine in DMF can be detected by colour reaction with 1-fluoro-2,4-dinitrobenzene. It has also been purified by drying overnight over KOH pellets and then distd from BaO through a 10 cm Vigreux column [*Exp Cell Res* **100** 213 1976]. [For efficiency of desiccants in drying dimethylformamide see Burfield and Smithers [*J Org Chem* **43** 3966 1978, and for a review on purification, tests of purity and physical properties, see Juillard *Pure Appl Chem* **49** 885 1977].

It has been purified by distilling from K_2CO_3 under high vac and fractionated in an all-glass apparatus. The middle fraction is collected, degassed (seven or eight freeze-thaw cycles) and redistd under as high a vacuum as possible [Mohammad and Kosower *J Am Chem Soc* **93** 2713 1971].

Rapid purification: Stir over CaH_2 (5% w/v) overnight, filter, then distil at 20mmHg. Store the distd DMF over 3A or 4A molecular sieves. For solid phase synthesis, the DMF used must be of high quality and free from amines.

***d,l*-2,4-Dimethylglutaric acid** [2121-67-7] M 160.2, m 144-145° $pK_{Est(1)} \sim 4.4$, $pK_{Est(2)} \sim 5.4$. Distd in steam and crystd from ether/pet ether.

3,3-Dimethylglutaric acid [4839-46-7] M 160.2, m 103-104°, b 89-90°/2mm, 126-127°/4.5mm, $pK_1^{25} 3.85$, $pK_2^{25} 6.45$. Crystd from water, *benzene or ether/pet ether. Dried in a vacuum.

3,3-Dimethylglutarimide [1123-40-6] M 141.2, m 144-146°. Recrystd from EtOH [Arnett and Harrelson *J Am Chem Soc* **109** 809 1987].

***N,N*-Dimethylglycinehydrazide hydrochloride** [539-64-0] M 153.6, m 181°. Crystd by adding EtOH to a conc aqueous soln.

Dimethylglyoxime [95-45-4] M 116.1, m 240°, $pK_1^{25} 10.60$, $pK_2^{25} 11.85$. Crystd from EtOH (10mL/g) or aqueous EtOH. **TOXIC.**

2,5-Dimethyl-2,4-hexadiene [764-13-6] M 110.2, f 14.5°, b 132-134°, d 0.773, n 1.4796. Distd, then repeatedly fractionally crystd by partial freezing. Immediately before use, the material was passed through a column containing Woelm silica gel (activity I) and Woelm alumina (neutral) in separate layers.

2,2-Dimethylhexane [590-73-8] M 114.2, m -121.2°, b 107°, d 0.695. Dried over type 4A molecular sieves and distd.

2,5-Dimethylhexane [592-13-2] M 114.2, m -91.2°, b 109°, d 0.694. Dried over type 4A molecular sieves and distd.

2,5-Dimethylhexane-2,5-diol [110-03-2] M 146.2, m 88-90°. Purified by fractional crystn. Then the diol was dissolved in hot acetone, treated with activated charcoal, and filtered while hot. The soln was cooled and the diol was filtered off and washed well with cold acetone. The crystn process was repeated several times and the crystals were dried under a vac in a freeze-drying apparatus [Goates et al. *J Chem Soc, Faraday Trans 1* **78** 3045 1982].

5,5-Dimethylhydantoin [77-71-4] M 128.1, m 177-178° $pK^{24} 9.19$. Crystd from EtOH and sublimed *in vacuo*.

1,1-Dimethylhydrazine [57-14-7] M 60.1, b 60.1°/702mm, d 0.790, n 1.408 $pK^{30} 7.21$. Fractionally distd through a 4-ft column packed with glass helices. Pptd as its oxalate from diethyl ether soln. After crystn from 95% EtOH, the salt was decomposed with aqueous saturated NaOH, and the free base was distd, dried over BaO and redistd [McBride and Kruse *J Am Chem Soc* **79** 572 1957]. Distn and storage should be under nitrogen.

4,6-Dimethyl-2-hydroxypyrimidine [108-79-2] M 124.1, m 198-199°, $pK_1^{20} 3.77$, $pK_2^{20} 10.50$. Crystd from absolute EtOH (charcoal).

1,2-Dimethylimidazole [1739-84-0] M 96.1, m 38-40°, b 206°/760mm, d 1.084, $pK_{Est} \sim 8.1$. Crystd from *benzene and stored at 0-4°. [Gorun et al. *J Am Chem Soc* **109** 4244 1987.]

1,1-Dimethyl-1*H*-indene [18636-55-0] M 144.2, b 57°/4.8mm, 115°/20mm. Purified by gas chromatography or by fractional distn.

Dimethyl itaconate [617-52-7] M 158.2, m 38°, b 208°, d 1.124. Crystd from MeOH by cooling to -78°.

Dimethylmaleic anhydride [766-39-2] M 126.1, m 96°, b 225°/760mm. Distd from *benzene/ligroin and sublimed in a vacuum.

Dimethylmalonic acid [595-46-0] M 132.1, m 192-193° pK₁²⁵ 3.03, pK₂²⁵ 5.73. Crystd from *benzene/pet ether and sublimed in a vacuum with slight decomposition.

1,5-Dimethylnaphthalene [571-61-9] M 156.2, m 81-82°, b 265-266°. Crystd from 85% aq EtOH.

2,3-Dimethylnaphthalene [581-40-8] M 156.2, m 104-104.5°. Steam distd and crystd from EtOH.

2,6-Dimethylnaphthalene [581-42-0] M 156.2, m 110-111°, b 122.5-123.5°/10mm, 261-262°/760mm. Distd in steam and crystd from EtOH.

3,3'-Dimethylnaphthidine (4,4'-diamino-3,3'-dimethyl-1,1'-binaphthyl) [13138-48-2] M 312.4, m 213°. Recrystd from EtOH or pet ether (b 60-80°).

***N,N*-Dimethyl-*m*-nitroaniline** [619-31-8] M 166.1, m 60°, pK²⁵ 2.63. Crystd from EtOH.

***N,N*-Dimethyl-*p*-nitroaniline** [100-23-2] M 166.1, m 164.5-165.2°, pK²⁵ 0.61 (0.92). Crystd from EtOH or aqueous EtOH. Dried under vacuum.

Dimethylnitrosamine (*N*-nitrosodimethylamine) [62-75-9] M 74.0, m -28°, b 149-150°/atm, 153°/774mm, d. 1.006, n 1.4370. Dry over anhyd K₂CO₃ or dissolve in Et₂O, dry over solid KOH, filter, evap Et₂O and distil yellow oily residue through a 30cm fractionating column discarding the first fraction which may contain Me₂N. Also dried over CaCl₂ and distd at atm pressure. All should be done in an efficient fume cupboard as the vapors are **TOXIC** and **CARCINOGENIC**. [Fischer *Chem Ber* 8 1588 1875; Romberg *Recl Trav Chim, Pays-Bas* 5 248 1886; Hatt *Org Synth Coll Vol II* 211 1961; Krebs and Mandt *Chem Ber* 108 1130 1975.]

***N,N*-Dimethyl-*p*-nitrosoaniline** [138-89-6] M 150.2, m 86-87°. See 4-nitroso-*N,N*-dimethylaniline on p. 314.

***N,N*-Dimethyl-*p*-nitrosoaniline hydrochloride** [42344-05-8] M 186.7, m 177°. Crystd from hot water in the presence of a little HCl.

2,6-Dimethyl-2,4,6-octatriene [7216-56-0; *cis/trans* mixt 673-84-7; *trans, trans* 3016-19-1] M 136.2, b 80-82°/15mm, ε_{278nm} 42,870. Repeated distn at 15mm through a long column of glass helices, the final distn being from sodium under nitrogen. See *neo*-allocimene on p. 100.

Dimethylolurea [140-95-4] M 120.1, m 137-139°. Crystd from aqueous 75% EtOH.

Dimethyl oxalate [553-90-2] M 118.1, m 54°, b 163-165°, d 1.148. Crystd repeatedly from EtOH. Degassed under nitrogen high vacuum and distd.

3,3-Dimethyloxetane [6921-35-3] M 86.1, b 79.2-80.3°/760mm. Purified by gas chromatography using a 2m silicone oil column.

2,3-Dimethylpentane [565-59-3] M 100.2, b 89.8°, d 0.695, n 1.39197, n²⁵ 1.38946. Purified by azeotropic distn with EtOH, followed by washing out the EtOH with water, drying and distn [Streiff et al. *J Res Nat Bur Stand* 37 331 1946].

2,4-Dimethylpentane [108-08-7] **M 100.2, b 80.5°, d 0.763, n 1.3814, n²⁵ 1.37882.** Extracted repeatedly with conc H₂SO₄, washed with water, dried and distd. Percolated through silica gel (previously heated in nitrogen to 350°). Purified by azeotropic distn with EtOH, followed by washing out the EtOH with water, drying and distn.

4,4-Dimethyl-1-pentene [762-62-9] **M 98.2, b 72.5°/760mm, d 0.6827, n 1.3918.** Purified by passage through alumina before use [Traylor et al. *J Am Chem Soc* **109** 3625 1987].

Dimethyl peroxide [690-02-8] **M 62.1, b 13.5°/760mm, d 0.8677, n 1.3503.** Purified by repeated trap-to-trap fractionation until no impurities could be detected by gas IR spectroscopy [Haas and Oberhammer *J Am Chem Soc* **106** 6146 1984]. *All necessary precautions should be taken in case of EXPLOSION.*

2,9-Dimethyl-1,10-phenanthroline [484-11-7] **M 208.3, m 162-164°, pK²⁵ 5.85.** Purified as hemihydrate from water, and as anhydrous from *benzene.

R-(+)-N,N'-Dimethyl-1-phenethylamine [19342-01-9] and **S-(-)-N,N'-Dimethyl-1-phenethylamine** [17279-31-1] **M 149.2, b 81°/16mm, [α]_D²⁰ (+) and (-) 50.2° (c 1, MeOH), [α]_D²⁶ +61.8° and -64.4° (neat l 1), d 0.908, pK_{Est} ~9.0 (for RS).** The amine is mixed with aqueous 10N NaOH and extracted with toluene. The extract is washed with saturated aqueous NaCl and dried over K₂CO₃, and transferred to fresh K₂CO₃ until the soln is clear, and filtered. The filtrate is distd. If a short column packed with glass helices is used, the yield is reduced but a purer product is obtained. [*Org Synth* **25** 89 1945; *J Am Chem Soc* **71** 291 3929 3931 4165 1949.] The (-)-picrate has m 140-141° (cryst from EtOH). The racemate [1126-71-2] has b 88-89°/16mm, 92-94°/30mm, 194-195°/atm, d₄²⁰ 0.908.

2,3-Dimethylphenol [526-75-0] **M 122.2, m 75°, b 120°/20mm, 218°/760mm, pK²⁵ 10.54.** Crystd from aqueous EtOH.

2,5-Dimethylphenol [95-87-1] **M 122.2, m 73°, b 211.5°/762mm, pK²⁵ 10.41.** Crystd from EtOH/ether.

2,6-Dimethylphenol [576-26-1] **M 122.2, m 49°, b 203°/760mm, pK²⁵ 10.61.** Fractionally distd under nitrogen, crystd from *benzene or hexane, and sublimed at 38°/10mm.

3,4-Dimethylphenol [95-65-8] **M 122.2, m 65°, b 225°/757mm, pK²⁵ 10.36.** Heated with an equal weight of conc H₂SO₄ at 103-105° for 2-3h, then diluted with four volumes of water, refluxed for 1h, and either steam distd or extracted repeatedly with diethyl ether after cooling to room temperature. The steam distillate was also extracted and evaporated to dryness. (The purification process depends on the much slower sulfonation of 3,5-dimethylphenol than most of its likely contaminants.) It can also be crystd from water, hexane or pet ether, and vacuum sublimed. [Kester *Ind Eng Chem (Anal Ed)* **24** 770 1932; Bernasconi and Paschalis *J Am Chem Soc* **108** 2969 1986.]

3,5-Dimethylphenol [108-68-9] **M 122.2, m 68°, b 219°, pK²⁵ 10.19.** Purification as for 3,4-dimethylphenol.

Dimethyl phthalate [131-11-3] **M 194.2, b 282°, n 1.5149, d 1.190, d²⁵ 1.1865.** Washed with aqueous Na₂CO₃, then distilled water, dried (CaCl₂) and distd under reduced pressure (b 151-152°/0.1mm).

2,2-Dimethyl-1,3-propanediol (neopentyl glycol) [126-30-7] **M 104.2, m 128.4-129.4°, b 208°/760mm.** Crystd from *benzene or acetone/water (1:1).

2,2-Dimethyl-1-propanol (neo-pentyl alcohol) [75-84-3] **M 88.2, m 52°, b 113.1°/760mm.** Difficult to distil because it is a solid at ambient temperatures. Purified by fractional crystallisation and sublimation.

***N,N*-Dimethylpropionamide** [758-96-3] M 101.2, b 175-178°, d 0.920, n 1.440. Shaken over BaO for 1-2 days, then distd at reduced pressure.

2,5-Dimethylpyrazine [123-32-0] M 108.1, b 156°, d 0.990, n 1.502, $pK_1^{2.5}$ -4.6 (aq H_2SO_4), $pK_2^{2.5}$ 1.85. Purified *via* its picrate (m 150°) [Wiggins and Wise *J Chem Soc* 4780 1956].

3,5-Dimethylpyrazole [67-51-6] M 96.1, m 107-108°, $pK^{2.0}$ 4.16. Crystd from cyclohexane or water. [Barszez et al. *J Chem Soc, Dalton Trans* 2025 1986.]

2,3-Dimethylquinoxaline [2379-55-7] M 158.2, m 106°, $pK^{2.5}$ -3.84 (aq H_2SO_4). Crystd from distilled water.

2,4-Dimethylresorcinol [634-65-1] M 138.1, m 149-150° $pK_{Est(1)}$ ~9.8, $pK_{Est(2)}$ ~11.7. Crystd from pet ether (b 60-80°).

***meso*- α,β -Dimethylsuccinic acid** [608-40-2] M 146.1, m 211°, $pK_1^{2.5}$ 3.77, $pK^{2.5}$ 5.36. Crystd from EtOH/ether or EtOH/chloroform.

2,2-Dimethylsuccinic acid [597-43-3] M 146.1, m 141°, $pK_1^{2.0}$ 4.15, $pK_2^{2.0}$ 6.40. Crystd from EtOH/ether or EtOH/chloroform.

(\pm)-2,3-Dimethylsuccinic acid [13545-04-5] M 146.1, m 129°, $pK_1^{2.5}$ 3.82, $pK_2^{2.5}$ 5.98. Crystd from water.

Dimethyl sulfide [75-18-3] M 62.1, f -98.27°, b 0°/172mm, 37.5-38°/760mm, d^{21} 0.8458, n^{25} 1.4319. Purified *via* the Hg(II) chloride complex by dissolving 1 mole of Hg(II)Cl₂ in 1250mL of EtOH and slowly adding the boiling alcoholic soln of dimethyl sulfide to give the right ratio for 2(CH₃)₂S.3HgCl₂. After recrystn of the complex to constant melting point, 500g of complex is heated with 250mL conc HCl in 750mL of water. The sulfide is separated, washed with water, and dried with CaCl₂ and CaSO₄. Finally, it is distd under reduced pressure from sodium. Precautions should be taken (*efficient fume hood*) because of its very UNPLEASANT ODOUR.

2,4-Dimethylsulfolane [1003-78-7] M 148.2, b 128°/77mm, d^{25} 1.1314. Vacuum distd.

Dimethyl sulfone [67-71-0] M 94.1, m 109°. Crystd from water. Dried over P₂O₅.

Dimethyl sulfoxide (DMSO) [67-68-5] M 78.1, m 18.0-18.5°, b 75.6-75.8°/12mm, 190°/760mm, d 1.100, n 1.479. Colourless, odourless, very *hygroscopic* liquid, synthesised from dimethyl sulfide. The main impurity is water, with a trace of dimethyl sulfone. The Karl-Fischer test is applicable. It is dried with Linde types 4A or 13X molecular sieves, by prolonged contact and passage through a column of the material, then distd under reduced pressure. Other drying agents include CaH₂, CaO, BaO and CaSO₄. It can also be fractionally crystd by partial freezing. More extensive purification is achieved by standing overnight with freshly heated and cooled chromatographic grade alumina. It is then refluxed for 4h over CaO, dried over CaH₂, and then fractionally distd at low pressure. For efficiency of desiccants in drying dimethyl sulfoxide see Burfield and Smithers [*J Org Chem* 43 3966 1978; Sato et al. *J Chem Soc, Dalton Trans* 1949 1986].

Rapid purification: Stand over freshly activated alumina, BaO or CaSO₄ overnight. Filter and distil over CaH₂ under reduced pressure (~ 12 mm Hg). Store over 4A molecular sieves.

Dimethyl terephthalate [120-61-6] M 194.2, m 150°. Purified by zone melting.

***N,N*-Dimethylthiocarbamoyl chloride** [16420-13-6] M 123.6, m 42-43°, b 64-65°/0.1mm. Crystd twice from pentane.

***N,N*-Dimethyl-*o*-toluidine** [609-72-3] M 135.2, b 68°/10mm, 211-211.5°/760mm, d 0.937, n 1.53664, pK²⁵ 5.85. Isomers and other bases have been removed by heating in a water bath for 100h with two equivalents of 20% HCl and two and a half volumes of 40% aq formaldehyde, then making the soln alkaline and separating the free base. After washing well with water it was distd at 10mm pressure and redistd at ambient pressure [von Braun and Aust *Chem Ber* 47 260 1914]. Other procedures include drying with NaOH, distilling from zinc in an atmosphere of nitrogen under reduced pressure, and refluxing with excess of acetic anhydride in the presence of conc H₂SO₄ as catalyst, followed by fractional distn under vacuum.

***N,N*-Dimethyl-*m*-toluidine** [121-72-2] M 135.2, b 211.5-212.5°, d 0.93, pK²⁵ 5.22. See *m*-methyl-*N,N*-dimethylaniline on p. 291.

***N,N*-Dimethyl-*p*-toluidine** [99-97-8] M 135.2, b 93-94°/11mm, b 211°, d 0.937, n 1.5469, pK²⁵ 4.76. See *p*-methyl-*N,N*-dimethylaniline on p. 291.

1,3-Dimethyluracil [874-14-6] M 140.1, m 121-122°, pK -3.25 (aq H₂SO₄). Crystd from EtOH/ether.

***sym*-Dimethylurea** [96-31-1] M 88.1, m 106°. Crystd from acetone/diethyl ether by cooling in an ice bath. Also crystd from EtOH and dried at 50° and 5mm for 24h [Bloemendahl and Somsen *J Am Chem Soc* 107 3426 1985].

2,2'-Dinaphthylamine [532-18-3] M 269.3, m 170.5°, pK_{Est} <0. Crystd from *benzene.

2,4-Dinitroaniline [97-02-9] M 183.1, m 180°, ε₃₄₈ 12,300 in dil aq HClO₄, pK²⁵ -4.27 (aq H₂SO₄). Crystd from boiling EtOH by adding one-third volume of water and cooling slowly. Dried in a steam oven.

2,6-Dinitroaniline [606-22-4] M 183.1, m 139-140°, pK²⁵ -5.37 (aq H₂SO₄). Purified by chromatography on alumina, then crystd from *benzene or EtOH.

2,4-Dinitroanisole [5327-44-6] M 198.1, m 94-95°. Crystd from aq EtOH.

3,5-Dinitroanisole [119-27-7] M 198.1, m 105-106°. Purified by repeated crystn from water and dried in a vacuum desiccator over P₂O₅.

1,2-Dinitrobenzene [528-29-0] M 168.1, m 116.5°. Crystd from EtOH.

1,3-Dinitrobenzene [99-65-0] M 168.1, m 90.5-91°. Crystd from alkaline EtOH soln (20g in 750mL 95% EtOH at 40°, plus 100mL of 2M NaOH) by cooling and adding 2.5L of water. The ppte, after filtering off, washing with water and sucking dry, was crystd from 120mL, then 80mL of absolute EtOH [Callow, Callow and Emmens *Biochem J* 32 1312 1938]. Has also been crystd from MeOH, CCl₄ and ethyl acetate. Can be sublimed in a vacuum. [Tanner *J Org Chem* 52 2142 1987.]

1,4-Dinitrobenzene [100-25-4] M 168.1, m 173°. Crystd from EtOH or ethyl acetate. Dried under vacuum over P₂O₅. Can be sublimed in a vacuum.

2,4-Dinitrobenzenesulfonyl chloride [528-76-7] M 234.6, m 96°. Crystd from CCl₄.

2,4-Dinitrobenzenesulfonyl chloride [1656-44-6] M 266.6, m 102°. Crystd from *benzene or *benzene/pet ether.

2,4-Dinitrobenzoic acid [610-30-3] M 212.1, m 183°, pK²⁵ 1.42. Crystd from aqueous 20% EtOH (10mL/g), dried at 100°.

- 2,5-Dinitrobenzoic acid** [610-28-6] M 212.1, m 179.5-180°, pK^{25} 1.62. Crystd from distd water. Dried in a vacuum desiccator.
- 2,6-Dinitrobenzoic acid** [603-12-3] M 212.1, m 202-203°, pK^{25} 1.14. Crystd from water.
- 3,4-Dinitrobenzoic acid** [528-45-0] M 212.1, m 166°, pK^{25} 2.81. Crystd from EtOH by addition of water.
- 3,5-Dinitrobenzoic acid** [99-34-3] M 212.1, m 205°, pK^{25} 2.73 (2.79). Crystd from distilled water or 50% EtOH (4mL/g). Dried in a vacuum desiccator or at 70° over BaO under vacuum for 6h.
- 4,4'-Dinitrobenzoic anhydride** [902-47-6] M 406.2, m 189-190°. Crystd from acetone.
- 3,5-Dinitrobenzoyl chloride** [99-33-2] M 230.6, m 69.5°. Crystd from CCl₄ or pet ether (b 40-60°). It reacts readily with water, and should be kept in sealed tubes or under dry pet ether.
- 2,2'-Dinitrobiphenyl** [2436-96-6] M 244.2, m 123-124°. Crystd from EtOH.
- 2,4'-Dinitrobiphenyl** [606-81-5] M 244.2, m 92.7-93.7°. Crystd from EtOH.
- 4,4'-Dinitrobiphenyl** [1528-74-1] M 244.2, m 240.9-241.8°. Crystd from *benzene, EtOH (charcoal) or acetone. Dried under vacuum over P₂O₅.
- 2,6-Dinitro-*p*-cresol (2,6-dinitro-4-methylphenol)** [609-93-3] M 198.1, m 78-79°, pK_{Est} ~3.7. Recrystd from EtOH and is steam volatile. **TOXIC IRRITANT.**
- 4,6-Dinitro-*o*-cresol** [534-52-1] M 198.1, m 85-86°, 87°, pK^{25} 4.70. Crystd from aqueous EtOH.
- 2,4-Dinitrodiphenylamine** [961-68-2] M 259.2, m 157°, pK_{Est} <0. Crystd from aqueous EtOH.
- 4,4'-Dinitrodiphenylurea** [587-90-6] M 302.2, m 312°(dec). Crystd from EtOH. Sublimes in vac.
- 2,4-Dinitrofluorobenzene (Sanger's reagent)** [70-34-8] M 186.1, m 25-27°, b 133°/2mm, 140-141°/5mm, d 1.483. Crystd from ether or EtOH. Vacuum distd through a Todd Column (see p. 174). If it is to be purified by distn *in vacuo*, the distn unit must be allowed to cool before air is allowed into the apparatus otherwise the residue carbonises spontaneously and an **EXPLOSION** may occur. The material is a **skin irritant** and may cause serious dermatitis.
- 1,8-Dinitronaphthalene** [602-38-0] M 218.2, m 170-171°. Crystd from *benzene.
- 2,4-Dinitro-1-naphthol (Martius Yellow)** [605-69-6] M 234.2, m 81-82°, pK_{Est} ~3.7. Crystd from *benzene or aqueous EtOH.
- 2,4-Dinitrophenetole** [610-54-8] M 240.2, m 85-86°. Crystd from aqueous EtOH.
- 2,4-Dinitrophenol** [51-28-5] M 184.1, m 114°, pK^{25} 4.12. Crystd from *benzene, EtOH, EtOH/water or water acidified with dil HCl, then recrystd from CCl₄. Dried in an oven and stored in a vac desiccator over CaSO₄.
- 2,5-Dinitrophenol** [329-71-5] M 184.1, m 108°, pK^{25} 5.20. Crystd from H₂O with a little EtOH.
- 2,6-Dinitrophenol** [573-56-8] M 184.1, m 63.0-63.7°, pK^{25} 3.73. Crystd from *benzene/cyclohexane, aqueous EtOH, water or *benzene/pet ether (b 60-80°, 1:1).

3,4-Dinitrophenol [577-71-9] M 184.1, m 138°, pK²⁵ 5.42. Steam distd and crystd from water and air-dried. **CAUTION - EXPLOSIVE** when dry, store with 10% water.

3,5-Dinitrophenol [586-11-8] M 184.1, m 126°, pK²⁵ 6.68. Crystd from *benzene or CHCl₃/pet ether. Should be stored with 10% water because it is **EXPLOSIVE** when dry.

2,4-Dinitrophenylacetic acid [643-43-6] M 226.2, m 179°(dec), pK²⁵ 3.50. Crystd from water.

2,4-Dinitrophenylhydrazine (DNPH) [119-26-6] M 198.1, m 200°(dec), pK_{Est} ~2.0. Crystd from butan-1-ol, dioxane, EtOH, *C₆H₆ or ethyl acetate. HCl has m 186°(dec).

2,2-Dinitropropane [595-49-3] M 162.1, m 53.5°. Crystd from EtOH or MeOH. Dried over CaCl₂ or under vacuum for 1h just above the melting point.

2,4-Dinitroresorcinol [519-44-8] M 200.1, m 149°, pK²⁵ 3.05. Crystd from aq EtOH. **Explosive.**

3,5-Dinitrosalicylic acid [609-99-4] M 228.1, m 173-174°, pK₁²⁵ 0.70, pK₂²⁵ 7.40. Crystd from water.

2,6-Dinitrothymol [303-21-9] M 240.2, m 53-54°. Crystd from aq EtOH.

2,3-Dinitrotoluene [602-01-7] M 182.1, m 63°. Distd in steam and crystd from water or *benzene/pet ether. Stored with 10% water. *Could be EXPLOSIVE when dry.*

2,4-Dinitrotoluene [121-14-2] M 182.1, m 70.5-71.0°. Crystd from acetone, isopropanol or MeOH. Dried under vacuum over H₂SO₄. Purified by zone melting. *Could be EXPLOSIVE when dry.*

2,5-Dinitrotoluene [619-15-8] M 182.1, m 51.2°. Crystd from *benzene.

2,6-Dinitrotoluene [606-20-2] M 182.1, m 64.3°. Crystd from acetone.

3,4-Dinitrotoluene [610-39-9] M 182.1, m 61°. Distil in steam and cryst from *benzene/pet ether. Store with 10% of water to avoid **EXPLOSION**.

3,5-Dinitro-*o*-toluic acid [28169-46-2] M 226.2, m 206°, pK_{Est} ~3.0. Crystd from H₂O or aq EtOH.

2,4-Dinitro-*m*-xylene [603-02-1] M 196.2, m 83-84°. Crystd from EtOH.

Dinonyl phthalate (mainly 3,5,5-trimethylhexyl phthalate isomer) [28553-12-0; 14103-61-8] M 418.6, m 26-29°, b 170°/2mm, d 0.9640, n 1.4825. Washed with aqueous Na₂CO₃, then shaken with water. Ether was added to break the emulsion, and the soln was washed twice with water, and dried (CaCl₂). After evaporating the ether, the residual liquid was distd three times under reduced pressure. It was stored in a vacuum desiccator over P₂O₅

Diocetyltrimethylammonium bromide [3700-67-2] M 630.9, m 161-163°. Crystd from acetone then MeOH [Lukac *J Am Chem Soc* 106 4387 1984]. Also purified by chromatography on alumina by washing with *C₆H₆ and eluting with Me₂CO, evap and cryst from MeCN [Swain and Kreevoy *J Am Chem Soc* 77 1126 1955].

Diosgenin [512-04-9] M 294.5, m 204-207°, [α]_D²⁵ -129° (in Me₂CO). Crystd from acetone.

1,3-Dioxane [505-22-6] M 88.1, b 104.5°/751mm, d 1.040, n 1.417. Dried with sodium and fractionally distd.

1,4-Dioxane [123-91-1] **M 88.1, f 11.8°, b 101.3°, d²⁵ 1.0292, n¹⁵ 1.4236, n²⁵ 1.42025.**

Prepared commercially either by dehydration of ethylene glycol with H₂SO₄ and heating ethylene oxide or bis(β-chloroethyl)ether with NaOH. Usual impurities are acetaldehyde, ethylene acetal, acetic acid, water and peroxides. Peroxides can be removed (and the aldehyde content decreased) by percolation through a column of activated alumina (80g per 100-200mL solvent), by refluxing with NaBH₄ or anhydrous stannous chloride and distilling, or by acidification with conc HCl, shaking with ferrous sulfate and leaving in contact with it for 24h before filtering and purifying further.

Hess and Frahm [*Chem Ber* 71 2627 1938] refluxed 2L of dioxane with 27mL conc HCl and 200mL water for 12h with slow passage of nitrogen to remove acetaldehyde. After cooling the soln KOH pellets were added slowly and with shaking until no more would dissolve and a second layer had separated. The dioxane was decanted, treated with fresh KOH pellets to remove any aq phase, then transferred to a clean flask where it was refluxed for 6-12h with sodium, then distd from it. Alternatively, Kraus and Vingee [*J Am Chem Soc* 56 511 1934] heated on a steam bath with solid KOH until fresh addition of KOH gave no more resin (due to acetaldehyde). After filtering through paper, the dioxane was refluxed over sodium until the surface of the metal was not further discoloured during several hours. It was then distd from sodium.

The acetal (b 82.5°) is removed during fractional distn. Traces of *benzene, if present, can be removed as the *benzene/MeOH azeotrope by distn in the presence of MeOH. Distn from LiAlH₄ removes aldehydes, peroxides and water. Dioxane can be dried using Linde type 4X molecular sieves. Other purification procedures include distn from excess C₂H₅MgBr, refluxing with PbO₂ to remove peroxides, fractional crystn by partial freezing and the addition of KI to dioxane acidified with aq HCl. Dioxane should be stored out of contact with air, preferably under N₂.

A detailed purification procedure is as follows: Dioxane was stood over ferrous sulfate for at least 2 days, under nitrogen. Then water (100mL) and conc HCl (14mL) / litre of dioxane were added (giving a pale yellow colour). After refluxing for 8-12h with vigorous N₂ bubbling, pellets of KOH were added to the warm soln to form two layers and to discharge the colour. The soln was cooled rapidly with more KOH pellets being added (magnetic stirring) until no more dissolved in the cooled soln. After 4-12h, if the lower phase was not black, the upper phase was decanted rapidly into a clean flask containing sodium, and refluxed over sodium (until freshly added sodium remained bright) for 1h. The middle fraction was collected (and checked for minimum absorbency below 250nm). The distillate was fractionally frozen three times by cooling in a refrigerator, with occasional shaking or stirring. This material was stored in a refrigerator. For use it was thawed, refluxed over sodium for 48h, and distilled into a container for use. All joints were clad with Teflon tape.

Coetzee and Chang [*Pure Appl Chem* 57 633 1985] dried the solvent by passing it slowly through a column (20g/L) of 3A molecular sieves activated by heating at 250° for 24h. Impurities (including peroxides) were removed by passing the effluent slowly through a column packed with type NaX zeolite (pellets ground to 0.1mm size) activated by heating at 400° for 24h or chromatographic grade basic Al₂O₃ activated by heating at 250° for 24h. After removal of peroxides the effluent was refluxed several hours over sodium wire, excluding moisture, distilled under nitrogen or argon and stored in the dark.

One of the best tests of purity of dioxane is the formation of the purple disodium benzophenone complex during reflux and its persistence on cooling. (Benzophenone is better than fluorenone for this purpose, and for the storing of the solvent.) [Carter, McClelland and Warhurst *Trans Faraday Soc* 56 343 1960]. **TOXIC.**

Rapid purification: Check for peroxides (see Chapter 1 and Chapter 2 for test under ethers). Pre-dry with CaCl₂ or better over Na wire. Then reflux the pre-dried solvent over Na (1% w/v) and benzophenone (0.2% w/v) under an inert atmosphere until the blue colour of the benzophenone ketyl radical anion persists. Distil, and store over 4A molecular sieves in the dark.

1,3-Dioxolane [646-06-0] **M 74.1, b 75.0-75.2°, d 1.0600, n²¹ 1.3997.** Dried with solid NaOH, KOH or CaSO₄, and distd from sodium or sodium amalgam. Barker et al. [*J Chem Soc* 802 1959] heated 34mL of dioxolane under reflux with 3g of PbO₂ for 2h, then cooled and filtered. After adding xylene (40mL) and PbO₂ (2g) to the filtrate, the mixture was fractionally distd. Addition of xylene (20mL) and sodium wire to the main fraction (b 70-71°) led to vigorous reaction, following which the mixture was again fractionally distd. Xylene and sodium additions were made to the main fraction (b 73-74°) before it was finally distd.

4,4'-Di-*n*-pentyloxyazoxybenzene [64242-26-8] **M 370.5.** Crystd from Me₂CO, and dried by heating under vacuum.

Diphenic acid [482-05-3] M 242.2, m 228-229°, pK²⁵ 3.46. Crystd from water.

Diphenic anhydride [6050-13-1] M 466.3, m 217°. After removing free acid by extraction with cold aq Na₂CO₃, the residue has been crystd from acetic anhydride and dried at 100°. Acetic anhydride also converts the acid to the anhydride.

***N,N*-Diphenylacetamide** [621-09-0] M 210.3, m 131°. Crystd from EtOH, then sublimed under vacuum at ca 96° onto a "finger" cooled in solid CO₂/MeOH, with continuous pumping to free it from occluded solvent.

Diphenylacetic acid [117-34-0] M 212.3, m 147.4-148.4°, pK²⁵ 3.94. Crystd from *benzene, H₂O or aq 50% EtOH.

Diphenylacetonitrile [86-29-3] M 193.3, m 73-75°. Crystd from EtOH or pet ether (b 90-100°).

Diphenylacetylene (tolan) [501-65-5] M 178.2, m 62.5°, b 90-97°/0.3mm. Crystd from EtOH.

Diphenylamine [122-39-4] M 169.2, m 62.0-62.5°, pK²⁵ 0.77 (aq H₂SO₄). Crystd from pet ether, MeOH, or EtOH/water. Dried under vacuum.

Diphenylamine-2-carboxylic acid [91-40-7] M 213.2, m 184°, pK₁²⁵ -1.28 (aq H₂SO₄), pK₂²⁵ 3.86. See *N*-phenylanthranilic acid on p. 327.

Diphenylamine-2,2'-dicarboxylic acid (2,2'-iminodibenzoic acid) [579-92-0] M 257.2, m 298°(dec), pK_{Est} ~3.7. Crystd from EtOH.

9,10-Diphenylanthracene [1499-10-1] M 330.4, m 248-249°. Crystd from acetic acid or xylene [Baumstark et al. *J Org Chem* 52 3308 1987].

***N,N'*-Diphenylbenzidine** [531-91-9] M 336.4, m 245-247°, 251-252°, pK²⁵ 0.30. Crystd from toluene or ethyl acetate. Stored in the dark.

***trans-trans*-1,4-Diphenylbuta-1,3-diene** [538-81-8] M 206.3, m 153-153.5°. Its soln in pet ether (b 60-70°) was chromatographed on an alumina-Celite column (4:1) and the column was washed with the same solvent. The main zone was cut out, eluted with ethanol and transferred to pet ether, which was then dried and evaporated [Pinckard, Wille and Zechmesiter *J Am Chem Soc* 70 1938 1948]. Recrystd from hexane.

***sym*-Diphenylcarbazine** [140-22-7] M 242.3, m 172°. A common impurity is phenylsemicarbazide which can be removed by chromatography [Willems et al. *Anal Chim Acta* 51 544 1970]. Crystd from EtOH or glacial acetic acid.

1,5-Diphenylcarbazon [538-62-5] M 240.3, m 124-127°. Crystd from EtOH (ca 5mL/g), and dried at 50°. A commercial sample, nominally *sym*-diphenylcarbazon but of m 154-156°, was a mixture of diphenylcarbazine and diphenylcarbazon. The former was removed by dissolving 5g of the crude material in 75mL of warm EtOH, then adding 25g Na₂CO₃ dissolved in 400mL of distd water. The alkaline soln was cooled and extracted six times with 50mL portions of diethyl ether (discarded). Diphenylcarbazon was then pptd by acidifying the alkaline soln with 3M HNO₃ or glacial acetic acid. It was filtered on a Büchner funnel, air dried, and stored in the dark [Gerlach and Frazier *Anal Chem* 30 1142 1958]. Other impurities were phenylsemicarbazide and diphenylcarbodiazone. Impurities can be detected by chromatography [Willems et al. *Anal Chim Acta* 51 544 1970].

Diphenyl carbonate [102-09-0] M 214.2, m 80°. Purified by sublimation, and by preparative gas chromatography with 20% Apiezon on Embacel, and crystn from EtOH.

Diphenylcyclopropenone (Diphencyprone) [886-38-4] M 206.2, m 87-90°(hydrate), 119-121°(anhydr). Crystd from cyclohexane. UV (MeCN): λ_{\max} 226, 282, 297nm.

Diphenyl disulfide (phenyl disulfide) [882-33-7] M 218.3, m 60.5°. Crystd from MeOH. [Alberti et al. *J Am Chem Soc* 108 3024 1986]. Crystd repeatedly from hot diethyl ether, then vac dried at 30° over P₂O₅, fused under nitrogen and re-dried, the whole procedure being repeated, with a final drying under vac for 24h. Also recrystd from hexane/EtOH soln. [Burkey and Griller *J Am Chem Soc* 107 246 1985.]

1,1-Diphenylethanol [599-67-7] M 198.3, m 80-81°. Crystd from *n*-heptane. [Bromberg et al. *J Am Chem Soc* 107 83 1985.]

1,1-Diphenylethylene [530-48-3] M 180.3, b 268-270°, d 1.024, n 1.6088. Distd under reduced pressure from KOH. Dried with CaH₂ and redistd.

***N,N'*-Diphenylethylenediamine (Wanzlick's reagent)** [150-61-8] M 212.3, m 67.5°, b 178-182°/2mm $pK_{\text{Est}(1)} \sim 0.5$, $pK_{\text{Est}(2)} \sim 3.8$. Crystd from aqueous EtOH or MeOH.

***N,N'*-Diphenylformamidine** [622-15-1] M 196.2, m 142°, 137°, 136-139°. Crystd from absolute EtOH, gives the hydrate with aqueous EtOH.

1,3-Diphenylguanidine [102-06-7] M 211.3, m 148°, pK^{25} 10.12. Crystd from toluene, aqueous acetone or EtOH, and vacuum dried.

1,6-Diphenyl-1,3,5-hexatriene [1720-32-7] M 232.3, m 200-203°. Crystd from CHCl₃ or EtOH/CHCl₃ (1:1).

5,5-Diphenylhydantoin [57-41-0] M 252.3, m 293-295°. Crystd from EtOH.

1,1-Diphenylhydrazine (hydrazobenzene) [122-66-7] M 184.2, m 34°, 44°, 175°/10mm, 222°/40mm, $pK_{\text{Est}} \sim 1.7$. Crystd from hot EtOH containing a little ammonium sulfide or H₂SO₃ (to prevent atmospheric oxidation), preferably under nitrogen. Dried in a vacuum desiccator. Also crystd from pet ether (b 60-100°) to constant absorption spectrum. *HCl*, from EtOH has m 163-164°(dec). *Picrate*, from *C₆H₆, has m 123°(dec).

1,3-Diphenylisobenzofuran [5471-63-6] M 270.3, m 129-130°. Recrystd from EtOH or EtOH/CHCl₃ (1:1) under red light (as in photographic dark rooms) or from *benzene in the dark.

Diphenylmethane [101-81-5] M 168.2, m 25.4°. Sublimed under vacuum, or distd at 72-75°/4mm. Crystd from EtOH. Purified by fractional crystn of the melt.

1,1-Diphenylmethanamine [91-00-9] M 183.2, m 34°, $pK_{\text{Est}} \sim 9.1$. Crystd from water.

Diphenylmethyl chloride (benzhydryl chloride) [90-99-3] M 202.7, m 17.0°, b 167°/17mm, n 1.5960. Dried with Na₂SO₄ and fractionally distd under reduced pressure.

***all-trans*-1,8-Diphenyl-1,3,5,8-octatetraene** [3029-40-1] M 258.4, m 235-237°. Crystd from EtOH.

2,5-Diphenyl-1,3,4-oxadiazole (PPD) [725-12-2] M 222.3, m 70° (hydrate), 139-140° (anhydrous), b 231°/13mm, 248°/16mm. Crystd from EtOH and sublimed *in vacuo*.

2,5-Diphenyloxazole (PPO) [92-71-7] M 221.3, m 74°, b 360°/760mm. Distd in steam and crystd from ligroin.

***N,N'*-Diphenyl-*p*-phenylenediamine** [74-31-7] M 260.3, m 148-149°, b 219-224°/0.7mm, pK_{Est} <0. Crystd from EtOH, chlorobenzene/pet ether or *benzene. Has also been crystd from aniline, then extracted three times with absolute EtOH.

1,1-Diphenyl-2-picrylhydrazine [1707-75-1] M 395.3, m 174°(dec), 178-179.5°(dec). Crystd from CHCl₃, or *benzene/pet ether (1:1), then degassed at 100° and <10⁻⁵mm Hg for ca 50h to decompose the 1:1 molar complex formed with *benzene.

2,2-Diphenylpropionic acid [5558-66-7] M 226.3, m 173-174°, pK_{Est} ~3.8. Crystd from EtOH.

3,3-Diphenylpropionic acid [606-83-7] M 226.3, m 155°, pK_{Est} ~4.5. Crystd from EtOH.

Diphenyl sulfide [139-66-2] M 186.3, b 145°/8mm, d 1.114, n 1.633. Washed with aqueous 5% NaOH, then water. Dried with CaCl₂, then with sodium. The sodium was filtered off and the diphenyl sulfide was distd under reduced pressure.

Diphenyl sulfone [127-63-9] M 218.3, m 125°, b 378°(dec). Crystd from diethyl ether. Purified by zone melting.

***sym*-Diphenylthiourea (thiocarbanilide)** [102-08-9] M 228.3, m 154°. Crystd from boiling EtOH by adding hot water and allowing to cool.

1,1-Diphenylurea [603-54-3] M 212.3, m 238-239°. Crystd from MeOH.

Dipicolinic acid (pyridine-2,6-dicarboxylic acid) [499-83-2] M 167.1, m 255°(dec), λ_{max} 270nm, pK₁²⁰ 2.10, pK₂²⁰ 4.68. Crystd from water, and sublimed in a vacuum.

***N,N*-Di-*n*-propylaniline** [2217-07-4] M 177.3, b 127°/10mm, 238-241°/760mm, pK²³ 5.68. Refluxed for 3hr with acetic anhydride, then fractionally distd under reduced pressure.

Dipropylene glycol (octan-4,5-diol) [110-98-5] M 134.2, b 109-110°/8mm, d 1.022, n 1.441. Fractionally distd below 15mm pressure, using packed column and taking precautions to avoid absorption of water.

Di-*n*-propyl ketone [123-19-3] M 114.2, b 143.5°, d 0.8143, n 1.40732. Dried with CaSO₄, then distd from P₂O₅ under nitrogen.

Di-*n*-propyl sulfide [111-47-7] M 118.2, b 141-142°, d 0.870, n 1.449. Washed with aqueous 5% NaOH, then water. Dried with CaCl₂ and distd from Na [Dunstan and Griffiths *J Chem Soc* 1344 1962].

Di-(4-pyridoyl)hydrazine [4329-75-3] M 246.2, m 254-255°. Crystd from water.

2,2'-Dipyridylamine [1202-34-2] M 171.2, m 84° and remelts at 95° after solidifying, b 176-178°/13mm, 307-308°/760mm, pK 6.69 (in 20% EtOH). Crystd from *benzene or toluene [Blakley and De Armond *J Am Chem Soc* 109 4895 1987].

2,2'-Dipyridyl disulfide (2,2'dithiopyridine) [2127-03-9] M 220.3, m 53°, 56-58°, 57-58°, pK₁²⁵ 0.35, pK₂²⁵ 2.45. Recrystd from *C₆H₆/pet ether (6:7), ligroin or *C₆H₆. *Picrate* has m 119° (from EtOH). [Walter et al. *Justus Liebigs Ann Chem* 695 7785 1966; Marckwald et al. *Chem Ber* 33 1556 1900; Brocklehurst and Little *Biochem J* 133 67,78 1973.]

1,2-Di-(4-pyridyl)-ethane [4916-57-8] M 184.2, pK_{Est(1)} ~3.8, pK_{Est(2)} ~5.4. Crystd from cyclohexane/*benzene (5:1).

trans-1,2-Di-(4-pyridyl)-ethylene [13362-78-2] M 182.2, m 153-154°, 155.5-156.5°, $pK_1^{2.5}$ 3.65, $pK_2^{2.5}$ 5.6. Crystd from water (1.6g/100mL at 100°). *Di-HCl* has m 347°, from EtOH

1,3-Di-(4-pyridyl)-propane [17252-51-6] M 198.3, m 60.5-61.5°, $pK_{Est(1)}$ ~4.5, $pK_{Est(2)}$ ~5.5. Crystd from *n*-hexane/*benzene (5:1).

S-1,2-Distearin [1188-58-5] M 625.0, m 76-77°, $[\alpha]_D^{20}$ -2.8° (c 6.3, CHCl₃), $[\alpha]_{546}^{20}$ +1.4° (c 10, CHCl₃/MeOH, 9:1). Crystd from chloroform/pet ether.

2,5-Distyrylpyrazine [14990-02-4] M 284.3, m 219°. Recrystd from xylene; chromatographed on basic silica gel (60-80 mesh) using CH₂Cl₂ as eluent, then vac sublimed on to a cold surface at 10⁻³ torr [Ebied *J Chem Soc, Faraday Trans 1* 78 3213 1982]. Operations should be carried out in the dark.

1,3-Dithiane [505-23-7] M 120.2, m 54°. Crystd from 1.5 times its weight of MeOH at 0°, and sublimed at 40-50°/0.1mm.

2,2'-Dithiobis(benzothiazole) [120-78-8] M 332.2, m 180°. Crystd from *benzene.

4,4'-Dithiodimorpholine [103-34-4] M 236.2, m 124-125°. Crystd from hot aq dimethylformamide.

1,4-Dithioerythritol (DTE, erythro-2,3-dihydroxy-1,4-dithiobutane) [6892-68-8] M 154.3, m 82-84°, pK_1 9.0, pK_2 9.9. Crystd from ether/hexane and stored in the dark at 0°.

Dithiooxamide (rubeanic acid) [79-40-3] M 120.2, m >300°. Crystd from EtOH and sublimed in a vacuum.

RS-1,4-Dithiothreitol (DTT, Cleland's reagent) [27565-41-9] M 154.3, m 42-43°, pK_1 8.3, pK_2 9.5. Crystd from ether and sublimed at 37°/0.005mm. Should be stored at 0°.

Dithizone (diphenylthiocarbazone) [60-10-6] M 256.3, ratio of $\epsilon_{620nm}/\epsilon_{450nm}$ should be ≥ 1.65 , ϵ_{620} 3.4×10^4 (CHCl₃), pK_2 4.6. The crude material is dissolved in CCl₄ to give a concentrated soln. This is filtered through a sintered glass funnel and shaken with 0.8M aq ammonia to extract dithizonate ion. The aqueous layer is washed with several portions of CCl₄ to remove undesirable materials. The aqueous layer is acidified with dil H₂SO₄ to precipitate pure dithizone. It is dried in a vacuum. When only small amounts of dithizone are required, purification by paper chromatography is convenient. [Cooper and Hibbits *J Am Chem Soc* 75 5084 1933.] Instead of CCl₄, CHCl₃ can be used, and the final extract, after washing with water, can be evapd in air at 40-50° and dried in a desiccator. Complexes with Cd, Hg, Ni and Zn.

Di-*p*-tolyl carbonate [621-02-3] M 242.3, m 115°. Purified by GLC with 20% Apiezon on Embacel followed by sublimation *in vacuo*.

***N,N'*-Di-*o*-tolylguanidine** [97-39-2] M 239.3, m 179° (175-176°), pK_{Est} ~10.3. Crystd from aqueous EtOH.

Di-*p*-tolylphenylamine [20440-95-3] M 273.4, m 108.5°, pK_{Est} ~5.0. Crystd from EtOH.

Di-*p*-tolyl sulfone [599-66-6] M 278.3, m 158-159°, b 405°. Crystd repeatedly from diethyl ether. Purified by zone melting.

Djenkolic acid (S,S'-methylene-bis-L-cysteine) [498-59-9] M 254.3, m 300-350°(dec), $[\alpha]_D^{20}$ -65° (c 2, HCl) [See pK of S-methyl-L-cysteine]. Crystd from a large volume of water (sol 0.5g%).

***cis*-4,7,10,13,16,19-Docosahexaenoic acid** [6217-54-5] M 328.5, m -44/1°, -44.1°, n_D^{20} 1.5017, pK_{Est} ~4.6. Its solubility in CHCl₃ is 5%. It has been purified from fish oil by GLC using Ar as mobile phase and EGA as stationary phase with an ionisation detector [UV: Stoffel and Ahrens *J Lipid Res* 1

139 1959], and *via* the ester by evaporative "molecular" distillation using a 'continuous molecular still' at 10^{-4} mm with the highest temperature being 110° , and a total contact time with the hot surface being 60sec [Farmer and van den Heuvel *J Chem Soc* 427 1938]. The *methyl ester* has **b** 208-211 $^{\circ}$ /2mm, d_4^{20} 0.9398, n_D^{20} 1.5035. With Br_2 it forms a *dodecabromide* **m ca** 240° dec. Also the acid was converted to the methyl ester and purified through a three stage molecular still [as described by Sutton *Chem Ind (London)* 11383 1953] at 96° with the rate adjusted so that one third of the material was removed each cycle of three distillations. The distillate (numbered 4) (13g) was dissolved in EtOH (100mL containing 8g of KOH) at -70° and set aside for 4h at 30° with occasional shaking under a vac. Water (100mL) is added and the soln is extracted with pentane, washed with HCl, dried (MgSO_4), filtered and evapd to give a clear oil (11.5g) **m** -44.5° to -44.1° . In the catalytic hydrogenation of the oil six mols of H_2 were absorbed and *docosanoic acid (behenic acid)* was produced with **m** 79.0 - 79.3° undepressed with an authentic sample (see docosanoic acid below) [Whitcutt *Biochem J* 67 60 1957].

Docosane [629-97-0] **M** 310.6, **m** 47° , **b** 224° /15mm. Crystd from EtOH or ether.

Docosanoic acid (behenic acid) [112-85-6] **M** 340.6, **m** 81 - 82° , pK_{Est} ~ 4.9 . Crystd from ligroin. [Francis and Piper *J Am Chem Soc* 61 577 1939].

1-Docosanol (behenyl alcohol) [661-19-8] **M** 182.3, **m** 70.8° . Crystd from ether or chloroform/ether.

***n*-Dodecane** [112-40-3] **M** 170.3, **b** 97.5 - 99.5° /5mm, 216° /760mm, **d** 0.748, **n** 1.42156. Passed through a column of Linde type 13X molecular sieves. Stored in contact with, and distd from, sodium. Passed through a column of activated silica gel. Has been crystd from diethyl ether at -60° . Unsaturated dry material which remained after passage through silica gel has been removed by catalytic hydrogenation (Pt_2O) at 45lb/in 2 (3.06 atmospheres), followed by fractional distn under reduced pressure [Zook and Goldey *J Am Chem Soc* 75 3975 1953]. Also purified by partial crystn from the melt.

Dodecane-1,10-dioic acid (decane-1,10-dicarboxylic acid) [693-23-2] **M** 230.3, **m** 129° , **b** 245° /10mm, pK_{Est} ~ 4.8 . Crystd from water, 75% or 95% EtOH (sol 10%), or glacial acetic acid.

1-Dodecanol (dodecyl alcohol) [112-53-8] **M** 186.3, **m** 24° , **b** 91° /1mm, 135° /10mm, 167° /40mm, 213° /200mm, 259° /atm, d_4^{24} 0.8309 (liquid). Crystd from aqueous EtOH, and vacuum distd in a spinning-band column. [Ford and Marvel *Org Synth* 10 62 1930.]

1-Dodecanthiol [112-55-0] **M** 202.4, **b** 111 - 112° /3mm, 153 - 155° /24mm, **d** 0.844, **n** 1.458, pK_{Est} ~ 10.8 . Dried with CaO for several days, then distd from CaO.

Dodecylammonium butyrate [17615-97-3] **M** 273.4, **m** 39 - 40° , pK^{25} 10.63 (for free base). Recrystd from *n*-hexane.

Dodecylammonium propionate [17448-65-6] **M** 259.4, **m** 55 - 56° . Recrystd from hexanol/pet ether (b 60 - 80°).

Dodecyltrimethylamine oxide [1643-20-5] **M** 229.4, **m** 102° . Crystd from acetone or ethyl acetate. [Bunton et al. *J Org Chem* 52 3832 1987].

Dodecyl ether [4542-57-8] **M** 354.6, **m** 33° . Vacuum distd, then crystd from MeOH/*benzene.

1-Dodecylpyridinium chloride [104-74-5] **M** 301.9, **m** 68 - 70° . Purified by repeated crystn from acetone (charcoal); twice recrystd from EtOH [Chu and Thomas *J Am Chem Soc* 108 6270 1986].

Dodecyltrimethylammonium bromide [1119-94-4] **M** 308.4, **m** 246° (dec). Purified by repeated crystn from acetone. Washed with diethyl ether and dried in a vacuum oven at 60° [Dearden and Wooley *J Phys Chem* 91 2404 1987].

Dodecyltrimethylammonium chloride [112-00-5] M 263.9, m 246°(dec). Dissolved in MeOH, treated with active charcoal, filtered and dried *in vacuo* [Waldenburg *J Phys Chem* 88 1655 1984], or recrystd several times from 10% EtOH in acetone. Also repeatedly crystd from EtOH/ether or MeOH. [Cella et al. *J Am Chem Soc* 74 2062 1952.]

Dulcitol [608-66-2] M 182.2, m 188-189°, b 276-280°/1.1mm. Crystd from water by addition of EtOH.

Duroquinone (tetramethylbenzoquinone) [527-17-3] M 164.2, m 110-111°. Crystd from 95% EtOH. Dried under vacuum.

α -Ecdyson [3604-87-3] M 464.7, m 239-242°, 242°, $[\alpha]_D^{20} +72^\circ$ (c 1, EtOH). Recrystd from tetrahydrofuran-pet ether, and from H₂O as a hydrate. It has been purified by chromatography on Al₂O₃ and elution with EtOAc-MeOH. It has λ_{\max} at 242nm (ϵ 12.400). Its *acetate* has m 214-216° from EtOAc-pet ether, and the 2,4-dinitrophenylhydrazone has m 170-175° (dec) from EtOAc. [Karlson and Hoffmeister *Justus Liebigs Ann Chem* 662 1 1963; Karlson *Pure Appl Chem* 14 75 1967.]

β -Ecdyson (β -echdysterone) [5289-74-7] M 480.7, m 245-247°, $[\alpha]_D^{20} +66^\circ$ (c 1, MeOH). Crystd from water or tetrahydrofuran/pet ether.

Echinenone [432-68-8] M 550.8, m 178-179°, $A_{1\text{cm}}^{1\%}$ (λ_{\max}) 2160 (458nm) in pet ether. Purified by chromatography on partially deactivated alumina or magnesia, or by using a thin layer of silica gel G with 4:1 cyclohexane/diethyl ether as the developing solvent. Stored in the dark at -20°.

Eicosane [112-95-8] M 282.6, m 36-37°, b 205°/15mm, $d^{36.7} 0.7779$, $n^{40} 1.43453$. Crystd from EtOH.

Elaidic (*trans*-oleic) acid [112-79-8] M 282.5, m 44.5°, $pK^{25} 4.9$. Crystd from acetic acid, then EtOH.

Ellagic acid (2H₂O) [476-66-4] M 338.2, m >360°, $pK_{\text{Est}(1)} \sim 8$, $pK_{\text{Est}(2)} \sim 11$. Crystd from pyridine.

Elymoclavine (8,9-didehydro-6-methylergoline-8-methanol) [548-43-6] M 254.3, m 249-253°(dec), $[\alpha]_D^{20} -59^\circ$ (c 1, EtOH). Crystd from MeOH.

Embonic acid (Pamoic acid, 4,4'-methylene bis[3-hydroxy-2-naphthalenecarboxylic acid]) [130-85-8] M 388.4, m 295°, >300°, $pK_{\text{Est}(1)} \sim 2.2$, $pK_{\text{Est}(2)} \sim 13.2$. Forms crystals from dilute pyridine which decomposition above 280° without melting. It is almost insoluble in H₂O, EtOH, Et₂O, *C₆H₆, CH₃CO₂H, sparingly soluble in CHCl₃ but soluble in nitrobenzene, pyridine and alkalis [Barber and Gaimster *J Appl Chem (London)* 2 565 1952]. Used for making salts of organic bases.

Emetine hydrochloride hydrate [316-42-7] M 553.6 + aq, m 235-240°, 235-250°, 240-250°, 248-250° (depending on H₂O content), $[\alpha]_D^{20} -49.2^\circ$ (free base, c 4, CHCl₃), +18° (c 6, H₂O, dry salt), $pK_1 5.77$, $pK_2 6.64$. It crystallises from MeOH-Et₂O, MeOH or Et₂O-EtOAc. The *free base* has m 104-105°, and the (-)-*phenyl thiourea derivative* has m 220-221° [from EtOAc-pet ether, $[\alpha]_D^{25} -29.3^\circ$ (CHCl₃)]. IR: 3413 (OH) and 2611 (NH⁺) cm⁻¹; UV λ_{\max} 230nm (ϵ 16 200) and 282nm (ϵ 6 890) [Brossi et al. *Helv Chim Acta* 42 1515 1959; Barash et al. *J Chem Soc* 3530 1959].

Emodine (1,3,8-trihydroxy-6-methyl-9,10-anthracenedione, archin) [518-82-1] M 270.2, m 253-257°, 255-256°, 256-257°, 262°, 264° (phenolic pKs 7-10). Forms orange needles from EtOH, Et₂O, *C₆H₆, toluene or pyridine. It sublimes above 200° at 12mm. [Tutin and Clewer *J Chem*