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2-(*N*-Morpholino)ethanesulfonic acid (MES) [4432-31-9] M 213.3, m >300°(dec), pK²⁵ 6.15. Crystd from hot EtOH containing a little water.

Mucochloric acid (2,3-dichloro-4-oxo-2-butenic acid) [87-56-9] M 169.0, m 124-126°, pK²⁵ 4.20. Crystd twice from water (charcoal).

***trans,trans*-Muconic acid (hexa-2,4-dienedioic acid)** [3588-17-8] M 142.1, m 300°, pK²⁵ 4.51, for *cis,cis* pK²⁵ 4.49. Cryst from H₂O.

Muramic acid (H₂O) (3-*O*- α -carboxyethyl-D-glucosamine) [1114-41-6] M 251.2, m 152-154°(dec). See muramic acid on p. 549 in Chapter 6.

Murexide (ammonium purpurate) [3051-09-0] M 284.2, m >300°, λ_{\max} 520nm (ϵ 12,000), pK₂ 9.2, pK₃ 10.9. The sample may be grossly contaminated with uramil, alloxanthine, etc. Difficult to purify. It is better to synthesise it from pure alloxanthine [Davidson *J Am Chem Soc* 58 1821 1936]. Crystd from water.

Myristic acid (tetradecanoic acid) [544-63-8] M 228.4, m 58°, pK²⁰ 6.3 (50% EtOH), pK_{Est} ~4.9 (H₂O). Purified *via* the *methyl ester* (b 153-154°/10mm, n²⁵ 1.4350), as for capric acid. [Trachtman and Miller *J Am Chem Soc* 84 4828 1962.] Also purified by zone melting. Crystd from pet ether and dried in a vacuum desiccator containing shredded wax.

Naphthacene (benz[b]anthracene, 2,3-benzanthracene, rubene) [92-24-0] M 228.3, m >300°, 341° (open capillary), 349°, 357°. Crystd from EtOH or *benzene. Dissolved in sodium-dried *benzene and passed through a column of alumina. The *benzene was evaporated under vacuum, and the chromatography was repeated using fresh *benzene. Finally, the naphthacene was sublimed under vacuum. [Martin and Ubbelohde *J Chem Soc* 4948 1961.] Also recrystd in orange needles from xylene and sublimes *in vacuo* at 186°. [UV: *Chem Ber* 65 517 1932, 69 607 1936; IR: *Spectrochim Acta* 4 373 1951.]

2-Naphthaldehyde [66-99-9] M 156.2, m 59°, b 260°/19mm, pK²⁰ -7.04 (aq H₂SO₄). Distilled with steam and crystd from water or EtOH.

Naphthalene [91-20-3] M 128.2, m 80.3°, b 87.5°/10mm, 218.0°/atm, d 1.0253, d¹⁰⁰ 0.9625, n⁸⁵ 1.5590. Crystd one or more times from the following solvents: EtOH, MeOH, CCl₄, *benzene, glacial acetic acid, acetone or diethyl ether, followed by drying at 60° in an Abderhalden drying apparatus. Also purified by vacuum sublimation and by fractional crystn from its melt. Other purification procedures include refluxing in EtOH over Raney Ni, and chromatography of a CCl₄ soln on alumina with *benzene as eluting solvent. Baly and Tuck [*J Chem Soc* 1902 1908] purified naphthalene for spectroscopy by heating with conc H₂SO₄ and MnO₂, followed by steam distn (repeating the process), and formation of the picrate which, after recrystallisation, was decomposed and the naphthalene was steam distd. It was then crystd from dilute EtOH. It can be dried over P₂O₅ under vacuum. Also purified by sublimation and subsequent crystn from cyclohexane. Alternatively, it has been washed at 85° with 10% NaOH to remove phenols, with 50% NaOH to remove nitriles, with 10% H₂SO₄ to remove organic bases, and with 0.8g AlCl₃ to remove thianaphthalenes and various alkyl derivatives. Then it was treated with 20% H₂SO₄, 15% Na₂CO₃ and finally distd. [Gorman et al. *J Am Chem Soc* 107 4404 1985.]

Zone refining purified naphthalene from anthracene, 2,4-dinitrophenylhydrazine, methyl violet, benzoic acid, methyl red, chrysene, pentacene and indoline.

Naphthalene-2,5-disulfonic acid [92-41-1] M 288.2, pK_{Est} <0. Crystd from conc HCl.

Naphthalene-1-sulfonic acid [85-47-2] M 208.2, m (2H₂O) 90°, (anhydrous) 139-140°, pK²⁰ -0.17. Crystd from conc HCl and twice from water.

Naphthalene-2-sulfonic acid [120-18-3] M 208.2, m 91°, pK_{Est} <1. Crystd from conc HCl.

Naphthalene-1-sulfonyl chloride [85-46-1] M 226.7, m 64-67°, 68°, b 147.5°/0.9 mm, 147.5°/13mm. If the IR indicates the presence of OH then treat with an equal weight of PCl₅ and heat at ca 100° for 3h, cool and pour into ice + H₂O, stir well and filter off the solid. Wash the solid with cold H₂O and dry the solid in a vacuum desiccator over P₂O₅ + solid KOH. Extract the solid with pet ether (b 40-60°) filter off any insoluble solid and cool. Collect the crystalline sulfonyl chloride and recryst from pet ether or *C₆H₆ pet ether. If large quantities are available then it can be distd under high vacuum. [Fierz-Davaid and Weissenbach *Helv Chim Acta* 3 2312 1920.] The *sulfonamide* has m 150° (from EtOH or H₂O).

Naphthalene-2-sulfonyl chloride [93-11-8] M 226.7, m 74-76°, 78°, 79°, b 148°/0.6 mm, 201°/13mm. Crystd (twice) from *benzene/pet ether (1:1 v/v). Purified as the 2-sulfonyl chloride. [Fierz-Davaid and Weissenbach *Helv Chim Acta* 3 2312 1920.] The *sulfonamide* has m 217° (from EtOH).

1,8-Naphthalic acid (naphthalene-1,8-dicarboxylic acid) [518-05-8] M 216.9, m 270°, pK_{Est(1)} ~ 2.1, pK_{Est(2)} ~ 4.5. Crystd from EtOH or aq EtOH.

1,8-Naphthalic anhydride [81-84-5] M 198.2, m 274°. Extracted with cold aqueous Na₂CO₃ to remove free acid, then crystd from acetic anhydride.

Naphthamide [2243-82-5] M 171.2, m 195°, pK²⁰ -2.30 (H₀ scale, aq H₂SO₄). Crystd from EtOH.

Naphthazarin (5,8-dihydroxy-1,4-naphthoquinone) [475-38-7] M 190.2, m ~ 220-230°(dec), m 225-230°, pK_{Est(1)} ~ 9.5, pK_{Est(2)} ~ 11.1. Red-brown needles with a green shine from EtOH. Also recrystd from hexane and purified by vacuum sublimation. [Huppert et al. *J Phys Chem* 89 5811 1985.] It is sparingly soluble in H₂O but soluble in alkalis. It sublimates at 2-10mm. The *diacetate* forms golden yellow prisms from CHCl₃, m 192-193° and the 5,8-*dimethoxy* derivative has m 157° (155°) (from pet ether) [Bruce and Thompson *J Chem Soc* 1089 1955; IR: Schmand and Boldt *J Am Chem Soc* 97 447 1975; NMR: Brockmann and Zeeck *Chem Ber* 101 4221 1968]. The *monothiosemicarbazone* has m 168°(dec) from EtOH [Gardner et al. *J Am Chem Soc* 74 2106 1952].

Naphthionic acid (4-aminonaphthalene-1-sulfonic acid) [84-86-6] M 223.3, m > 300°(dec), pK²⁵ 2.68. It crystallises from H₂O as needles of the 0.5 hydrate. Salt solns fluoresce strongly blue.

1-Naphthoic acid [86-55-5] M 172.2, m 162.5-163.0°, pK²⁵ 3.60. Crystd from toluene (3mL/g) (charcoal), pet ether (b 80-100°), or aqueous 50% EtOH.

2-Naphthoic acid [93-09-4] M 172.2, m 184-185°, pK²⁵ 4.14. Crystd from EtOH (4mL/g), or aqueous 50% EtOH. Dried at 100°.

1-Naphthol [90-15-3] M 144.2, m 95.5-96°, pK²⁵ 9.34. Sublimed, then crystd from aqueous MeOH (charcoal), aq 25% or 50% EtOH, *benzene, cyclohexane, heptane, CCl₄ or boiling water. Dried over P₂O₅ under vacuum. [Shizuka et al. *J Am Chem Soc* 107 7816 1985.]

2-Naphthol [135-19-3] M 144.2, m 122.5-123.5°, pK²⁵ 9.57. Crystd from aqueous 25% EtOH (charcoal), water, *benzene, toluene or CCl₄, e.g. by repeated extraction with small amounts of EtOH, followed by dissolution in a minimum amount of EtOH and pptn with distilled water, then drying over P₂O₅ under vacuum. Has also been dissolved in aqueous NaOH, and ppted by adding acid (repeated several times), then ppted from *benzene by addition of heptane. Final purification can be by zone melting or sublimation *in vacuo*. [Bardez et al. *J Phys Chem* 89 5031 1985; Kikuchi et al. *J Phys Chem* 91 574 1987.]

Naphthol AS-D (3-hydroxy-2-naphthoic-o-toluide) [135-61-5] M 277.3, m 1196-198°. Purified by recrystn from xylene. Gives yellow-green fluorescent solutions at pH 8.2-9.5, [IR: Schnopper et al. *Anal Chem* 31 1542 1959.] With AcCl *naphthol AS-D acetate* is obtained m 168-169°, and with

chloroacetyl chloride *naphthol AS-D-chloroacetate* is obtained [Moloney et al. *J Histochem Cytochem* **8** 200 1960; Burstone *Arch Pathology* **63** 164 1957].

α -Naphtholbenzein [bis-(α -{4-hydroxynaphth-1-yl})-benzyl alcohol] [6948-88-5] **M 392.5, m 122-125^o, pK_{Est} ~ 9.3.** Crystd from EtOH, aqueous EtOH or glacial acetic acid.

1-Naphthol-2-carboxylic acid (1-hydroxy-2-naphthoic acid) [86-48-6] **M 188.2, m 203-204^o, pK_{Est(1)}~2.5, pK_{Est(2)}~12.** Successively crystd from EtOH/water, diethyl ether and acetonitrile, with filtration through a column of charcoal and Celite. [Tong and Glesmann *J Am Chem Soc* **79** 583 1957.]

2-Naphthol-3-carboxylic acid (2-hydroxy-3-naphthoic acid) [92-70-6] **M 188.2, m 222-223^o, pK₁²⁵ 2.79, pK₂²⁵ 12.84.** Crystd from water or acetic acid.

1,2-Naphthoquinone [524-42-5] **M 158.2, m 140-142^o(dec).** Crystd from ether (red needles) or *benzene (orange leaflets).

1,4-Naphthoquinone [130-15-4] **M 158.2, m 125-125.5^o.** Crystd from diethyl ether (charcoal). Steam distd. Crystd from *benzene or aqueous EtOH. Sublimed in a vacuum.

β -Naphthoxyacetic acid [120-23-0] **M 202.2, m 156^o, pK_{Est} ~3.0.** Crystd from hot water or *benzene.

β -Naphthoyltrifluoroacetone (4,4,4-trifluoro-2-naphthylbutan-1,3-dione) [893-33-4] **M 266.2, m 70-71^o, 74-76^o, pK²⁰ 6.35.** Crystd from EtOH. The *mono oxime* crystd from H₂O or aq EtOH has m 137-138^o. [Reid and Calvin *J Am Chem Soc* **72** 2948 1950.]

Naphthvalene [34305-47-0] **M 104.1, m dec at 175^o to benzvalene.** Purified by chromatography on alumina and eluting with pentane. It is stable at room temp [Abelt et al. *J Am Chem Soc* **107** 4148 1985]. The ¹H NMR in CCl₄ has τ 3.18 (4H), 6.17 (t J 1.5Hz, 2H), 7.60 (t J 1.5Hz 2H).

1-Naphthyl acetate [830-81-9] **M 186.2, m 45-46^o.** Chromatographed on silica gel and crystd as the 2-isomer below.

2-Naphthyl acetate [1523-11-1] **M 186.2, m 71^o.** Crystd from pet ether (b 60-80^o) or dilute aq EtOH.

1-Naphthylacetic acid [86-87-3] **M 186.2, m 132^o, pK²⁵ 4.23.** Crystd from EtOH or water.

2-Naphthylacetic acid [581-96-4] **M 186.2, m 143.1-143.4^o, pK²⁵ 4.30.** Crystd from water or *benzene.

1-Naphthylamine [134-32-7] **M 143.2, m 50.8-51.2^o, b 160^o, pK²⁵ 3.94.** Sublimed at 120^o in a stream of nitrogen, then crystd from pet ether (b 60-80^o), or abs EtOH then diethyl ether. Dried under vacuum in an Abderhalden pistol. Has also been purified by crystn of its hydrochloride from water, followed by liberation of the free base and distn; finally purified by zone melting. **CARCINOGEN.**

1-Naphthylamine hydrochloride [552-46-5] **M 179.7, m sublimes on heating.** Crystd from water (charcoal).

2-Naphthylamine [91-59-8] **M 143.2, m 113^o, pK²⁵ 4.20.** Sublimed at 180^o in a stream of nitrogen. Crystd from hot water (charcoal) or *benzene. Dried under vacuum in an Abderhalden pistol. **CARCINOGEN.**

1-Naphthylamine-5-sulfonic acid [84-89-9] **M 223.3, m >200^o(dec), pK_{Est(1)}<1, pK₂²⁵ 3.69 (NH₂)** Crystd under nitrogen from boiling water and dried in a steam oven [Bryson *Trans Faraday Soc* **47** 522, 527 1951].

2-Naphthylamine-1-sulfonic acid [81-16-3] M 223.3, m >200°(dec), $pK_1^{25} < 1$, $pK_2^{25} 2.35$ (NH₂). Crystd under nitrogen from boiling water and dried in a steam oven [Bryson *Trans Faraday Soc* 47 522, 527 1951].

2-Naphthylamine-6-sulfonic acid [93-00-5] M 223.3, m >200°(dec). Crystd from a large volume of hot water.

1-(1-naphthyl) ethanol [R-(+)- 42177-25-3; S-(-)- 15914-84-8] M 172.2, m 46°, 45-47.5°, 48°, $[\alpha]_{546}^{20}$ (+) and (-) 94°, $[\alpha]_D^{20}$ (+) and (-) 78° (c 1, MeOH). Purified by recrystn from Et₂O-pet ether, Et₂O, hexane [Balfe et al. *J Chem Soc* 797 1946; IR, NMR: Theisen and Heathcock *J Org Chem* 53 2374 1988; see also Fredga et al. *Acta Chem Scand* 11 1609 1957]. The *RS*-alcohol [57605-95-5] has m 63-65°, 65-66° from hexane.

1-(1-Naphthyl)ethylamine [R-(+)- 3886-70-2; S-(-)- 10420-89-0] M 171.2, b 153°/11mm, 178-181°/20mm, $d_4^{20} 1.067$, $n_D^{20} 1.624$, $[\alpha]_{546}^{20}$ (+) and (-) 65°, $[\alpha]_D^{20}$ (+) and (-) 55° (c 2, MeOH); $[\alpha]_D^{17}$ (+) and (-) 82.8° (neat), $pK_{Est} \sim 9.3$. Purified by distn in a good vacuum. [Mori et al. *Tetrahedron* 37 1343 1981; cf Wilson in *Top Stereochem* (Allinger and Eliel eds) vol 6 135 1971; Fredga et al. *Acta Chem Scand* 11 1609 1957.] The *hydrochlorides* crystallises from H₂O $[\alpha]_D^{18} \pm 3.9^\circ$ (c 3, H₂O) and the *sulfates* recrystallises from H₂O as *tetrahydrates* m 230-232°. The *RS*-amine has b 153°/11mm, 156°/15mm, 183.5°/41mm [Blicke and Maxwell *J Am Chem Soc* 61 1780 1939].

2-Naphthylethylene (2-vinylnaphthalene) [827-54-3] M 154.2, m 66°, b 95-96°/2.1mm, 135-137°/18mm. Crystd from aqueous EtOH.

***N*-(α -Naphthyl)ethylenediamine dihydrochloride** [1465-25-4] M 291.2, m 188-190°, $pK_{Est(1)} \sim 3.8$, $pK_{Est(2)} \sim 9.4$. Crystd from water.

1-Naphthyl isocyanate [86-84-0] M 169.2, m 3-5°, b 269-270°/atm, $d_4^{20} 1.18$. Distd at atmospheric pressure or in a vacuum. Can be crystd from pet ether (b 60-70°) at low temperature. *It has a pungent odour, is TOXIC and is absorbed through the skin.*

1-Naphthyl isothiocyanate [551-06-4] M 185.3, m 58-59°. Crystd from hexane (1g in 9 mL). White needles soluble in most organic solvents but is insoluble in H₂O. *It is absorbed through the skin and may cause dermatitis.* [*Org Synth Coll Vol IV* 700 1963.]

2-Naphthyl lactate [93-43-6] M 216.2. Crystd from EtOH.

2-(2-Naphthyloxy)ethanol [93-20-9] M 188.2, m 76.7°. Crystd from *benzene/pet ether.

***N*-1-Naphthylphthalamic acid** [132-66-1] M 291.3, m 203°. Crystd from EtOH.

2-Naphthyl salicylate [613-78-5] M 264.3, m 95°, $pK_{Est} \sim 10.0$. Crystd from EtOH.

1-Naphthyl thiourea (ANTU) [86-88-4] M 202.2, m 198°. Crystd from EtOH.

1-Naphthyl urea [6950-84-1] M 186.2, m 215-220°. Crystd from EtOH.

2-Naphthyl urea [13114-62-0] M 186.2, m 219-220°. Crystd from EtOH.

1,5-Naphthyridine [254-79-5] M 130.1, m 75°, b 112°/15mm, $pK^{20} 2.84$. Purified by repeated sublimation.

Narcein {6-[6-(2-dimethylaminoethyl)]-2-methoxy-3,4-(methylenedioxy)phenylacetyl]-2,3-dimethoxybenzoic acid} [131-28-2] M 445.4, m 176-177° (145° anhydrous), pK_1^{15} 3.5, pK_2^{15} 9.3. Crystd from water (as trihydrate).

Naringenin (4',5,7-trihydroxyflavanone) [480-41-1] M 272.3, m 251° (phenolic pK_s ~ 8-11). Crystd from aqueous EtOH.

Naringin (naringenin 7-rhamnoglucoside) [10236-47-2] M 580.5, m 171° (2H₂O), $[\alpha]_D^{19}$ -90° (c 1, EtOH), $[\alpha]_{546}^{20}$ -107° (c 1, EtOH). Crystd from water. Dried at 110°(to give the dihydrate).

Neopentane (2,2-dimethylpropane) [463-82-1] M 72.2, b 79.3°, d 0.6737, n 1.38273. Purified from isobutene by passage over conc H₂SO₄ or P₂O₅, and through silica gel.

Neostigmine [(3-dimethylcarbamoylphenyl)trimethylammonium] bromide [114-80-7] M 303.2, m 176°(dec). Crystd from EtOH/diethyl ether. (*Highly TOXIC*).

Neostigmine methyl sulfate [51-60-5] M 334.4, m 142-145°. Crystd from EtOH. (*Highly TOXIC.*)

Nerolidol (3,7,11-trimethyl-1,6,10-dodecatrien-3-ol) M 222.4 [*cis/trans* 7212-44-4] b 122°/3mm, n 1.477, d 0.73, [*cis* 3790-78-1] b 70°/0.1mm, [*trans* 40716-66-3] b 78°/0.2mm, 145-146°/2mm. Purified by TLC on plates of Kieselguhr G [McSweeney *J Chromatogr* 17 183 1965] or silica gel impregnated with AgNO₃, using 1,2-CH₂Cl₂/CHCl₃/EtOAc/PrOH (10:10:1:1) as solvent system. Also by GLC on butanediol succinate (20%) on Chromosorb W. Stored under N₂ at ~5° in the dark.

Neutral Red (2-amino-8-dimethylamino-3-methylphenazine HCl, **Basic Red 5**, **CI 50040**) [553-24-2] M 288.8, m 290°(dec), pK^{25} 6.5. Crystd from *benzene/MeOH (1:1). In aq sol it is red at pH 6.8 and yellow at pH 8.0.

Nicotinaldehyde thiosemicarbazone [3608-75-1] M 180.2, m 222-223°. Crystd from water.

Nicotinamide [98-92-0] M 122.1, m 128-131°, pK_1^{20} 0.5, pK_2^{20} 3.33. Crystd from *benzene.

Nicotinic acid (niacin, 2-yridine-3-carboxylic acid) [59-67-6] M 123.1, m 232-234°, pK_1^{25} 2.00, pK_2^{25} 4.82. Crystd from *benzene.

Nicotinic acid hydrazide [553-53-7] M 137.1, m 158-159°, pK_1^{20} 3.3, pK_2^{20} 11.49(NH). Crystd from aqueous EtOH or *benzene.

Nile Blue A (a benzophenoxazinium sulfate dye) [3625-57-8] M 415.5, m >300°(dec), **CI 51180**, pK^{25} 2.4. Crystd from pet ether.

Ninhydrin (1,2,3-triketohydrindene hydrate) [485-47-2] M 178.1, m 241-243°(dec), pK^{30} 8.82. Crystd from hot water (charcoal). Dried under vacuum and stored in a sealed brown container.

Nitrilotriacetic acid [tris(carboxymethyl)amine, NTA, **Complexone 1**] [139-13-9] M 191.1, m 247°(dec), pK_1 0.8, pK_2 1.71, pK_3 2.47, pK_4 9.71. Crystd from water. Dried at 110°.

2-Nitroacetanilide [552-32-9] M 180.2, m 93-94°, pK_{Est} <0. Crystd from water.

4-Nitroacetanilide [104-04-1] M 180.2, m 217°, pK_{Est} <0. Ppted from 80% H₂SO₄ by adding ice, then washed with water, and crystd from EtOH. Dried in air.

3-Nitroacetophenone [121-89-1] M 165.2, m 81°, b 167°/18mm, 202°/760mm. Distilled in steam and crystd from EtOH.

4-Nitroacetophenone [100-19-6] M 165.2, m 80-81°, b 145-152°/760mm. Crystd from EtOH or aqueous EtOH.

3-Nitroalizarin (1,2-dihydroxy-3-nitro-9,10-anthraquinone, Alizarin Orange) [568-93-4] M 285.2, m 244° (dec), $pK_{Est(1)} \sim 4.6$, $pK_{Est(2)} \sim 9.6$. Crystd from acetic acid.

***o*-Nitroaniline** [88-74-4] M 138.1, m 72.5-73.0°, $pK^{25} -0.25 (-0.31)$. Crystd from hot water (charcoal), then crystd from water, aqueous 50% EtOH, or EtOH, and dried in a vacuum desiccator. Has also been chromatographed on alumina, then recrystd from *benzene.

***m*-Nitroaniline** [99-09-2] M 138.1, m 114°, $pK^{25} 2.46$. Purified as for *o*-nitroaniline. **Warning: it is absorbed through the skin.**

***p*-Nitroaniline** [100-01-6] M 138.1, m 148-148.5°, $pK^{25} 1.02$. Purified as for *o*-nitroaniline. Also crystd from acetone. Freed from *o*- and *m*-isomers by zone melting and sublimation.

***o*-Nitroanisole (2-methoxynitrobenzene)** [91-23-6] M 153.1, f 9.4°, b 265°/737mm, d 1.251, n 1.563. Purified by repeated vacuum distn in the absence of oxygen.

***p*-Nitroanisole (4-methoxynitrobenzene)** [100-17-4] M 153.1, m 54°. Crystd from pet ether or hexane and dried *in vacuo*.

9-Nitroanthracene [602-60-8] M 223.2, m 142-143°. Purified by recrystn from EtOH or MeOH. Further purified by sublimation or TLC.

5-Nitrobarbituric acid (dilituric acid) [480-68-2] M 173.1, m 176°, 176-183°, $pK^{20} 10.25$. Crystd from water.

***o*-Nitrobenzaldehyde** [552-89-6] M 151.1, m 44-45°, b 120-144°/3-6mm. Crystd from toluene (2-2.5mL/g) by addition of 7mL pet ether (b 40-60°) for 1mL of soln. Can also be distd at reduced pressures.

***m*-Nitrobenzaldehyde** [99-61-6] M 151.1, m 58°. Crystd from water or EtOH/water, then sublimed twice at 2mm pressure at a temperature slightly above its melting point.

***p*-Nitrobenzaldehyde** [555-16-8] M 151.1, m 106°. Purification as for *m*-nitrobenzaldehyde above.

Nitrobenzene [98-95-3] M 123.1, f 5.8°, b 84-86.5°/6.5-8mm, 210.8°/760mm, d 1.206, $n^{15} 1.55457$, $n 1.55257$, $pK^{18} -11.26$ (aq H_2SO_4). Common impurities include nitrotoluene, dinitrothiophene, dinitrobenzene and aniline. Most impurities can be removed by steam distn in the presence of dilute H_2SO_4 , followed by drying with $CaCl_2$, and shaking with, then distilling at low pressure from BaO, P_2O_5 , $AlCl_3$ or activated alumina. It can also be purified by fractional crystn from absolute EtOH (by refrigeration). Another purification process includes extraction with aqueous 2M NaOH, then water, dilute HCl, and water, followed by drying ($CaCl_2$, $MgSO_4$ or $CaSO_4$) and fractional distn under reduced pressure. The pure material is stored in a brown bottle, in contact with silica gel or CaH_2 . It is very *hygroscopic*.

4-Nitrobenzene-azo-resorcinol (magneson II) [74-39-5] M 259.2, m 199-200°. Crystd from EtOH.

2-Nitrobenzenesulfonyl chloride (NPS-Cl) [7669-54-7] M 189.6, m 73-74.5°, 74.5-75°, 74-76°. Recrystd from CCl_4 (2mL/g), filter off the soln at 5° (recovery 75%). Also recrystd from pet ether (b 40-60°), dried rapidly at 50° and stored in a brown glass bottle, sealed well and stored away from moisture. [Hubacher *Org Synth Coll Vol II* 455 1943; Ito et al. *Chem Pharm Bull (Jpn)* 26 296 1978.]

4-Nitrobenzhydrazide [606-26-8] M 181.1, m 213-214°. Crystd from EtOH.

4'-Nitrobenzo-15-crown-5 [60835-69-0] M 313.3, m 84-85°, 93-95°. Recrystd from EtOH, MeOH or *C₆H₆-hexane as for the 18-crown-6 compound below. It complexes with Na⁺, K⁺, NH₄⁺, Ca²⁺, Mg²⁺ and Cd²⁺. NMR (CDCl₃) has δ: 3.6-4.4 (m 16CH₂), 6.8 (d 1H arom), 7.65 (d 1H arom), 7.80 (dd 1H arom *J*_{ab} 9Hz and *J*_{bc} 3Hz) [Shmid et al. *J Am Chem Soc* 98 5198 1976; Kikukawa et al. *Bull Chem Soc Jpn* 50 2207 1977; Toke et al. *Justus Liebigs Ann Chem* 349 349, 761 1988; Lindner et al. *Z Anal Chem* 322 157 1985].

4'-Nitrobenzo-18-crown-6 [53408-96-1] M 357.4, m 83-84°, 83-84°. If impure and discoloured then chromatograph on Al₂O₃ and eluting with *C₆H₆ (1:1) with 1% MeOH added. The fractions are followed by TLC on Al₂O₃ (using detection with Dragendorff's reagent R_F 0.6 in the above solvent system). Recrystallise the residues from the fractions containing the product from *C₆H₆-hexane to give yellowish leaflets. It complexes with Na or K ions with logK_{Na} 3.95 and logK_K 4.71. [Petranek and Ryba *Collect Chem Czech Chem Commun* 39 2033 1974.]

2-Nitrobenzoic acid [552-16-9] M 167.1, m 146-148°, pK²⁵ 2.21. Crystd from *benzene (twice), *n*-butyl ether (twice), then water (twice). Dried and stored in a vacuum desiccator. [Le Noble and Wheland *J Am Chem Soc* 80 5397 1958.] Has also been crystd from EtOH/water.

3-Nitrobenzoic acid [121-92-6] M 167.1, m 143-143.5°, pK²⁵ 3.46. Crystd from *benzene, water, EtOH (charcoal), glacial acetic acid or MeOH/water. Dried and stored in a vacuum desiccator.

4-Nitrobenzoic acid [62-23-7] M 167.1, m 241-242°, pK²⁵ 3.43. Purification as for 3-nitrobenzoic acid above.

4-Nitrobenzoyl chloride [122-04-3] M 185.6, m 75°, b 155°/20mm. Crystd from dry pet ether (b 60-80°) or CCl₄. Distilled under vacuum. **IRRITANT**.

4-Nitrobenzyl alcohol [619-73-8] M 153.1, m 93°. Crystd from EtOH and sublimed in a vacuum. Purity should be at least 99.5%. Sublimed samples should be stored in the dark over anhydrous CaSO₄ (Drierite). If the IR contains OH bands then the sample should be resublimed before use. [Mohammed and Kosower *J Am Chem Soc* 93 2709 1979.]

4-Nitrobenzyl bromide [100-11-8] M 216.0, m 98.5-99.0°. Recrystd four times from abs EtOH, then twice from cyclohexane/hexane/*benzene (1:1:1), followed by vac sublimation at 0.1mm and a final recrystn from the same solvent mixture. [Lichtin and Rao *J Am Chem Soc* 83 2417 1961.] Has also been crystd from pet ether (b 80-100°, 10mL/g, charcoal). It slowly decomposes even when stored in a desiccator in the dark. **IRRITANT**.

***m*-Nitrobenzyl chloride** [619-23-8] M 171.6, m 45°. Crystd from pet ether (b 90-120°). **IRRITANT**.

***p*-Nitrobenzyl chloride** [100-14-1] M 171.6, m 72.5-73°. Crystd from CCl₄, dry diethyl ether, 95% EtOH or *n*-heptane, and dried under vacuum. **IRRITANT**.

***p*-Nitrobenzyl cyanide** [555-21-5] M 162.2, m 117°. Crystd from EtOH. **TOXIC**.

4-(4-Nitrobenzyl)pyridine (PNBP) [1083-48-3] M 214.2, m 70-71°, pK_{Est} ~5.0. Crystd from cyclohexane.

2-Nitrobiphenyl [86-00-0] M 199.2, m 36.7°. Crystd from EtOH (seeding required). Sublimed under vacuum.

3-Nitrocinnamic acid [555-68-0] M 193.2, m 200-201°, pK²⁵ 2.58 (*trans*). Crystd from *benzene or EtOH.

4-Nitrocinnamic acid [882-06-4] M 193.2, m 143° (*cis*), 286° (*trans*), $pK_{\text{Est}} \sim 2.6$ (*trans*). Crystd from water.

4-Nitrodiphenylamine [836-30-6] M 214.2, m 133-134°, $pK^{25} -2.5$. Crystd from EtOH.

2-Nitrodiphenyl ether [2216-12-8] M 215.2, b 106-108°/0.01mm, 137-138°/0.5mm, 161-162°/4mm, 188-189°/12mm, 195-200°/25mm, $d_4^{20} 1.241$, $n_D^{25} 1.600$. Purified by fractional distn. UV (EtOH): 255, 315nm (ϵ 6200 and 2800); IR (CS₂): 1350 (NO₂) and 1245, 1265 (COC) cm⁻¹ [UV, IR: Dahlgard and Brewster *J Am Chem Soc* **80** 5861 1958; Tomita and Takase *Yakugaku Zasshi (J Pharm Soc Japan)* **75** 1077 1955; Fox and Turner *J Chem Soc* 1115 1930, Henley *J Chem Soc* 1222 1930].

Nitrodurene (1-nitro-2,3,5,6-tetramethylbenzene) [3463-36-3] M 179.2, m 53-55°, b 143-144°/10mm. Crystd from EtOH, MeOH, acetic acid, pet ether or chloroform.

Nitroethane [79-24-3] M 75.1, b 115°, d 1.049, n 1.3920, $n^{25} 1.39015$, $pK^{25} 8.60$ (8.46, pH equilibrium requires *ca* 5 min). Purified as described for *nitromethane*. A spectroscopic impurity has been removed by shaking with activated alumina, decanting and rapidly distilling.

2-Nitrofluorene [607-57-8] M 211.2, m 156°. Crystd from aqueous acetic acid.

Nitroguanidine [556-88-7] M 104.1, m 246-246.5°(dec), 257°, $pK_1^{25} -0.55$, $pK_2^{25} 12.20$. Crystd from water (20mL/g).

5-Nitroindole [6146-52-7] M 162.1, m 141-142°, $pK^{25} -7.4$ (aq H₂SO₄). Decolorised (charcoal) and recrystd twice from aqueous EtOH.

Nitromesitylene (2-nitro-1,3,5-trimethylbenzene) [603-71-4] M 165.2, m 44°, b 255°. Crystd from EtOH.

Nitromethane [75-52-5] M 61.0, f -28.5°, b 101.3°, d 1.13749, $d^{30} 1.12398$, n 1.3819, $n^{30} 1.37730$, $pK^{25} 10.21$. Nitromethane is generally manufactured by gas-phase nitration of methane. The usual impurities include aldehydes, nitroethane, water and small amounts of alcohols. Most of these can be removed by drying with CaCl₂ or by distn to remove the water/nitromethane azeotrope, followed by drying with CaSO₄. Phosphorus pentoxide is not suitable as a drying agent. [Wright et al. *J Chem Soc* 199 1936.] The purified material should be stored by dark bottles, away from strong light, in a cool place. Purifications using extraction are commonly used. For example, Van Looy and Hammett [*J Am Chem Soc* **81** 3872 1959] mixed about 150mL of conc H₂SO₄ with 1L of nitromethane and allowed it to stand for 1 or 2 days. The solvent was washed with water, aqueous Na₂CO₃, and again with water, then dried for several days with MgSO₄, filtered again with CaSO₄. It was fractionally distd before use. Smith, Fainberg and Winstein [*J Am Chem Soc* **83** 618 1961] washed successively with aqueous NaHCO₃, aqueous NaHSO₃, water, 5% H₂SO₄, water and dilute NaHCO₃. The solvent was dried with CaSO₄, then percolated through a column of Linde type 4A molecular sieves, followed by distn from some of this material (in powdered form). Buffagni and Dunn [*J Chem Soc* 5105 1961] refluxed for 24h with activated charcoal while bubbling a stream of nitrogen through the liquid. The suspension was filtered, dried (Na₂SO₄) and distd, then passed through an alumina column and redistd. It has also been refluxed over CaH₂, distd and kept under argon over 4A molecular sieves.

Can be purified by zone melting or by distn under vacuum at 0°, subjecting the middle fraction to several freeze-pump-thaw cycles. An impure sample containing higher nitroalkanes and traces of cyanoalkanes was purified (on the basis of its NMR spectrum) by crystn from diethyl ether at -60° (cooling in Dry-ice)[Parrett and Sun *J Chem Educ* **54** 448 1977].

Fractional crystn was more effective than fractional distn from Drierite in purifying nitromethane for conductivity measurements. [Coetzee and Cunningham *J Am Chem Soc* **87** 2529 1965.] Specific conductivities around 5×10^{-9} ohm⁻¹cm⁻¹ were obtained.

Nitron [1,4-diphenyl-3-phenylamino-(1*H*)-1,2,4-triazolium (hydroxide) inner salt] [2218-94-2] **M 312.4, m 189°(dec)**. Crystd from EtOH, chloroform or EtOH/*C₆H₆.

1-Nitronaphthalene [86-57-7] **M 173.2, m 57.3-58.3°, b 30-40°/0.01mm**. Fractionally distd under reduced pressure, then crystd from EtOH, aqueous EtOH or heptane. Chromatographed on alumina from *benzene/pet ether. Sublimes *in vacuo*.

2-Nitronaphthalene [581-89-5] **M 173.2, m 79°, b 165°/15mm**. Crystd from aqueous EtOH and sublimed in a vacuum.

1-Nitro-2-naphthol [550-60-7] **M 189.2, m 103°, pK²⁵ 5.93**. Crystd (repeatedly) from *benzene-pet ether (b 60-80°)(1:1).

2-Nitro-1-naphthol [607-24-9] **M 189.2, m 127-128°, pK²⁵ 5.89**. Crystd (repeatedly) from EtOH.

5-Nitro-1,10-phenanthroline [4199-88-6] **M 225.2, m 197-198°, pK²⁵ 3.33**. Crystd from *benzene/pet ether, until anhydrous.

2-Nitrophenol [88-75-5] **M 139.1, m 44.5-45.5°, pK²⁵ 7.23**. Crystd from EtOH/water, water, EtOH, *benzene or MeOH/pet ether (b 70-90°). Can be steam distd. Petrucci and Weygandt [*Anal Chem* **33** 275 1961] crystd from hot water (twice), then EtOH (twice), followed by fractional crystn from the melt (twice), drying over CaCl₂ in a vacuum desiccator and then in an Abderhalden drying pistol.

3-Nitrophenol [554-84-7] **M 139.1, m 96°, b 160-165°/12mm, pK²⁵ 8.36**. Crystd from water, CHCl₃, CS₂, EtOH or pet ether (b 80-100°), and dried under vacuum over P₂O₅ at room temperature. Can also be distd at low pressure.

4-Nitrophenol [100-02-7] **M 139.1, m 113-114°, pK²⁵ 7.16**. Crystd from water (which may be acidified, e.g. *N* H₂SO₄ or 0.5*N* HCl), EtOH, aqueous MeOH, CHCl₃, *benzene or pet ether, then dried under vacuum over P₂O₅ at room temperature. Can be sublimed at 60°/10⁻⁴mm.

2-Nitrophenoxyacetic acid [1878-87-1] **M 197.2, m 150-159°, pK²⁵ 2.90**. Crystd from water, and dried over P₂O₅ under vacuum..

***p*-Nitrophenyl acetate** [830-03-5] **M 181.2, m 78-79°**. Recrystd from absolute EtOH [Moss et al. *J Am Chem Soc* **108** 5520 1986].

2-Nitrophenylacetic acid [3740-52-1] **M 181.2, m 120°, pK²⁵ 3.95**. Crystd from EtOH/water and dried over P₂O₅ under vacuum.

4-Nitrophenylacetic acid [104-03-0] **M 181.2, m 80.5°, pK²⁵ 3.92**. Crystd from EtOH/water (1:1), then from sodium-dried diethyl ether and dried over P₂O₅ under vacuum.

4-Nitro-1,2-phenylenediamine [99-56-9] **M 153.1, m 201°, pK₁²⁵ 1.39 (1-NH₂), pK₂²⁵ 2.61 (2-NH₂)**. Crystd from water.

1-(4-Nitrophenyl)ethylamine hydrochloride [*R*-(+)- 57233-86-0; *S*-(-)- 132873-57-5] **M 202.6, m 225°, 240-242° (dec), 243-245° (dec), 248-250°, [α]_D²⁰ (+) and (-) 72° (c 1, 0.05 M NaOH), (+) and (-) 0.3° (H₂O), pK_{Est} ~8.6**. To ensure dryness the hydrochloride (*ca* 175 g) is extracted with EtOH (3x100mL) and evaporated to dryness (any residual H₂O increases the solubility in EtOH and lowers the yield). The hydrochloride residue is triturated with absolute EtOH and dried *in vacuo*. The product is further purified by refluxing with absolute EtOH (200 mL for 83g) for 1h, cool to 10° to give 76.6g of hydrochloride **m 243-245° (dec)**. The *free base* is prepd by dissolving in *N* NaOH, extract with CH₂Cl₂ (3 x 500mL), dry (Na₂CO₃), filter, evaporate and distil, **m 27°, b 119-120°/0.5mm (105-107°/0.5mm, 157-159°/9mm, d₄²⁰**

1.1764, n_D^{20} 1.5688, $[\alpha]_D^{24} \pm 17.7^\circ$ (neat)[Perry et al. *Synthesis* 492 1977; ORD: Nerdel and Liebig *Justus Liebigs Ann Chem* 621 142 1959].

4-Nitrophenylhydrazine [100-16-3] M 153.1, m 158^o(dec), pK_1^{25} -9.2 (aq H₂SO₄), pK_2^{25} 3.70. Crystd from EtOH.

3-Nitrophenyl isocyanate [3320-87-4] M 164.1, m 52-54^o. Crystd from pet ether (b 28-38^o).

4-Nitrophenyl isocyanate [100-28-7] M 164.1, m 53^o. Crystd from pet ether (b 28-38^o).

2-Nitrophenylpropionic acid [530-85-8] M 191.1, m 157^o(dec), pK^{25} 2.83. Crystd from water.

4-Nitrophenyl trifluoroacetate [658-78-6] M 235.1, m 37-39^o, b 120^o/12mm. Recrystd from CHCl₃/hexane [Margolis et al. *J Biol Chem* 253 7891 1078].

4-Nitrophenyl urea [556-10-5] M 181.2, m 238^o. Crystd from EtOH and hot water.

3-Nitrophthalic acid [603-11-2] M 211.1, m 216-218^o, pK^{25} 3.93. Crystd from hot water (1.5mL/g). Air dried.

4-Nitrophthalic acid [610-27-5] M 211.1, m 165^o, pK^{25} 4.12. Crystd from ether or ethyl acetate.

3-Nitrophthalic anhydride [641-70-3] M 193.1, m 164^o. Crystd from *benzene, *benzene/pet ether, acetic acid or acetone. Dried at 100^o.

1-Nitropropane [108-03-2] M 89.1, b 131.4^o, d 1.004, n 1.40161, n^{25} 1.39936, pK^{25} 8.98. Purified as *nitromethane*.

2-Nitropropane [79-46-9] M 89.1, b 120.3^o, d 0.989, n 1.3949, n^{25} 1.39206, pK^{25} 7.68. Purified as *nitromethane*.

5-Nitro-2-*n*-propoxyaniline [553-79-7] M 196.2, m 47.5-48.5^o, pK_{Est} ~2.32. Crystd from *n*-propyl alcohol/pet ether.

3-Nitro-2-pyridinesulfonyl chloride [68206-45-1] M 190.2, m 217-222^o(dec). Crystallises as yellow needles from CH₂Cl₂. When pure it is stable for several weeks at room temperature, and no decomposition was observed after 6 months at <0^o. UV (MeCN) has λ_{max} at 231nm (ϵ 12,988), 264nm (ϵ 5,784) and 372nm (ϵ 3,117). [NMR and UV: Matsuda and Aiba *Chem Lett* 951 1978; Wagner et al. *Chem Ber* 75 935 1942.]

5-Nitroquinoline [607-34-1] M 174.2, m 70^o, pK^{20} 2.69. Crystd from pentane, then from *benzene.

8-Nitroquinoline [706-35-2] M 174.2, m 88-89^o, pK^{20} 2.55. Crystd from hot water, MeOH, EtOH or EtOH/diethyl ether (3:1).

4-Nitroquinoline 1-oxide [56-57-5] M 190.2, m 157^o. Recrystd from aqueous acetone [Seki et al. *J Phys Chem* 91 126 1987].

2-Nitroresorcinol [601-89-8] M 155.1, m 81-81^o, pK_1^{20} 6.37, pK_2^{20} 9.46. Crystd from aq EtOH.

4-Nitrosalicylic acid [619-19-1] M 183.1, m 277-288^o, pK^{25} 2.23. Crystd from water.

5-Nitrosalicylic acid [96-97-9] M 183.1, m 233^o, pK_1^{25} 2.32, pK_2^{25} 10.34. Crystd from acetone (charcoal), then twice more from acetone alone.

Nitrosobenzene [586-96-9] M 107.1, m 67.5-68°, b 57-59°/18mm. Steam distd, then cryst from a small volume of EtOH with cooling below 0°, dried over CaCl₂ in a dessicator at atm pressure, and stored under N₂ at 0°. Alternatively it can be distd onto a cold finger cooled with brine at ~-10° in a vac at 17mm (water pump), while heating in a water bath at 65-70° [Robertson and Vaughan *J Chem Educ* 27 605 1950].

N-Nitrosodiethanolamine [1116-54-7] M 134.4, b 125°/0.01mm, n 1.485. Purified by dissolving the amine (0.5g) in 1-propanol (10mL) and 5g of anhydrous Na₂SO₄ added with stirring. After standing for 1-2h, it was filtered and passed through a chromatographic column packed with AG 50W x 8 (H⁺form, a strongly acidic cation exchanger). The eluent and washings were combined and evapd to dryness at 35°. [Fukuda et al. *Anal Chem* 53 2000 1981.] Possible **CARCINOGEN**.

4-Nitroso-N,N-dimethylaniline [138-89-6] M 150.2, m 86-87°, 92.5-93.5°, b 191-192°/100mm, pK²⁵ 4.54. Recryst from pet ether or CHCl₃-CCl₄ and dried in air. Alternatively suspend in H₂O, heat to boiling and add HCl until it dissolves. Filter, cool and collect the *hydrochloride* [42344-05-8], m 177° after recrystn from H₂O containing a small amount of HCl. The *hydrochloride* (e.g. 30g) is made into a paste with H₂O (100mL) in a separating funnel. Add cold aq 2.5 NaOH or Na₂CO₃ to a pH of ~ 8.0 (green color due to free base) and extracted with toluene, CHCl₃ or Et₂O. Dry extract (K₂CO₃), filter, distil off the solvent, cool residue and collect the crystalline free base. Recryst as above and dried in air.

N-Nitrosodiphenylamine [156-10-5] M 198.2, m 144-145°(dec). Crystd from *benzene.

1-Nitroso-2-naphthol [131-91-9] M 173.2, m 110.4-110.8°, pK²⁵ 7.63. Crystd from pet ether (b 60-80°, 7.5mL/g).

2-Nitroso-1-naphthol [132-53-6] M 173.2, m 158°(dec), pK²⁵ 7.24. Purified by recrystn from pet ether (b 60-80°) or by dissolving in hot EtOH, followed by successive addition of small volumes of water.

4-Nitroso-1-naphthol [605-60-7] M 173.2, m 198°, pK²⁵ 8.18. Crystd from *benzene.

2-Nitroso-1-naphthol-4-sulfonic acid (3H₂O) [3682-32-4] M 316.3, m 142-146°(dec), pK_{Est} ~6.3 (OH). Crystd from dilute HCl soln. Crystals were dried over CaCl₂ in a vacuum desiccator. Also purified by dissolution in aqueous alkali and pptn by addition of water. Reagent for cobalt.

4-Nitrosophenol [104-91-6] M 123.1, m >124°(dec), pK²⁵ 6.36. Crystd from xylene.

N-Nitroso-N-phenylbenzylamine [612-98-6] M 212.2, m 58°. Crystd from absolute EtOH and dried in air.

trans-β-Nitrostyrene [5153-67-3] M 149.2, m 60°. Crystd from absolute EtOH, or three times from *benzene/pet ether (b 60-80°) (1:1).

4-Nitrostyrene [100-13-0] M 149.2, m 20.5-21°. Crystd from CHCl₃/hexane. Purified by addition of MeOH to ppte the polymer, then crystd at -40° from MeOH. Also crystd from EtOH. [Bernasconi et al. *J Am Chem Soc* 108 4541 1986.]

2-Nitro-4-sulfobenzoic acid [552-23-8] M 247.1, m 111°, pK_{Est} ~1.65. Crystd from dilute HCl. Hygroscopic.

2-Nitrotoluene [88-72-2] M 137.1, m -9.55° (α-form), -3.85° (β-form), b 118°/16mm, d 1.163, 222.3°/760mm, n 1.545. Crystd (repeatedly) from absolute EtOH by cooling in a Dry-ice/alcohol mixture, Further purified by passage of an alcoholic soln through a column of alumina.

3-Nitrotoluene [99-08-1] M 137.1, m 16°, b 113-114°/15mm, 232.6°, d 1.156, n 1.544. Dried with P₂O₅ for 24h, then fractionally distd under reduced pressure. [*Org. Synth Coll Vol I* 416 1948.]

4-Nitrotoluene [99-99-0] M 137.1, m 52°. Crystd from EtOH, MeOH/water, EtOH/water (1:1) or MeOH. Air dried, then dried in a vac desiccator over H₂SO₄. [Wright and Grilliom *J Am Chem Soc* 108 2340 1986.]

5-Nitrouracil (2,4-dihydroxy-5-nitropyrimidine) [611-08-5] M 157.1, m 280-285°, >300°, pK₁²⁰ 0.03, pK₂²⁰ 5.55, pK₃²⁰ 11.3. Recrystallises as prisms from boiling H₂O as the monohydrate and loses H₂O on drying *in vacuo*. [UV: Brown *J Chem Soc* 3647 1959; Brown *J Appl Chem* 2 239 1952; Johnson *J Am Chem Soc* 63 263 1941.]

Nitrourea [556-89-8] M 105.1, m 158.4-158.8°(dec). Crystd from EtOH/pet ether.

5-Nitrovanillin (nitroveratric aldehyde) [6635-20-7] M 197.2, m 172-175°, 176°, 178°. Forms yellow plates from AcOH, and needles from EtOH [Slotta and Szyszke *Chem Ber* 68 184 1935]. With diazomethane, 5-nitro-3,4-dimethoxyacetophenone is formed [Brady and Manjunath *J Chem Soc* 125 1067 1924]. The *methyl ether* crystallises from EtOAc or AcOH, m 88°, 90-91°, and the *phenylhydrazone* has m 108-110° (from aqueous EtOH). [Finger and Schott *J Prakt Chem* [2] 115 288 1927.] For *oxime* m 216° (from EtOH or AcOH) and the *oxime acetate* has m 147° (from aq EtOH) [Vogel *Monatsh Chem* 20 384 1899; Brady and Dunn *J Chem Soc* 107 1861 1915].

n-Nonane [111-84-2] M 126.3, b 150.8°, d 0.719, n 1.40542, n²⁵ 1.40311. Fractionally distd, then stirred with successive volumes of conc H₂SO₄ for 12h each until no further colouration was observed in the acid layer. Then washed with water, dried with MgSO₄ and fractionally distd. Alternatively, it was purified by azeotropic distn with 2-ethoxyethanol, followed by washing out the alcohol with water, drying and distilling. [Forziati et al. *J Res Nat Bur Stand* 36 129 1946].

2,5-Norbornadiene [121-46-0] M 92.1, b 89°, d 0.854, n 1.4707. Purified by distn from activated alumina [Landis and Halpern *J Am Chem Soc* 109 1746 1987].

cis-endo-5-Norbornene-2,3-dicarboxylic anhydride (carbic anhydride, 3 α ,4,7,7, $\alpha\alpha$ -tetrahydro-4 α ,7 α -methanoisobenzofuran-1,3-dione) [129-64-6] M 164.2, m 164.1°, 164-165°, 164-167°, d 1.417. Forms crystals from pet ether, hexane or cyclohexane. It is hydrolysed by H₂O to form the acid [Diels and Alder *Justus Liebigs Ann Chem* 460 98 1928; Maitte *Bull Soc Chim Fr* 499 1959]. The *exo-exo-isomer* has m 142-143° (from *C₆H₆-pet ether) [Alder and Stein *Justus Liebigs Ann Chem* 504 216 1933].

Norbornylene [498-66-8] M 94.2, m 44-46°, b 96°. Refluxed over Na, and distd [Gilliom and Grubbs *J Am Chem Soc* 108 733 1986]. Also purified by sublimation *in vacuo* onto an ice-cold finger [Woon et al. *J Am Chem Soc* 108 7990 1986].

Norcamphor (bicyclo[2.2.1]heptan-2-one) [497-38-1] M 110.2, m 94-95°. Crystd from water.

Norcholanic acid [511-18-2] M 346.5, m 177°, 186°, [α]_D²⁰ +32° (EtOH), pK_{Est} ~4.8. Crystd from acetic acid.

Norcodeine [467-15-2] M 285.3, m 185°, 186°, pK 9.10. Crystd from acetone or ethyl acetate.

Nordihydroguaiaretic [1,4-bis(3,4-dihydroxyphenyl)-2,3-dimethylbutane] acid [500-38-9] M 302.4, m 184-185°, pK_{Est(1)} ~9.7, pK_{Est(2)} ~12. Crystd from dilute acetic acid.

Norleucine (α -amino-n-caproic acid) [R(+)- 327-56-0; S(-)- 327-57-1] M 117.2, m 301° [α]₅₄₆²⁰ (+) and (-) 28° (c 5, 5M HCl); [RS: 616-06-8] m 297-300° (sublimes partially at ~280°), pK₁ 2.39, pK₂ 9.76 (for RS). Crystd from water.

Norvaline (*R*- α -amino-*n*-valeric acid) [*R*(+)- 2031-12-9; *S*(-)- 6600-40-4] M 117.2, m 305°(dec), $[\alpha]_{546}^{20}$ (+) and (-) 25° (c 10, 5M HCl), pK_1^{25} 2.36, pK_2^{25} 9.87 (9.72). Crystd from aqueous EtOH or water.

Nylon powder. Pellets were dissolved in ethylene glycol under reflux. Then pptd as a white powder on addition of EtOH at room temperature. This was washed with EtOH and dried at 100° under vacuum.

***n*-Octacosane** [630-02-4] M 394.8, m 62.5°. Purified by forming its adduct with urea, washing and crystallising from acetone/water. [McCubbin *Trans Faraday Soc* 58 2307 1962.] Crystd from hot, filtered isopropyl ether soln (10mL/g).

***n*-Octacosanol** (octacosyl alcohol) [557-61-9] M 410.8, m 83.4°, 84°. Recryst from large vols of Me₂CO. It sublimates at 200-250°/1mm instead of distilling.

***n*-Octadecane** [593-45-3] M 254.5, m 28.1°, b 173.5°/10mm, 316.1°/760mm, d_4^{20} 0.7768, n 1.4390. Crystd from acetone and distd under reduced pressure from sodium.

Octadecyl acetate [822-23-1] M 312.5, m 32.6°. Distd under vac, then crystd from diethyl ether/MeOH.

***n*-Octadecyl alcohol** (stearyl alcohol) [112-92-5] M 270.5, m 61°, b 153-154°/0.3mm. Crystd from MeOH, or dry diethyl ether and *benzene, then fractionally distd under reduced pressure. Purified by column chromatography. Freed from cetyl alcohol by zone melting.

Octadecyl ether (dioctadecyl ether) [6297-03-6] M 523.0, m 59.4°. Vacuum distd, then crystd from MeOH/*benzene.

Octadecyltrimethylammonium bromide [1120-02-1] M 392.5, m 250°(dec). See entry on p. 446 in Chapter 6.

2,3,7,8,12,13,17,18-Octaethyl-21*H*,23*H*-porphine [2683-82-1] M 534.8, m 322°, 326°. Chromatographed on SiO₂ using CHCl₃ as eluent. It crystallises from CHCl₃ (dark red), MeOH (blue violet), pyridine (m 318°) and *C₆H₆ (deep red). [Fischer and Bämle *Justus Liebigs Ann Chem* 468 58, 85 1929.]

Octafluoropropane (profluorane) [76-19-7] M 188.0, m -183°, b -38°. Purified for pyrolysis studies by passage through a copper vessel containing CoF₃ at about 270°, then fractionally distd. [Steunenberg and Cady *J Am Chem Soc* 74 4165 1952.] Also purified by several trap-to-trap distns at low temperatures [Simons and Block *J Am Chem Soc* 59 1407 1937].

1,2,3,4,6,7,8,9-Octahydroanthracene [1079-71-6] M 186.3, m 78°. Crystd from EtOH, then purified by zone melting.

Octamethylcyclotetrasiloxane [556-67-2] M 296.6, m 17.3°, b 175-176°, d_4^{20} 0.957, n 1.396. Purified by zone melting.

Octan-1,8-diol (octamethylene glycol) [629-41-4] M 146.2, m 59-61°, b 172°/20mm. Recrystd from EtOH and distd in a vac.

***n*-Octane** [111-65-9] M 114.2, b 126.5°, d_4^{20} 0.704, n 1.39743, n^{25} 1.39505. Extracted repeatedly with conc H₂SO₄ or chlorosulfonic acid, then washed with water, dried and distd. Also purified by azeotropic distn with EtOH, followed by washing with water to remove the EtOH, drying and distilling. For further details, see *n*-heptane. Also purified by zone melting.

1-Octanethiol [111-88-6] M 146.3, b 86°/15mm, 197-200°/760mm, d_4^{20} 0.8433, n 1.4540, pK^{25} 10.72 (dil *t*-BuOH). Passed through a column of alumina [Battacharyya et al. *J Chem Soc, Faraday Trans 1* 82 135 1986; Fletcher *J Am Chem Soc* 68 2727 1946].

1-Octene [111-66-0] M 112.2, b 121°/742mm, d_4^{20} 0.716, n 1.4087. Distd under nitrogen from sodium which removes water and peroxides. Peroxides can also be removed by percolation through dried, acid washed alumina. Stored under nitrogen in the dark. [Strukul and Michelin *J Am Chem Soc* 107 7563 1985.]

(trans)-2-Octene [13389-42-9] M 112.2, b 124-124.5°/760mm, d_4^{20} 0.722, n 1.4132. Purification as for 1-octene above.

***n*-Octyl alcohol** [111-87-5] M 130.2, b 98°/19mm, 195.3°/760mm, d 0.828, n 1.43018. Fractionally distd under reduced pressure. Dried with sodium and again fractionally distd or refluxed with boric anhydride and distd (b 195-205°/5mm), the distillate being neutralised with NaOH and again fractionally distd. Also purified by distn from Raney nickel and by preparative GLC.

***n*-Octylammonium 9-anthranilate** [88020-99-9] M 351.5, m 134-135°, pK^{25} 10.65 (for octylamine). Recrystd several times from ethyl acetate.

***n*-Octylammonium hexadecanoate** [88020-97-7] M 385.7, m 52-53°. Purified by several recrystns from *n*-hexane or ethyl acetate. The solid was then washed with cold anhydrous diethyl ether, and dried *in vacuo* over P₂O₅.

***n*-Octylammonium octadecanoate** [32580-92-0] M 413.7, m 56-57°. Purified as for the hexadecanoate above.

***n*-Octylammonium tetradecanoate** [17463-35-3] M 358.6, m 46-48°. Purified as for the hexadecanoate above.

4-Octylbenzoic acid [3575-31-3] M 234.3, m 99-100°, pK^{25} 6.5 (80% aq EtOH), pK_{Est} ~4.5 (H₂O). Crystd from EtOH has m 139°; crystd from aq EtOH has m 99-100°. Forms liquid crystals.

***n*-Octyl bromide** [111-83-1] M 193.1, b 201.5°, d_4^{20} 1.118, n^{25} 1.4503. Shaken with H₂SO₄, washed with water, dried with K₂CO₃ and fractionally distd.

4-(*tert*-Octyl)phenol [140-66-9] M 206.3, m 85-86°, b 166°/20mm, pK_{Est} ~ 10.4. Crystd from *n*-hexane.

1-Octyne [629-05-0] M 110.2, b 126.2°/760mm, d_4^{20} 0.717, n^{25} 1.4159. Distd from NaBH₄ to remove peroxides.

α-Oestradiol [57-91-0] M 272.4, m 220-230°, $[\alpha]_D^{20}$ +55° (c 1, dioxane). Crystd from aq EtOH.

β-Oestradiol-3-benzoate [50-50-0] M 376.5, m 194-195°, $[\alpha]_{546}^{20}$ +70° (c 2, dioxane). Crystd from EtOH

Oleic acid [112-80-1] M 282.5, m 16°, b 360°(dec), d_4^{20} 0.891, n^{30} 1.4571, pK^{25} 6.42 (50% aq EtOH), pK_{Est} ~4.8 (H₂O). Purified by fractional crystn from its melt, followed by molecular distn at 10⁻³mm, or by conversion to its methyl ester, the free acid can be crystd from acetone at -40° to -45° (12mL/g). For purification by the use of lead and lithium salts, see Keffler and McLean [*J Soc Chem Ind (London)* 54 176T 1935]. Purification based on direct crystn from acetone is described by Brown and Shinowara [*J Am Chem Soc* 59 6 1937; *pK White J Am Chem Soc* 72 1857 1950].

Oleyl alcohol [143-28-2] M 268.5, b 182-184°/1.5mm, d_4^{20} 0.847, $n_D^{27.5}$ 1.4582. Purified by fractional crystn at -40° from acetone, then distd under vacuum.

Opianic acid (2-formyl-4,5-dimethylbenzoic acid) [519-05-1] M 210.2, m 150°, pK_1^{25} 3.07. Crystd from water.

Orcinol (5-methylresorcinol) [504-15-4] M 124.2, m 107.5°, m 59-61° (hydrate), pK_1^{20} 9.36 (9.48), pK_2^{20} 11.6 (11.20). Crystd from $CHCl_3$ /*benzene (2:3).

L-Ornithine [70-26-8] M 132.2, m 140°, $[\alpha]_D^{25} +16^\circ$ (c 0.5, H_2O), pK_2^{20} 8.75, pK_3^{25} 10.73. Crystd from water containing 1mM EDTA (to remove metal ions).

L-Ornithine monohydrochloride [3184-13-2] M 168.6, m 233°(dec), $[\alpha]_D^{25} +28.3^\circ$ (5M HCl). Likely impurities are citrulline, arginine and D-ornithine. Crystd from water by adding 4 volumes of EtOH and dried in a vacuum desiccator over fused $CaCl_2$.

Orotic acid (H_2O) [50887-69-9] M 174.1, m 235-346°(dec), pK_1^{25} 1.8, pK_2^{25} 9.55. Crystd from water.

Orthanilic acid (2-aminobenzenesulfonic acid) [88-21-1] M 173.2, m >300°(dec), pK_1^{25} 2.49. Crystd from aqueous soln, containing 20mL of conc HCl per L, then crystd from distilled water, and dried in a vacuum desiccator over Sicapent.

Ouabain {3-[(6-deoxy- α -L-mannopyranosyl)oxy]-1,5,11a,14,19-pentahydroxycard-20(22)-enolide} [630-60-4] M 728.8, m 180°(dec), $[\alpha]_{546}^{20} -30^\circ$ (c 1, H_2O). Crystd from water as the octahydrate. Dried at 130°. Stored in the dark.

Oxalic acid ($2H_2O$) [6153-56-6] M 90.0, m 101.5°; [anhydrous 144-62 -7] m 189.5°, pK_1^{25} 1.08 (1.37), pK_2^{25} 3.55 (3.80). Crystd from distilled water. Dried in vacuum over H_2SO_4 . The anhydrous acid can be obtained by drying at 100° overnight.

Oxaloacetic acid [328-42-7] M 132.1, m 160°(decarboxylates), pK_1^{25} 2.22, pK_2^{25} 3.89, pK_3^{25} 13.0. Crystd from boiling ethyl acetate, or from hot acetone by addition of hot *benzene.

2-Oxoglutaric acid (2-oxopentane-1,5-dioic, α -ketoglutaric acid) [328-50-7] M 146.1, m 114°, 115-117°, (pK_{Est} see oxaloacetic acid above). Crystd repeatedly from Me_2CO /*benzene, EtOAc or ethyl propionate.

Oxalylindigo [2533-00-8] M 316.3. Recrystd twice from nitrobenzene and dried by heating *in vacuo* for several hours. [Sehanze et al. *J Am Chem Soc* 108 2646 1986.]

Oxamide [471-46-5] M 88.1, m >320°(dec). Crystd from water, ground and dried in an oven at 150°.

2-Oxazolidinone [497-25-6] M 87.1, m 89-90°, 91°, b 152°/0.4mm. Crystd from *benzene or dichloroethane.

Oxetane (1.3-trimethylene oxide) [503-30-0] M 58.1, b 45-46°/736mm, 47-49°/atm, 48°/760mm, d_4^{20} 0.892, n_D^{20} 1.395. Distd twice from sodium metal and then fractionated through a small column at atmospheric pressure, b 47.0-47.2°. Also purified by preparative gas chromatography using a 2m silica gel column. Alternatively add KOH pellets (50g for 100g of oxetane) and distil through an efficient column or a column packed with 1/4in Berl Saddles and the main portion boiling at 45-50° is collected and redistilled over fused KOH. [Noller *Org Synth Coll Vol III* 835 1955; Dittmer et al. *J Am Chem Soc* 79 4431 1957.]

Oxine Blue [3-(4-hydroxyphenyl)-3-(8-hydroxy-6-quiniliny)-1(3*H*)-isobenzofuranone] [3733-85-5] **M 369.4, m 134-135°**. Recrystd from EtOH. Dried over H₂SO₄.

Palmitic acid anhydride (hexadecanoic anhydride) [623-65-4] **M 494.9, m 63-64°, 64°, d⁸² 0.838, n⁶⁸ 1.436**. It is moisture sensitive and hydrolyses in water. Purified by refluxing with acetic anhydride for 1hr, evaporating and freeing the residue of acetic acid and anhydride by drying the residue at high vac and crystallising from pet ether at low temperature.

[2.2]-Paracyclophane (tricyclo[8.2.2.2^{4,7}]hexadeca-4,6,10,12,13,15-hexaene) [1633-22-3] **M 208.3, m 284°, 285-287°, 286-288°, 288-290°**. Purified by recrystn from AcOH. ¹H-NMR δ: 1.62 (Ar-H) and -1.71 (CH₂) [Waugh and Fessenden *J Am Chem Soc* **79** 846 1957; IR and UV: Cram et al. *J Am Chem Soc* **76** 6132 1954, Cram and Steinberg *J Am Chem Soc* **73** 5691 1951; complex with unsaturated compounds: Cram and Bauer *J Am Chem Soc* **81** 5971 1959; Syntheses: Brink *Synthesis* 807 1975, Givens et al. *J Org Chem* **44** 16087 1979, Kaplan et al. *Tetrahedron Lett* 3665 1976].

Paraffin (oil) [8012-95-1] **d 0.880, n 1.482**. Treated with fuming H₂SO₄, then washed with water and dilute aqueous NaOH, then percolated through activated silica gel.

Paraffin Wax. Melted in the presence of NaOH, washed with water until all of the base had been removed. The paraffin was allowed to solidify after each wash. Finally, 5g of paraffin was melted by heating on a water-bath, then shaken for 20-30min with 100mL of boiling water and fractionally crystd.

Parafuchsin (4,4',4''-triaminotriptyllium [triphenylmethane] carbonium ion, pararosaniline, paramagenta) [467-62-9] **M 305.4, pK 7.57 and free base has pK >13**. Dissolve in EtOH (1.16g in 30mL), filter and add aqueous NH₃ till neutral and ppte by adding H₂O giving 0.8g **m 247° dec** (sintering at 230°). Dissolve in EtOH neutralise with NH₃ add 0.1g of charcoal filter, and repeat, then add H₂O (100mL) to ppte the colourless *carbinol* dry, **m 257° dec** (sintering at 232°). [Weissberger and Theile *J Chem Soc* 148 1934.] The *carbinol* (pseudo-base) was said to have **m 232°** (186° dec), and is slightly sol in H₂O but sol in acids and EtOH [pK: Goldacre and Phillips *J Chem Soc* 172 1949]. The *perchlorate* (dark red with a green shine) has **m 300°** and explodes at 317° [Dilthey and Diaklage *J Prakt Chem* [2] 129 1931].

Paraldehyde (acetaldehyde trimer, 2,4,6-trimethyl-1,3,5-trioxane) [123-63-7] **M 132.2, m 12.5°, 124°, d 0.995, n 1.407**. Washed with water and fractionally distd.

Patulin {4-hydroxy-4*H*-furo[3.2-*c*]pyran-2(6*H*)-one} [149-29-1] **M 154.1, m 110°**. Crystd from diethyl ether or chloroform. (*Highly TOXIC*).

Pavatrine hydrochloride [548-65-2] **M 333.7, m 143-144°**. Recrystd from isopropanol, and dried over P₂O₅ under vacuum.

Pelargonic acid (nonanoic acid) [112-05-0] **M 158, m 15°, b 98.9°/1mm, 225°/760mm, pK²⁵ 4.96**. Esterified with ethylene glycol and distd. (This removes dibasic acids as undistillable residues.) The acid was regenerated by hydrolysing the ester.

Pelargonitrile (octyl cyanide) [2243-27-8] **M 139.2, m -34°, b 92°/10mm, 224°, d 0.818, n 1.4255**. Stirred with P₂O₅ (~5%), distd from it and redistd under vac. IR should have CN but no OH bands.

Pelargonyl chloride (nonanoyl chloride) [764-85-2] **M 176.7, b 88°/12mm, d 0.941, n 1.436**. Refluxed with acetyl chloride (~ 3 vols) for 1h, then distil off the AcCl followed by the nanoyl chloride under reduced pressure. It is moisture sensitive and should be stored in sealed ampules.

Penicillic acid [90-65-3] **M 158.2, m 58-64° (monohydrate), 83-84° (anhydrous, lactone)**. Crystd from water as the monohydrate, or from pet ether. Free acid is in equilibrium with the lactone.