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Galactaric Acid **(mucic acid)** *[526-99-6]* **M 210.1, m 212-213°(dec) pK_1^{25} 3.09 (3.29), pK_2^{25} 3.63 (4.41).** Dissolved in the minimum volume of dil aq NaOH, and ppted by adding dil HCl. The temperature should be kept below 25°.

D-Galactonic acid *[576-36-3]* **M 196.2, m 148°, pK_{Est} ~3.5.** Crystd from EtOH. Cyclises to *D-galactonono-1,4-lactone*, **m** 134-136°, $[\alpha]_{546}^{30}$ mutarotates in 1h to -92° (c 5,H_2O).

D(-)-Galactono-1,4-lactone *[2782-07-2]* **M 178.1, m 134-137°, $[\alpha]_D^{20}$ -78° (in H_2O).** Crystd from EtOH.

D(+)-Galactosamine hydrochloride *[1772-03-8]* **M 215.6, m 181-185°, $[\alpha]_D^{25}$ +96.4° (after 24h, c 3.2 in H_2O), pK_{Est} ~7.7 (free base).** Dissolved in a small volume of H_2O. Then added three volumes of EtOH, followed by acetone until faintly turbid and stood overnight in a refrigerator. [Roseman and Ludoweig *J Am Chem Soc* **76** 301 *1954*.]

α-D-Galactose *[59-23-4]* **M 180.2, m 167-168°, $[\alpha]_D^{20}$ +80.4° (after 24h, c 4 in H_2O), pK^{25} 12.48.** Crystd twice from aqueous 80% EtOH at -10°, then dried under vacuum over P_2O_5.

Gallic acid (H_2O) (3,4,5-trihydroxybenzoic acid) *[5995-86-8 (H_2O), 149-91-7 (anhydr)]* **M 188.1, m 253°(dec), pK_1^{25} 4.27, pK_2^{25} 8.68.** Crystd from water.

Galvinoxyl [2,6-di-*tert*-butyl-α-(3,5-di-*tert*-butyl-4-oxo-2,5-cyclohexadiene-1-ylidene)-*p*-tolyloxy] *[2370-18-5]* **M 421.65, m 153.2-153.6°, 158-159°.** A stable free radical scavanger of short-lived free radicals with odd electrons on C or O. Best prepared freshly by oxidation of 3,3',5,5'-tetra-*tert*-butyl-4,4'-dihydroxydiphenyl-methane [**m** 154°, 157.1-157.6°; obtained by gently heating for 10-15min 2,6-di-*tert*-butylphenol, formaldehyde and NaOH in EtOH and recryst from EtOH (20g/100mL) as colorless plates, Karasch and Joshi *J Org Chem* **22** 1435 *1957*; Bartlett et al. *J Am Chem Soc* **82** 1756 *1960* and **84** 2596 *1962*.] Oxidation is carried out under N_2 with PbO_2 in Et_2O or isooctane [Galvin A. Coppinger *J Am Chem Soc* **79** 501 *1957* ; Bartlett et al. above] or with alkaline potassium ferricyanide [Karasch and Joshi, above], whereby Galvinoxyl separates as deep blue crystals, and is recrystd twice under N_2 from $*C_6H_6$ soln by suction evaporation at 30°. The VIS spectrum has λmax 407nm (ε 30,000), 431nm (ε 154,000) and weak absorption at 772.5nm, and IR: ν 1577 and 2967cm^{-1}, and is estimated by iodometric titration. It is sensitive to O_2 in presence of OH^- ions and to traces of strong acid in hydroxylic or hydrocarbon solvents. At 62.5° in a 0.62mM soln in $*C_6H_6$ the radical decays with a first order $k = 4 \times 10^{-8}$ sec^{-1} (half life 1.7×10^{17} sec, ~200 days) as observed by the change in OD at 550nm [see also Green and Adam *J Org Chem* **28** 3550 *1963*].

Genistein (4',5,7-trihydroxyisoflavone) *[446-72-0]* **M 270.2, m 297-298°, $[\alpha]_D^{20}$ -28° (c 0.6, 20mM NaOH), (phenolic pKs 8-10).** Crystd from 60% aqueous EtOH or water.

Genistin (genistein-7-D-glucoside) *[529-59-9]* **M 432.4, m 256°.** Crystd from 80% EtOH/water.

α-Gentiobiose (amygdalose, 6-O-α-D-glucopyranosyl-D-glucopyranose) *[5995-99-5 (bi-pyranose)]* **M 342.3, m 86°, $[\alpha]_{546}^{20}$ +11° (after 24h, c 4, H_2O).** Crystd from MeOH (retains solvent of crystn).

β-Gentiobiose (see above) *[5996-00-9 (bi-pyranose); 554-91-6 (one open ring)]* **M 342.3, m 190-195°, $[\alpha]_{546}^{20}$ +8° (after 6h, c 3, H_2O).** Crystd from MeOH or EtOH.

Geraniol *[106-24-1]* **M 154.3, b 230°, d 0.879, n 1.4766.** Purified by ascending chromatography or by thin layer chromatography on plates of kieselguhr G with acetone/water/liquid paraffin (130:70:1) as solvent system. Hexane/ethyl acetate (1:4) is also suitable. Also purified by GLC on a silicone-treated column of Carbowax 20M (10%) on Chromosorb W (60-80 mesh). [Porter *Pure Appl Chem* **20** 499 *1969*.] Stored in full, tightly sealed containers in the cool, protected from light.

Gibberillic acid (GA_3) *[77-06-5]* **M 346.4, m 233-235°(dec), $[\alpha]_{546}^{20}$ +92° (c 1, MeOH), pK 4.0.** Crystd from ethyl acetate.

Girard Reagent T (2-hydrazino-*N,N,N*-trimethyl-2-oxo-ethanaminium chloride) *[123-46-6]* **M 167.6, m 192°.** Should be crystd from absolute EtOH (slight decomposition) when it has a slight odour, and stored in tightly stoppered containers because it is hygroscopic.

Glucamine *[488-43-7]* **M 181.2, m 127°, $[\alpha]_D^{20}$ -8° (c 10, H_2O), pK_{Est} ~9.0.** Crystd from MeOH.

D-Gluconamide *[3118-85-2]* **M 197.2, m 144°, $[\alpha]_D^{23}$ +31° (c 2, H_2O).** Crystd from EtOH.

D-Glucono-δ-lactone *[90-80-2]* **M 178.1, m 152-153°, $[\alpha]_{546}^{20}$ +76° (c 4, H_2O).** Crystd from ethylene glycol monomethyl ether and dried for 1h at 110°.

D-Glucosamine *[3416-24-8]* **M 179.2, m 110°(dec), $[\alpha]_D^{20}$ +28° → +48° (c 5, H_2O), pK^{24} 7.71.** Crystd from MeOH. *N-Acetyl deriv*, **m** 205° from MeOH/Et_2O has $[\alpha]_D^{20}$ +64° → +41° (c 5, H_2O).

D-Glucosamine hydrochloride *[66-84-2]* **M 215.6, m >300°, $[\alpha]_D^{25}$ +71.8° (after 20h, c 4, H_2O).** Crystd from 3M HCl, water, and finally water/EtOH/acetone as for galactosamine hydrochloride.

α-D-Glucose *[492-62-6]* **M 180.2, m 146°, $[\alpha]_D^{20}$ +52.5° (after 24h, c 4, H_2O), pK^{25} 12.46.** Recrysts slowly from aqueous 80% EtOH, then vacuum dried over P_2O_5. Alternatively, crystd from water at 55°, then dried for 6h in a vacuum oven between 60-70° at 2mm.

β-D-Glucose *[50-99-7]* **M 180.2, m 148-150°.** Crystd from hot glacial acetic acid.

α-D-Glucose pentaacetate *[604-68-2]* **M 390.4, m 110-111°, 112°, $[\alpha]_{546}^{20}$ +119° (c 5, $CHCl_3$).** Crystd from MeOH or EtOH.

β-D-Glucose pentaacetate *[604-69-3]* **M 390.4, m 131-132°, $[\alpha]_{546}^{20}$ +5° (c 5, $CHCl_3$).** Crystd from MeOH or EtOH.

D-Glucose phenylhydrazone *[534-97-4]* **M 358.4, m 208°.** Crystd from aqueous EtOH.

D-Glucuronic acid *[6556-12-3]* **M 194.1, m 165°, $[\alpha]_D^{20}$ +36° (c 3, H_2O), pK_2^{20} 3.18.** Crystd from EtOH or ethyl acetate.

D-Glucuronolactone *[32449-92-6]* **M 176.1, m 175-177°, $[\alpha]_{546}^{20}$ +22° (after 24h, c 10, H_2O).** Crystd from water.

L-Glutamic acid *[56-86-0]* **M 147.1, m 224-225°(dec), $[\alpha]_D^{25}$ +31.4° (c 5, 5M HCl), pK_1^{20}, p K_2^{20} 2.06,4.35, pK_3^{20} 9.85.** Crystd from H_2O acidified to pH 3.2 by adding 4 volumes of EtOH, and dried at 110°. Likely impurities are aspartic acid and cysteine.

L-Glutamic acid-γ-benzyl ester *[1676-73-9]* **M 237.3, m 179-181°, $[\alpha]_{589}^{20}$ 19.3° (c 1, HOAc), pK_1^{25} 2.17, pK_2^{25} 9.00.** Recrystd from H_2O and stored at 0°. [Estrin *Biochem Prep* **13** 25 *1971*.]

L-Glutamine *[56-85-9]* **M 146.2, m 184-185°, $[\alpha]_D^{25}$ +31.8° (M HCl), pK_1^{25} 2.17, pK_2^{25} 9.13.** Likely impurities are glutamic acid, ammonium pyroglutamate, tyrosine, asparagine, isoglutamine, arginine. Crystd from water.

Glutaraldehyde *[111-30-8]* **M 100.1, b 71°/10mm, as 50% aq soln.** Likely impurities are oxidation products - acids, semialdehydes and polymers. It can be purified by repeated washing with activated charcoal (Norit) followed by vacuum filtration, using 15-20g charcoal/100mL of glutaraldehyde soln.
Vacuum distn at 60-65°/15mm, discarding the first 5-10%, was followed by dilution with an equal volume of freshly distilled water at 70-75°, using magnetic stirring under nitrogen. The soln is stored at low temp (3-4°),

in a tightly stoppered container, and protected from light. Standardised by titration with hydroxylamine. [Anderson *J Histochem Cytochem* **15** 652 *1967*.]

Glutaric acid *[110-94-1]* **M 132.1, m 97.5-98°, pK_1^{25} 4.35, pK_2^{25} 5.40.** Crystd from *benzene, $CHCl_3$, distilled water or *benzene containing 10% (w/w) of diethyl ether. Dried under vacuum.

***dl*-Glyceraldehyde** *[56-82-6]* **M 90.1, m 145°.** Crystd from EtOH/diethyl ether.

Glycerol *[56-81-5]* **M 92.1, m 18.2°, b 182°/20mm, 290°/760mm, d 1.261, n^{25} 1.47352, pK 14.4.** Glycerol was dissolved in an equal volume of *n*-butanol (or *n*-propanol, amyl alcohol or liquid ammonia) in a water-tight container, cooled and seeded while slowly revolving in an ice-water slurry. The crystals were collected by centrifugation, then washed with cold acetone or isopropyl ether. [Hass and Patterson *Ind Eng Chem (Anal Ed)* **33** 615 *1941*.] Coloured impurities can be removed from substantially dry glycerol by extraction with 2,2,4-trimethylpentane. Alternatively, glycerol can be decolorised and dried by treatment with activated charcoal and alumina, followed by filtering. Glycerol can be distd at 15mm in a stream of dry nitrogen, and stored in a desiccator over P_2O_5. Crude glycerol can be purified by digestion with conc H_2SO_4 and saponification with a lime paste, then re-acidified with H_2SO_4, filtered, treated with an anion exchange resin and fractionally distd under vacuum.

Glycidol (oxirane-2-methanol) *[RS-(±)- 556-52-5; R-(+)- 57044-25-4; S-(-)- 60456-23-7]* **M 74.1, b 61-62°/15mm, d 1.117, n 1.433 (±) , b 49-50°/7mm, 66-67°/19mm, $[\alpha]_D^{20}$ -15° (neat) (*S-isomer*, § also available on polymer support), b 56-56.5°/11mm, d 1.117, n 1.429, $[\alpha]_D^{20}$ +15° (neat).** Purified by fractional distn. The *4-nitrobenzoates* have **m** 56° (±); **m** 60-62°, $[\alpha]_D^{20}$ -37.9° (c 3.38 $CHCl_3$) for *R-(-)*-isomer *[106268-95-5]*; **m** 60-62°, $[\alpha]_D^{20}$ +38° (c 1 $CHCl_3$) for *S-(+)*-isomer **m** 60-62°, $[\alpha]_D^{20}$ -38° (c 1 $CHCl_3$) *[115459-65-9]*, and are recrystd from Et_2O or Et_2O/pet ether (b 40-60°) [*S-isomer*: Burgos et al. *J Org Chem* **52** 4973 *1987*; Sowden and Fischer *J Am Chem Soc* **64** 1291 *1942*.]

Glycinamide hydrochloride *[1668-10-6]* **M 110.5, m 186-189° (207-208°), pK_1^{25} -6.10, pK_2^{25} -1.78, pK_3^{25} 7.95.** Crystd from EtOH.

Glycine see **aminoacetic acid.**

Glycine ethyl ester hydrochloride *[623-33-6]* **M 136.9, m 145-146°, pK^{25} 7.69.** Crystd from absolute EtOH.

Glycine hydrochloride *[6000-43-7]* **M 111.5, m 176-178°.** Crystd from absolute EtOH.

Glycine methyl ester hydrochloride *[5680-79-5]* **M 125.6, m 174°(dec), pK^{25} 7.66.** Crystd from MeOH.

Glycine *p*-nitrophenyl ester hydrobromide. *[7413-60-7]* **M 277.1, m 214° (dec).** Recryst from MeOH by adding diethyl ether. [Alners et al. *Biochem Preps* **13** 22 *1971*].

Glycocholic acid (*N*-cholylglycine) *[475-31-0]* **M 465.6, m 154-155°, 165-168°, $[\alpha]_{546}^{20}$ +37° (c 1, EtOH), pK 4.4.** Crystd from hot water as sesquihydrate. Dried at 100°.

Glycolic (α-hydroxyacetic) acid *[79-14-1]* **M 76.1, m 81°, pK^{25} 3.62.** Crystd from diethyl ether.

***N*-Glycylanilide** *[555-48-6]* **M 150.2, m 62°, pK_{Est}~8.0.** Crystd from water, sol in Et_2O..

Glycylglycine *[556-50-3]* **M 132.1, m 260-262°(dec), pK^{20} 8.40, pK^{30} 8.04.** Crystd from aqueos 50% EtOH or water at 50-60° by addition of EtOH. Dried at 110°.

Glycylglycine hydrochloride *[13059-60-4]* **M 168.6, m 215-220°, 235-236°, 260-262°, pK_1^{25} 3.12, pK_2^{25} 8.17.** Crystd from 95% EtOH.

Glycyl-L-proline *[704-15-4]* **M 172.2, m 185°, pK_1^{25} 2.81, pK_2^{25} 8.65.** Crystd from water at 50-60° by addition of EtOH.

***dl*-Glycylserine** *[687-38-7]* **M 162.2, m 207°(dec), pK_1^{25} 2.92, pK_2^{25} 8.10.** Crystd from H_2O (charcoal) by addition of EtOH.

Glycyrrhizic acid ammonium salt ($3H_2O$) *[53956-04-0]* **M 823.0, m 210°(dec), $[\alpha]_{546}^{20}$ +60° (c 1, 50% aq EtOH), pK_{Est} ~4.0.** Crystd from glacial acetic acid, then dissolved in ethanolic ammonia and evaporated.

Glyoxal bis(2-hydroxyanil) *[1149-16-2]* **M 240.3, m 210-213°, ε_{294nm} 9880.** Crystd from MeOH or EtOH.

Glyoxylic acid *[298-12-4]* **M 74.0, m 98°(anhydr), 50-52°(monohydrate), pK^{25} 2.98.** Crystd from water as the monohydrate.

Gramine (3-dimethylaminoethylindole) *[87-52-5]* **M 174.3, m 134°, pK^{25} 16.00 (NH acidic).** Crystd from diethyl ether, ethanol or acetone.

Griseofulvin *[126-07-8]* **M 352.8, m 220°, $[\alpha]_D^{22}$ +365° (c 1, acetone).** Crystd from *benzene.

Guaiacic acid [4,4'-(2,3-dimethyl-1-butene-(1,4-diyl)-bis-(2-methoxyphenol)] *[500-40-3]* **M 328.4, m 99-100.5°, pK_{Est} ~10.0.** Crystd from EtOH.

Guaiacol (2-methoxyphenol) *[90-05-1]* **M 124.1, m 32°, b 106°/24mm, 205°/746mm, pK^{25} 9.90.** Crystd from *benzene/pet ether or distd.

Guaiacol carbonate *[553-17-3]* **M 274.3, m 88.1°.** Crystd from EtOH.

Guanidine *[113-00-8]* **M 59.1, m ~50°, pK^{25} 13.6.** Crystd from water/EtOH under nitrogen. Very deliquescent and absorbs CO_2 from the air readily.

Guanidine carbonate *[593-85-1]* **M 180.2, m 197°.** Crystd from MeOH.

Guanidine hydrochloride *[50-01-1]* **M 95.5, m 181-183°.** Crystd from hot methanol by chilling to about -10°, with vigorous stirring. The fine crystals were filtered through fritted glass, washed with cold (-10°) methanol, dried at 50° under vacuum for 5h. (The product is more pure than that obtained by crystn at room temperature from methanol by adding large amounts of diethyl ether.) [Kolthoff et al. *J Am Chem Soc* **79** 5102 *1957*].

Guanosine (H_2O) *[118-00-3]* **M 283.2, m 240-250°(dec), $[\alpha]_{546}^{20}$ -86° (c 1, 0.1M NaOH), pK_1^{25} 1.9, pK_2^{25} 9.24, pK_3^{25} 12.33.** Crystd from water. Dried at 110°.

Guanylic acid (guanosine-5'-monophosphoric acid) *[85-32-5]* **M 363.2, m 208°(dec), pK_2^{25} 2.4, pK_3^{25} 6.66 (6.1), pK_4^{25} 9.4.** Crystd from water. Dried at 110°.

Harmine *[442-51-3]* **M 212.3, m 261°(dec), pK^{20} 7.61.** Crystd from MeOH.

Harmine hydrochloride (hydrate) *[343-27-1]* **M 248.7, m 280°(dec).** Crystd from water.

Hecogenine acetate *[915-35-5]* **M 472.7, m 265-268°, $[\alpha]_D^{23}$ -4.5° (c 1, $CHCl_3$).** Crystd from MeOH.

Heptadecanoic acid (margaric) *[506-12-7]* **M 270.5, m 60-61°, b 227°/100mm, pK_{Est} ~4.9.** Crystd from MeOH or pet ether.

1-Heptadecanol *[1454-85-9]* **M 256.5, m 54°.** Crystd from acetone.

Heptafluoro-2-iodopropane *[677-69-0]* **M 295.9, b 41°.** Purified by gas chromatography on a triacetin (glyceryl triacetate) column, followed by bulb-to-bulb distn at low temperature. Stored over Cu powder to stabilise it.

***n*-Heptaldehyde** *[111-71-7]* **M 114.2, b 40.5°/12mm, 152.8°/760mm, d 0.819, n^{25} 1.4130.** Dried with $CaSO_4$ or Na_2SO_4 and fractionally distd under reduced pressure. More extensive purification by pptn as the bisulfite compound (formed by adding the aldehyde to saturated aqueous $NaHSO_3$) which was filtered off and recrystd from hot H_2O. The crystals, after being filtered and washed well with H_2O, were hydrolysed by adding 700mL of aqueous Na_2CO_3 (12.5% w/w of anhydrous Na_2CO_3) per 100g of aldehyde. The aldehyde was then steam distd, separated, dried with $CuSO_4$ and distd under reduced pressure in a slow stream of nitrogen. [McNesby and Davis *J Am Chem Soc* **76** 2148 *1954*].

***n*-Heptaldoxime** *[629-31-2]* **M 129.2, m 53-55°.** Crystd from 60% aqueous EtOH.

***n*-Heptane** *[142-18-5]* **M 100.2, b 98.4°, d 0.684, n 1.38765, n^{25} 1.38512.** Passage through a silica gel column greatly reduces the ultraviolet absorption of *n*-heptane. (The silica gel is previously heated to 350° before use.) For more extensive purification, heptane is shaken with successive small portions of conc H_2SO_4 until the lower (acid) layer remains colourless. The heptane is then washed successively with water, aq 10% Na_2CO_3, water (twice), and dried with $CaSO_4$, $MgSO_4$ or $CaCl_2$. It is distd from sodium. *n*-Heptane can be distd azeotropically with methanol, then the methanol can be washed out with water and, after drying, the heptane is redistd. Other purification procedures include passage through activated basic alumina, drying with CaH_2, storage with sodium, and stirring with 0.5*N* $KMnO_4$ in 6N H_2SO_4 for 12h after treatment with conc H_2SO_4. Carbonyl-containing impurities have been removed by percolation through a column of impregnated Celite made by dissolving 0.5g of 2,4-dinitrophenylhydrazine in 6mL of 85% H_3PO_4 by grinding together, then adding 4mL of distilled water and 10g Celite. [Schwartz and Parks *Anal Chem* **33** 1396 *1961*].

Hept-1-ene *[592-76-7]* **M 98.2, b 93°/771mm, d 0.698, n 1.400.** Distd from sodium, then carefully fractionally distd using an 18-in gauze-packed column. Can be purified by azeotropic distn with EtOH. Contained the 2- and 3-isomers as impurities. These can be removed by gas chromatography using a Carbowax column at 70°.

***n*-Heptyl alcohol** *[111-70-6]* **M 116.2, b 175.6°, d 0.825, n 1.425.** Shaken with successive lots of alkaline $KMnO_4$ until the colour persisted for 15min, then dried with K_2CO_3 or CaO, and fractionally distd.

***n*-Heptylamine** *[111-68-2]* **M 115.2, b 155°, d 0.775, n 1.434, pK^{25} 10.66.** Dried in contact with KOH pellets for 24h, then decanted and fractionally distd.

***n*-Heptyl bromide** *[629-04-9]* **M 179.1, b 70.6°/19mm, 180°/760mm, d 1.140, n 1.45.** Shaken with conc H_2SO_4, washed with water, dried with K_2CO_3, and fractionally distd.

Heptyl-β-D-glucopyranoside *[78617-12-6]* **M 278.4, m 74-77°, 76-77°, $[\alpha]_D^{20}$ -34.2° (c 5, H_2O).** Purified by several recrystns from M_2CO which is a better solvent than EtOAc. The *acetate* has **m** 66-68.5°, $[\alpha]_D^{20}$ -20.5° (c 4, $CHCl_3$) [Pigman and Richtmyer *J Am Chem Soc* **64** 369 *1942*].

Heptyl-β-D-1-thioglucopyranoside *[85618-20-8]* **M 294.4, m 98-99°.** The *tetra-acetyl derivative* is purified by silica gel column chromatography and eluted with a *C_6H_6-Me_2CO (gradient up to 5% of Me_2CO) and recrystd from *n*-hexane as colourless needles **m** 72-74° (Erbing and Lindberg *Acta Chem Scand*

B30 611 *1976* gave **m** 69-70°). Hydrolysis using an equivalent of base in methanol gave the desired glucoside. This is a non-ionic detergent for reconstituting membrane proteins and has a critical micelle concentration of 30 mM. [Shimamoto et al. *J Biochem (Tokyo) 97* 1807 *1985*; Saito and Tsuchiya *Chem Pharm Bull Jpn* **33** 503 *1985*].

Hesperetin (3',5,7-trihydroxy-4'-methoxyflavanone) *[520-33-2]* **M 302.3, m 227-228°, pK_{Est} ~8.5-10.5 (phenolic).** Crystd from EtOAc or ethanol. The natural (-) form has $[\alpha]_D^{20}$ -38° (c 2, EtOH). Note that C2 is chiral.

Hesperidin (hesperetin 7-rhamnoside) *[520-26-3]* **M 610.6, m 258-262°, $[\alpha]_{546}^{20}$ -82° (c 2, pyridine).** Dissolved in dilute aqueous alkali and ppted by adjusting the pH to 6-7.

Hexachlorobenzene *[118-74-1]* **M 284.8, m 230.2-231.0°.** Crystd repeatedly from *benzene. Dried under vacuum over P_2O_5.

Hexachloro-1,3-butadiene *[87-68-3]* **M 260.8.** See perchlorobutadiene on p. 323.

1,2,3,4,5,6-Hexachlorocyclohexane *[α-319-84-6; γ-58-89-9]* **M 290.8, m 158° (α-), 312° (ß-), 112.5° (γ-isomer).** Crystd from EtOH. Purified by zone melting. **Possible CANCER AGENT, TOXIC.**

Hexachlorocyclopentadiene *[77-47-4]* **M 272.8, b 80°/1mm, d 1.702, n^{25} 1.5628.** Dried with $MgSO_4$. Distd under vacuum in nitrogen.

Hexachloroethane *[67-72-1]* **M 236.7, m 187°.** Steam distd, then crystd from 95% EtOH. Dried in the dark under vacuum.

Hexacosane *[630-01-3]* **M 366.7, m 56.4°, b 169°/0.05mm, 205°/1mm, 262°/15mm.** Distd under vacuum and crystd from diethyl ether.

Hexacosanoic acid (cerotinic acid) *[506-46-7]* **M 396.7, m 86-87°, 88-89°, pK_{Est} ~4.9.** Crystd from EtOH, aq EtOH and pet ether+Me_2CO.

1,14-Hexadecanedioic acid (thaspic acid). *[505-54-4]* **M 286.4, m 126°, $pK_{Est(1)}$~4.5, $pK_{Est(2)}$ ~5.5.** Crystd from EtOH, ethyl acetate or *C_6H_6.

***n*-Hexadecane (Cetane)** *[544-76-3]* **M 226.5, m 18.2°, b 105°/0.1mm, d 0.773, n 1.4345, n^{25} 1.4325.** Passed through a column of silica gel and distd under vacuum in a column packed with Pyrex helices. Stored over silica gel. Crystd from acetone, or fractionally crystd by partial freezing.

Hexadecanoic acid (palmitic acid) *[57-10-3]* **M 256.4, m 62-63°, b 215°/15mm, pK^{25} 6.46 (50% aq EtOH), 5.0 (H_2O).** Purified by slow (overnight) recrystn from hexane. Some samples were also crystd from acetone, EtOH or EtOAc. Crystals were stood in air to lose solvent, or were pumped dry of solvent on a vacuum line. [Iwahashi et al. *J Chem Soc, Faraday Trans 1* **81** 973 *1985*; pK: White *J Am Chem Soc* **72** 1858 *1950*].

Hexadecyl 3-hydroxynaphthalene-2-carboxylate *[531-84-0]* **M 412.6, m 73-74°.** Recrystd from hot EtOH and sublimed in a vacuum. [Oshima and Hayashi *J Soc Chem Ind Jpn* **44** 821 *1941*.]

1,5-Hexadiene *[592-42-7]* **M 82.2, b 59.6°, d 0.694, n 1.4039.** Distd from $NaBH_4$.

Hexaethylbenzene *[604-88-6]* **M 246.3, m 128.7-129.5°.** Crystd from *benzene or *benzene/EtOH.

Hexafluoroacetone *[684-16-2, 34202-69-2 ($3H_2O$)]* **M 166.1, m -129°, (trihydrate m 18-21°), b -28°.** Dehydrated by passage of the vapour over P_2O_5. Ethylene was removed by passing the dried vapour

through a tube containing Pyrex glass wool moistened with conc H_2SO_4. Further purification was by low temperature distn using Warde-Le Roy stills. Stored in the dark at -78°. [Holmes and Kutschke *Trans Faraday Soc* **58** 333 *1962*].

Hexafluoroacetylacetone (1,1,1,5,5,5-hexafluoro-2,4-pentanedione) *[1522-22-1]* **M 208.1, b 68°/736mm, 70-70.2°/760mm, 68-71°/atm, d_4^{20} 1.490, n_D^{20} 1.333.** It forms a dihydrate which has no UV spectrum compared with λmax ($CHCl_3$) 273nm (ε 7,800) for the anhydrous ketone. The dihydrate dec at ~90°. The hydrate (10g) plus anhyd $CaSO_4$ (Drierite, 30g) are heated and distd; the distillate is treated with more $CaSO_4$ and redist. When the distillate is treated with aqueous NaOH and heated, the dihydrate crystallises on cooling. The Cu complex has **m** 135° (after sublimation). [Gilman et al. *J Am Chem Soc* **78** 2790 *1956*; Belford et al. *J Inorg Nucl Chem* **2** 11 *1956*].

Hexafluorobenzene *[392-56-3]* **M 186.1, m 5.1°, b 79-80°, d 1.61, n 1.378.** Main impurities are incompletely fluorinated benzenes. Purified by standing in contact with oleum for 4h at room temperature, repeating until the oleum does not become coloured. Washed several times with water, then dried with P_2O_5. Final purification was by repeated fractional crystn.

Hexafluoroethane *[76-16-4]* **M 138.0, b -79°.** Purified for pyrolysis studies by passage through a copper vessel containing CoF_3 at *ca* 270°, and held for 3h in a bottle with a heated (1300°) platinum wire. It was then fractionally distd. [Steunenberg and Cady *J Am Chem Soc* **74** 4165 *1962*.]

1,1,1,3,3,3-Hexafluoropropan-2-ol *[920-66-1]* **M 168.1, b 57-58°/760mm, d 1.4563, n^{22} 1.2750.** Distd from 3A molecular sieves, retaining the middle fraction.

Hexahydro-1*H*-azepine (hexamethyleneimine, Azepane) *[111-49-9]* **M 99.2, b 70-72°/30mm, 135-138°/atm, d 0.879, n 1.466, pK^{25} 11.10 (pK° 9.71, pK^{75} 9.71).** Purified by dissolving in Et_2O and adding ethanolic HCl until all the base separates as the white *hydrochloride* , filter, wash with Et_2O and dry (**m** 236°). The salt is dissolved in the minimum vol of H_2O and basified to pH ~ 14 with 10N KOH. The soln is extracted with Et_2O, the extract is dried over KOH, evapd and distd. The base is a **FLAMMABLE** and **TOXIC** liquid, and best kept as the salt. The *nitrate* has **m** 120-123°, *Picrate* has **m** 145-147°, and the *Tosylate* has **m** 76.5° (ligroin). [Müller and Sauerwald *Monatsh Chem* **48** 727 *1027*; Hjelt and Agback *Acta Chem Scand* **18** 194 *1964*].

Hexahydromandelic acid *[R-(-)- 53585-93-6; S-(+)- 61475-31-8]* **M 158.2, m 127-129°, 128-129°, 129.7°, $[\alpha]_d^{20}$ (-) and (+) 25.5° (c 1, AcOH) and $[\alpha]_d^{20}$ (-) and (+) 13.6° (c 7.6, EtOH).** For hexagonal clusters by recrystallisation from CCl_4 or Et_2O. [Wood and ComLey *J Chem Soc* 2638 *1924*; Lettré et al. *Chem Ber* **69** 1594 *1936*]. The *racemate* has **m** 137.2-137.6° (134-135°) [Smith et al. *J Am Chem Soc* **71** 3772 *1949*].

Hexamethylbenzene *[87-85-4]* **M 162.3, m 165-165.5°.** Sublimed, then crystd from abs EtOH, *benzene, EtOH/*benzene or EtOH/cyclohexane. Also purified by zone melting. Dried under vac over P_2O_5.

Hexamethyl(Dewar)benzene *[7641-77-2]* **M 162.3, m 7°, b 60°/20mm, d 0.803, n 1.4480.** Purified by passage through alumina [Traylor and Miksztal *J Am Chem Soc* **109** 2770 *1987*].

Hexamethylenediamine *[124-09-4]* **M 116.2, m 42°, b 46-47°/1mm, 84.9°/9mm, 100°/20mm, 204-205°/760mm, pK_1^{25} 10.24, pK_2^{25} 11.02.** Crystd in a stream of nitrogen. Sublimed in a vacuum.

Hexamethylenediamine dihydrochloride *[6055-52-3]* **M 189.2, m 248°.** Crystd from water or EtOH.

Hexamethylene glycol (1,6-hexanediol) *[629-11-8]* **M 118.2, m 41.6°, 43-45°, b 134°/10mm, 250°, n 1.458.** Fractionally crystd from its melt or from water. Distils *in vacuo*.

Hexamethylenetetramine (Urotropine, hexamine, HMTA) *[100-97-0]* **M 140.1, m 280° (subln), 290-292° (sealed tube, CARE), d 1.331, pK^{25} 4.85 (6.30).** It is soluble in H_2O (67%), $CHCl_3$ (10%), EtOH (8%) and Et_2O (0.3%), and a 0.2M soln has a pH of 8.4. Dissolve in hot abs EtOH (reflux, Norite), filter using a heated funnel, cool at room temp first then in ice. Wash crysts with cold Et_2O, dry in air or under a vacuum. A further crop can be obtained by adding Et_2O to the filtrate. It sublimes above 260° without melting. The *picrate* has **m** 179° (dec). [pK 4.85:Reilley and Schmid *Anal Chem* **30** 947 *1958*; pK 6.30: Pummerer and Hofmann *Chem Ber* **56** 1255 *1923*.]

***n*-Hexane** *[110-54-3]* **M 86.2, b 68.7°, d 0.660, n 1.37486, n^{25} 1.37226.** Purification as for *n*-heptane. Modifications include the use of chlorosulfonic acid or 35% fuming H_2SO_4 instead of conc H_2SO_4 in washing the alkane, and final drying and distn from sodium hydride. Unsatd compounds can be removed by shaking the hexane with nitrating acid (58% H_2SO_4, 25% conc HNO_3, 17% water, or 50% HNO_3, 50% H_2SO_4), then washing the hydrocarbon layer with conc H_2SO_4, followed by H_2O, drying, and distg over sodium or *n*-butyl lithium. Also purified by distn under nitrogen from sodium benzophenone ketyl solubilised with tetraglyme. Also purified by passage through a silica gel column followed by distn [Kajii et al. *J Phys Chem* **91** 2791 *1987*]. FLAMMABLE liquid and possible nerve toxin.
Rapid purification: Distil, discarding the first forerun and stored over 4A molecular sieves.

1,2-Hexanediol *[6920-22-5]* **M 118.2, b 214-215, d 0.951, n 1.442.** Fractionally distd.

1-Hexene *[592-41-6]* **M 84.2, b 63°, d 0.674, n 1.388.** Purified by stirring over Na/K alloy for at least 6h, then fractionally distd from sodium under nitrogen.

***cis*-2-Hexene** *[7688-21-3]* **M 84.2, b 68-70°, d 0.699, n 1.399.** Purification as for 1-hexene above.

***trans*-2-Hexene** *[4050-45-7]* **M 84.2, b 65-67°, n 1.390.** Purifn as for 1-hexene above.

***trans*-3-Hexene** *[13269-52-8]* **M 84.2, b 67-69°, d 0.678, n 1.393.** Purifn as for 1-hexene above.

***meso*-Hexoestrol** *[84-16-2]* **M 270.4, m 185-188°.** Crystd from *benzene or aqueous EtOH.

***n*-Hexyl alcohol (1-hexanol)** *[111-27-3]* **M 102.2, b 157.5°, d 0.818, n^{15} 1.4198, n^{25} 1.4158.** Commercial material usually contains other alcohols which are difficult to remove. A suitable method is to esterify with hydroxybenzoic acid, recrystallise the ester and saponify. [Olivier *Recl Trav Chim, Pays-Bas* **55** 1027 *1936*.] Drying agents include K_2CO_3 and $CaSO_4$, followed by filtration and distn. (Some decomposition to the olefin occurred when Al amalgam was used as drying agent at room temperature, even though the amalgam was removed prior to distn.) If the alcohol is required anhydrous, the redistd material can be refluxed with the appropriate alkyl phthalate or succinate, as described under *Ethanol*.

***n*-Hexylamine** *[111-26-2]* **M 101.2, b 131°, d 0.765, n 1.419, pK^{25} 10.64.** Dried with, and fractionally distd from, KOH or CaH_2.

***n*-Hexyl bromide** *[111-25-1]* **M 165.1, b 87-88°/90mm, 155°/743mm, d 1.176, n 1.448.** Shaken with H_2SO_4, washed with water, dried with K_2CO_3 and fractionally distd.

***n*-Hexyl methacrylate** *[142-09-6]* **M 154.2, b 65-66°/4mm,88-88.5°/14mm, d 0.8849, n 1.4320.** Purified as for *methyl methacrylate*. [IR: Hughes and Walton *J Am Chem Soc* **79** 3985 *1957*.]

Hexyltrimethylammonium bromide *[2650-53-5]* **M 224.3, m 186°.** Recrystd from acetone. Extremely *hygroscopic* salt. [McDowell and Kraus *J Am Chem Soc* **73** 2170 *1951*.]

1-Hexyne *[693-02-7]* **M 82.2, b 12.5°/75mm, 71°/760mm, d 0.7156, n 1.3989.** Distd from $NaBH_4$ to remove peroxides. Stood with sodium for 24h, then fractionally distd under reduced pressure. Also dried by repeated vac transfer into freshly activated 4A molecular sieves, followed by vacuum transfer into Na/K alloy and stirring for 1h before fractionally distilling.

2-Hexyne *[764-35-2]* **M 82.2, b 83.8°/760mm, d 0.73146, n 1.41382.** Purification as for 1-hexyne above.

3-Hexyne *[928-49-4]* **M 82.2, b 81°/760mm, d 0.7231, n 1.4115.** Purification as for 1-hexyne above.

Histamine *[51-45-6]* **M 111.2, m 86° (sealed tube), b 167°/0.8mm, 209°/18mm, pK_1^{25} 6.02, pK_2^{25} 9.70.** Crystd from *benzene or chloroform.

Histamine dihydrochloride *[56-92-8]* **M 184.1, m 249-252° (244-245°).** Crystd from aq EtOH.

***S*-Histidine** *[71-00-1]* **M 155.2, m 287°(dec), $[\alpha]_D^{25}$ -39.7° (c 1, H_2O), +13.0° (6M HCl), pK_1^{25} 1.96, pK_2^{25} 6.12, pK_3^{25} 9.17.** Likely impurity is arginine. Adsorbed from aqueous soln on to Dowex 50-H^+ ion-exchange resin, washed with 1.5M HCl (to remove other amino acids), then eluted with 4M HCl as the dihydrochloride. Histidine is also purified as the dihydrochloride which is finally dissolved in water, the pH adjusted to 7.0, and the free zwitterionic base crystallises out on addition of EtOH. Sol in H_2O is 4.2% at 25°.

***S*-Histidine dihydrochloride** *[1007-42-7]* **M 242.1, m 245°, $[\alpha]_D^{20}$ +47.5° (c 2, H_2O).** Crystd from water or aqueous EtOH, and washed with acetone, then diethyl ether. Converted to the histidine di-(3,4-dichlorobenzenesulfonate) salt by dissolving 3,4-dichlorobenzenesulfonic acid (1.5g/10mL) in the aqueous histidine soln with warming, and then the soln is cooled in ice. The resulting crystals (**m** 280° dec) can be recrystd from 5% aqueous 3,4-dichlorobenzenesulfonic acid, then dried over $CaCl_2$ under vacuum, and washed with diethyl ether to remove excess reagent. The dihydrochloride can be regenerated by passing the soln through a Dowex-1 (Cl^- form) ion-exchange column. The solid is obtained by evapn of the soln on a steam bath or better in a vacuum. [Greenstein and Winitz, The Amino Acids **Vol 3** p. 1976 *1961*.]

***S*-Histidine monohydrochloride (H_2O)** *[5934-29-2 (H_2O); 7048-02-4]* **M 209.6, m 80° monohydrate, 254°(dec, anhyd), $[\alpha]_D^{25}$ +13.0° (6M HCl).** Crystd from aqueous EtOH.

***dl*-Homocysteine** *[6027-13-0]* **M 135.2, pK_2^{25} 8.70, pK_3^{30} 10.46.** Crystd from aqueous EtOH.

Homocystine *[462-10-2]* **M 268.4, m 260-265°(dec), pK_1^{25} 1.59, pK_2^{25} 2.54, pK_3^{25} 8.52, pK_4^{25} 9.44.** Crystd from water.

Homophthalic acid *[89-51-0]* **M 180.2, m 182-183°, 189-190°, (depends on heating rate) $pK_{Est(1)}$ ~3.5, $pK_{Est(2)}$~4.3.** Crystd from boiling water (25mL/g). Dried at 100°.

Homopiperazine (1,4-diazepane) *[505-66-8]* **M 100.2, m 38-40°, 43°, b 60°/10mm, 92°/50mm, 169°/atm, pK_1^{20} 6.70, pK_2^{20} 10.41.** Purified by fractionation through a column of 10 theoretical plates with a reflux ratio of 3:1. It boiled at 169° and the cool distillate crystallises in plates **m** 43°. [Poppelsdorf and Myerly *J Org Chem* **26** 131 *1961*.] Its pKa values are 6.89 and 10.65 at 40°, and 6.28 and 9.86 at 40° [Pagano et al. *J Phys Chem* **65** 1062 *1961*]. The *1,4-bis(4-bromobenzoyl* derivative has **m** 194-198° (from EtOH); the *hydrochloride* has **m** 270-290° (from EtOH) and the *picrate* has **m** 265° (dec) [Lloyd et al. *J Chem Soc (C)* 780 *1966*].

L-Homoserine (2-amino-4-hydroxybutyric acid) *[672-15-1]* **M 119.1, m 203°, $[\alpha]_D^{26}$ +18.3° (in 2M HCl), $pK_{Est(1)}$ ~2.1, $pK_{Est(2)}$~9.3.** Likely impurities are *N*-chloroacetyl-L-homoserine, *N*-chloroacetyl-D-homoserine, L-homoserine, homoserine lactone, homoserine anhydride (formed in strong solns of homoserine if slightly acidic). Cyclises to the lactone in strongly acidic soln. Crystd from water by adding 9 volumes of EtOH.

Homoveratronitrile (3,4-dimethoxybenzylnitrile) *[93-17-4]* **M 177.2, m 62-64°, 68°, b 184°/20mm, 195-196°/2mm, 208°/atm.** Its solubility is 10% in MeOH. and has been recrystd from

EtOH or MeOH. Purified by distillation followed by recrystn. [Niederl and Ziering *J Am Chem Soc* **64** 885 *1952*; Julian and Sturgis *J Am Chem Soc* **57** 1126 *1935*.]

Homoveratrylamine (2-[3,4-dimethoxyphenyl]ethylamine) *[120-20-7]* **M 181.2, b 99.3-101.3°/0.5mm, 168-170°/15mm, d_4^{20} 1.091, n_D^{20} 1.5460, pK_{Est} ~9.8.** Purified by fractionation through an efficient column in an inert atmosphere as it is a relatively strong base. [Horner and Sturm *Justus Liebigs Ann Chem* **608** 12819 *1957*; Jung et al. *J Am Chem Soc* **75** 4664 *1953*.] The *hydrochloride* has **m** 152°, 154°, 156° (from EtOH, Me_2CO or EtOH/Et_2O) and the *picrate* has **m** 165-167° dec, and the *4-nitrobenzoyl* derivative has **m** 147° [Buck *J Am Chem Soc* **55** 2593 *1933*].

Hordenine {4-[(2-dimethylamino)ethyl]phenol} *[539-15-1]* **M 165.2, m 117-118°, pK^{25} 9.46 (OH).** Crystd from EtOH or water.

Humulon *[26472-41-3]* **M 362.5, m 65-66.5°, $[\alpha]_D^{26}$ -212° (95% EtOH).** Crystd from Et_2O.

Hyamine 1622 [(diisobutylphenoxyethoxyethyl)dimethylbenzylammonium chloride, benzethionium chloride] *[121-54-0]* **M 448.1, m 164-166° (sinters at 120°, monohydrate).** Crystd from boiling acetone after filtering, or from $CHCl_3$-pet ether. The ppte was filtered off, washed with diethyl ether and dried for 24h in a vacuum desiccator.

Hydantoin (2,4-dihydroxyimidazole) *[461-72-3]* **M 100.1, m 216°, 220°, pK^{25} 9.15.** Crystd from MeOH. The *diacetate* has **m** 104-105°.

Hydrazine *N,N'*-dicarboxylic acid diamide *[110-21-4]* **M 116.1, m 248°.** Crystd from water and dried in vac over P_2O_5.

4-Hydrazinobenzoic acid *[619-67-0]* **M 152.2, m 217° (dec), pK^{25} 4.13.** Crystd from water.

1-Hydrazinophthalazine hydrochloride (hydralazine hydrochloride) *[304-20-1]* **M 196.6, m 172-173°, pK^{20} 6.57.** Crystd from MeOH.

2-Hydrazinopyridine *[4930-98-7]* **M 109.1, m 41-44°, 46-47°, 49-50°, b 105°/0.5mm, 128-135°/13mm.** Purified by distn and by recrystn from Et_2O-hexane. [Kauffmann et al. *Justus Liebigs Ann Chem* **656** 103 *1962*, Potts and Burton *J Org Chem* **31** 251 *1966*.] The *mono-hydrochloride* has **m** 183° (dec) from aq HCl and the *di-hydrochloride* has **m** 214-215°.

Hydrazobenzene *[122-66-7]* **M 184.2.** See 1,1-diphenylhydrazine on p. 225.

Hydrobenzamide [1-phenyl-*N,N'*-bis(phenylmethylene)-methanediamine] *[92-29-5]* **M 298.4, m 101-102°, 107-108°.** Crystd from absolute EtOH or cyclohexane/*benzene. Dried under vacuum over P_2O_5. [Pirrone *Gazz Chim Ital* **67** 534 *1937*.]

***dl*-Hydrobenzoin** *[492-70-6]* **M 214.3, m 120°.** Crystd from diethyl ether/pet ether.

***meso*-Hydrobenzoin** *[579-43-1]* **M 214.3, m 139°.** Crystd from EtOH or water.

Hydroquinone (1,4-dihydroxybenzene, quinol) *[123-31-9]* **M 110.1, m 175.4, 176.6°, pK_1^{20} 9.91, p K_2^{20} 11.56.** Crystd from acetone, *benzene, EtOH, EtOH/*benzene, water or acetonitrile (25g in 30mL), preferably under nitrogen. Dried under vacuum. [Wolfenden et al. *J Am Chem Soc* **109** 463 *1987*.]

4'-Hydroxyacetanilide *[103-90-2]* See 4-acetamidophenol on p. 83.

***p*-Hydroxyacetophenone** *[99-93-4]* **M 136.2, m 109°, pK^{25} 8.01.** Crystd from diethyl ether, aqueous EtOH or *benzene/pet ether.

4-Hydroxyacridine *[18123-20-1]* **M 195.2, m 116.5° pK_1^{15} 5.28, pK_2^{15} 9.75.** Crystd from EtOH.

5-Hydroxyanthranilic acid *[548-93-6]* **M 153.1, m >240°(dec), λ_{max} 298nm, log ε 3000 (0.1M HCl), pK_1^{20} 2.7, pK_2^{20} 5.37, pK_3^{20} 10.12.** Crystd from water. Sublimes below its melting point in a vacuum.

***erythro*-3-Hydroxy-*RS*-aspartic acid** *[6532-76-9]* **M 149.1, pK_1^{25} 1.91, pK_2^{25} 3.51, pK_3^{25} 9.11.** Likely impurities are 3-chloromalic acid, ammonium chloride, *threo*-3-hydroxyaspartic acid. Crystd from water.

***m*-Hydroxybenzaldehyde** *[100-83-4]* **M 122.1, m 108° pK_1^{25} 8.98, pK_2^{25} 15.81.** Crystd from water.

***p*-Hydroxybenzaldehyde** *[123-08-0]* **M 122.1, m 115-116°, pK^{25} 7.61.** Crystd from water (containing some H_2SO_4). Dried over P_2O_5 under vacuum.

***m*-Hydroxybenzoic acid** *[99-06-9]* **M 138.1, m 200.8°, pK_1^{25} 4.08, pK_2^{25} 9.98.** Crystd from absolute EtOH.

***p*-Hydroxybenzoic acid** *[99-96-7]* **M 138.1, m 213-214°, pK_1^{25} 4.50, pK_2^{25} 9.11.** Crystd from water.

***p*-Hydroxybenzonitrile** *[767-00-0]* **M 119.1, m 113-114°.** See *p*-cyanophenol on p. 176.

4-Hydroxybenzophenone *[1137-42-4]* **M 198.2, m 133.4-133.8°, pK^{25} 7.95.** See *p*-benzoylphenol on p. 126.

2-Hydroxybenzothiazole *[934-34-9]* **M 183.1, m 117-118°** Crystd from aqueous EtOH or water. [Dryland and Sheppard *J Chem Soc Perkin Trans 1* 125 *1986.*]

1-Hydroxybenzotriazole hydrate (HOBt) *[2592-95-2]* **M 135.1, m 159-160°.** Crystd from aqueous EtOH or water. [Dryland and Sheppard *J Chem Soc Perkin Trans 1* 125 *1986.*]
§ A polystyrene supported version is available.

2-Hydroxybenzyl alcohol *[90-01-7]* **M 124.1, m 87°, pK^{25} 9.84.** Crystd from water or *benzene.

3-Hydroxybenzyl alcohol *[620-24-6]* **M 124.1, m 71°, pK_{Est} ~9.8.** Crystd from *benzene.

4-Hydroxybenzyl alcohol *[623-05-2]* **M 124.1, m 114-115°, pK^{25} 9.73.** Crystd from water.

2-Hydroxybiphenyl *[90-43-7]* **M 170.2, m 56°, b 145°/14mm, 275°/760mm, pK^{20} 10.01.** Crystd from pet ether.

4-Hydroxybiphenyl (4-phenylphenol) *[92-69-3]* **M 170.2, m 164-165°, b 305-308°/760mm, pK^{23} 9.55.** Crystd from aqueous EtOH, aq EtOH, $*C_6H_6$, and vac dried over $CaCl_2$ [Buchanan et al. *J Am Chem Soc* **108** 7703 *1986*].

3-Hydroxy-2-butanone (acetoin) *[513-86-0]* **M 88.1, b 144-145°, [m 100-105° dimer].** Washed with EtOH until colourless, then with diethyl ether or acetone to remove biacetyl. Air dried by suction and further dried in a vacuum desiccator.

(±)-α-Hydroxy-γ-butyrolactone *[19444-84-9]* **M 102.1, b 84°/0.2mm, 133°/10mm, d_4^{20} 1.310, n_D^{20} 1.4656.** It has been purified by repeated fractionation, forms a colourless liquid. It has to be distd at high vacuum otherwise it will dehydrate. The *acetoxy* derivative has **b** 94°/0.2mm, [NMR: Daremon and Rambaud *Bull Soc Chim Fr* 294 *1971*; Schmitz et al. *Chem Ber* **108** 1010 *1975.*]

4-Hydroxycinnamic acid (*p*-**coumaric acid**) *[501-98-4]* **M 164.2, m 210-213°, 214-215°, 215° pK_1^{25} 4.64, pK_2^{25} 9.45.** Crystd from H_2O (charcoal). Needles from conc aqueous solutions as the *anhydrous acid*, but from hot dilute solutions the *monohydrate acid* separates on slow cooling. The acid (33g) has been recrystd from 2.5L of H_2O (1.5g charcoal) yielding 28.4g of recrystd acid, **m** 207°. It is insol in *C_6H_6 or pet ether. The UV in 95% EtOH has λ_{max} 223 and 286nm (ε 14,450 and 19000 $M^{-1}cm^{-1}$). [UV Wheeler and Covarrubias *J Org Chem* **28** 2015 *1963*; Corti *Helv Chim Acta* **32** 681 *1949*.]

4-Hydroxycoumarin *[1076-38-6]* **M 162.1, m 206°, pK_{Est} ~9.0.** Crystd from water and dried in a vacuum desiccator over Sicapent.

3-(4-Hydroxy-3,5-dimethoxyphenyl)acrylic acid *[530-59-6]* **M 234.1, m 204-205°(dec), $pK_{Est(1)}$~4.6, $pK_{Est(2)}$ ~9.3.** Crystd from water.

4-Hydroxydiphenylamine *[122-37-2]* **M 185.2, m 72-73°, pK_{Est} ~10.0.** Crystd from chlorobenzene/pet ether.

12-Hydroxydodecanoic acid *[505-95-3]* **M 216.3, m 86-88°, pK_{Est} ~4.8.** Crystd from toluene [Sadowik et al. *J Am Chem Soc* **108** 7789 *1986*].

2-Hydroxy-4-(*n*-dodecyloxy)benzophenone *[2985-59-3]* **M 382.5, m 50-52°, pK_{Est} ~7.1.** Recryst from *n*-hexane and then 10% (v/v) EtOH in acetonitrile [Valenty et al. *J Am Chem Soc* **106** 6155 *1984*].

***N*-[2-Hydroxyethyl]ethylenediamine [2-(2-aminoethylamino)ethanol]** *[111-41-1]* **M 104.1, b 91.2°/5mm, 238-240°/752mm, n 1.485, d 1.030, pK_1^{20} 3.75, pK_2^{20} 9.15.** Distilled twice through a Vigreux column. Redistilled from solid NaOH, then from CaH_2. Alternatively, it can be converted to the dihydrochloride and recrystallised from water. It is then dried, mixed with excess of solid NaOH and the free base distilled from the mixture. It is finally redistilled from CaH_2. [Drinkard, Bauer and Bailar *J Am Chem Soc* **82** 2992 *1960*.]

***N*-[2-Hydroxyethyl]ethylenediaminetriacetic acid (HEDTA)** *[150-39-0]* **M 278.3, m 212-214°(dec), pK_1^{20} 2.51, pK_2^{20} 5.31, pK_3^{20} 9.86.** Crystd from warm H_2O, after filtering, by addition of 95% EtOH and allowing to cool. The crystals, collected on a sintered-glass funnel, were washed three times with cold absolute EtOH, then again crystd from H_2O. After leaching with cold H_2O, the crystals were dried at 100° under vacuum. [Spedding, Powell and Wheelwright *J Am Chem Soc* **78** 34 *1956*.]

***N*-Hydroxyethyliminodiacetic acid (HIMDA)** *[93-62-9]* **M 177.2, m 181°(dec), pK_1^{25} 2.16, pK_2^{25} 8.72, pK_3^{25} 13.7 (OH).** Crystd from water.

2-Hydroxyethylimino-tris(hydroxymethyl)methane (MONO-TRIS) *[7343-51-3]* **M 165.2, m 91°, pK_{Est} ~9.8.** Crystd twice from EtOH. Dried under vacuum at 25°.

2-Hydroxyethyl methacrylate *[868-77-9]* **M 130.1, b 67°/3.5mm, d 1.071, n 1.452.** Dissolved in water and extracted with *n*-heptane to remove ethylene glycol dimethacrylate (checked by gas-liquid chromatography and by NMR) and distilled twice under reduced pressure [Strop, Mikes and Kalal *J Phys Chem* **80** 694 *1976*].

***N*-2-Hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid (HEPES)** *[7365-45-9]* **M 238.3, pK^{20} 7.55.** Crystd from hot EtOH and water.

3-Hydroxyflavone *[577-85-5]* **M 238.2, m 169-170°, 171-172°.** Recrystd from MeOH, EtOH or hexane. Also purified by repeated sublimation under high vacuum, and dried by high vacuum pumping for at least one hour [Bruker and Kelly *J Phys Chem* **91** 2856 *1987*].

ß-Hydroxyglutamic acid *[533-62-0]* **M 163.1, m 100°(dec), pK_1^{25} 2.27, pK_2^{25} 4.29, pK_3^{25} 9.66.** Crystd from water.

4-Hydroxyindane *[1641-41-1]* **M 134.2, m 49-50°, b 120°/12mm, pK^{25} 10.32.** Crystd from pet ether. *Acetyl* deriv has **m** 30-32° (from EtOH), **b** 127°/14mm. [Dallacker et al. *Chem Ber* **105** 2568 *1972*.]

5-Hydroxyindane *[1470-94-6]* **M 134.2, m 55°, b 255°/760mm, pK_{Est} ~10.4.** Crystd from pet ether.

5-Hydroxy-L-lysine monohydrochloride *[32685-69-1]* **M 198.7, $[\alpha]_D^{25}$ +17.8° (6M HCl), pK_2^{25} 8.85, pK_3^{25} 9.83.** Likely impurities are 5-*allo*-hydroxy-(D and L)-lysine, histidine, lysine, ornithine. Crystd from water by adding 2-9 volumes of EtOH stepwise.

4-Hydroxy-3-methoxyacetophenone *[498-02-2]* **M 166.2, m 115°, pK_{Est} ~7.9.** Crystd from water, or EtOH/pet ether.

4-Hydroxy-3-methoxycinnamic acid (ferulic acid) *[1135-24-6]* **M 194.2, m 174°, pK_1^{25} 4.58, pK_2^{25} 9.39.** Crystd from H_2O.

1-Hydroxymethyladamantane *[770-71-8]* **M 166.3, m 115°.** Dissolve in Et_2O, wash with aqueous 0.1N NaOH and H_2O, dry over $CaCl_2$, evaporate and recryst residue from aqueous MeOH. [*Chem Ber* **92** 1629 *1959*.]

17β-Hydroxy-17α-methyl-3-androsterone (Mestanolone) *[521-11-9]* **M 304.5°, m 192-193°.** Crystd from ethyl acetate.

4-Hydroxy-2-methylazobenzene *[1435-88-7]* **M 212.2, m 100-101°, pK_{Est} ~9.5.** Crystd from hexane.

4-Hydroxy-3-methylazobenzene *[621-66-9]* **M 212.2, m 125-126°.** Crystd from hexane.

3-Hydroxy-4-methylbenzaldehyde *[57295-30-4]* **M 136.1, m 116-117°, b 179°/15mm, pK_{Est} ~10.2.** Crystd from water.

***dl*-2-Hydroxy-2-methylbutyric acid** *[3739-30-8]* **M 118.1, m 72-73°, pK^{25} 3.73.** Crystd from *benzene, and sublimed at 90°.

***dl*-2-Hydroxy-3-methylbutyric (α-hydroxyisovaleric) acid** *[600-37-3]* **M 118.1, m 86°, pK_{Est} ~3.9.** Crystd from ether/pentane.

***R*-γ-Hydroxymethyl-γ-butyrolactone** *[52813-63-5]* **M 116.1, b 101-102°/0.048mm, d_4^{20} 1.2238, n_D^{20} 1.471, $[\alpha]_{546}^{20}$ -38°, $[\alpha]_D^{20}$ -33° (c 3, EtOH), $[\alpha]_D^{30}$ -53.5° (c 3, EtOH).** Purified by column chromatography in Silica gel 60 (Merck 70-230 mesh) and eluting with 7% EtOH-73% $CHCl_3$. IR (film): 3400 (OH), 1765 (C=O) and 1180 (COC) cm^{-1}. [Eguchi and Kakuta *Bull Chem Soc Jpn* **47** 1704 *1974*; IR and NMR: Ravid et al. *Tetrahedron* **34** 1449 *1978*.]

7-Hydroxy-4-methylcoumarin (4-methylumbelliferone) *[90-33-5]* **M 176.2, m 185-186°, pK_{Est} ~10.0.** Crystd from absolute EtOH. (See also entry on p. 548 in Chapter 6.)

2-Hydroxymethyl-12-crown-4 *[75507-26-5]* **M 206.2, d_4^{20} 1.186, n_D^{20} 1.480.** Purified by chromatography on Al_2O_3 with EtOAc as eluent to give a *hygroscopic* colourless oil with IR 3418 (OH) and 1103 (COC) cm^{-1}, NMR δ 3.70 (s). [Pugia et al. *J Org Chem* **52** 2617 *1987*.]

***S*-(-)-5-Hydroxymethyl-2(5*H*)-furanone** *[78508-96-0]* **M 114.1, 39-42°, 40-44°, b 130°/0.3mm, $[\alpha]_{546}^{20}$ -180°, $[\alpha]_D^{20}$ -148° (c 1.4, H_2O).** It has been purified by chromatography on

Silica gel using hexane-EtOAc (1:1) to give a colourless oil which was distd using a Kügelrohr apparatus and the distillate crystallises on cooling. It has R_F 0.51 on Whatman No 1 paper using pentan-1-ol and 85% formic acid (1:1) and developing with ammoniacal $AgNO_3$. [Boll *Acta Chem Scand* **22** 3245 *1968*; NMR: Oppolzer et al. *Helv Chim Acta* **68** 2100 *1985*.]

5-(Hydroxymethyl)furfural *[67-47-0]* **M 126.1, m 33.5°, b 114-116°/1mm.** Crystd from diethyl ether/pet ether.

3-Hydroxy-3-methylglutaric acid (Meglutol) *[503-49-1]* **M 162.1, m 99-102°, 108-109°, 100°, $pK_{Est(1)}$ ~4.0, $pK_{Est(2)}$~5.0.** Recrystd from diethyl ether/hexane and dried under vac at 60° for 1h.

***dl*-3-Hydroxy-*N*-methylmorphinan** *[297-90-5]* **M 257.4, m 251-253°.** Crystd from anisole + aqueous EtOH.

6-Hydroxy-2-methyl-1,4-naphthaquinone *[633-71-6]* **M 188.2, pK_{Est} ~10.0.** Crystd from aqueous EtOH. Sublimes on heating.

4-Hydroxy-4-methyl-2-pentanone *[123-42-2]* **M 116.2, b 166°, d 0.932, n 1.4235, n^{25} 1.4213.** Loses water when heated. Can be dried with $CaSO_4$, then fractionally distd under reduced pressure.

17α-Hydroxy-6α-methylprogesterone (Medroxyprogesterone) *[520-85-4]* **M 344.5, m 220°, $[\alpha]_D^{25}$ +75°.** Crystd from chloroform.

2-Hydroxy-2-methylpropionic acid (α-hydroxyisobutyric acid, 2-methyllactic acid)) *[594-61-6]* **M 104.1, m 79°, b 114°/12mm, 212°/760mm, pK^{25} 3.78.** Distd in steam, crystd from diethyl ether or *benzene, sublimed at 50° and dried under vacuum.

8-Hydroxy-2-methylquinoline *[826-81-3]* **M 159.2, m 74-75°, b 266-267°, pK_1^{25} 5.61, pK_2^{25} 10.16.** Crystd from EtOH or aqueous EtOH.

2-Hydroxy-1-naphthaldehyde *[708-06-5]* **M 172.2, m 82°, b 192°/27mm, pK_{Est} ~7.8.** Crystd from EtOH (1.5mL/g), ethyl acetate or water.

2-Hydroxy-1-naphthaleneacetic acid *[10441-45-9]* **M 202.2, $pK_{Est(1)}$ ~4.2, $pK_{Est(2)}$~8.3.** Treated with activated charcoal and crystd from EtOH/water (1:9, v/v). Dried under vacuum, over silica gel, in the dark. Stored in the dark at -20° [Gafni, Modlin and Brand *J Phys Chem* **80** 898 *1976*]. Forms a lactone (**m** 107°) readily.

6-Hydroxy-2-naphthalenepropionic acid *[553-39-9]* **M 216.2, m 180-181°, $pK_{Est(1)}$ ~4.6, $pK_{Est(2)}$~9.0** Crystd from aqueous EtOH or aqueous MeOH.

3-Hydroxy-2-naphthalide *[92-77-3]* **M 263.3, m 248.0-248.5°, CI 37505.** Crystd from xylene [Schnopper, Broussard and La Forgia *Anal Chem* **31** 1542 *1959*].

3-Hydroxy-2-naphtho-4'-chloro-*o*-toluidide *[92-76-2]* **M 311.8, m 243.5-244.5°.** Crystd from xylene [Schnopper, Broussard and La Forgia *Anal Chem* **31** 1542 *1959*].

3-Hydroxy-2-naphthoic-1'-naphthylamide *[123-68-3]* **M 314.3, m 217-.5-218.0°.** Crystd from xylene [Schnopper, Broussard and La Forgia *Anal Chem* **31** 1542 *1959*].

3-Hydroxy-2-naphthoic-2'-naphthylamide *[136-64-8]* **M 305.3, m 243.5-244.5°, and other naphthol AS derivatives.** Crystd from xylene [Schnopper, Broussard and La Forgia *Anal Chem* **31** 1542 *1959*].

2-Hydroxy-1,4-naphthaquinone *[83-72-7]* **M 174.2, m 192°(dec), pK_1^{25}-5.6 (C=O protonation), pK_2^{25} 4.00 (phenolic OH).** Crystd from *benzene.

5-Hydroxy-1,4-naphthaquinone (Juglone) *[481-39-0]* **M 174.2, m 155°, 164-165°, pK 8.7.** Crystd from *benzene/pet ether or pet ether.

6-Hydroxy-2-naphthyl disulfide *[6088-51-3]* **M 350.5, m 221-222°, 226-227°, pK_{Est} ~9.0.** Crystallises as leaflets from AcOH and is slightly soluble in EtOH, and AcOH, but is soluble in $*C_6H_6$ and in alkalis to give a yellow soln. [Zincke and Dereser *Chem Ber* **51** 352 *1918*.] The *acetoxy* derivative has **m** 198-200° (from AcOH or dioxane-MeOH) and the *diacetyl* derivative has **m** 167-168° (from AcOH). A small amount of impure disulfide can be purified by dissolving in a small volume of Me_2CO and adding a large volume of toluene, filter rapidly and concentrate to one third of its volume. The hot toluene soln is filtered rapidly from any tarry residue, and crystals separate on cooling. After recrystn from hot acetic acid gives crystals **m** 220-223° [Barrett and Seligman *Science* **116** 323 *1952*].

2-Hydroxy-5-nitrobenzyl bromide *[772-33-8]* **M 232.0, m 147°, pK_{Est} ~8.0.** Crystd from *benzene or *benzene/ligroin.

4-Hydroxy-2-*n*-nonylquinoline *N*-oxide *[316-66-5]* **M 287.4, m 148-149°, pK_{Est} ~6.0.** Crystd from EtOH.

***N*-Hydroxy-5-norbornene-2,3-dicarboxylic acid imide** *[21715-90-2]* **M 179.2, m 165-166°, 166-169°, pK_{Est}~6** Dissolve in $CHCl_3$, filter, evaporate and recrystallise from EtOAc. IR (nujol): 1695, 1710 and 1770 (C=O), and 3100 (OH) cm^{-1}. *O-Acetyl* derivative has **m** 113-114° (from EtOH) with IR bands at 1730, 1770 and 1815 cm^{-1} only, and the *O-benzoyl* derivative has **m** 143-144° (from propan-2-ol or $*C_6H_6$). [Bauer and Miarka *J Org Chem* **24** 1293 *1959*; Fujino et al. *Chem Pharm Bull Jpn* **22** 1857 *1974*].

DL-*erythro*-3-Hydroxynorvaline (2-amino-3-hydroxypentanoic acid) *[34042-00-7]* **M 133.2, m 257-259° (dec), 263° (dec), pK_1^{20} 2.32, pK_2^{20} 9.12.** Purified by recrystn from aqueous EtOH. The *Cu salt* has **m** 255-256° (dec), the *benzoyl* derivative has **m** 181°, and the *N-phenylcarbamoyl* derivative has **m** 164°. [Buston et al. *J Biol Chem* **204** 665 *1953*].

2-Hydroxyoctanoic acid (2-hydroxycaprylic acid) *[617-73-2]* **M 160.2, m 69.5°, b 160-165°/10mm, pK_{Est} ~3.7.** Crystd from EtOH/pet ether or ether/ligroin.

1-Hydroxyphenazine (Hemipyocyanine) *[528-71-2]* **M 196.2, m 157-158°, pK_1^{25} 1.61, pK_2^{15} 8.33.** Chromatographed on acidic alumina with *benzene/ether. Crystd from *benzene/heptane, and sublimed.

2-Hydroxyphenylacetic acid *[614-75-5]* **M 152.2, m 148-149°, b 240-243°/760mm, $pK_{Est(1)}$~4.3, $pK_{Est(2)}$~10.1.** Crystd from ether or chloroform (**m** from latter is always lower).

3-Hydroxyphenylacetic acid *[621-37-4]* **M 152.2, m 137°, $pK_{Ext(1)}$~4.3, $pK_{Ext(2)}$~10.** Crystd from *benzene/ligroin.

4-Hydroxyphenylacetic acid *[156-38-7]* **M 152.2, m 150-151°, 152°, pK_1 4.28, pK_2 10.1.** Crystd from water or Et_2O/pet ether.

2-(2-Hydroxyphenyl)benzothiazole *[3411-95-8]* **M 227.2, m 132-133°, b 173-179°/3mm.** Recrystd several times from aqueous EtOH and by sublimation. [Itoh and Fujiwara *J Am Chem Soc* **107** 1561 *1985*.]

2-(2-Hydroxyphenyl)benzoxazole *[835-64-3]* **M 211.2, m 127°, b 338°/760mm.** Recrystd several times from aqueous EtOH and by sublimation. [Itoh and Fujiwara *J Am Chem Soc* **107** 1561 *1985*.]

3-Hydroxy-2-phenylcinchoninic acid *[485-89-2]* **M 265.3, m 206-207°(dec).** Crystd from EtOH.

***N*-(*p*-Hydroxyphenyl)glycine** *[22818-40-2]* **M 167.2, m >240°(dec), $pK_{Est(1)}$~2, $pK_{Est(2)}$~4.5, $pK_{Est(3)}$~10.3.** Crystd from water.

***N*-(4-Hydroxyphenyl)-3-phenylsalicylamide** *[550-57-2]* **M 305.3, m 183-184°, pK_{Est} ~9.5.** Crystd from aqueous MeOH.

L-2-Hydroxy-3-phenylpropionic acid (3-phenyl lactic acid) *[20312-36-1]* **M 166.2, m 125-126°, $[\alpha]_D^{12}$ -18.7° (EtOH), pK see below.** Crystd from water, MeOH, EtOH or *benzene.

***dl*-2-Hydroxy-3-phenylpropionic acid** *[828-01-3]* **M 166.2, m 97-98°, b 148-150°/15mm, pK_{Est} ~3.7.** Crystd from *benzene or chloroform.

3-*p*-Hydroxyphenylpropionic acid (phloretic acid) *[501-97-3]* **M 166.2, m 129-130°, 131-133°, $pK_{Est(1)}$~4.7, $pK_{Est(2)}$~10.1.** Crystd from ether or H_2O.

***p*-Hydroxyphenylpyruvic acid** *[156-39-8]* **M 180.2, m 220°(dec), pK_{Est} ~2.3.** Crystd three times from 0.1M HCl/EtOH (4:1, v/v) immediately before use [Rose and Powell *Biochem J* **87** 541 *1963*], or from Et_2O. The *3,4-Dinitrophenylhydrazone* has **m** 178°.

***N*-Hydroxyphthalimide** *[524-38-9]* **M 163.1, m 230°, ~235° (dec), 237-240°, pK^{30} 7.0.** Dissolve in H_2O by adding Et_3N to form the salt and while hot acidify, cool and pour into a large volume of H_2O. Filter off the solid, wash with H_2O, dry over P_2O_5 in vacuum. [Nefken And Teser *J Am Chem Soc* **83** 1263 *1961*; Fieser **1** 485 *1976;* Nefkens et al. *Recl Trav Chim Pays-Bas* **81** 683 *1962*] The *O-acetyl* derivative has **m** 178-180° (from EtOH).

3-β-Hydroxy-5-pregnen-20-one (pregnenolone) *[145-13-1]* **M 316.5, m 189-190°, $[\alpha]_D^{20}$ +30° (EtOH), $[\alpha]_{546}$ +34° (c 1, EtOH).** Crystd from MeOH.

17α-Hydroxyprogesterone *[604-09-1]* **M 330.5, m 222-223°, $[\alpha]_{546}^{20}$ +141° (c 2, dioxane), λ_{max} 240nm.** Crystd from acetone or EtOH. *Acetate:* **m** 239-240° and *caproate*: **m** 119-121° crystallised from $CHCl_3$/MeOH.

***R*-(+)-3-Hydroxyprolidine** *[2799-21-5]* **M 87.1, b 215-216°, d_4^{20} 1.078, n_D^{20} 1.490, $[\alpha]_D^{20}$ +6.5° (c 1.5, MeOH), pK_{Est} ~10.1.** Purify by repeated distn. The *hydrochloride* has -ve rotation and the *dimethiodide* has **m** 230° and $[\alpha]_D^{24}$ -8.02°. [Suyama and Kanno *Yakugaku Zasshi (J Pharm Soc Japan)* **85** 531 *1965*; Uno et al. *J Heterocycl Chem* **24** 1025 *1987*; Flanagan and Joullie *Heterocycles* **26** 2247 *1987.*]

***trans*-L-4-Hydroxyproline** *[51-35-4]* **M 131.1, m 274°, $[\alpha]_D^{20}$ -76.0° (c 5, H_2O), pK_1^{25} 1.86, pK_2^{25} 9.79.** Crystd from MeOH/EtOH (1:1). Separation from normal *allo*-isomer can be achieved by crystn of the copper salts [see *Biochem Prep* **8** 114 *1961*].

4'-Hydroxypropiophenone *[70-70-2]* **M 150.2, m 149°, pK_{Est} ~10.** Crystd from water.

2-(α-Hydroxypropyl)piperidine (2-piperidinepropanol) *[24448-89-3]* **M 143.2, m 121°, b 226°, pK_{Est} ~10.2.** Crystd from ether.

7-(2-Hydroxypropyl)theophylline (Proxyphylline) *[603-00-9]* **M 238.2, m 135-136°.** Crystd from EtOH.

6-Hydroxypurine (hypoxanthine) *[68-94-0]* **M 136.1, m 150°(dec), pK_1^{20} 8.96, pK_2^{20} 12.18.** Crystd from hot water. Dried at 105°.

2-Hydroxypyridine (2-pyridone) *[142-08-5]* **M 95.1, m 105-107°, b 181-185°/24mm, ε_{293nm} 5900 (H_2O) pK_1^{25} 1.25, pK_2^{25} 11.99.** Distd under vacuum to remove coloured impurity, then crystd from

*benzene, CCl_4, EtOH or $CHCl_3$/diethyl ether. It can be sublimed under high vacuum. [DePue et al. *J Am Chem Soc* **107** 2131 *1985*.]

3-Hydroxypyridine *[109-00-2]* **M 95.1, m 129° pK_1^{25} 5.10, pK_2^{25} 8.6.** Crystd from water or EtOH.

4-Hydroxypyridine (4-pyridone) *[626-64-2]* **M 95.1, m 65°(hydrate), 148.5° (anhydr), b >350°/760mm, pK_1^{20} 3.20, pK_2^{20} 11.12.** Crystd from H_2O. Loses H_2O on drying *in vacuo* over H_2SO_4. Stored over KOH because it is *hygroscopic*.

2(6)-Hydroxypyridine-5(3)-carboxylic acid (6-hydroxynicotinic acid) *[5006-66-6]* **M 139.1, m 304°(dec), pK^{20} 3.82.** Crystd from water.

4-Hydroxypyridine-2,6-dicarboxylic acid (chelidamic acid) *[138-60-3]* **M 183.1, m 254°(dec), pK_1^{22} 1.9, pK_2^{22} 3.18, pK_3^{22} 10.85.** Crystd from water.

2-Hydroxypyrimidine *[557-01-7]* **M 96.1, m 179-180°, pK_1^{20} 2.15, pK_2^{20} 9.2.** Crystd from EtOH or ethyl acetate.

4-Hydroxypyrimidine *[4562-27-0]* **M 96.1, m 164-165°, pK_1^{20} 1.66, pK_2^{20} 8.63.** Crystd from *benzene or ethyl acetate.

2-Hydroxypyrimidine hydrochloride *[38353-09-2]* **M 132.5, m 205°(dec).** Crystd from EtOH.

2-Hydroxyquinoline (carbostyril) *[59-31-4]* **M 145.2, m 199-200°, pK_1^{20} -0.31, pK_2^{20} 11.76.** Crystd from MeOH.

8-Hydroxyquinoline (oxine, 8-quinolinol) *[148-24-3]* **M 145.2, m 71-73°, 75-76°, 76°, b ~ 267° pK_1^{25} 4.91, pK_2^{25} 9.81.** Crystd from hot EtOH, acetone, pet ether (b 60-80°) or water. Crude oxine can be purified by pptn of copper oxinate, followed by liberation of free oxine with H_2S or by steam distn after acidification with H_2SO_4. Stored in the dark. Forms metal complexes. [Manske et al. *Can J Research* **27F** 359 *1949;* Phillips *Chem Rev* **56** 271 *1956*.]

8-Hydroxyquinoline-5-sulfonic acid (H_2O) *[84-88-8]* **M 243.3, m >310° pK_1^{25} 4.09, pK_2^{25} 8.66.** Crystd from water or dil HCl (*ca* 2% by weight).

5-Hydroxysalicylic acid *[490-79-9]* **M 154.1.** See 2,5-dihydroxybenzoic acid on p. 207.

***trans*-4-Hydroxystilbene** *[6554-98-9]* **M 196.3, m 189°.** Crystd from *benzene or acetic acid.

***N*-Hydroxysuccinimide** *[6066-82-6]* **M 115.1, m 96-98°, pK 6.0.** Recrystd from EtOH/ethyl acetate [Manesis and Goodmen *J Org Chem* **52** 5331 *1987*].

***dl*-2-Hydroxytetradecanoic acid** *[2507-55-3]* **M 244.4, m 81-82°, pK_{Est} ~3.7.** Crystd from chloroform.

***R*-2-Hydroxytetradecanoic acid** *[26632-17-7]* **M 244.4, m 88-2-88.5°, $[\alpha]_D^{20}$ -31° ($CHCl_3$).** Crystd from chloroform.

4-Hydroxy-2,2,6,6-tetramethylpiperidine *[2403-88-5]* **M 157.3, m 130-131°, pK 10.05.** Crystd from water as hydrate, and crystd from ether as the anhydrous base.

Hydroxy(tosyloxy)iodobenzene [phenyl(hydroxyl)tosyloxyiodine, hydroxy(4-methylbenzenesulfonato-O)phenyliodine, Koser's reagent] *[27126-76-7]* **M 392.2, m 134-136°, 135-138°, 134-136°, 136-138.5°.** Possible impurities are tosic acid (removed by washing with Me_2CO) and acetic acid (removed by washing with Et_2O). It is purified by dissolving in the minimum vol of MeOH, adding

Et_2O to cloud point and setting aside for the prisms to separate [Koser and Wettach *J Org Chem* **42** 1476 *1977*; NMR: Koser et al. *J Org Chem* **41** 3609 *1976*]. It has also been crystd from CH_2Cl_2 (needles, **m** 140-142°) [Neiland and Karele *J Org Chem, USSR (Engl Transl)* **6** 889 *1970*].

4(6)-Hydroxy-2,5,6(2,4,5)-triaminopyrimidine sulfate *[35011-47-3]* **M 257.22, m >340°, pK_1 2.0, pK_2 5.1, pK_3 10.1.** This salt has very low solubility in H_2O. It is best purified by conversion into the dihydrochloride salt which is then reconverted to the insoluble sulfate salt. The sulfate salt (2.57g, 10mmoles) is suspended in H_2O (20mL) containing $BaCl_2$ (10mmoles) and stirred in a boiling water bath for 15min. After cooling the insoluble $BaSO_4$ is filtered off and washed with boiling H_2O (10mL). The combined filtrate and washings are made acidic with HCl and evaporated to dryness. The residual hydrochloride salt is recrystd from H_2O by adding conc HCl whereby the *dihydrochloride salt* separates as clusters which darken at 260° and dec > 300° [Baugh and Shaw *J Org Chem* **29** 3610 *1964*; King and Spengley *J Chem Soc* 2144 *1952*]. The hydrochloride is then dissolved in H_2O and while hot an equivalent of H_2SO_4 is added when the sulfate separates as a white microcrystalline solid which is filtered off washed liberally with H_2O and dried in vacuum over P_2O_5. [Albert and Wood *J Appl Chem London* **3** 521 *1953*; UV: Cavalieri et al. *J Am Chem Soc* **70** 3875 *1948*; see also Pfleiderer *Chem Ber* **90** 2272 *1957*; Traube *Chem Ber* **33** 1371 *1900*].

9-Hydroxytriptycene *[73597-16-7]* **M 270.3, m 245-246.5°.** Crystd from *benzene/pet ether. Dried at 100° in a vacuum [Imashiro et al. *J Am Chem Soc* **109** 729 *1987*].

5-Hydroxy-L-tryptophan *[4350-09-8]* **M 220.2, m 273°(dec), $[\alpha]_D^{22}$ -32.5°, $[\alpha]_{546}^{20}$ -73.5° (c 1, H_2O), $pK_{Est(1)}$~2.4, $pK_{Est(2)}$~9.0, $pK_{Est(3)}$~9.4, $pK_{Est(4)}$ 16 (NH).** Likely impurities are 5-hydroxy-D-tryptophan and 5-benzyloxytryptophan. Crystd under nitrogen from water by adding EtOH. Stored under nitrogen.

Hydroxyurea *[127-07-1]* **M 76.1, m 70-72° (unstable form), m 135-140°, 141° (stable form).** See hydroxyurea on p. 431 in Chapter 5.

3-Hydroxyxanthone *[3722-51-8]* **M 212.2, m 246°.** Purified by chromatography on SiO_2 gel with pet ether/*benzene). Recrystd from *benzene or EtOH [Itoh et al. *J Am Chem Soc* **107** 4819 *1985*].

α-Hyodeoxycholic acid *[83-49-8]* **M 392.6, m 196-197°, $[\alpha]_{546}^{20}$ +8° (c 2, EtOH), pK_{Est} ~4.9.** Crystd from ethyl acetate.

Hyoscine (scopolamine, atroscine) *[51-34-3]* **M 321.4, m 59°, $[\alpha]_D^{20}$ -18° (c 5, EtOH), -28° (c 2, H_2O), $[\alpha]_{546}^{20}$ -30° (c 5, $CHCl_3$), pK^{25} 7.55.** Crystd from *benzene/pet ether. Racemate has **m** 56-57° (H_2O), 37-38° ($2H_2O$), syrup (anhydr), *l* and *d* isomers can separate as syrups when anhydrous.

Hypericin *[548-04-9]* **M 504.4, m 320°(dec).** Crystd from pyridine by addition of methanolic HCl.

Ibogaine

[83-74-9] **M 300.3, m 152-153°, $[\alpha]_D^{20}$ -54° (EtOH), pK 8.1 (80% aq $MeOCH_2CH_2OH$).** Crystd from aqueous EtOH and sublimes at 150°/0.01mm.

Imidazole (glyoxaline) *[288-32-4]* **M 68.1, m 89.5-91°, b 256°, pK_1^{25} 6.99, pK_2^{25} 14.44.** Crystd from *benzene, CCl_4, CH_2Cl_2, EtOH, pet ether, acetone/pet ether and distd deionized water. Dried at 40° under vacuum over P_2O_5. Distd at low pressure. Also purified by sublimation or by zone melting. [Caswell and Spiro *J Am Chem Soc* **108** 6470 *1986*.] ^{15}N-imidazole was crystd from *benzene [Scholes et al. *J Am Chem Soc* **108** 1660 *1986*].

4'-(Imidazol-1-yl)acetophenone *[10041-06-2]* **M 186.2, m 104-107°, pK 4.54.** Twice recrystd from CH_2Cl_2/hexane [Collman et al. *J Am Chem Soc* **108** 2588 *1986*].

Iminodiacetic acid *[142-73-4]* **M 133.1, m 225°(dec), pK_1^{25} 2.50, pK_2^{25} 9.40.** Crystd from water.

1,3-Indandione *[606-23-5]* **M 146.2, m 129-132°, pK^{18} 7.2 (1% aq EtOH).** Recrystd from EtOH [Bernasconi and Paschalis *J Am Chem Soc* **108** 2969 *1986*].

Indane *[496-11-7]* **M 118.1, b 177°, d 0.960, n 1.538.** Shaken with conc H_2SO_4, then water, dried and fractionally distd.

Indanthrene *[81-77-6]* **M 442.4, m 470-500°.** Crystd repeatedly from 1,2,4-trichlorobenzene.

Indazole *[271-44-3]* **M 118.1, m 147°, pK$_1^{20}$ 1.32, p K$_2^{25}$ 13.80 (acidic NH).** Crystd from water, sublimed under a vacuum, then pet ether (b 60-80°).

Indene *[95-13-6]* **M 116.2, f -1.5°, b 114.5°/100mm, d 0.994, n 1.5763.** Shaken with 6M HCl for 24h (to remove basic nitrogenous material), then refluxed with 40% NaOH for 2h (to remove benzonitrile). Fractionally distd, then fractionally crystd by partial freezing. The higher-melting portion was converted to its sodium salt by adding a quarter of its weight of sodamide under nitrogen and stirring for 3h at 120°. Unreacted organic material was distd off at 120°/1mm. The sodium salts were hydrolysed with water, and the organic fraction was separated by steam distn, followed by fractional distn. Before use, the distillate was passed, under nitrogen, through a column of activated silica gel. [Russell *J Am Chem Soc* **78** 1041 *1956*.]

Indigo *[482-89-3]* **M 262.3, sublimes at ~300°, m 390°(dec), and halogen-substituted indigo dyes.** Reduced in alkaline soln with sodium hydrosulfite, and filtered. The filtrate was then oxidised by air, and the resulting ppte was filtered off, dried at 65-70°, ground to a fine powder, and extracted with $CHCl_3$ in a Soxhlet extractor. Evapn of the $CHCl_3$ gave the purified dye. [Brode, Pearson and Wyman *J Am Chem Soc* **76** 1034 *1954*; spectral characteristics are listed.]

Indole *[120-72-9]* **M 117.2, m 52°, b 124°/5mm, 253-254°/760mm, pK$_1^{25}$ -2.47 (H_0 scale), pK$_2^{25}$ 16.97 (acidic NH).** Crystd from *benzene, hexane, water or EtOH/water (1:10). Further purified by sublimation in a vacuum or zone melting.

Indole-3-acetic acid *[87-51-4]* **M 175.2, m 167-169°, pK$_1^{25}$ -6.13 (aq H_2SO_4), pK$_2^{25}$ 4.54 (CO_2H).** Recrystd from EtOH/water [James and Ware *J Phys Chem* **89** 5450 *1985*].

3-Indoleacetonitrile *[771-51-7]* **M 156.2, m 33-36°, 36-38°, b 157°/0.2mm, 158-160°/0.1mm, viscous oil n$_D^{20}$ 1.6097.** Distil in very high vacuum and the viscous distillate crystallises on standing after a few days; the *picrate* has **m** 127-128° (from EtOH) [Coker et al. *J Org Chem* **27** 850 *1962*; Thesing and Schülde *Chem Ber* **85** 324 *1952*]. The *N-acetate* has **m** 118° (from MeOH) and has R_F = 0.8, on Silica Gel F_{254} in $CHCl_2$-MeOH 19:1 [Buzas et al. *Synthesis* 129 *1977*].

Indole-3-butanoic acid *[133-32-4]* **M 203.2, m 124-125°.** See 3-indolylbutyric acid on p. 543 in Chapter 6.

Indole-3-propionic acid *[830-96-6]* **M 189.2, m 134-135°, pK$_{Est}$ ~4.7.** Recrystd from EtOH/water [James and Ware *J Phys Chem* **89** 5450 *1985*].

Indolizine [pyrrocoline, pyrrolo(1,2-*a*)pyridine] *[274-40-8]* **M 117, m 73-74°, 75°, pK^{20} 3.94 (C-protonation).** Purified through an alumina column in *C_6H_6 and eluted with *C_6H_6 (toluene could be used instead). The eluate contained in the fluorescent band (using UV light λ 365mn) was collected, evapd and the cryst residues sublimed twice at 40-50°/0.2-0.5mm. The colourless crystals darkend on standing and should be stored in dark sealed containers. If the original sample is dark in color then is should be covered with water and steam distd. The crysts in the distillate are collected and, dried between filter paper and sublimed. It protonates on C3 in aqueous acid. It should give one fluorescent spot on paper chromatography (Whatman 1) in 3% aq ammonia and in *n*-BuOH, AcOH, H_2O (4:1:1). The *picrate* has **m** 101° from EtOH. [Armarego *J Chem Soc* 226 *1944*; Armarego *J Chem Soc (B)* 191 *1966;* Scholtz *Chem Ber* **45** 734 *1912*.]

(-)-Inosine *[58-63-9]* **M 268.2, m 215°,** $[\alpha]_{546}^{20}$ **-76° (c 1, 0.1M NaOH),** pK_1^{25} **1.06,** pK_2^{25} **8.96,** pK_3^{25} **11.36.** Crystd from aqueous 80% EtOH.

***i*-Inositol (*myo*)** *[87-89-8]* **M 180.2, m 228°.** See entry on p. 543 in Chapter 6.

Inositol monophosphate *[15421-51-9]* **M 260.1, m 195-197°(dec).** Crystd from water and EtOH.

Iodinin (1,6-phenazine-5,10-dioxide) *[68-81-5]* **M 244.1, m 236°(dec), pK 12.5.** Crystd from $CHCl_3$.

Inulin *[9005-80-5]* **M** $(162.14)_n$. Crystd from water.

Iodoacetamide *[144-48-9]* **M 185.0, m** *ca* **143°(dec).** Crystd from water or CCl_4.

Iodoacetic acid *[64-69-7]* **M 160.6, m 78°,** pK^{25} **3.19.** Crystd from pet ether (b 60-80°) or $CHCl_3$/CCl.

2-Iodoaniline *[615-43-0]* **M 219.0, m 60-61°,** pK^{25} **2.54.** Distd with steam and crystd from *benzene/pet ether.

4-Iodoaniline *[540-37-4]* **M 219.0, m 62-63°,** pK^{25} **3.81.** Crystd from pet ether (b 60-80°) by refluxing, then cooling in an ice-salt bath freezing mixture. Dried in air. Also crystd from EtOH and dried in a vacuum for 6h at 40° [Edidin et al. *J Am Chem Soc* **109** 3945 *1987*].

4-Iodoanisole *[696-62-8]* **M 234.0, m 51-52°, b 139°/35mm, 237°/726mm.** Crystd from aqueous EtOH.

Iodobenzene *[591-50-4]* **M 204.0, b 63-65°/10mm, 188°/atm, d 1.829,** n^{25} **1.6169.** Washed with dilute aqueous $Na_2S_2O_3$, then water. Dried with $CaCl_2$ or $CaSO_4$. Decolorised with charcoal. Distd under reduced pressure and stored with mercury or silver powder to stabilise it.

***o*-Iodobenzoic acid** *[88-67-5]* **M 248.4, m 162°,** pK^{20} **2.93.** Crystd repeatedly from water and EtOH. Sublimed under vacuum at 100°.

***m*-Iodobenzoic acid** *[618-51-9]* **M 248.4, m 186.6-186.8°,** pK^{25} **3.85.** Crystd repeatedly from water and EtOH. Sublimed under vacuum at 100°.

***p*-Iodobenzoic acid** *[619-58-9]* **M 248.4, m 271-272°,** pK^{25} **4.00.** Crystd repeatedly from water and EtOH. Sublimed under vacuum at 100°.

4-Iodobiphenyl *[1591-31-7]* **M 280.1, m 113.7-114.3°.** Crystd from EtOH/*benzene and dried under vacuum over P_2O_5.

2-Iodobutane *[513-48-4]* **M 184.0, b 120.0, d 1.50,** n^{25} **1.4973.** Purified by shaking with conc H_2SO_4, then washing with water, aq Na_2SO_3 and again with water. Dried with $MgSO_4$ and distd. Alternatively, passed through a column of activated alumina before distn, or treated with elemental bromine, followed by extraction of the free halogen with aqueous $Na_2S_2O_3$, thorough washing with water, drying and distilling. It is stored over silver powder and distd before use.

1-Iodo-2,4-dinitrobenzene *[709-49-9]* **M 294.0, m 88°.** Crystd from ethyl acetate.

Iodoform *[75-47-8]* **M 393.7, m 119°.** Crystd from MeOH, EtOH or EtOH/EtOAc. Steam volatile.

1-Iodo-4-nitrobenzene *[636-98-6]* **M 249.0, m 171-172°.** Ppted from acetone by addition of water, then recrystd from EtOH.

o-**Iodophenol** *[533-58-4]* **M 280.1, m 42°, pK^{25} 8.51.** Crystd from $CHCl_3$ or diethyl ether.

p-**Iodophenol** *[540-38-5]* **M 280.1, m 94°, 138-140°/5mm, pK^{25} 9.30.** Crystd from pet ether (b 80-100°) or distd *in vacuo*. If material has a brown or violet color, dissolve in $CHCl_3$, shake with 5% sodium thiosulfate soln until the $CHCl_3$ is colorless. Dry (Na_2SO_4), extract, evap and dist residue *in vacuo*. [Dains and Eberly *Org Synth* Coll Vol II, 355 *1948*.]

5-Iodosalicylic acid (2-hydroxy-5-iodobenzoic acid) *[119-30-2]* **M 264.0, m 197° pK$_1^{25}$ 2.65, pK$_2^{25}$ 13.05.** Crystd from water.

o-**Iodosobenzoic acid** *[304-91-6]* **M 264.0, m >200°, pK$_{Est}$ ~2.6.** Crystd from EtOH.

N-**Iodosuccinimide** *[516-12-1]* **M 225.0, m 200-201°.** Crystd from dioxane/CCl_4.

p-**Iodotoluene** *[624-31-7]* **M 218.0, m 35°, b 211-212°.** Crystd from EtOH.

3-Iodo-L-tyrosine *[70-78-0]* **M 307.1, m 205-208°(dec), [α]$_D^{25}$ -4.4° (c 5, 1M HCl), pK$_{Est(2)}$~2.1, pK$_{Est(3)}$~6.4, pK$_4^{25}$ 8.7.** Likely impurities are tyrosine, diiodotyrosine and iodide. Crystd by soln in dilute ammonia, at room temperature, followed by addition of dilute acetic acid to pH 6. Stored at 0°.

α-Ionone *[127-41-3]* **M 192.3, b 131°/13mm, d 0.931, n 1.520, [α]$_D^{23}$ +347° (neat).** Purified on a spinning band fractionating column.

β-Ionone *[79-77-6]* **M 192.3, b 150-151°/24mm, d 0.945, n 1.5211, ε$_{296nm}$ 10,700.** Converted to the *semicarbazone* (**m** 149°) by adding 50g of semicarbazide hydrochloride and 44g of potassium acetate in 150mL of water to a soln of 85g of ß-ionone in EtOH. (More EtOH was added to redissolve any ß-ionone that ppted.) The semicarbazone crystallised on cooling in an ice-bath and was recrystallised from EtOH or 75% MeOH to constant **m** (148-149°). The semicarbazone (5g) was shaken at room temperature for several days with 20mL of pet ether and 48mL of M H_2SO_4, then the ether layer was washed with water and dilute aqueous $NaHCO_3$, dried and the solvent was evaporated. The ß-ionone was distilled under vacuum. (The customary steam distillation of ß-ionone semicarbazone did not increase the purity.) [Young et al. *J Am Chem Soc* **66** 855 *1944*].

Iproniazid (isonicotinic acid 2-isopropylhydrazide) phosphate *[305-33-9]* **M 277.2, m 178-179°, 180-182°, pK$_{Est}$ ~3.5 (free base).** Crystd from H_2O and Me_2CO. *Free base* has **m** 113-114° from *C_6H_6/pet ether.

(±)-Irone (6-methyl-ionone, ±-*trans*-(α)-4*t*-[2,5,6,6-tetramethyl-cyclohex-2-yl]but-3*t*-en-2-one) *[79-69-6]* **M 206.3, b 85-86°/0.05mm, 109°/0.7mm, d$_4^{20}$ 0.9340, n$_D^{20}$ 1.4998.** If large amounts are available then fractionate through a Podbielniak column (see p. 141) or an efficient spinning band column, but small amounts are distilled using a Kügelrohr apparatus. The *4-phenyl-semicarbazone* has **m** 174-175° (165-165.5°). [IR: Seidel and Ruzocka *Helv Chim Acta* **35** 1826 *1952*; Naves *Helv Chim Acta* **31** 1280 *1948*; Lecomte and Naves *J Chim Phys* **53** 462 *1956*.]

Isatin (indole-2,3-dione) *[91-56-5]* **M 147.1, m 201-203°, 205°, pK >12 (acidic NH).** Crystd from amyl alcohol and sublimed at 180°/1mm. In aq NaOH the ring opens to yield sodium *o*-aminobenzoylformate.

Isatoic anhydride (3,1-benzoxazin-2,4[1-*H*]-dione) *[118-48-9]* **M 163.1, m 235-240°, 240-243°, 243°, 243-245°.** Recryst from EtOH or 95% EtOH (30mL/g) or dioxane (10mL/g) and dried in a vacuum. [Wagner and Fegley *Org Synth* Coll Vol III 488 *1955*; Ben-Ishai and Katchalski *J Am Chem Soc* **74** 3688 *1952*; UV: Zentmyer and Wagner *J Org Chem* **14** 967 *1949*.]

Isoamyl acetate (1-butyl-3-methyl acetate) *[123-92-2]* **M 130.2, b 142.0°, d 0.871, n 1.40535.** Dried with finely divided K_2CO_3 and fractionally distd.

Isoamyl alcohol (1-butyl-3-methyl alcohol) *[123-51-3]* **M 88.2, b 132°/760mm, d^{15} 0.8129, n^{15} 1.4085.** See 3-methyl-1-butanol on p. 290.

Isoamyl bromide (1-butyl-3-methyl bromide) *[107-82-4]* **M 151.1, f -112°, b 119.2°/737mm, d 1.208, n 1.444.** Shaken with conc H_2SO_4, washed with water, dried with K_2CO_3 and fractionally distd.

Isoamyl chloride (1-butyl-3-methyl chloride) *[513-36-0]* **M 106.6, b 99°/734mm, d 0.8704, n 1.4084.** Shaken vigorously with 95% H_2SO_4 until the acid layer no longer became coloured during 12h, then washed with water, saturated aq Na_2CO_3, and more water. Dried with $MgSO_4$, filtered and fractionally distd. Alternatively, a stream of oxygen containing 5% of ozone was passed through the chloride for a time, three times longer than was necessary to cause the first coloration of starch iodide paper by the exit gas. Subsequent washing of the liquid with aqueous $NaHCO_3$ hydrolysed the ozonides and removed organic acids. After drying and filtering, the isoamyl chloride was distd. [Chien and Willard *J Am Chem Soc* **75** 6160 *1953*.]

Isoamyl ether [diisopentyl ether, di-(1-butyl-3-methyl) ether] *[544-01-4]* **M 158.3, b 173.4°, d 0.778, n 1.40850.** This is a mixture of 2- and 3-methylbutyl ether. It is purified by refluxing with sodium for 5h, then distilled under reduced pressure, to remove alcohols. Isoamyl ether can also be dried with $CaCl_2$ and fractionally distd from P_2O_5.

D(-)-Isoascorbic acid (araboascorbic acid) *[89-65-6]* **M 176.1, m 174°(dec), $[\alpha]_D^{25}$ -16.8° (c 2, H_2O), pK^{18} 3.99.** Crystd from H_2O or dioxane.

***dl*-Isoborneol** *[124-76-5]* **M 154.3, m 212° (sealed tube).** Crystd from EtOH or pet ether (b 60-80°). Sublimes in a vacuum.

Isobutane *[75-28-5]* **M 58.1, b -10.2°, d 0.557.** Olefines and moisture can be removed by passage at 65° through a bed of silica-alumina catalyst which has previously been evacuated at about 400°. Alternatively, water and CO_2 can be taken out by passage through P_2O_5 then asbestos impregnated with NaOH. Treatment with anhydrous $AlBr_3$ at 0° then removes traces of olefins. Inert gases can be separated by freezing the isobutane at -195° and evacuating out the system.

Isobutene *[115-11-7]* **M 56.1, b -6.6°/760mm.** Dried by passage through anhydrous $CaSO_4$ at 0°. Purified by freeze-pump-thaw cycles and trap-to-trap distn.

Isobutyl alcohol (2-methyl-1-propanol) *[78-83-1]* **M 74.1, b 108°/760mm, d 0.801, n 1.396.** Dried with K_2CO_3, $CaSO_4$ or $CaCl_2$, filtered and fractionally distd. For further drying, the redistd alcohol can be refluxed with the appropriate alkyl phthalate or succinate as described under *ethanol* (see also p. 271).

Isobutyl bromide (1-bromo-2-methylpropane) *[78-77-3]* **M 137.0, b 91.2°, d 1.260, n 1.437.** Partially hydrolysed to remove any tertiary alkyl halide, then fractionally distd, washed with conc H_2SO_4, water and aqueous K_2CO_3, then redistd from dry K_2CO_3. [Dunbar and Hammett *J Am Chem Soc* **72** 109 *1950*.]

Isobutyl chloride (1-chloro-2-methylpropane) *[513-36-0]* **M 92.3, b 68.8°/760mm, d 0.877, n 1.398.** Same methods as described under *isoamyl chloride*.

Isobutyl chloroformate *[543-27-1]* **M 136.6, b 123-127°/atm, 128.8°/atm, d 1.053, n 1.4070.** It can be dried over $CaCl_2$ and fractionated at atm press while keeping moisture out. Its purity can be checked by conversion to the *phenyl urethane derivative* with PhNCO [Saunders et al. *J Am Chem Soc* **73** 3796 *1951*.] IR: ν 1780cm^{-1} [Thompson and Jameson *Spectrochim Acta* **13** 236 *1959*; Röse *Justus Liebigs Ann Chem* **205** 227 *1880*].

Isobutyl formate *[542-55-2]* **M 102.1, b 98.4°, d 0.885, n 1.38546.** Washed with saturated aqueous $NaHCO_3$ in the presence of saturatedd NaCl, until no further reaction occurred, then with saturated aqueous NaCl, dried ($MgSO_4$) and fractionally distd.

Isobutyl iodide (1-iodo-2-methylpropane) *[513-38-2]* **M 184.0, b 83°/250mm, 120°/760mm, d 1.60, n 1.495.** Shaken with conc H_2SO_4, and washed with water, aqueous Na_2SO_3, and water, dried with $MgSO_4$ and distd. Alternatively, passed through a column of activated alumina before distn. Stored under nitrogen with mercury in a brown bottle or in the dark.

Isobutyl vinyl ether *[109-53-5]* **M 100.2, b 108-110°, d 0.768, n 1.398.** Washed three times with equal volumes of aqueous 1% NaOH, dried with CaH_2, refluxed with sodium for several hours, then fractionally distd from sodium.

Isobutyraldehyde *[78-84-2]* **M 72.1, b 62.0°, d 0.789, n 1.377.** Dried with $CaSO_4$ and used immediately after distn under nitrogen because of the great difficulty in preventing oxidation. Can be purified through its acid bisulfite derivative.

Isobutyramide *[563-83-7]* **M 87.1, m 128-129°, b 217-221°.** Crystd from acetone, *benzene, $CHCl_3$ or water, then dried under vacuum over P_2O_5 or 99% H_2SO_4. Sublimed under vacuum.

Isobutyric acid *[79-31-2]* **M 88.1, b 154-154.5°, d 0.949, n 1.393, pK^{25} 4.60.** Distd from $KMnO_4$, then redistd from P_2O_5.

Isobutyronitrile (2-methylpropionitrile, isopropyl cyanide) *[78-82-0]* **M 69.1, b 103.6°, d^{25} 0.7650, n 1.378.** Shaken with conc HCl (to remove isonitriles), then with water and aq $NaHCO_3$. After a preliminary drying with silica gel or Linde type 4A molecular sieves, it is shaken or stirred with CaH_2 until hydrogen evolution ceases, then decanted and distd from P_2O_5 (not more than 5g/L, to minimize gel formation). Finally it is refluxed with, and slowly distd from CaH_2 (5g/L), taking precautions to exclude moisture.

(-)-γ-Isocaryophyllene (8-methylene-4,11,11-trimethylbicyclo[7.2.0]undec-4-ene) *[118-65-0]* **M 204.4, b 122-124°/12mm, 131-133°/16mm, 130-131°/24mm, 271-273°/atm, d_4^{20} 0.8959, n_D^{20} 1.496, $[\alpha]_{546}^{20}$ -31°, $[\alpha]_D^{20}$ -27° (neat).** Purified by vac dist or GLC using a nitrile-silicone column [Corey et al. *J Am Chem Soc* **86** 485 *1964*; Ramage and Simonsen *J Chem Soc* 741 *1936*; Kumar et al. *Synthesis* 461 *1976*].

L-Isoleucine *[73-32-5]* **M 131.2, m 285-286°(dec), $[\alpha]_D^{20}$ +40.6° (6M HCl) pK_1^{25} 2.66, pK_2^{25} 9.69.** Crystd from water by addition of 4 volumes of EtOH.

(-)-β-Isolongifolene (1-*R*-(-)- 2,2,7,7-tetramethyltricyclo[6.2.1.$0^{1,6}$]undec-5-ene) *[1135-66-6]* **M 204.4, b 82-83°/0.4mm, 144-146°/30mm, 255-256°/atm, d_4^{20} 0.930, n_D^{20} 1.4992, $[\alpha]_{546}^{20}$ -166°, $[\alpha]_D^{20}$ -138° (c 1, EtOH).** Refluxed over and distd from Na. [Zeiss and Arakawa *J Am Chem Soc* **76** 1653 *1954*; IR: Reinaecker and Graafe *Angew Chem, Int Ed Engl* **97** 348 *1985*; UV and NMR: Ranganathan et al. *Tetrahedron* **26** 621 *1970*.]

Isolysergic acid *[478-95-5]* **M 268.3, m 218°(dec), $[\alpha]_D^{20}$ +281° (c 1, pyridine) pK_1^{24} 3.33, pK_2^{24} 8.46.** Crystd from water.

Isonicotinamide *[1453-82-3]* **M 122.1, m 155.5-156°, pK_1^{20} -1.0 (protonation of $CONH_2$), pK_2^{20} 3.61, pK_3^{25} 11.47 (acidic $CONH_2$).** Recrystd from hot water.

Isonicotinic acid (pyridine-4-carboxylic acid) *[55-22-1]* **M 123.1, m 320°, pK^{25} 4.90.** Crystd repeatedly from water. Dried under vac at 110°.

Isonicotinic acid hydrazide (isoniazide) *[54-85-3]* **M 137.1, m 172°, pK_1 1.75 ($NHNH_2$), pK_2 3.57 (=N-), pK_3 10.75 (-NH).** Crystd from 95% EtOH.

1-Isonicotinyl-2-isopropylhydrazide *[54-92-2]* **M 179.2, m 112.5-113.5°.** Crystd from *benzene/pet ether.

1-Isonicotinyl-2-salicylidenehydrazide *[495-84-1]* **M 241.2, m 232-233°.** Crystd from EtOH.

Isonitrosoacetone (*anti*-pyruvic aldehyde-1-oxime) *[31915-82-9]* **M 87.1, m 69°.** Crystd from ether/pet ether or CCl_4.

Isonitrosoacetophenone (phenylglyoxaldoxime) *[532-54-7]* **M 149.2, m 126-128°.** Crystd from water.

5-Isonitrosobarbituric acid (violuric acid) *[26851-19-9]* **M 175.1, m 221-223°, 245-250°, pK_1 4.41, pK_2 9.66 (10.1).** Crystd from water or EtOH. *1,1-Dimethylvioluric acid*, **m** 144-147° has pK 4.72 [Taylor and Robinson *Talanta* **8** 518 *1961*].

Isononane *[34464-40-9]* **M 128.3, b 142°/760mm.** Passed through columns of activated silica gel and basic alumina (activity 1). Distd under high vacuum from Na/K alloy.

Isopentyl formate *[110-45-2]* **M 116.2, b 121-123°/atm, 123-123.6°/atm, 123-124°/atm, d_4^{20} 0.8713, n_D^{20} 1.391.** Colourless liquid which is soluble in 300 volumes of H_2O and is soluble in common organic solvents. It is purified by repeated distn using an efficient column at atmospheric pressure.

Isophorone *[78-59-1]* **M 138.2, b 94°/16mm, d 0.921, n^{18} 1.4778.** Washed with aqueous 5% Na_2CO_3 and then distd under reduced pressure, immediately before use. Alternatively, can be purified *via* the semicarbazone. [Erskine and Waight *J Chem Soc* 3425 *1960*.]

Isophthalic acid (benzene-1,3-dicarboxylic acid) *[121-91-5]* **M 166.1, m 345-348°, pK_1^{25} 3.70, pK_2^{25} 4.60.** Crystd from aqueous EtOH.

Isopinocampheol (pinan-3-ol, 2,6,6-trimethylbicyclo[3.1.1]heptan-3-ol) *[1S,2S,3S,5R-(+)- 27779-29-9; 1R,2R,3R,5S-(-)- 25465-65-0]* **M 154.25, m 52-55°, 55-56°, 55-57°, b 103°/11mm, n_D^{20} 1.4832, $[\alpha]_{546}^{20}$ (+) and (-) 43°, $[\alpha]_D^{20}$ (+) and (-) 36° (c 20, EtOH).** Dissolve in Et_2O, dry $MgSO_4$, filter, evaporate, then recryst from pet ether. Also recryst from aqueous EtOH and has been distd in a vacuum. [Kergomard and Geneix *Bull Soc Chim Fr* 394 *1958*; Zweifel and Brown *J Am Chem Soc* **86** 393 *1964*.] The *3,4-dinitrobenzoyl* deriv has **m** 100-101°, the *phenylcarbamoyl* derivative has **m** 137-138° and the *acid -phthalate* has **m** 125-126°.

Isoprene (2-methyl-1,3-butadiene) *[78-79-5]* **M 68.1, b 34.5-35°/762mm, d 0.681, n^{25} 1.4225.** Refluxed with sodium. Distd from sodium or $NaBH_4$ under nitrogen, then passed through a column containing KOH, $CaSO_4$ and silica gel. *tert*-Butylcatechol (0.02% w/w) was added, and the isoprene was stored in this way until redistd before use. The inhibitor (*tert*-butylcatechol) in isoprene can be removed by several washings with dil NaOH and water. The isoprene is then dried over CaH_2, distd under nitrogen at atmospheric pressure, and the fraction distilling at 32° is collected. Stored under nitrogen at -15°.

Isopropanol *[67-63-0]* **M 60.1, b 82.5°, d 0.783, $n^{25.8}$ 1.3739, pK^{25} 17.1.** Isopropyl alcohol is prepared commercially by dissolution of propene in H_2SO_4, followed by hydrolysis of the sulfate ester. Major impurities are water, lower alcohols and oxidation products such as aldehydes and ketones. Purification of isopropanol follows substantially the same procedure as for *n*-propyl alcohol.
Isopropanol forms a constant-boiling mixture, **b** 80.3°, with water. Most of the water can be removed from this 91% isopropanol by refluxing with CaO (200g/L) for several hours, then distilling. The distillate can be dried further with CaH_2, magnesium ribbon, BaO, $CaSO_4$, calcium, anhydrous $CuSO_4$ or Linde type 5A molecular sieves. Distn from sulfanilic acid removes ammonia and other basic impurities. Peroxides [indicated by liberation of iodine from weakly acid (HCl) solns of 2% KI] can be removed by refluxing with solid stannous chloride or with $NaBH_4$ then fractionally distilling. To obtain isopropanol containing only 0.002M of water, sodium (8g/L) has been dissolved in material dried by distn from $CaSO_4$, 35mL of isopropyl benzoate has been

added and, after refluxing for 3h, the alcohol has been distd through a 50-cm Vigreux column. [Hine and Tanabe *J Am Chem Soc* **80** 3002 *1958*.] Other purification steps for isopropanol include refluxing with solid aluminium isopropoxide, refluxing with $NaBH_4$ for 24h, and the removal of acetone by treatment with, and distn from 2,4-dinitrophenylhydrazine. Peroxides re-form in isopropanol if it is stood for several days.

Isopropenylcyclobutane *[3019-22-5]* **M 98.1, b 98.7°, d 0.7743, n 1.438.** Purified by preparative chromatography (silicon oil column), or fractionally distd. Dried with molecular sieves.

Isopropyl acetate *[108-22-5]* **M 102.1, b 88.4°, d 0.873, n 1.3773.** Washed with 50% aq K_2CO_3 (to remove acid), then with saturated aq $CaCl_2$ (to remove any alcohol). Dried with $CaCl_2$ and fractionally distd.

Isopropyl bromide (2-bromopropane) *[75-26-3]* **M 123.0, b 0°/69.2mm, 59.4°/760mm, d 1.31, n^{15} 1.42847, n 1.4251.** Washed with 95% H_2SO_4 (conc acid partially oxidised it) until a fresh portion of acid did not become coloured after several hours, then with water, aq $NaHSO_3$, aq 10% Na_2CO_3 and again with water. (The H_2SO_4 can be replaced by conc HCl.) Prior to this treatment, isopropyl bromide has been purified by bubbling a stream of oxygen containing 5% ozone through it for 1h, followed by shaking with 3% hydrogen peroxide soln, neutralising with aq Na_2CO_3, washing with distilled water and drying. Alternatively, it has been treated with elemental bromine and stored for 4 weeks, then extracted with aq $NaHSO_3$ and dried with $MgSO_4$. After the acid treatment, isopropyl bromide can be dried with Na_2SO_4, $MgSO_4$ or CaH_2, and fractionally distd.

***N*-Isopropylcarbazole** *[1484-09-9]* **M 209.3, m 120°.** Crystd from isopropanol. Sublimed under vacuum. Zone refined. The *picrate* has **m** 143° after recrystn from EtOH.

Isopropyl chloride (2-chloropropane) *[75-29-6]* **M 78.5, b 34.8°, d 0.864, n 1.3779, n^{25} 1.3754.** Purified with 95% H_2SO_4 as described for *isopropyl bromide*, then dried with $MgSO_4$, P_2O_5 or CaH_2, and fractionally distd from Na_2CO_3 or CaH_2. Alternatively, a stream of oxygen containing *ca* 5% ozone has been passed through the chloride for about three times as long as was necessary to obtain the first coloration of starch iodide paper by the exit gas, and the liquid was then washed with $NaHCO_3$ soln to hydrolyse ozonides and remove organic acids before drying and distilling.

Isopropyl ether (diisopropyl ether) *[108-20-3]* **M 102.2, b 68.3°, d 0.719, n 1.3688, n^{25} 1.36618.** Common impurities are water and peroxides [detected by the liberation of iodine from weakly acid (HCl) solns of 2% KI]. Peroxides can be removed by shaking with aqueous Na_2SO_3 or with acidified ferrous sulfate (0.6g $FeSO_4$ and 6mL conc H_2SO_4 in 110mL of water, using 5-10g of soln per L of ether), or aqueous $NaBH_4$ soln. The ether is then washed with water, dried with $CaCl_2$ and distd. Alternatively, refluxing with $LiAlH_4$ or CaH_2, or drying with $CaSO_4$, then passage through an activated alumina column, can be used to remove water and peroxides. Other dehydrating agents used with isopropyl ether include P_2O_5, sodium amalgam and sodium wire. (The ether is often stored in brown bottles, or in the dark, with sodium wire.) Bonner and Goishi (*J Am Chem Soc* **83** 85 *1961*) treated isopropyl ether with dil sodium dichromate/sulfuric acid soln, followed by repeated shaking with a 1:1 mixture of 6M NaOH and saturated $KMnO_4$. The ether was washed several times with water, dilute aqueous HCl, and water, with a final washing with, and storage over, ferrous ammonium sulfate acidified with H_2SO_4. Blaustein and Gryder (*J Am Chem Soc* **79** 540 *1957*), after washing with alkaline $KMnO_4$, then water, treated the ether with ceric nitrate in nitric acid, and again washed with water. Hydroquinone was added before drying with $CaCl_2$ and $MgSO_4$, and refluxing with sodium amalgam (108g Hg/100g Na) for 2h under nitrogen. The distillate (nitrogen atmosphere) was made 2 x 10^{-5}M in hydroquinone to inhibit peroxide formation (which was negligible if the ether was stored in the dark). Catechol (pyrocatechol) and resorcinol are alternative inhibitors.

4,4'-Isopropylidenediphenol *[80-05-7]* **M 228.3, m 158°, pK_{Est} ~10.3.** Crystd from acetic acid/water (1:1).

Isopropyl iodide (2-iodopropane) *[75-30-9]* **M 170.0, b 88.9°, d 1.70, n 1.4987.** Treated with bromine, followed by extraction of free halogen with aqueous $Na_2S_2O_3$ or $NaHSO_3$, washing with water, drying ($MgSO_4$ or $CaCl_2$) and distn. (The treatment with bromine is optional.) Other purification methods include

passage through activated alumina, or shaking with copper powder or mercury to remove iodine, drying with P_2O_5 and distn. Washing with conc H_2SO_4 or conc HCl (to remove any alcohol), water, aqueous Na_2SO_3, water and aqueous Na_2CO_3 has also been used. Treatment with silica gel causes some liberation of iodine. Distillations should be carried out at slightly reduced pressure. Purified isopropyl iodide is stored in the dark in the presence of a little mercury.

Isopropyl methyl ether *[598-53-8]* **M 74.1, b 32.5°/777mm, d^{15} 0.724, n 1.3576.** Purified by drying with $CaSO_4$, passage through a column of alumina (to remove peroxides) and fractional distn.

Isopropyl *p*-nitrobenzoate *[13756-40-6]* **M 209.2, m 105-106°.** Dissolved in diethyl ether, washed with aqueous alkali, then water and dried. Evapn of the ether and recrystn from EtOH gave pure material.

***p*-Isopropyl toluene (*p*-cymene)** *[99-87-6]* **M 134.2, b 176.9°/744mm, d 0.8569, n 1.4902.** See entry on p. 183.

Isoquinoline *[119-65-3]* **M 129.2, m 24°, b 120°/18mm, d 1.0986, n 1.6148, pK^{25} 5.40.** Dried with Linde type 5A molecular sieves or Na_2SO_4 and fractionally distd at reduced pressure. Alternatively, it was refluxed with, and distd from, BaO. Also purified by fractional crystn from the melt and distd from zinc dust. Converted to its *phosphate* (**m** 135°) or *picrate* (**m** 223°), which were purified by crystn and the free base recovered and distd. [Packer, Vaughn and Wong *J Am Chem Soc* **80** 905 *1958*.] The procedure for purifying *via* the picrate comprises the addition of quinoline to picric acid dissolved in the minimum volume of 95% EtOH to yield yellow crystals which are washed with EtOH and air dried before recrystn from acetonitrile. The crystals are dissolved in dimethyl sulfoxide (previously dried over 4A molecular sieves) and passed through a basic alumina column, on which picric acid is adsorbed. The free base in the effluent is extracted with *n*-pentane and distd under vacuum. Traces of solvent are removed by vapour phase chromatography. [Mooman and Anton *J Phys Chem* **80** 2243 *1976*.]

Isovaleric acid *[502-74-2]* **M 102.1, b 176.5°/762mm, d 0.927, n^{15} 1.4064, n 1.40331, pK^{25} 4.77.** Dried with Na_2SO_4, then fractionally distd.

L-Isovaline (2-amino-2-methylbutyric acid) *[595-40-4]* **M 117.2, m *ca* 300° (sublimes in vac), $[\alpha]_D^{25}$ +10° (5M HCl), $pK_{Est(1)}$~2.4, $pK_{Est(2)}$~9.7.** Crystd from aqueous acetone.

Isovanillin (3-hydroxy-4-methoxybenzaldehyde) *[621-59-0]* **M 152.2, m 117°, b 175°/14mm, pK^{25} 8.89.** Cryst from H_2O or *C_6H_6. The *oxime* has **m** 147°.

Isoviolanthrone *[128-64-3]* **M 456.5, m 510-511°(uncorrected).** Dissolved in 98% H_2SO_4 and ppted by adding water to reduce the acid concentration to about 90%. Sublimes *in vacuo*. [Parkyns and Ubblehode *J Chem Soc* 4188 *1960*.]

Itaconic acid (2-propen-1,2-dicarboxylic acid) *[97-65-4]* **M 130.1, m 165-166°, pK_1^{25} 3.63, pK_2^{25} 5.00.** Crystd from EtOH, EtOH/water or EtOH/*benzene.

Itaconic anhydride (2-propen-1,2-dicarboxylic anhydride) *[2170-03-8]* **M 112.1, m 66-68°, 67-68°, 68°, b 139-140°/30mm.** Crystd from $CHCl_3$/pet ether. Can be distd under reduced press. Distn at atm press, or prolonged distn causes rearrangement to citraconic anhydride (2-methylmaleic anhydride). If the material (as seen in the IR spectrum) contains much free acid then heat with acetyl chloride or $SOCl_2$, evaporate and distil at as high a vacuum as possible. The crude anhydride deposits crystals of itaconic acid on standing probably due to hydrolysis by H_2O — store in sealed ampoules under dry N_2. [*Org Synth* Coll Vol II 369 *1943*; IR: Nagai *Bull Chem Soc Jpn* **37** 369 *1964*; Kelly and Segura *J Am Chem Soc* **56** 2497 *1934*.]

Janus Green B **(3-dimethylamino-7-[4-dimethylaminoazo]-5-phenylphenazonium chloride)** *[2869-83-2]* **M 511.1, m >200°.** Dissolves in H_2O to give a bluish violet soln which

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