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**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 H<sub>2</sub>SO<sub>4</sub>.

**Palmitic acid anhydride** (hexadecanoic anhydride) [623-65-4] M 494.9, m 63-64°, 64°, d<sup>82</sup> 0.838, n<sup>68</sup> 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<sup>4,7</sup>]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. <sup>1</sup>H-NMR δ: 1.62 (Ar-H) and -1.71 (CH<sub>2</sub>) [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 H<sub>2</sub>SO<sub>4</sub>, 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 NH<sub>3</sub> till neutral and ppt by adding H<sub>2</sub>O giving 0.8g m 247° dec (sintering at 230°). Dissolve in EtOH neutralise with NH<sub>3</sub> add 0.1g of charcoal filter, and repeat, then add H<sub>2</sub>O (100mL) to ppt 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 H<sub>2</sub>O 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 P<sub>2</sub>O<sub>5</sub> under vacuum.

**Pelargonic acid** (nonanoic acid) [112-05-0] M 158, m 15°, b 98.9°/1mm, 225°/760mm, pK<sup>25</sup> 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 P<sub>2</sub>O<sub>5</sub> (~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.

**Pentabromoacetone** [79-49-2] M 452.6, m 76°, pK 8.0 (MeOH), pK<sub>Est</sub> ~4.6 (H<sub>2</sub>O). Crystd from diethyl ether or EtOH and sublimes.

**Pentabromophenol** [608-71-9] M 488.7, m 229°, pK<sub>Est</sub> ~ 4.5. Purified by crystn (charcoal) from toluene then from CCl<sub>4</sub>. Dried for 2 weeks at ca 75°.

**1-Pentacene** [135-48-8] M 278.4, m 300°. Crystd from \*benzene.

**Pentachloroethane (pentalin)** [76-01-7] M 202.3, b 69°/37mm, 152.2°/64mm, 162.0°, d 1.678, n<sup>15</sup> 1.50542. Usual impurities include trichloroethylene. Partially decomposes if distd at atmospheric pressure. Drying with CaO, KOH or sodium is unsatisfactory because of the elimination of the elements of HCl. It can be purified by steam distn, or by washing with conc H<sub>2</sub>SO<sub>4</sub>, water, and then aqueous K<sub>2</sub>CO<sub>3</sub>, drying with solid K<sub>2</sub>CO<sub>3</sub> or CaSO<sub>4</sub>, and fractionally distd under reduced pressure.

**Pentachloronitrobenzene** [82-68-8] M 295.3, m 146°. Crystd from EtOH.

**Pentachlorophenol** [87-86-5] M 266.3, m 190-191°, pK<sup>25</sup> 4.8. Twice crystd from toluene/EtOH. Sublimed *in vacuo*.

**Pentachloropyridine** [2176-62-7] M 251.3, m 122-124°, 123°, 124°, 124-125°, 125-126°, b 279-280°/atm, pK -6.02 (aq H<sub>2</sub>SO<sub>4</sub>). Purified by recryst from EtOH or aqueous EtOH. It sublimes at 150°/3mm. [den Hertog et al. *Recl Trav Chim Pays-Bas* 69 673 1950; Schikh et al. *Chem Ber* 69 2604 1936.]

**Pentachlorothiophenol** [133-49-3] M 282.4, m between 228° and 235°, pK<sub>Est</sub> ~1.1. Crystd from \*benzene.

**Pentadecafluoro octanoic acid (perfluorocaprylic acid)** [335-67-1] M 414.1, m 54.9-55.6°, b 189°/736mm, pK<sub>Est</sub> <0. Recrystd from CCl<sub>4</sub> and toluene, and can be distd. It forms micelles in H<sub>2</sub>O and the solubility is 1% in H<sub>2</sub>O. [Bernett and Zisman *J Phys Chem* 63 1911 1959; IR: Bro and Sperati *J Polym Sci* 38 289 1959.]

**Pentadecanoic acid** [1002-84-2] M 242.4, m 51-53°, 80°, b 158°/1mm, 257°/760mm, d<sup>80</sup> 0.8424, pK<sub>Est</sub> ~5.0. Cryst from Et<sub>2</sub>O and distd. Very hygroscopic. See purification of palmitic acid.

**Pentadecanolide (1-oxacyclohexadecan-2-one, pentadecanoic- $\omega$ -lactone, 15-hydroxypentadecanoic lactone, exaltolide, Tibetolide)** [106-02-5] M 240.4, m 34-36°, 37-37.5°, 37-38°, b 102-103°/0.03mm, 112-114°/0.2mm, 137°/2mm, 169°/10-11mm, d<sub>4</sub><sup>40</sup> 0.9401. It has been recrystd from MeOH (4 parts) at -15°. [Hundiecker and Erlbach *Chem Ber* 80 135 1947; Galli and Mandolini *Org Synth* 58 100 1978; Demole and Enggist *Helv Chim Acta* 11 2318 1978.]

**Penta-1,3-diene** [*cis*: 1574-41-0; *trans*: 2004-70-8] M 68.1, b 42°, d 0.680, n 1.4316. Distd from NaBH<sub>4</sub>. Purified by preparative gas chromatography. [Reimann et al. *J Am Chem Soc* 108 5527 1986.]

**Penta-1,4-diene** [591-93-5] M 68.1, b 25.8-26.2°/756mm, d 0.645, n 1.3890. Distd from NaBH<sub>4</sub>. Purified by preparative gas chromatography. [Reimann et al. *J Am Chem Soc* 108 5527 1986.]

**Pentaerythritol** [115-77-5] M 136.2, m 260.5°. Refluxed with an equal volume of MeOH, then cooled and the ppte dried at 90°. Crystd from dil aq HCl. Sublimed under vacuum at 200°.

**Pentaerythritol tetraacetate** [597-71-7] M 304.3, m 78-79°. Crystd from hot water, then leached with cold water until the odour of acetic acid was no longer detectable.

**Pentaerythrityl laurate** [13057-50-6] M 864.6, m 50°. Crystd from pet ether.

**Pentaerythritol tetranitrate.** [78-11-5] M 316.2, m 140.1°. Crystd from acetone or acetone/EtOH. **EXPLOSIVE.**

**Pentaethylenehexamine** [4067-16-7] M 232.4, d 0.950,  $n_D^{20}$  1.510,  $pK_1$  1.2,  $pK_2$  2.7,  $pK_3$  4.3,  $pK_4$  7.8,  $pK_5$  9.1,  $pK_6$  9.9 (all estimated). Fractionally distd twice at 10-20mm, the fraction boiling at 220-250° being collected. Its soln in MeOH (40mL in 250mL) was cooled in an ice-bath and conc HCl was added dropwise with stirring. About 50mL was added, and the ppted hydrochloride was filtered off, washed with acetone and diethyl ether, then dried in a vacuum desiccator. [Jonassen et al. *J Am Chem Soc* 79 4279 1957.]

**Pentafluorobenzene** [363-72-4] M 168.1, b 85°/atm, 85-86°/atm, 88-89°/atm,  $d_4^{20}$  1.524,  $n_D^{20}$  1.3931. Purified by distn and by gas chromatography. IR film: 1535 and 1512  $cm^{-1}$  (\*benzene ring). [UV: Stephen and Tatlow *Chem Ind (London)* 821 1957 ; Nield et al. *J Chem Soc* 166 1959.] See triethylenepentamine

**2,3,4,5,6-Pentafluorobenzoic acid** [602-94-8] M 212.1, m 101-103°, 103-104°, 104-105°, 106-107°,  $pK^{25}$  1.75. Dissolve in Et<sub>2</sub>O, treat with charcoal, filter, dry (CaSO<sub>4</sub>), filter, evaporate and recrystallise residue from pet ether (b 90-100°) after adding a little toluene to give large colourless plates. UV (H<sub>2</sub>O):  $\lambda_{max}$  265nm ( $\epsilon$  761). The *S*-benzylisothiuronium salt has m 187° after recrystn from H<sub>2</sub>O. [McBee and Rapkin *J Am Chem Soc* 73 1366 1951; Nield et al. *J Chem Soc* 166, 170 1959.]

***O*-(2,3,4,5,6-Pentafluorobenzyl)hydroxylamine hydrochloride (PFBHA)** [57981-02-9] M 249.6, m 215°, 215-216°,  $pK_{Est}$  ~1.1. Recrystd from EtOH to form colourless leaflets. Drying the compound at high vacuum and elevated temperature will result in losses by sublimation. [Youngdale *J Pharm Sci* 65 625 1976; Wehner and Handke *J Chromatog* 177 237 1979; Nambara et al. give incorrect m as 115-116° *J Chromatogr* 114 81 1975.]

**2,3,4,5,6-Pentafluorophenol** [771-61-9] M 184.1, m 33-35°, 38.5-39.5°, b 72-74°/48mm, 142-144°/atm, 143°/atm,  $n_D^{20}$  1.4270 (liquid prep),  $pK^{25}$  5.53. A *hygroscopic* low melting solid not freely soluble in H<sub>2</sub>O. Purified by distn, preferably in a vacuum [Forbes et al. *J Chem Soc* 2019 1959; IR and  $pK_a$ : Birchall and Haszeldine *J Chem Soc* 13 1959]. IR film: 3600 (OH) and 1575 (fluoroaromatic breathing)  $cm^{-1}$ . The *benzoyl* derivative has m 74-75°, *3,4-dinitrobenzoyl* derivative has m 107°, the *tosylate* has m 64-65° (from EtOH) and the *K salt* crystallises from Me<sub>2</sub>CO, m 242° dec, with 1H<sub>2</sub>O salt the m is 248° dec and the 2H<sub>2</sub>O salt has m 245° dec.

**1-(Pentafluorophenyl)ethanol** [*R*-(+)- 104371-21-3; *S*-(-)- 104371-20-2] M 212.1, m 41-42°, 42°, 42.5-43°,  $[\alpha]_{546}^{20}$  (+) and (-) 9°,  $[\alpha]_D^{20}$  (+) and (-) 7.5° (c 1, *n*-pentane). Recrystd from *n*-pentane at -40° and vacuum sublimed at room temp at 0.3mm (use ice cooled cold finger). It has also been purified by column chromatography through Kieselgel 60 (0.063-0.2mm mesh, Merck), eluted with EtOAc-*n*-hexane (1:5), then recrystd from *n*-pentane and vacuum sublimed. It has  $R_F$  on Kieselgel 60 F<sub>254</sub> TLC foil and eluting with EtOAc-*n*-hexane (1:5). [Meese *Justus Liebigs Ann Chem* 2004 1986.] The *racemate* [75853-08-6] has m 32-34°, b 77-79°/8mm, 80-82°/37mm,  $n_D^{20}$  1.4426 and the *3,4-dinitrobenzoate* has m 83° [Nield et al. *J Chem Soc* 166 1959].

**2,2,3,3,3-Pentafluoropropan-1-ol** [422-05-9] M 150.1, b 80°, d 1.507, n 1.288,  $pK^{25}$  12.74. Shaken with alumina for 24h, dried with anhydrous K<sub>2</sub>CO<sub>3</sub>, and distd, collecting the middle fraction (b 80-81°) and redistilling.

**Pentafluoropyridine** [700-16-3] M 169.1, m -41.5°, b 83.5°, 84°, 83-85°,  $d_4^{20}$  1.609,  $n_D^{20}$  1.3818,  $pK_{Est}$  ~<0. Distd through a concentric tube column; has  $\lambda$  max in cyclohexane at 256.8nm. [Chambers et al. *J Chem Soc* 3573 1964]; <sup>19</sup>F NMR: Bell et al. *J Fluorine Chem* 1 51 1971.] The *hexafluoroantimonate* has m 98-102° dec.

**Pentamethylbenzene** [700-12-9] M 148.3, m 53.5-55.1°. Successively crystd from absolute EtOH, toluene and MeOH, and dried under vacuum. [Rader and Smith *J Am Chem Soc* **84** 1443 1962.] It has also been crystd from \*benzene or aqueous EtOH, and sublimed.

**n-Pentane** [109-66-0] M 72.2, b 36.1°, d 0.626, n<sup>25</sup> 1.35472. Stirred with successive portions of conc H<sub>2</sub>SO<sub>4</sub> until there was no further coloration during 12h, then with 0.5N KMnO<sub>4</sub> in 3M H<sub>2</sub>SO<sub>4</sub> for 12h, washed with water and aqueous NaHCO<sub>3</sub>. Dried with MgSO<sub>4</sub> or Na<sub>2</sub>SO<sub>4</sub>, then P<sub>2</sub>O<sub>5</sub> and fractionally distd through a column packed with glass helices. It was also purified by passage through a column of silica gel, followed by distn and storage with sodium hydride. An alternative purification is by azeotropic distn with MeOH, which is subsequently washed out from the distillate (using water), followed by drying and distn. For removal of carbonyl-containing impurities, see *n-heptane*. Also purified by fractional freezing (ca 40%) on a copper coil through which cold air was passed, then washed with conc H<sub>2</sub>SO<sub>4</sub> and fractionally distd.

**Pentane-1-thiol** [110-66-7] M 104.2, m -76°, b 122.9°/697.5mm, d<sup>25</sup> 0.8375, pK<sub>Est</sub> ~10.1. Dissolved in aqueous 20% NaOH, then extracted with a small amount of diethyl ether. The soln was acidified slightly with 15% H<sub>2</sub>SO<sub>4</sub>, and the thiol was distd out, dried with CaSO<sub>4</sub> or CaCl<sub>2</sub>, and fractionally distd under nitrogen. [Ellis and Reid *J Am Chem Soc* **54** 1674 1932.]

**Pentan-2-ol** [6032-29-7] M 88.2, b 119.9°, d 0.810, n 1.41787, n<sup>25</sup> 1.4052. Refluxed with CaO, distd, refluxed with magnesium and again fractionally distd.

**Pentan-3-ol** [584-02-1] M 88.2, b 116.2°, d 0.819, n<sup>25</sup> 1.4072. Refluxed with CaO, distd, refluxed with magnesium and again fractionally distd.

**Pentan-3-one (diethyl ketone)** [96-22-0] M 86.1, b 102.1°, d 0.8099, n 1.392. See diethyl ketone on p. 204.

**Pentaquine monophosphate** [5428-64-8] M 395.6, m 189-190°, pK<sup>70</sup> 8.22. Crystd from 95% EtOH.

**Pent-2-ene (mixed isomers)** [109-68-2] M 70.1, b 36.4°, d 0.650, n 1.38003, n<sup>25</sup> 1.3839. Refluxed with sodium wire, then fractionally distd twice through a Fenske (glass helices packing) column.

**cis-Pent-2-ene** [627-20-3] M 70.1, b 37.1°, d 0.657, n<sup>25</sup> 1.3798. Dried with sodium wire and fractionally distd, or purified by azeotropic distn with MeOH, followed by washing out the MeOH with water, drying and distilling. Also purified by chromatography through silica gel and alumina [Klassen and Ross *J Phys Chem* **91** 3668 1987].

**trans-Pent-2-ene** [646-04-8] M 70.1, b 36.5°, d 0.6482, n 1.3793. It was treated as above and washed with water, dried over anhydrous Na<sub>2</sub>CO<sub>3</sub>, and fractionally distd. The middle cut was purified by two passes of fractional melting.

**Pentobarbital (5-ethyl-5-1'-methylbutyl barbituric acid, Nembutal)** [76-74-4] M 226.4, m ~127°(dec), pK<sub>Est(1)</sub> ~ 8.0, pK<sub>Est(2)</sub> ~12.7. Soln of the sodium salt in 10% HCl was prepared and the acid was extracted by addition of ether. Then purified by repeated crystn from CHCl<sub>3</sub>. [Bucket and Sandorfy *J Phys Chem* **88** 3274 1984.]

**Pentyl acetate (n-amyl acetate)** [628-63-7] M 130.2, b 147-149°/atm, 149.55°, 149.2°/atm, d<sub>4</sub><sup>20</sup> 0.8753, n<sub>D</sub><sup>20</sup> 1.4028. Purified by repeated fractional distn through an efficient column or spinning band column. [Timmermann and Hennant-Roland *J Chim Phys* **52** 223 1955; Mumford and Phillips *J Chem Soc* **75** 1950; <sup>1</sup>H NMR: Crawford and Foster *Can J Phys* **34** 653 1956.]

**Pent-2-yne** [627-21-4] M 68.1, b 26°/2.4mm, d 0.710, n<sup>25</sup> 1.4005. Stood with, then distd at low pressure from, sodium or NaBH<sub>4</sub>.

**Perbenzoic acid** [93-59-4] M 138.1, m 41-43°, b 97-110°/13-15mm, pK<sub>Est</sub> ~7.7. Crystd from \*benzene or pet ether. Readily sublimed and is steam volatile. Sol in CHCl<sub>3</sub>, CCl<sub>4</sub> and Et<sub>2</sub>O. [*Org Synth Coll Vol I* (2nd Edn) 431 1948.] **EXPLOSIVE.**

**Perchlorobutadiene** [87-68-3] M 260.8, b 144.1°/100mm, 210-212°/760mm, d 1.683, n 1.5556. Washed with four or five 1/10th volumes of MeOH (or until the yellow colour has been extracted), then stirred for 2h with H<sub>2</sub>SO<sub>4</sub>, washed with distilled water until neutral and filtered through a column of P<sub>2</sub>O<sub>5</sub>. Distd under reduced pressure through a packed column. [Rytner and Bauer *J Am Chem Soc* 82 298 1960.]

**Perfluorobutyric acid** [375-22-4] M 214.0, m -17.5°, b 120°/735mm, d 1.651, n<sup>16</sup> 1.295, pK<sup>25</sup> -0.17. Fractionally distd twice in an Oldershaw column with an automatic vapour-dividing head, the first distn in the presence of conc H<sub>2</sub>SO<sub>4</sub> as a drying agent.

**Perfluorocyclobutane** [115-25-3] M 200.0, m -40°, b -5°, d<sup>-20</sup> 1.654, d° 1.72. Purified by trap-to-trap distn, retaining the middle portion.

**Perfluorocyclohexane** [355-68-0] M 300.1, m 51° (sublimes), b 52°. Extracted repeatedly with MeOH, then passed through a column of silica gel (previously activated by heating at 250°).

**Perfluoro-1,3-dimethylcyclohexane** [335-27-3] M 400.1, b 101°, d 1.829, n 1.300. Fractionally distd, then 35mL was sealed with about 7g KOH pellets in a borosilicate glass ampoule and heated at 135° for 48h. The ampoule was cooled and opened, and the liquid was resealed with fresh KOH in another ampoule and heated as before. This process was continued until no further decomposition was observed. The substance was then washed with distilled water, dried (CaSO<sub>4</sub>) and distd. [Grafstein *Anal Chem* 26 523 1954.]

**Perfluoroheptane** [335-57-9] M 388.1, b 99-101°, d<sup>25</sup> 1.7200. Purified as for *perfluorodimethylhexane*. Other procedures include shaking with H<sub>2</sub>SO<sub>4</sub>, washing with water, drying with P<sub>2</sub>O<sub>5</sub> for 48h and fractionally distilling. Alternatively, it has been refluxed for 24h with saturated acid KMnO<sub>4</sub> (to oxidise and remove hydrocarbons), then neutralised, steam distd, dried with P<sub>2</sub>O<sub>5</sub>, and passed slowly through a column of dry silica gel. It has been purified by fractional crystn, using partial freezing.

**Perfluoro-*n*-hexane** [355-42-0] M 338.1, m -4°, b 58-60°, d 1.684. Purified by fractional freezing. The methods described for *perfluoroheptane* should be applicable here.

**Perfluoro(methylcyclohexane)** [355-02-2] M 350.1, b 76.3°, d<sup>25</sup> 1.7878. Refluxed for 24h with saturated acid KMnO<sub>4</sub> (to oxidise and remove hydrocarbons), then neutralised, steam distd, dried with P<sub>2</sub>O<sub>5</sub> and passed slowly through a column of dry silica gel. [Glew and Reeves *J Phys Chem* 60 615 1956.] Also purified by percolation through a 1m neutral activated alumina column, and <sup>1</sup>H-impurities checked by NMR.

**Perfluorononane** [375-96-2] M 488.1, b 126-127°, d 1.80, n 1.275. Purified as for *perfluorodimethylcyclohexane*.

**Perfluoropropyl iodide** [754-34-7] M 295.9, b 41°, d 2.13, n 1.339. Purified by fractional distn.

**Perfluorotributylamine (heptacosafuorotributylamine)** [311-89-7] M 671.1, b 177.6°/760mm, d 1.881, n 1.291, pK<sub>Est</sub> ~5.0. Purified as for *perfluorodimethylcyclopropane*, see also *perfluorotripropylamine* [Hazeldine *J Chem Soc* 102 1951].

**Perfluorotripropylamine (heneicosafuorotripropylamine)** [338-83-0] M 521.1, b 130°/atm, 129.5-130.5°/atm, d 1.822, n 1.279, pK<sub>Est</sub> ~5.6. Purified as for *perfluorodimethylcyclopropane*. [Hazeldine *J Chem Soc* 102 1951, for azeotropes see Simons and Linevsky *J Am Chem Soc* 74 4750 1972.] **IRRITANT.**

**Pericyazine** [10-{3-(4-hydroxy-1-piperidinyl)-propyl}-10*H*-phenothiazine-2-carbonitrile] [2622-26-6] M 365.4, m 116-117°. Recrystd from a saturated soln in cyclohexane. Antipsychotic and is a reagent for Pd and Rh.

**Perylene** [198-55-0] M 252.3, m 273-274°. Purified by silica-gel chromatography of its recrystd picrate. [Ware *J Am Chem Soc* 83 4374 1961.] Crystd from \*benzene, toluene or EtOH and sublimed in a flow of oxygen-free nitrogen. [Gorman et al. *J Am Chem Soc* 107 4404 1985; Johansson et al. *J Am Chem Soc* 109 7374 1987.]

**Petroleum ether** [8032-32-4] b 35-60°, d 0.640, n 1.363. Shaken several times with conc H<sub>2</sub>SO<sub>4</sub>, then 10% H<sub>2</sub>SO<sub>4</sub> and conc KMnO<sub>4</sub> (to remove unsatd, including aromatic, hydrocarbons) until the permanganate colour persists. Washed with water, aqueous Na<sub>2</sub>CO<sub>3</sub> and again with water. Dried with CaCl<sub>2</sub> or Na<sub>2</sub>SO<sub>4</sub>, and distd. It can be dried further using CaH<sub>2</sub> or sodium wire. Passage through a column of activated alumina, or treatment with CaH<sub>2</sub> or sodium, removes peroxides. For the elimination of carbonyl-containing impurities without using permanganate, see *n-heptane*. These procedures could be used for all fractions of pet ethers.

**Rapid purification:** Pass through an alumina column and fractionally distilling, collecting the desired boiling fraction.

**R(-)- $\alpha$ -Phellandrene** (*p-menta-1,5-diene*) [4221-98-1] M 136.2, b 61°/11mm, 175-176°/760mm, d 0.838, n 1.471. Purified by gas chromatography on an Apiezon column.

**Phenacylamine hydrochloride** [5468-37-1] M 171.6, m 194°(dec). See 2-aminoacetophenone hydrochloride on p. 103.

**Phenanthrene** [85-01-8] M 178.2, m 98°. Likely contaminants include, anthracene, carbazole, fluorene and other polycyclic hydrocarbons. Purified by distn from sodium, boiling with maleic anhydride in xylene, crystn from acetic acid, sublimation and zone melting. Has also been recrystd repeatedly from EtOH, \*benzene or pet ether (b 60-70°), with subsequent drying under vacuum over P<sub>2</sub>O<sub>5</sub> in an Abderhalden pistol. Feldman, Pantages and Orchin [*J Am Chem Soc* 73 4341 1951] separated from most of the anthracene impurity by refluxing phenanthrene (671g) with maleic anhydride (194g) in xylene (1.25L) under nitrogen for 22h, then filtered. The filtrate was extracted with aqueous 10% NaOH, the organic phase was separated, and the solvent was evaporated. The residue, after stirring for 2h with 7g of sodium, was vacuum distd, then recrystd twice from 30% \*benzene in EtOH, then dissolved in hot glacial acetic acid (2.2mL/g), slowly adding an aqueous soln of CrO<sub>3</sub> (60g in 72mL H<sub>2</sub>O added to 2.2L of acetic acid), followed by slow addition of conc H<sub>2</sub>SO<sub>4</sub> (30mL). The mixture was refluxed for 15min, diluted with an equal volume of water and cooled. The ppt was filtered off, washed with water, dried and distd, then recrystd twice from EtOH. Further purification is possible by chromatography from CHCl<sub>3</sub> soln on activated alumina, with \*benzene as eluent, and by zone refining.

**Phenanthrene-9-aldehyde** [4707-71-5] M 206.3, m 102-103°, pK -6.39 (aq H<sub>2</sub>SO<sub>4</sub>). Crystd from EtOH and sublimed at 95-98°/0.07mm.

**9,10-Phenanthrenequinone** [84-11-7] M 208.2, m 208°, pK -7.1 (aq H<sub>2</sub>SO<sub>4</sub>). Crystd from dioxane or 95% EtOH and dried under vacuum.

**Phenanthridine** [229-87-8] M 179.2, m 106.5°, 108-109°, b 350°, pK<sup>20</sup> 4.61 (4.48). Purified via the HgCl<sub>2</sub> addition compound formed when phenanthridine (20g) in 1:1 HCl (100mL) was added to aq HgCl<sub>2</sub> (60g in 3L), and the mixture was heated to boiling. Conc HCl was then added until all of the solid had dissolved. The compound separated on cooling, and was dec with aq NaOH (ca 5M). Phenanthridine was extracted with Et<sub>2</sub>O and crystd from pet ether (b 80-100°) or EtOAc. [Cumper et al. *J Chem Soc* 45218 1962.] Also purified by zone melting; sublimes in vac. [Slough and Ubbelohde *J Chem Soc* 911 1957.] See p. 124.

**1,10-Phenanthroline** (*o-phenanthroline*) [66-71-7 (anhydr); 5144-89-8 (H<sub>2</sub>O)] M 198.2, m 98-101°, 108-110° (hydr), 118° (anhydr), b >300°, pK<sub>1</sub><sup>25</sup> -0.7 (aq HClO<sub>4</sub>), pK<sub>2</sub><sup>25</sup> 4.86 (4.96). Crystd as its picrate (m 191°) from EtOH, then the free base was liberated, dried at 78°/8mm over P<sub>2</sub>O<sub>5</sub> and crystd from pet ether (b 80-100°). [Cumper, Ginman and Vogel *J Chem Soc* 1188 1962.] It can be purified by zone melting. Also crystd from hexane, \*benzene/pet ether (b 40-60°) or sodium-dried \*benzene, dried and stored over H<sub>2</sub>SO<sub>4</sub>. The monohydrate is obtained by crystn from aqueous EtOH or ethyl acetate. It has been crystd from H<sub>2</sub>O (300 parts) to give the monohydrate m 102-103° and sublimes at 10<sup>-3</sup>mm [Fielding and LeFevre *J*

*Chem Soc* 1811 1951.] The *anhydrous* compound has *m* 118° (after drying at high vacuum at 80°), also after recrystn from pet ether or \*C<sub>6</sub>H<sub>6</sub> (70 parts) and drying at 78°/8mm. [UV: Badger et al. *J Chem Soc* 3199 1951.] It has a pKa in H<sub>2</sub>O of 4.857 (25°) or 5.02 (20°) and 4.27 in 50% aq EtOH (20°) [Albert et al. *J Chem Soc* 2240 1948].

**1,10-Phenanthroline hydrochloride** (*o*-phenanthroline hydrochloride) [3829-86-5] *M* 243.7, *m* 212-219°. It crystallises from 95% EtOH, *m* 212-219° as the *monohydrate*, the *half hydrate* has *m* 217°. The *3HCl* has *m* 143-145° (sinters at 128°) [Thevenet et al. *Acta Cryst Sect B* 33 2526 1977].

**4,7-Phenanthroline-5,6-dione** [84-12-8] *M* 210.2, *m* 295°(dec). Crystd from MeOH.

**Phenazine** [92-82-0] *M* 180.2, *m* 171°, *pK*<sub>1</sub><sup>20</sup> -4.9 (aq H<sub>2</sub>SO<sub>4</sub>), *pK*<sub>2</sub><sup>20</sup> 1.21. Crystd from EtOH, CHCl<sub>3</sub> or ethyl acetate, after pre-treatment with activated charcoal. It can be sublimed *in vacuo*, and zone refined.

**Phenazine methosulfate** [299-11-6] *M* 306.3. See 5-methylphenazinium methyl sulfate on p. 547 in Chapter 6.

**Phenethylamine** [64-04-0] *M* 121.2, *b* 87°/13mm, *d* 0.962, *n* 1.535, *pK*<sup>25</sup> 9.88. Distd from CaH<sub>2</sub>, under reduced pressure, just before use.

**Phenethyl bromide** [103-63-9] *M* 185.1, *b* 92°/11mm, *d* 1.368, *n* 1.557. Washed with conc H<sub>2</sub>SO<sub>4</sub>, water, aq 10% Na<sub>2</sub>CO<sub>3</sub> and water again, then dried with CaCl<sub>2</sub> and fractionally distd just before use.

***N*-2-Phenethyl urea** [2158-04-5] *M* 164.2, *m* 173-174°. Crystd from water.

**Phenetole** [103-73-1] *M* 122.2, *b* 60°/9mm, 77.5°/31mm, 170.0°/760mm, *d* 0.967, *n* 1.50735, *n*<sup>46</sup> 1.50485. Small quantities of phenol can be removed by shaking with NaOH, but this is not a very likely contaminant of commercial material. Fractional distn from sodium, at low pressures, probably gives adequate purification. It can be dissolved in diethyl ether and washed with 10% NaOH (to remove phenols), then water. The ethereal soln was evaporated and the phenetole fractionally distd under vacuum.

**Phenocoll hydrochloride** (4-ethoxyaniline, *p*-phenetidine HCl) [536-10-6] *M* 230.7, *m* 234°, *pK*<sup>28</sup> 5.20. Crystd from water. Sublimes *in vacuo*.

**Phenol** [108-95-2] *M* 94.1, *m* 40.9°, *b* 85.5-86.0°/20mm, 180.8°/760mm, *d* 1.06, *n*<sup>41</sup> 1.54178, *n*<sup>46</sup> 1.53957, *pK*<sup>25</sup> 9.86 (10.02). Steam was passed through a boiling soln containing 1mole of phenol and 1.5-2.0moles of NaOH in 5L of H<sub>2</sub>O until all non-acidic material had distd. The residue was cooled, acidified with 20% (v/v) H<sub>2</sub>SO<sub>4</sub>, and the phenol was separated, dried with CaSO<sub>4</sub> and fractionally distd under reduced pressure. It was then fractionally crystd several times from its melt [Andon et al. *J Chem Soc* 5246 1960]. Purification *via* the benzoate has been used by Berliner, Berliner and Nelidow [*J Am Chem Soc* 76 507 1954]. The benzoate was crystd from 95% EtOH, then hydrolysed to the free phenol by refluxing with two equivalents of KOH in aq EtOH until the soln became homogeneous. It was acidified with HCl and extracted with diethyl ether. The ether layer was freed from benzoic acid by thorough extraction with aqueous NaHCO<sub>3</sub>, and, after drying and removing the ether, the phenol was distd.

Phenol has also been crystd from a 75% w/w soln in water by cooling to 11° and seeding with a crystal of the hydrate. The crystals were centrifuged off, rinsed with cold water (0-2°) satd with phenol, and dried. It can be crystd from pet ether [Berasconi and Paschalis *J Am Chem Soc* 108 2969 1986].

Draper and Pollard [*Science* 109 448 1949] added 12% water, 0.1% aluminium (can also use zinc), and 0.05% NaHCO<sub>3</sub> to phenol, and distd at atmospheric pressure until the azeotrope was removed. The phenol was then distd at 25mm. Phenol has also been dried by distn from the \*benzene soln to remove the water-\*benzene azeotrope and the excess \*benzene, followed by distn of the phenol at reduced pressure under nitrogen. Processes such as this are probably adequate for analytical grade phenol which has as its main impurity water. Phenol has also been crystd from pet ether/\*benzene or pet ether (*b* 40-60°). Purified material is stored in a vacuum desiccator over P<sub>2</sub>O<sub>5</sub> or CaSO<sub>4</sub>.

**Phenol-2,4-disulfonic acid** [96-77-5] M 254.2,  $pK_1 < 1$ ,  $pK_2 < 1$ ,  $pK_3 \sim 8.3$ . Crystd from EtOH/diethyl ether.

**Phenolphthalein** [77-09-8] M 319.2, m 263°,  $pK_{Est(1)} \sim 4.2$ ,  $pK_{Est(2)} \sim 9.8$ . Dissolved in EtOH (7mL/g), then diluted with eight volumes of cold water. Filtered. Heated on a water-bath to remove most of the alcohol and the pptd phenolphthalein was filtered off and dried under vacuum.

**Phenolphthalol** [81-92-5] M 306.3, m 201-202°,  $pK_{Est} \sim 9.8$ . Crystd from aqueous EtOH.

**Phenosafranin (3,7-diamino-5-phenylphenazinium chloride)** [81-93-6] M 322.8, m  $> 300^\circ$ ,  $\lambda_{max}$  530nm (H<sub>2</sub>O). Crystd from dilute HCl.

**Phenothiazine** [92-84-2] M 199.3, m 184-185°. Crystd from \*benzene or toluene (charcoal) after boiling for 10min under reflux. Filtered on a suction filter. Dried in an oven at 100°, then in a vacuum desiccator over paraffin chips. Also twice recrystd from water and dried in an oven at 100° for 8-10h.

**Phenoxazine** [135-67-1] M 199.2, m 156°, 156-158°, 158-159°, b 215°/4mm. Crystd from EtOH and sublimed *in vacuo*. If too impure then extract in a Soxhlet using toluene. Evaporate the solvent and dissolve residue (*ca* 100g) in \*C<sub>6</sub>H<sub>6</sub> (1L) **CARCINOGEN**, use an efficient fume cupboard) and chromatograph through an Al<sub>2</sub>O<sub>3</sub> column (50 x 450 mm). The eluent (*ca* 3L) is evaporated to *ca* 150mL and cooled when *ca* 103g of phenoxazine m 149-153° is obtained. Sublimation yields platelets m 158-159°. It forms a green *picrate* m 141.5-142°. [Gilman and Moore *J Am Chem Soc* 79 3485 1957; Müller et al. *J Org Chem* 24 37 1959.]

**Phenoxyacetic acid** [122-59-8] M 152.2, m 98-99°,  $pK^{25} 3.18$ . Crystd from water or aq EtOH.

**Phenoxyacetyl chloride** [701-99-5] M 170.6, b 112°/10mm, 102°/16mm, 225-226°/atm,  $d_4^{20} 1.235$ ,  $n_D^{20} 1.534$ . If it has no OH band in the IR then distil in a vacuum, taking precautions for the moisture-sensitive compound. If it contains free acid (due to hydrolysis, OH bands in the IR) then add an equal volume of redistilled SOCl<sub>2</sub>, reflux for 2-3h, evaporate and distil the residue in a vacuum as before. The *amide* has m 101°. [McElvain and Carney *J Am Chem Soc* 68 2592 1946.]

**4-Phenoxyaniline** [139-59-3] M 185.2, m 95°,  $pK^{20} 4.44$  (50% aq EtOH). Crystd from water.

**Phenoxybenzamine** [*N*-(2-chloroethyl)-*N*-(1-methyl-2-phenoxyethyl)benzylamine] [59-96-1] M 303.5, m 38-40°, **hydrochloride** [63-92-3] M 340.0, m 137.5-140°,  $pK_{Est} \sim 4.2$ . The free base is crystd from pet ether and the *HCl* is crystd from EtOH/diethyl ether.

**2-Phenoxybenzoic acid** [2243-42-7] M 214.2, m 113°, b 355°/760mm,  $pK^{15} 3.53$ . Crystd from aqueous EtOH.

**3-Phenoxybenzoic acid** [3739-38-6] M 214.2, m 145°,  $pK 3.59$ . Crystd from aqueous EtOH.

**Phenoxybutyric acid** [6303-58-8] M 180.2, m 64°, 65-66°, 82-83°, 99°, b 180-185°/12mm,  $pK 3.17$ . It has been purified by recrystn from pet ether, \*C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O-pet ether, EtOH and from H<sub>2</sub>O. It can be steam distd or distd in a good vac. [UV: Ramart-Lucas and Hoch *Bull Soc Chim Fr* [4] 51 824 1932; Dann and Arndt *Justus Liebigs Ann Chem* 587 38 1954.] The *acid chloride* has b 154-156°/20mm [Hamford and Adams *J Am Chem Soc* 57 921 1935]; and the *amide* crystallises from \*C<sub>6</sub>H<sub>6</sub> as needles m 113°.

**2-Phenoxypropionic acid (lactic acid *O*-phenylether)** [940-31-8] M 166.2, m 115-116°, b 105-106°/5mm, 265-266°/758mm,  $pK 3.11$ . Crystd from water.

**Phensuximide** (*N*-methyl-2-phenylsuccinimide) [86-34-0] M 189.2, m 71-73°. Crystd from hot 95% EtOH.



**Phenylacetamide** [103-81-1] M 135.2, m 158.5°. Crystd repeatedly from absolute EtOH. Dried under vacuum over P<sub>2</sub>O<sub>5</sub>.

**Phenyl acetate** [122-79-2] M 136.2, b 78°/10mm, d 1.079, n<sup>22</sup> 1.5039. Freed from phenol and acetic acid by washing (either directly or as a soln in pentane) with aqueous 5% Na<sub>2</sub>CO<sub>3</sub>, then with saturated aqueous CaCl<sub>2</sub>, drying with CaSO<sub>4</sub> or Na<sub>2</sub>SO<sub>4</sub>, and fractional distn at reduced pressure.

**Phenylacetic acid** [103-82-2] M 136.2, m 76-77°, b 140-150°/20mm, pK<sub>1</sub> -7.59 (aq H<sub>2</sub>SO<sub>4</sub>), pK<sub>2</sub> 4.31. Crystd from pet ether (b 40-60°), isopropyl alcohol, aq 50% EtOH or hot water. Dried under vac. It can be distd under reduced pressure.

**Phenylacetone (1-phenylpropan-2-one)** [103-79-9] M 134.2, b 69-71°/3mm, d 1.00, n 1.516. Converted to the semicarbazone and crystd three times from EtOH (m 186-187°). The semicarbazone was hydrolysed with 10% phosphoric acid and the ketone was distd. [Kumler, Strait and Alpen *J Am Chem Soc* 72 1463 1950.]

**4'-Phenylacetophenone** [92-91-1] M 196.3, m 120.3-121.2°, b 196-210°/18mm, 325-327°/760mm. Crystd from EtOH or acetone. Can also be distd under reduced or atmospheric pressure.

**Phenylacetylene** [536-74-3] M 102.1, b 75°/80mm, d 0.930, n<sup>25</sup> 1.5463, pK -19. Distd through a spinning band column. Should be filtered through a short column of alumina before use [Collman et al. *J Am Chem Soc* 108 2988 1986; for pK see Brandsma *Preparative Acetylenic Chemistry*, 1st Edn Elsevier 1971, p. 15, ISBN 0444409475].

**dl-Phenylalanine** [150-30-1] M 165.2, m 162°, pK<sub>1</sub><sup>25</sup> 2.58, pK<sub>2</sub><sup>25</sup> 9.24. Crystd from water and dried under vacuum over P<sub>2</sub>O<sub>5</sub>.

**L-Phenylalanine** [63-91-2] M 165.2, m 280°(dec), [α]<sub>D</sub><sup>25</sup> -34.0° (c 2, H<sub>2</sub>O). Likely impurities are leucine, valine, methionine and tyrosine. Crystd from water by adding 4 volumes of EtOH. Dried under vac over P<sub>2</sub>O<sub>5</sub>. Also crystd from satd refluxing aq solns at neutral pH, or 1:1 (v/v) EtOH/water soln, or conc HCl.

**Phenylalaninol (2-amino-3-phenylpropan-1-ol)** [*R*-(+)- 5267-64-1; *S*-(-)- 3182-95-4] M 151.2, m 91-92°, 91.5°, 92-94°, b 80°/11mm (Kügelrohr), [α]<sub>D</sub><sup>20</sup> (+) and (-) 28°, [α]<sub>D</sub><sup>20-25</sup> (+) and (-) 23-28.7° (c 1-5, EtOH), pK<sub>Est</sub> ~9.3. It can be recrystd from Et<sub>2</sub>O, \*C<sub>6</sub>H<sub>6</sub>-pet ether (b 40-60°) or toluene and distd in a vacuum. Has been purified by dissolving in Et<sub>2</sub>O, drying over K<sub>2</sub>CO<sub>3</sub>, filtering, evaporating to a small volume, cooling in ice and collecting the plates. Store in the presence of KOH (i.e. CO<sub>2</sub>-free atm). [Karrer and Ehrhardt *Helv Chim Acta* 34 3203 1951; Oeda *Bull Chem Soc Jpn* 13 465 1938.] The *picrate* has m 141-141.5° (from EtOH-pet ether). The *hydrogen oxalate* has m 177°, 161-162° [Hunt and McHale *J Chem Soc* 2073 1957]. The *racemate* has m 87-88° from \*C<sub>6</sub>H<sub>6</sub>-pet ether (75-77° from Et<sub>2</sub>O), and the *hydrochloride* has m 139-141° [Fodor et al. *J Chem Soc* 1858 1951].

**3-Phenylallyl chloride (cinnamyl chloride)** [*E*: 18685-01-3; *Z*: 39199-93-4] M 152.6, b 92-93°/3mm. Distd under vacuum three times from K<sub>2</sub>CO<sub>3</sub>.

**Phenyl 4-aminosalicylate** [133-11-9] M 229.2, m 153°, pK<sub>Est(1)</sub> ~2.0 (NH<sub>2</sub>), pK<sub>Est(2)</sub> ~9.7 (OH). Crystd from isopropanol.

**4-Phenylanisole (4-methoxybiphenyl)** [361-37-6] M 184.2, m 89.9-90.1°. Crystd from \*benzene/pet ether. Dried under vacuum in an Abderhalden pistol.

**9-Phenylanthracene** [602-55-1] M 254.3, m 153-154°, b 417°. Chromatographed on alumina in \*benzene and crystd from acetic acid.

**N-Phenylanthranilic acid** [91-40-7] M 213.2, m 182-183°, pK<sub>1</sub><sup>25</sup> -1.28 (aq H<sub>2</sub>SO<sub>4</sub>), pK<sub>2</sub><sup>25</sup> 3.86 (CO<sub>2</sub>H). Crystd from EtOH (5mL/g) or acetic acid (2mL/g) by adding hot water (1mL/g).

**2-Phenyl-1-azaindolizine** [56983-95-0] M 194.2, m 140°, pK<sub>Est</sub> ~1.9. Crystd from EtOH or \*benzene/pet ether.

**p-Phenylazobenzoyl chloride** [104-24-5] M 244.7, m 93°. Crystd from pet ether (b 60-80°).

**4-Phenylazo-1-naphthylamine** [131-22-6] M 247.3, m 125-125.5°. Crystd from cyclohexane or aq EtOH. [Brode et al. *J Am Chem Soc* 74 4641 1952.]

**1-Phenylazo-2-naphthylamine** [85-84-7] M 247.3, m 102-104°. See 1-benzeneazo-2-naphthylamine on p. 120.

**4-Phenylazophenacyl bromide** [62625-24-5] M 317.3, m 103-104°. Purified on a column of silica gel, using pet ether/diethyl ether (9:1 v/v) as solvent.

**4-Phenylazophenol (4-hydroxyazobenzene)** [1689-82-3] M 198.2, m 155°, pK<sub>1</sub><sup>25</sup> -0.93, pK<sub>2</sub><sup>25</sup> 8.2. Crystd from \*benzene or 95% EtOH.

**Phenyl benzenethiosulfonate (diphenyldisulfoxide)** [1212-08-4] M 250.3, m 36-37°, 45-46°, 45-47°. Recrystd from EtOH or MeOH. Also purified from phenylsulfide impurities by dissolving in CHCl<sub>3</sub>, washing with aq satd NaHCO<sub>3</sub>, drying (Na<sub>2</sub>SO<sub>4</sub>) evaporating and the residual oil was passed through a silica gel column (600g) and eluted with hexane-\*C<sub>6</sub>H<sub>6</sub> (1L, 4:1, eluting PhSSPh) then \*C<sub>6</sub>H<sub>6</sub> (1L) which elutes PhSSO<sub>2</sub>Ph. [Trost and Massiot *J Am Chem Soc* 99 4405 1977; Knoevenagel and Römer *Chem Ber* 56 215 1923.]

**Phenyl benzoate** [93-99-2] M 198.2, m 69.5°, b 198-199°. Crystd from EtOH using *ca* twice the volume needed for complete soln at 69°.

**Phenyl-1,4-benzoquinone** [363-03-1] M 184.2, m 114-115°. Crystd from heptane or pet ether (b 60-70°) and sublimed *in vacuo*. [Carlson and Miller *J Am Chem Soc* 107 479 1985.]

**1-Phenylbiguanide** [102-02-3] M 177.2, m 144-146°, pK<sub>1</sub><sup>32</sup> 2.16, pK<sub>2</sub><sup>32</sup> 10.74. Crystd from water or toluene.

**S-(-)-1-Phenylbutanol** [22135-49-5] M 150.2, m 46-47°, 46-48°, 49°, b 90-92°/2mm. [ $\alpha$ ]<sub>D</sub><sup>18</sup> -51.4° (c 5, CHCl<sub>3</sub>), -44.7° (c 5.13, \*C<sub>6</sub>H<sub>6</sub>). Purified by distn and crystallises on cooling. The *hydrochloride* has [ $\alpha$ ]<sub>D</sub><sup>20</sup> +45.1° (c 4.8, \*C<sub>6</sub>H<sub>6</sub>). The (-)-*hydroperoxide* has b 58°/0.005mm, n<sub>D</sub><sup>20</sup> 1.5123,  $\alpha$ <sub>D</sub><sup>18</sup> -2.14°, (l = 0.5dcm, neat). [Holding and Ross *J Chem Soc* 145 1954; Davies and Feld *J Chem Soc* 4637 1958.] The ( $\pm$ )-*racemate* has b 73°/0.05mm, and its 4-nitrophenylhydrazone has m 58°.

**Phenylbutazone (4-butyl-1,2-diphenylpyrazolidin-3,5-dione)** [50-33-9] M 308.4, m 105°, 106-108°. Crystd from EtOH.

**2-Phenylbutyramide** [90-26-6] M 163.2, m 86°. Crystd from water.

**2-Phenylbutyric acid** [R-(-)- 938-79-4; S-(+)- 4286-15-1] M 164.2, b 102-104°/atm, d<sub>4</sub><sup>20</sup> 1.056, n<sub>D</sub><sup>20</sup> 1.521, [ $\alpha$ ]<sub>D</sub><sup>20</sup> (-) and (+) 96° (c 2.5, \*C<sub>6</sub>H<sub>6</sub>), [ $\alpha$ ]<sub>D</sub><sup>23</sup> (-) and (+) 5.8° (neat), pK<sub>Est</sub> ~4.3. Purified by distn at atmospheric pressure using an efficient column. The *acid chlorides* have b 106-107°/20mm, [ $\alpha$ ]<sub>D</sub><sup>18</sup> (-) and (+) 108° (c 2, \*C<sub>6</sub>H<sub>6</sub>). [Levene et al. *J Biol Chem* 100 589 1933, Gold and Aubert *Helv Chim Acta* 41 1512 1958; ORD in heptane: Rothen and Levene *J Chem Phys* 7 975 1939.]

**3-Phenylbutyric acid** [R-(-)- 772-14-5; S-(+)- 772-15-6] M 164.2, b 94-95°/3mm, 134°/4mm, d<sub>4</sub><sup>26</sup> 1.066, n<sub>D</sub><sup>25</sup> 1.5167, [ $\alpha$ ]<sub>D</sub><sup>20</sup> (-) and (+) 57° (c 1, \*C<sub>6</sub>H<sub>6</sub>), pK<sub>25</sub> 4.40. Purified as the 2-isomer above, i.e. by distn, but under a good vacuum. [Prelog and Scherrer *Helv Chim Acta* 42 2227 1959; Levene and Marker *J Biol Chem* 93 761 1932, 100 685 1933; Cram *J Am Chem Soc* 74 2137 1952.] The *R-amide*

crystallises from H<sub>2</sub>O, **m** 101.5-102°, [ $\alpha$ ]<sub>D</sub><sup>20</sup> -16.5° (c 1.2, EtOH). The *racemic acid* has **m** 39-40°, **b** 134-136°/6mm, 158°/12mm [Marvel et al. *J Am Chem Soc* **62** 3499 1940].

**4-Phenylbutyric acid** [1821-12-1] **M** 164.2, **m** 50°, **pK**<sup>25</sup> 4.76. Crystd from pet ether (b 40-60°).

***o*-(Phenylcarbamoyl)-1-scopolamine methobromide** [138-10-3] **M** 518.4, **m** 200.5-201.5° (**dec**). Crystd from 95% EtOH.

**9-Phenylcarbazole** [1150-62-5] **M** 243.3, **m** 94-95°. Crystd from EtOH or isopropanol and sublimed *in vacuo*.

***O*-Phenyl chlorothionoformate** [1005-56-7] **M** 172.6, **b** 81-83°/6mm, 91°/10mm, **d**<sub>4</sub><sup>20</sup> 1.276, **n**<sub>D</sub><sup>20</sup> 1.585. Purified by dissolving in CHCl<sub>3</sub>, washing with H<sub>2</sub>O, drying (CaCl<sub>2</sub>), filtering, evaporating and distilling twice under vacuum to give a clear yellow liquid. **It is reactive and POISONOUS - work in a fume cupboard.** Store in sealed ampoules under N<sub>2</sub>. Possible impurity is *O,O'*-diphenyl thiocarbonate which has **m** 106° which remains behind in the distilling flask. [Bögemann et al. in *Methoden Der Organischen Chemie (Houben-Weyl)* 4th edn (E. Müller Ed.) **Vol 9 Schwefel-Selen-Tellur Verbindungen** pp. 807-808 1955; Rivier and Schalch *Helv Chim Acta* **6** 612 1932; Kalson *Chem Ber* **20**, 2384 1987; Rivier and Richard *Helv Chim Acta* **8** 490 1925; Schönberg and Varga *Justus Liebigs Ann Chem* **483** 176 1930; *Chem Ber* **64** 1390 1931.]

**Phenyl cinnamate** [2757-04-2] **M** 224.3, **m** 75-76°, **b** 205-207°/15mm. Crystd from EtOH (2mL/g). It can also be distd under reduced pressure.

**$\alpha$ -Phenylcinnamic acid** [91-48-5] **M** 224.3, **m** 174°(*cis*), **m** 138-139°(*trans*), **pK** 4.8 (60% aq EtOH). Crystd from ether/pet ether.

***o*-Phenylenediamine** [95-54-5] **M** 108.1, **m** 100-101°, **pK**<sub>1</sub><sup>25</sup> 0.67 (aq H<sub>2</sub>SO<sub>4</sub>), **pK**<sub>2</sub><sup>25</sup> 4.47 (4.85). Crystd from aqueous 1% sodium hydrosulfite (charcoal), washed with ice-water and dried in a vacuum desiccator, or sublimed *in vacuo*. It has been purified by recrystn from toluene and zone refined [Anson et al. *J Am Chem Soc* **108** 6593 1986]. Purification by refluxing a CH<sub>2</sub>Cl<sub>2</sub> solution containing charcoal was also carried out followed by evaporation and recrystn [Koola and Kochi *J Org Chem* **52** 4545 1987], protect from light.

***m*-Phenylenediamine** [108-45-2] **M** 108.1, **m** 61-63°, 62-63°, 62.5°, 63-64°, **b** 146°/22mm, 282-284°/760mm, 284-287°/atm, **d**<sub>10</sub><sup>10</sup> 1.1422, **n**<sub>D</sub><sup>57.7</sup> 1.6340, **pK**<sub>1</sub><sup>25</sup> 2.41, **pK**<sub>2</sub><sup>25</sup> 4.98. Purified by distn under vac followed by recryst from EtOH (rhombs) and if necessary redistn. It should be protected from light otherwise it darkens rapidly. [Neilson et al. *J Chem Soc* 371 1962; IR: Katritzky and Jones *J Chem Soc* 3674, 2058 1959; UV: Forbes and Leckie *Can J Chem* **36** 1371 1958.] The *hydrochloride* has **m** 277-278°, and the *bis-4-chlorobenzenesulfonyl* derivative has **m** 220-221° from H<sub>2</sub>O (214-215°, from MeOH-H<sub>2</sub>O) [Runge and Pfeiffer *Chem Ber* **90** 1737 1957].

***p*-Phenylenediamine** [106-50-3] **M** 108.1, **m** 140°, **pK**<sub>1</sub><sup>25</sup> 2.89, **pK**<sub>2</sub><sup>25</sup> 6.16. Crystd from EtOH or \*benzene, and sublimed *in vacuo*, protect from light.

***o*-Phenylenediamine dihydrochloride** [615-28-1] **M** 181.1, **m** 180°. Crystd from dilute HCl (60mL conc HCl, 40mL water, with 2g stannous chloride), after treatment of the hot soln with charcoal by adding an equal volume of conc HCl and cooling in an ice-salt mixture. The crystals were washed with a small amount of conc HCl and dried in a vacuum desiccator over NaOH.

**2-Phenyl-1,3-diazahexahydroazulene** [2161-31-1] **M** 212.3. Recrystd three times from de-aerated cyclohexane in the dark.

**1,4-Phenylene diisothiocyanate (bitoscanate)** [4044-65-9] M 192.3, m 129-131°, 130-131°, 132°. Purified by recrystn from AcOH, pet ether (b 40-60°), Me<sub>2</sub>CO or aq Me<sub>2</sub>CO. [van der Kerk et al. *Recl Trav Chim Pays-Bas* 74 1262 1955; Leiber and Slutkin *J Org Chem* 27 2214 1962.]

**1-Phenyl-1,2-ethanediol** [*R*-(-)- 16355-00-3; *S*-(+)- 25779-13-9] M 138.2, m 64-67°, 65-66°,  $[\alpha]_D^{24}$  (-) and (+) 40.5° (c 2.8, H<sub>2</sub>O),  $[\alpha]_D^{20}$  (-) and (+) 39° (c 3, EtOH). Purified by recryst from \*C<sub>6</sub>H<sub>6</sub>-ligroin and sublimed at 1-2mm. [Arpesella et al. *Gazetta* 85 1354 1955; Prelog et al. *Helv Chim Acta* 37 221 1954.]

***dl*-1-Phenylethanol** [13323-81-4] M 122.2, b 60.5-61.0°/3mm, 106-107°/22-23mm, d 1.01, n<sup>25</sup> 1.5254. Purified *via* its hydrogen phthalate. [See Houssa and Kenyon *J Chem Soc* 2260 1930.] Shaken with a soln of ferrous sulfate, and the alcohol layer was washed with distilled H<sub>2</sub>O, dried (MgSO<sub>4</sub>) and fractionally distd.

**2-Phenylethanol** [60-12-8] M 122.2, b 215-217°, d 1.020. Purified by shaking with a soln of ferrous sulfate, and the alcohol layer was washed with distd water and fractionally distd.

**Phenyl ether (diphenyl ether)** [101-84-8] M 170.2, m 27.0°, d 1.074, n<sup>30.7</sup> 1.57596. Crystd from 90% EtOH. Melted, washed with 3M NaOH and water, dried with CaCl<sub>2</sub> and fractionally distd under reduced pressure. Fractionally crystd from its melt and stored over P<sub>2</sub>O<sub>5</sub>.

**1-Phenylethyl isocyanate (α-methylphenyl isocyanate)** [*R*-(+)- 33375-06-3; *S*-(-)- 14649-03-7] M 147.2, b 82-83°/12-14mm, d<sub>4</sub><sup>20</sup> 1.045, n<sub>D</sub><sup>20</sup> 1.513,  $[\alpha]_D^{24}$  (+) and (-) 2° (c 3.5, \*C<sub>6</sub>H<sub>6</sub>), (+) and (-) 10.5° (neat). Purified by fractional distn under vacuum. With ammonia it gives the *ureido* derivative which crystallises from H<sub>2</sub>O, m 121-122°,  $[\alpha]_D^{25}$  (+) and (-) 48.8°. [Cairns *J Am Chem Soc* 63 870 1941.] The *racemate* has b 90-94°/3mm, 96°/18mm [Seifan *Justus Liebigs Ann Chem* 562 75 1949].

***p*-α-Phenylethylphenol** [1988-89-2] M 198.3, m 56.0-56.3°, pK<sub>Est</sub> ~10.3. Crystd from pet ether.

**5-(α-Phenylethyl)semioxamazine** [93-95-8] M 207.1, m 167-168° (*l*-), 157° (*dl*-). Crystd from EtOH.

**9-Phenyl-3-fluorone** [975-17-7] M 320.3, m >300°(dec), λ<sub>max</sub> 462nm (ε 4.06 x 10<sup>4</sup>, in 1M HCl aq EtOH). Recrystd from warm, acidified EtOH by addition of ammonia. The crude material (1g) can be extracted with EtOH (50mL) in a Soxhlet apparatus for 10h to remove impurities. Impurities can be detected by paper electrophoresis. [Petrova et al. *Anal Lett* 5 695 1972.]

***L*-α-Phenylglycine** [2935-35-5] M 151.2, m 305-310°,  $[\alpha]_{546}$  +185° (c 1, M HCl), pK<sub>1</sub><sup>2.5</sup> 1.83, pK<sub>2</sub><sup>2.5</sup> 4.39 (for *dl*). Crystd from EtOH.

**Phenylglycine-*o*-carboxylic acid** [612-42-0] M 195.2, m 208°. Crystd from hot water (charcoal).

**Phenylhydrazine** [100-63-0] M 108.1, m 23°, b 137-138°/18mm, 241-242°/760mm, d 1.10, n 1.607, pK<sub>1</sub><sup>2.0</sup> -5.2 (aq H<sub>2</sub>SO<sub>4</sub>), pK<sub>2</sub><sup>2.5</sup> 5.27. Purified by chromatography, then crystd from pet ether (b 60-80°)/\*benzene. [Shaw and Stratton *J Chem Soc* 5004 1962.]

**Phenylhydrazine hydrochloride** [59-88-1] M 144.5, m 244°. One litre of boiling EtOH was added to 100g of phenylhydrazine hydrochloride dissolved during 1-3h (without heating) in 200mL of warm water (60-70°). The soln was filtered off, while still hot, through Whatman No 2 filter paper and cooled in a refrigerator. The ppt was collected on a medium sintered-glass filter and recrystd twice this way, then washed with cold EtOH, dried thoroughly and stored in a stoppered brown bottle. [Peterson, Karrer and Guerra *Anal Chem* 29 144 1957.] Hough, Powell and Woods [*J Chem Soc* 4799 1956] boiled the hydrochloride with three times its weight of water, filtered hot (charcoal), added one-third volume of conc HCl and cooled to 0°. The crystals were washed with acetone, and dried over P<sub>2</sub>O<sub>5</sub> under vacuum. The salt has also been crystd from 95% EtOH.

**Phenylhydroxylamine (N-hydroxyaniline)** [100-65-2] M 109.1, m 82°, pK 3.2. Impure base deteriorates rapidly. Crystd from H<sub>2</sub>O, \*C<sub>6</sub>H<sub>6</sub> or \*C<sub>6</sub>H<sub>6</sub>/pet ether (40-60°). Picrate has m 186°.

**2-Phenyl-1,3-indandione** [83-12-5] M 222.2, m 149-151°, pK<sup>20</sup> 4.12 (1% aq MeOH). Crystd from EtOH.

**2-Phenylindolizine** [25379-20-8] M 193.2, m 214°(dec), pK<sub>Est</sub> ~4.4. Crystd from EtOH.

**Phenylisocyanate** [103-71-9] M 119.1, b 45-47°/10mm, d 1.093, n 1.536. Distd under reduced pressure from P<sub>2</sub>O<sub>5</sub>.

**Phenylisothiocyanate (phenyl mustard oil)** [103-72-0] M 135.2, m -21°, b 95°/12mm, 117.1°/33mm, 221°/760mm, d<sub>4</sub><sup>25</sup> 1.1288, n<sub>D</sub><sup>23.4</sup> 1.64918. It is insol in H<sub>2</sub>O, but sol in Et<sub>2</sub>O and EtOH. If impure (due to formation of thiourea) then steam dist into a receiver containing 5-10mL of N H<sub>2</sub>SO<sub>4</sub>. Separate the oil, dry over CaCl<sub>2</sub> and distil under vacuum. [Dains et al. *Org Synth Coll Vol I* 447 1941.]

**8-Phenylmenthol** [1R,2S,5R(-)- 65253-04-5; 1S,2R,5S(+)- 57707-91-2] M 232.4, [α]<sub>D</sub><sup>20</sup> (-) and (+) 26° (c 2, EtOH). Dissolve in toluene, dry (Na<sub>2</sub>SO<sub>4</sub>), evap and chromatograph on a silica gel column and eluting with 5% Et<sub>2</sub>O in pet ether to give an oil with the desired rotation. IR has ν 3420cm<sup>-1</sup> (OH) with consistent <sup>1</sup>H NMR [Corey and Ensley *J Org Chem* 43 1610 1978; Whitesell et al. *Tetrahedron* 42 2993 1986; Bednarski and Danishefsky *J Am Chem Soc* 108 7060 1986.]

**1-Phenyl-5-mercaptotetrazole** [86-93-1] M 178.2, m 150° (dec), 155° (dec), 157-158°, pK<sup>25</sup> 3.65 (5% aq EtOH). Purified by recryst from EtOH or CHCl<sub>3</sub> (m 152°) [Tautomerism: Kauer and Sheppard *J Org Chem* 32 3580 1967; UV: Leiber et al. *Can J Chem* 37 563 1959]. The ammonium salt crystallises from EtOH and dec at 176°, and the sodium salt crystallises from EtOH-\*C<sub>6</sub>H<sub>6</sub>, melts at 96° and dec at 145° [Stollé *J Prakt Chem* [2] 133 60 1932].

**Phenyl methanesulfonate** [16156-59-5] M 172.1, m 61-62°. Crystd from MeOH.

**2-Phenyl-naphthalene** [612-94-2] M 204.3, m 103-104°. Chromatographed on alumina in \*benzene and crystd from aqueous EtOH.

**N-Phenyl-1-naphthylamine** [90-30-2] M 219.3, m 63.7-64.0°, pK<sub>Est</sub> ~0.1. Crystd from EtOH, pet ether or \*benzene/EtOH. Dried under vacuum in an Abderhalden pistol.

**N-Phenyl-2-naphthylamine** [135-88-6] M 219.3, m 107.5-108.5°, 110°, pK<sub>Est</sub> ~0.5. Crystd from EtOH, MeOH, glacial acetic acid or \*benzene/hexane.

**4-Phenylphenacyl bromide** [135-73-9] M 275.2, m 126°. Crystd (charcoal) from EtOH (15mL/g), or ethyl acetate/pet ether (b 90-100°).

**2-Phenylpropanal** [93-53-8] M 134.2, b 206°/760mm, d 1.001, n 1.5183. May contain up to 15% of acetophenone. Purified via the bisulfite addition compound [Lodge and Heathcock *J Am Chem Soc* 109 3353 1987] and see Chapter 2 for prepn, and decompn, of bisulfite adduct.

**Phenylpropionic acid** [637-44-5] M 146.2, m 137.8-138.4°, pK<sup>25</sup> 2.23. Crystd from \*benzene, CCl<sub>4</sub> or aqueous EtOH.

**RS-2-Phenylpropionic acid** [492-37-5] M 150.2, m 16-16.5°, b 153-155°/20mm, 189°/48mm, 260-262°/atm, d 1.10, n 1.522, pK<sup>25</sup> 4.3. Fractionally distd, or recrystd from pet ether (b 40-60°) strong cooling (see references below).

**2-Phenylpropionic acid** [R(-)- 7782-26-5; S(+)- 7782-24-3] M 150.2, m 30.3-31°, 30-32°, b 115°/1-2mm, 142°/12mm, [α]<sub>D</sub><sup>20</sup> (-) and (+) 99.7° (l = 1 dcm, neat), (-) and (+) 89.1° (c

**1.7, EtOH), (-) and (+) 75° (c 1.6, CHCl<sub>3</sub>).** Purified by vacuum distn and by recrystn from pet ether. The *S*-anilide has **m 103-104°** (from H<sub>2</sub>O or CHCl<sub>3</sub>/*S*-C<sub>6</sub>H<sub>5</sub>), [ $\alpha$ ]<sub>D</sub><sup>25</sup> +47° (c 9, Me<sub>2</sub>CO) [Argus and Kenyon *J Chem Soc* 916 1939; Campbell and Kenyon *J Chem Soc* 25 1946; Levene et al. *J Biol Chem* 88 27, 34 1930; Beilstein 9, 3rd Suppl p 2417].

**3-Phenylpropionic acid (hydrocinnamic acid) [501-52-0] M 150.2, m 48-48.5°, pK<sup>25</sup> 4.56.** Crystd from \*benzene, CHCl<sub>3</sub> or pet ether (b 40-60°). Dried in a vacuum.

**3-Phenylpropyl bromide [637-59-2] M 199.1, b 110°/12mm, 128-129°/29mm, d 1.31.** Washed successively with conc H<sub>2</sub>SO<sub>4</sub>, water, 10% aqueous Na<sub>2</sub>CO<sub>3</sub> and again with water, then dried with CaCl<sub>2</sub> and fractionally distd just before use.

**Phenyl 2-pyridyl ketoxime [1826-28-4] M 198.2, m 151-152°.** Crystd from EtOH (charcoal).

**Phenylpyruvic acid [156-06-9] M 164.2, m 150-154°, 158-159°, pK<sub>Est</sub> ~2.1.** Recrystd from \*C<sub>6</sub>H<sub>6</sub>. The *phenylhydrazone* has **m 173°** [Zeller *Helv Chim Acta* 26 1614 1943; Hopkins and Chisholm *Can J Research [B]* 24 89 1946]. The *2,4-dinitrophenylhydrazone* has **m 162-164°** (189°, 192-194°) [Fones *J Org Chem* 17 1952].

**6-Phenylquinoline [612-95-3] M 205.3, m 110.5-111.5°, pK<sub>Est</sub> ~5.2.** Crystd from EtOH (charcoal).

**2-Phenylquinoline-4-carboxylic acid (cinchophen) [132-60-5] M 249.3, m 215°, pK<sub>Est(1)</sub> ~0.5 (CO<sub>2</sub>H), pK<sub>Est(2)</sub> ~5.1 (N).** Crystd from EtOH (*ca* 20mL/g).

**Phenyl salicylate (Salol) [118-55-8] M 214.2, m 41.8-42.6°, pK<sub>Est</sub> ~9.9.** Fractionally crystd from its melt, then crystd from \*benzene.

**3-Phenylsalicylic acid [304-06-3] M 214.3, m 186-187.5°, pK<sub>Est(1)</sub> ~2.8 (CO<sub>2</sub>H), pK<sub>Est(2)</sub> ~11.0 (OH).** Dissolved in *ca* 1 equivalent of saturated aqueous Na<sub>2</sub>CO<sub>3</sub>, filtered and ppted by adding 0.8 equivalents of M HCl. Crystd from ethylene dichloride (charcoal), and sublimed at 0.1mm. [Brooks, Eglington and Norman *J Chem Soc* 661 1961.]

**1-Phenylsemicarbazide [103-03-7] M 151.2, m 172°.** Crystd from water and dried in vac over KOH.

**4-Phenylsemicarbazide [537-47-3] M 151.2, m 122°.** Crystd from water and dried in vac over KOH.

**Phenylsuccinic acid [R(-)- 46292-93-7; S-(+)- 4036-30-1] M 194.2, m 173-176°, 178.5-179°, 179-180°, [ $\alpha$ ]<sub>D</sub><sup>25</sup> (-) and (+) 171° (c 2, Me<sub>2</sub>CO), [ $\alpha$ ]<sub>D</sub><sup>26-30</sup> (-) and (+) 148° (c 0.27-5, EtOH), pK<sub>1</sub><sup>25</sup> 3.78, pK<sub>2</sub><sup>25</sup> 5.55.** Purified by repptn from alkali and recrystn from H<sub>2</sub>O. [Naps and Johns *J Am Chem Soc* 62 2450 1940; Fredga and Matell *Bull Soc Chim Belg* 62 47 1953; Wren and Williams *J Chem Soc* 109 572 1916.] The *racemate* [635-51-8] has **m 166-168°**, 168° after recrystn from H<sub>2</sub>O or MeCN; its *S*-benzylthiouronium salt has **m 164-165°** (from EtOH) [Griediger and Pedersen *Acta Chem Scand* 9 1425 1955].

**1-Phenyl-5-sulfanilamidopyrazole [526-08-9] M 314.3, m 179-183°.** Crystd from EtOH.

**1-Phenylthiosemicarbazide [645-48-7] M 167.2, m 200-201°(dec).** Crystd from EtOH.

**4-Phenylthiosemicarbazide [5351-69-9] M 167.2, m 140°.** Crystd from EtOH.

**1-Phenyl-2-thiourea [103-85-5] M 152.1, m 154°.** Crystd from water and dried at 100° in air.

**Phenyltoloxamine [2-(2-dimethylaminoethoxy)-diphenylmethane] hydrochloride [6152-43-8] M 291.8, m 119-120°, pK<sup>25</sup> 9.3 (free base).** Crystd from isobutyl methyl ketone.