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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), $[\alpha]_D^{25} +10^\circ$ (5M HCl), $pK_{Est(1)} \sim 2.4$, $pK_{Est(2)} \sim 9.7$. 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

becomes colourless when made 10M in NaOH. Dissolve in EtOH to give a blue-violet colour, filter from insoluble material then add dry Et₂O whereby the dye separates out leaving a small amount of blue colour in soln. Filter off the solid and dry in vacuum. Store in a dark bottle.

Janus Red B {3-[(2-hydroxy-1-naphthol)azo-2-methylphenylazo]N,N,N-trimethylbenzenaminium chloride} [2636-31-9] M 460.0. Crystd from EtOH/H₂O (1:1 v/v) and dry in vacuum. Store in a dark bottle.

Jervine (3β,23β-17,23-epoxy-2-hydroxyvertraman-11-one, a steroidal alkaloid) [469-59-0] M 425.6, m 243-245°, [α]_D²⁰ -150° (in EtOH), pK_{Est}~9.4 Crystd from MeOH/H₂O. The hydrochloride has m 300-302°. [Kutney et al. *Can J Chem* 53 1796 1975.]

Julolidine (2,3,6,7-tetrahydro-1H,5H-benzo[*ij*]quinolizidine) [479-59-4] M 173.3, m 34-36°, 40°, b 105-110°/1mm, 155-156°/17mm, 280° (dec), pK_{Est} ~7.0. Purified by dissolving in dilute HCl, steam is bubbled through the soln and the residual acidic soln is basified with 10N NaOH, extracted with Et₂O, washed with H₂O, dried (NaOH pellets), filtered, evaporated and distd *in vacuo*. The distillate crystallises on standing (m 39-40°). On standing in contact with air for several days it develops a red colour. The colour can be removed by distilling or dissolving in 2-3 parts of hexane, adding charcoal, filtering and cooling in Me₂CO-Dry-ice when julolidine crystallises (85-90% yield). The hydrochloride [83646-41-7] has m 218° (239-242°), the picrate has m 165° and the methiodide crystallises from MeOH, m 186° [Glass and Weisberger *Org Synth Coll Vol III* 304 1955.] **Highly TOXIC.**

Kainic acid monohydrate (2S,3S,4S-2-carboxy-4-isoprenyl-3-pyrrolidine-acetic acid) [487-79-6] M 231.4, m 235-245° (dec), 251° (dec), [α]_D²⁰ -14.6° (c 1.46, H₂O), pK₁ 2.09, pK₂ 4.58, pK₃ 10.21. Purified by adsorbing on to a strongly acidic ion exchange resin (Merck), elution of the diacid with aqueous M NaOH, the eluate is evaporated, H₂O is added, and filtered through a weakly acidic ion exchange resin (Merck). The filtrate is then evaporated and recrystd from EtOH. Its solubility is 0.1g in 1mL of 0.5N HCl. (±)-α-Kainic acid recryst from H₂O, m 230-260°. UV (MeOH): λ_{max} 219 (log ε 3.9); ¹H NMR (CCl₄, 100MHz, Me₄Si standard) δ: 1.64 (s 1H), 1.70 (s 3H), 3.24 (d J 7.5, 2H), 3.3-4.2 (1H), 3.70 (s 3H), 3.83 (s 3H), 4.35 (dd J 7.5, J 14.5, 1H), 5.21 (t J 7.5, 1H), 7.26 (t J 7.5, 1H). [Oppolzer and Andres *Helv Chim Acta* 62 2282 1979.]

Kerosene [8008-20-6] (mixture of hydrocarbons) b ~175-325°, d 0.75-0.82, n 1.443. Stirred with conc H₂SO₄ until a fresh portion of acid remains colourless, then washed with water, dried with solid KOH and distd in a Claisen flask. For more complete drying, the kerosene can be refluxed with, and distd from Na.

Ketanserine [3(4-*p*-fluorobenzoylpiperidinyl)-*N*-ethylquinazolin-2,4-dione] [74050-98-9] M 395.4, m 227-235°, pK 7.5. Solubility is 0.001% in H₂O, 0.038% in EtOH and 2.34 in Me₂NCHO. It has been purified by recrystn from 4-methyl-3-pentanone [Peeters et al. *Cryst Structure Commun* 11 375 1982; Kacprowicz et al. *J Chromatogr* 272 417 1983; Davies et al. *J Chromatogr* 275 232 1983].

Ketene [463-51-4] M 42.0, b 127-130°, d 1.093, n 1.441. Prepared by pyrolysis of acetic anhydride. Purified by passage through a trap at -75° and collected in a liquid-nitrogen-cooled trap. Ethylene was removed by evacuating the ethylene in an isopentane-liquid-nitrogen slush pack at -160°. Stored at room temperature in a suitable container in the dark. See diketene on p. 209.

2-Keto-L-gulonic acid [526-98-7] M 194.1, m 171°. Crystd from water and washed with acetone.

Ketone moschus (4-*tert*-butyl-2,6-dimethyl-3,5-dinitroacetophenone) [81-14-1] M 234.1, m 134-137°, 137-138°. Purified by recryst from MeOH. [Fuson et al. *J Org Chem* 12 587 1947.]

Khellin (4,9-dimethoxy-7-methyl-5-oxofuro[3,2-*g*]-1,2-chromene) [82-02-0] M 260.3, m 154-155°, b 180-200°/0.05mm. Crystd from MeOH or diethyl ether.

Kojic acid [(2-hydroxy-5-hydroxymethyl)-4H-pyran-4-one] [501-30-4] M 142.1, m 154-155°, pK_1^{25} -1.38, pK_2^{25} 7.66. Crystd from MeOH (charcoal) by adding Et₂O. Sublimed at 0.1 torr.

Kynurenic acid (4-hydroxyquinoline-2-carboxylic acid) [492-27-3] M 189.1, m 282-283°, $pK_{Est(1)} \sim 2$, $pK_{Est(2)} \sim 10$. Crystd from absolute EtOH.

L-Kynurenine [343-65-7] M 208.2, m 190°(dec), 210°(dec), $[\alpha]_D^{20}$ -30° (c 0.4, H₂O), $pK_{Est(1)} \sim 2.3$, $pK_{Est(2)} \sim 3.5$, $pK_{Est(3)} \sim 9.2$. Crystd from H₂O or aq AcOH. *Picrate* has m 188.5-189°(dec) after crystn from H₂O.

L-Kynurenine sulfate [16055-80-4] M 306.3, m 194°, monohydrate m 178°, $[\alpha]_D^{25}$ +9.6° (H₂O). Crystd from water by addition of EtOH.

L(+)-Lactic acid [79-33-4] M 90.1, m 52.8°, b 105°/0.1mm, $[\alpha]_D^{20}$ +3.82° (H₂O), pK^{31} 3.83. Purified by fractional distn at 0.1mm pressure, followed by fractional crystn from diethyl ether/isopropyl ether (1:1, dried with sodium). [Borsook, Huffman and Liu *J Biol Chem* 102 449 1933.] The solvent mixture, *benzene/diethyl ether (1:1) containing 5% pet ether (b 60-80°) has also been used.

Lactobionic acid [96-82-2] M 358.3, m 128-130°, $[\alpha]_{546}^{20}$ +28° (c 3, after 24h in H₂O), $pK_{Est} \sim 3.6$. Crystd from water by addition of EtOH.

α -Lactose (H₂O) [63-42-3] M 360.3, m 220°(dec), $[\alpha]_D^{20}$ +52.3° (c 4.2, H₂O), pK 12.2 (OH). Crystd from water below 93.5°.

Lactulose [4618-18-2] M 342.2, m 167-169°(dec), $[\alpha]_{546}^{20}$ -57° (c 1, H₂O). Crystd from MeOH.

Lanatoside A [17575-20-1] M 969.1, m 245-248°, $[\alpha]_D^{20}$ +32° (EtOH). Crystd from MeOH.

Lanatoside B [17575-21-2] M 985.1, m 233°(dec), $[\alpha]_D^{20}$ +35° (MeOH). Crystd from MeOH.

Lanatoside C [17575-22-3] M 297.1, m 246-248°, $[\alpha]_D^{20}$ +34° (EtOH). Crystd from MeOH.

Lanosterol [79-63-0] M 426.7, m 138-140°, $[\alpha]_D^{20}$ +62.0° (c 1, CHCl₃). Recrystd from anhydrous MeOH. Dried *in vacuo* over P₂O₅ for 3h at 90°. Purity checked by proton magnetic resonance.

Lanthanide shift reagents A variety of these reagents are available commercially and they are generally quite stable and should not deteriorate on long storage in a dry state and in the absence of light. [See G.R.Sullivan in *Top Stereochem* (Eliel and Allinger Eds) J Wiley & Sons Vol 10 287 1978; T.C.Morrill Ed. *Lanthanide Shift Reagents* Deerfield Beach Florida 1986, ISBN 0895731193.]

Lapachol [84-79-7] M 226.3, m 140°. Crystd from EtOH or diethyl ether.

dl- and l-Laudanosine [(±) 1699-51-0; (-) 2688-77-9] M 357.4, m 114-115°. Crystd from EtOH. The (-)-isomer has m 83-85° and $[\alpha]_D^{20}$ -85° (c 0.5, EtOH).

Lauraldehyde (1-dodecanal) [112-54-9] M 184.3, b 99.5-100°/3.5mm, $n^{24.7}$ 1.4328. Converted to the addition compound by shaking with saturated aqueous NaHSO₃ for 1h. The ppte was filtered off, washed with ice cold water, EtOH and ether, then decomposed with aqueous Na₂CO₃. The aldehyde was extracted into diethyl ether which, after drying and evap, gave an oil which was fractionally distd under vacuum.

Lauric acid (1-dodecanoic acid) [143-07-7] M 200.3, m 44.1°, b 141-142°/0.6-0.7mm, 225°/100mm, pK^{20} 5.3. Vacuum distd. Crystd from absolute EtOH, or from acetone at -25°.

Alternatively, purified *via* its *methyl ester* (b 140.0°/15mm), as described for *capric acid*. Also purified by zone melting.

Lauryl peroxide (dodecyl peroxide) [105-74-8] M 398.6, m 53-54°. Crystd from *n*-hexane or *benzene and stored below 0°. Potentially **EXPLOSIVE**.

L-Leucine [61-90-5] M 131.2, m 293-295°(dec), $[\alpha]_D^{25} +15.6^\circ$ (5M HCl), $pK_1^{25} 2.33$, $pK_2^{25} 9.74$. Likely impurities are isoleucine, valine, and methionine. Crystd from water by adding 4 volumes of EtOH.

Leucomalachite Green [129-73-7] M 330.5, m 92-93°, $pK^{25} 6.90$ (several pK 's). Crystd from 95% EtOH (10mL/g), then from *benzene/EtOH, and finally from pet ether.

Lithocholic acid [434-13-9] M 376.6, m 184-186°, $[\alpha]_D^{23} +35^\circ$ (c 1, EtOH), $pK_{Est} \sim 4.8$. Crystd from EtOH or acetic acid.

Lumichrome [1086-80-2] M 242.2, m >290°, $pK_{Est(1)} \sim -0.1$ (basic), $pK_{Est(2)} \sim 9.9$ (acidic), Recrystd twice from glacial AcOH and dried at 100° in a vacuum.

Luminol (5-aminophthalazin-1,4-dione) [521-31-3] M 177.2, m 329-332°, $pK_1 3.37$, $pK_2 6.35$. Dissolved in KOH soln, treated with Norit (charcoal), filtered and ppted with conc HCl. [Hardy, Sietz and Hercules *Talanta* 24 297 1977.] Stored in the dark in an inert atmosphere, because its structure changes during its luminescence. It has been recrystd from 0.1M KOH [Merenyi et al. *J Am Chem Soc* 108 77716 1986].

dl-Lupinane [10248-30-3] M 169.3, m 98-99°. Crystd from acetone.

Lupulon [468-28-0] M 414.6, m 92-94°. Crystd from 90% MeOH.

Lutein (α -carotene-3,3'-diol, xanthophyll) [127-40-2] M 568.9, m 196°, $\epsilon_{1cm}^{1\%} 1750$ (423nm), 2560 (446nm), 2340 (477.5nm) in EtOH; λ_{max} in CS₂ 446, 479 and 511nm. Crystd from MeOH (copper-coloured prisms) or from diethyl ether by adding MeOH. Also purified by chromatography on columns of magnesia or calcium hydroxide, and crystd from CS₂/EtOH. May be purified *via* the dipalmitate ester. Stored in the dark, in an inert atmosphere.

Lutidine (mixture). For the preparation of pure 2,3-, 2,4- and 2,5-lutidine from commercial "2,4- and 2,5-lutidine" see Coulson et al. *J Chem Soc* 1934 1959, and Kyte, Jeffery and Vogel *J Chem Soc* 4454 1960.

2,3-Lutidine [583-61-9] M 107.2, f -14.8°, b 160.6°, d 0.9464, n 1.50857, $pK^{25} 6.57$. Steam distd from a soln containing about 1.2 equivalents of 20% H₂SO₄, until *ca* 10% of the base has been carried over with the non-basic impurities. The acid soln was then made alkaline, and the base was separated, dried over NaOH or BaO, and fractionally distd. The distd lutidine was converted to its urea complex by stirring 100g with 40g of urea in 75mL of H₂O, cooling to 5°, filtering at the pump, and washing with 75mL of H₂O. The complex, dissolved in 300mL of H₂O was steam distd until the distillate gave no turbidity with a little solid NaOH. The distillate was then treated with excess solid NaOH, and the upper layer was removed: the aqueous layer was then extracted with diethyl ether. The upper layer and the ether extract were combined, dried (K₂CO₃), and distd through a short column. Final purification was by fractional crystn using partial freezing. [Kyte, Jeffery and Vogel *J Chem Soc* 4454 1960].

2,4-Lutidine [108-47-4] M 107.2, b 157.8°, d 0.9305, n 1.50087, $n^{25} 1.4985$, $pK^{25} 6.77$. Dried with Linde type 5A molecular sieves, BaO or sodium, and fractionally distd. The distillate (200g) was heated with *benzene (500mL) and conc HCl (150mL) in a Dean and Stark apparatus on a water bath until water no longer separated, and the temperature just below the liquid reached 80°. When cold, the supernatant *benzene was decanted and the 2,4-lutidine hydrochloride, after washing with a little *benzene, was dissolved in water (350mL). After removing any *benzene by steam distn, an aqueous soln of NaOH (80g) was added, and the free

lutidine was steam distd. It was isolated by saturating the distillate with solid NaOH, and distd through a short column. The pptn cycle was repeated, then the final distillate was partly frozen in an apparatus at -67.8 - 68.5° (cooled by acetone/ CO_2). The crystals were then melted and distd. [Kyte, Jeffery and Vogel *J Chem Soc* 4454 1960.] Alternative purifications are *via* the picrate [Clarke and Rothwell *J Chem Soc* 1885 1960], or the hydrobromide [Warnhoff *J Org Chem* 27 4587 1962]. The latter is ppted from a soln of lutidine in *benzene by passing dry HBr gas: the salt is recrystd from CHCl_3 /methyl ethyl ketone, then decomposed with NaOH, and the free base is extracted into diethyl ether, dried, evaporated and the residue distd.

2,5-Lutidine [589-93-5] M 107.2, m -15.3° , b $156.7^{\circ}/759\text{mm}$, d 0.927, n^{25} 1.4982, pK^{25} 6.40. Steam distd from a soln containing 1-2 equivalents of 20% H_2SO_4 until about 10% of the base had been carried over with the non-basic impurities, then the acid soln was made alkaline, and the base separated, dried with NaOH and fractionally distd twice. Dried with Na and fractionally distd through a Todd column packed with glass helices (see p. 174).

2,6-Lutidine [108-48-5] M 107.2, m -59° , b 144.0° , d 0.92257, n 1.49779, pK^{25} 6.72. Likely contaminants include 3- and 4-picoline (similar boiling points). However, they are removed by using BF_3 , with which they react preferentially, by adding 4mL of BF_3 to 100mL of dry fractionally distd 2,6-lutidine and redistilling. Distn of commercial material from AlCl_3 (14g per 100mL) can also be used to remove picolines (and water). Alternatively, lutidine (100mL) can be refluxed with ethyl benzenesulfonate (20g) or ethyl *p*-toluenesulfonate (20g) for 1h, then the upper layer is cooled, separated and distd. The distillate is refluxed with BaO or CaH_2 , then fractionally distd, through a glass helices-packed column.

2,6-Lutidine can be dried with KOH or sodium, or by refluxing with (and distilling from) BaO, prior to distn. For purification *via* its picrate, 2,6-lutidine, dissolved in abs EtOH, is treated with an excess of warm ethanolic picric acid. The ppt is filtered off, recrystd from acetone (to give m 163 - 164.5°), and partitioned between ammonia and CHCl_3 /diethyl ether. The organic soln, after washing with dilute aqueous KOH, is dried with Na_2SO_4 and fractionally distd. [Warnhoff *J Org Chem* 27 4587 1962.] Alternatively, 2,6-lutidine can be purified *via* its urea complex, as described under 2,3-lutidine. Other purification procedures include azeotropic distn with phenol [Coulson et al. *J Appl Chem (London)* 2 71 1952], fractional crystn by partial freezing, and vapour-phase chromatography using a 180-cm column of polyethylene glycol-400 (Shell, 5%) on Embacel (May and Baker) at 100° , with argon as carrier gas [Bamford and Block *J Chem Soc* 4989 1961].

3,5-Lutidine [591-22-0] M 107.2, f -6.3° , b $172.0^{\circ}/767\text{mm}$, d 0.9419, n 1.50613, n^{25} 1.5035, pK^{25} 6.15. Dried with sodium and fractionally distd through a Todd column packed with glass helices (see p. 174). Dissolved (100mL) in dil HCl (1:4) and steam distd until 1L of distillate was collected. Excess conc NaOH was added to the residue which was again steam distd. The base was extracted from the distillate, using diethyl ether. The extract was dried with K_2CO_3 , and distd. It was then fractionally crystd by partial freezing.

Lycopene [502-65-8] M 536.9, m 172 - 173° , $\epsilon_{1\text{cm}}^{1\%}$ 2250 (446nm), 3450 (472nm), 3150 (505nm) in pet ether. Crystd from CS_2 /MeOH, diethyl ether/pet ether, or acetone/pet ether, and purified by column chromatography on deactivated alumina, CaCO_3 , calcium hydroxide or magnesia. Stored in the dark, in an inert atmosphere.

Lycorine [476-28-8] M 552.9, m 275 - $280^{\circ}(\text{dec})$ $[\alpha]_{\text{D}}^{20}$ -130° (c 0.16, EtOH). Crystd from EtOH.

Lycoxanthin (Ψ, Ψ -carotene-16-ol) [19891-74-8] M 268.3, m 173 - 174° , $\epsilon_{1\text{cm}}^{1\%}$ 3360 (472.5nm), also λ_{max} 444 and 503nm in pet ether. Crystd from diethyl ether/light petroleum, *benzene/pet ether or CS_2 . Purified by chromatography on columns of CaCO_3 , $\text{Ca}(\text{OH})_2$ or deactivated alumina, washing with *benzene and eluting with 3:1 *benzene/MeOH. Stored in the dark, in an inert atmosphere, at -20° .

Lysergic acid [82-58-6] M 268.3, m $240^{\circ}(\text{dec})$, $[\alpha]_{\text{D}}^{20}$ $+40^{\circ}$ (pyridine), pK_1^{25} 3.32, pK_2^{25} 7.82. Crystd from water.

L-Lysine [56-87-1] M 146.2, m >210°(dec), pK₁ 2.18, pK₂ 8.95, pK₃ 10.53. Crystd from aqueous EtOH.

L-Lysine dihydrochloride [657-26-1] M 219.1, m 193°, [α]_D²⁵ +25.9° (5M HCl). Crystd from MeOH, in the presence of excess HCl, by adding diethyl ether.

L-Lysine monohydrochloride [657-27-2] M 182.7, [α] as above. Likely impurities are arginine, D-lysine, 2,6-diaminoheptanedioic acid and glutamic acid. Crystd from water at pH 4-6 by adding 4 volumes of EtOH. Above 60% relative humidity it forms a dihydrate.

β-D-Lyxose [1114-34-7] M 150.1, m 118-119°, [α]_D²⁰ -14° (c 4, H₂O). Crystd from EtOH or aqueous 80% EtOH. Dried under vacuum at 60°, and stored in a vacuum desiccator over P₂O₅ or CaSO₄.

Malachite Green (carbinol) [510-13-4] M 346.4, m 112-114°, CI 42000, pK²⁴ 6.84. The oxalate was recrystd from hot water and dried in air. The carbinol was pptd from the oxalate (1g) in distd water (100mL) by adding M NaOH (10mL). The ppte was filtered off, recrystd from 95% EtOH containing a little dissolved KOH, then washed with ether, and crystd from pet ether. Dried in a vacuum at 40°. An acid soln (2 x 10⁻⁵M in 6 x 10⁻⁵M H₂SO₄) rapidly reverted to the dye. [Swain and Hedberg *J Am Chem Soc* 72 3373 1950.]

Z-Maleamic acid (cis-maleic acid monoamide) [557-24-4] M 115.1, m 158-161°, 172-173°(dec), pK_{Est} ~2.65. Crystd from EtOH. IRRITANT.

Maleic acid [110-16-7] M 116.1, m 143.5°, pK₁²⁵ 1.91, pK₂²⁵ 6.33. Crystd from acetone/pet ether (b 60-80°) or hot water. Dried at 100°.

Maleic anhydride [108-31-6] M 98.1, m 54°, b 94-96°/20mm, 199°/760mm. Crystd from *benzene, CHCl₃, CH₂Cl₂ or CCl₄. Sublimed under reduced pressure. [Skell et al. *J Am Chem Soc* 108 6300 1986.]

Maleic hydrazide [123-33-1] M 112.1, m 144°(dec), pK₁²⁵ 5.67, pK₂²⁵ 13.3. Crystd from water.

Maleimide (pyrrol-2,5-dione) [541-59-3] M 97.1, m 91-93°, 92.6-93°, d_D^{105.5} 1.2493, n_D^{110.7} 1.49256. Purified by sublimation in a vacuum. The UV has λ_{max} at 216 and 280nm in EtOH. [de Wolf and van de Straete *Bull Soc Chim Belg* 44 288 1935; UV: Rondestvedt et al. *J Am Chem Soc* 78 6115 1956; IR: Chiorboli and Mirone *Ann Chim (Rome)* 42 681 1952.]

Maleuric acid [105-61-3] M 158.1, m 167-168°(dec). Crystd from hot water.

dl-Malic acid [617-48-1 and 6915-15-7] M 134.1, m 128-129°. Crystd from acetone, then from acetone/CCl₄, or from ethyl acetate by adding pet ether (b 60-70°). Dried at 35° under 1mm pressure to avoid formation of the anhydride.

L-Malic acid [97-67-6] M 134.1, m 104.5-106°, [α]_D²⁰ -2.3° (c 8.5, H₂O), pK₁²⁵ 3.46, pK₂²⁵ 5.10. Crystd (charcoal) from ethyl acetate/pet ether (b 55-56°), keeping the temperature below 65°. Or, dissolved by refluxing in fifteen parts of anhydrous diethyl ether, decanted, concentrated to one-third volume and crystd at 0°, repeatedly to constant melting point.

Malonamide [108-13-4] M 102.1, m 170°. Crystd from water.

Malonic acid [141-82-2] M 104.1, m 136°, pK₁²⁵ 2.58, pK₂²⁵ 5.69. Crystd from *benzene/diethyl ether (1:1) containing 5% of pet ether (b 60-80°), washed with diethyl ether, then recrystd from H₂O or acetone. Dried under vac over conc H₂SO₄.