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Pyronin Y [3,6-bis(dimethylamino)xanthylium chloride] *[92-32-0]* **M 302.8, m 250-260°, CI 45005, λmax 522nm, pK_{Est} ~7.6.** Commercial material contained a large quantity of zinc. Purified by dissolving 1g in 50mL of hot water containing 5g NaEDTA. Cooled to 0°, filtered, evapd to dryness and the residue extracted with EtOH. The soln was evaporated to 5-10mL, filtered, and the dye pptd by addition of excess dry diethyl ether. It was centrifuged and the crystals were washed with dry ether. The procedure was repeated, then the product was dissolved in $CHCl_3$, filtered and evapd. The dye was stored in a vacuum.

Pyrrole *[109-97-7]* **M 67.1, m 23.4°, b 66°/80mm, 129-130°/atm, d 0.966, n 1.5097, pK_1^{25} -4.4 (Protonation on carbon), pK_2^{25} 17.51 (aq KOH, H. scale).** Dried with NaOH, CaH_2 or $CaSO_4$. Fractionally distd under reduced pressure from CaH_2. Stored under nitrogen, turns brown in air. Redistd immediately before use.

Pyrrolidine *[123-75-1]* **M 71.1, b 87.5-88.5°, d 0.860, n 1.443, pK^{25} 11.31.** Dried with BaO or sodium, then fractionally distd, under N_2, through a Todd column packed with glass helices (see p. 174).

Pyrrolidine-1-carbodithioic acid ammonium salt *[5108-96-3]* **M 164.3, m 128-130°, pK^{25} 3.25 (free acid).** Purified by recryst twice by dissolving in MeOH and adding Et_2O. Also by recrystn from EtOH. [Synth and Polarography: Kitagawa and Taku *Bull Chem Soc Jpn* **64** 2151 *1973*; Malissa and Schöffmann *Mikrochim Acta* 187 *1955*.]

Pyruvic acid *[127-17-3]* **M 88.1, m 13°, b 65°/10mm, pK^{25} 2.39 (2.60).** Distd twice, then fractionally crystd by partial freezing.

p-Quaterphenyl *[135-70-6]* M 306.4, m 312-314°.

Recrystd from dimethyl sulfoxide at *ca* 50°.

Quercetin ($2H_2O$) (3,3',4',5,6-pentahydroxyflavone) *[6151-25-3 ($2H_2O$); 117-39-3 (anhydr)]* **M 338.3, m *ca* 315°(dec), (phenolic pKs 7—10).** Crystd from aq EtOH and dried at 100°.

Quercitrin (quercetin glycoside) *[522-12-3]* **M 302.2, m 168°, 176-178°.** Crystd from aq EtOH and dried at 135° to give the higher melting form.

Quinaldic (quinoline-2-carboxylic) acid *[93-10-7]* **M 173.2, m 156-157°, pK_1^{25} 1.45, pK_2^{25} 2.49 (2.97).** Crystd from *benzene.

Quinalizarin (1,2,5,8-tetrahydroxy-9,10-anthraquinone) *[81-61-8]* **M 272.2, m 275°, $pK_{Est(1)}$~7.1 (1-OH), $pK_{Est(2)}$~9.9 (8-OH), $pK_{Est(3)}$~11.1 (5-OH), $pK_{Est(4)}$~11.8 (2-OH).** Crystd from acetic acid or nitrobenzene. It can be sublimed *in vacuo*.

Quinazoline *[253-82-7]* **M 130.2, m 48.0-48.5°, b 120-121°/17-18mm, pK_1^{20} -4.51 (aq H_2SO_4, anhydrous dication) , pK_2^{20} 2.01 (anhydrous monocation), pK_3^{20} 4.3 (equilibrium with 3,4-hydrated species), pK_4^{20} 12.1 (hydrated anion).** Purified by passage through an activated alumina column in *benzene or pet ether (b 40-60°). Distd under reduced pressure, sublimed under vacuum and crystd from pet ether. [Armarego *J Appl Chem* **11** 70 *1961*.]

Quinhydrone *[106-34-3]* **M 218.2, m 168°.** Crystd from H_2O at 65°, then dried in a vac desiccator.

1*R*,3*R*,4*R*,5*R*-Quinic acid (1,3,4,5-tetrahydroxy-cyclohexane-carboxylic acid) *[77-95-2]* **M 192.3, m 172°(dec), $[\alpha]_{546}^{20}$ -51° (c 20, H_2O), pK^{25} 3.58.** Crystd from water.

Quinidine *[56-54-2]* **M 324.4, m 171°, $[\alpha]_{546}^{20}$ +301.1° ($CHCl_3$ contg 2.5% (v/v) EtOH), pK_1^{15} 4.13, pK_2^{15} 8.77.** Crystd from *benzene or dry $CHCl_3$/pet ether (b 40-60°), discarding the initial, oily crop of crystals. Dried under vacuum at 100° over P_2O_5.

Quinine *[130-95-0]* **M 324.4, m 177°(dec), $[\alpha]_{546}^{20}$ -160° (c 1, $CHCl_3$), pK_1^{20} 4.13 (quinoline N), pK_2^{20} 8.52 (piperidine N).** Crystd from abs EtOH.

Quinine bisulfate *[6183-68-2 ($7H_2O$); 549-56-4 (anhydr)]* **M 422.4, m 160° (anhydrous).** Crystd from 0.1M H_2SO_4, forms heptahydrate when crystd from water

Quinine sulfate ($2H_2O$) *[6119-70-6 (H_2O); 804-63-7 (anhydr)]* **M 783.0, m 205°.** Crystd from water, dried at 110°.

Quinizarin (1,4-dihydroxy-9,10-anthraquinone) *[81-64-1]* **M 240.2, m 200-202°, pK_1^{25} 9.90 (9.5), pK_2^{25} 11.18.** Crystd from glacial acetic acid.

Quinoline *[91-22-5]* **M 129.2, m -16°, b 111.5°, 236°/758mm, d 1.0937, n 1.625, pK^{25} 4.80 (4.93).** Dried with Na_2SO_4 and vac distd from zinc dust. Also dried by boiling with acetic anhydride, then fractionally distilling. Calvin and Wilmarth [*J Am Chem Soc* **78** 1301 *1956*] cooled redistd quinoline in ice and added enough HCl to form its hydrochloride. Diazotization removed aniline, the diazo compound being broken down by warming the soln to 60°. Non-basic impurities were removed by ether extraction. Quinoline was liberated by neutralising the hydrochloride with NaOH, then dried with KOH and fractionally distd at low pressure. Addition of cuprous acetate (7g/L of quinoline) and shaking under hydrogen for 12h at 100° removed impurities due to the nitrous acid treatment. Finally the hydrogen was pumped off and the quinoline was distd. Other purification procedures depend on conversion to the phosphate (**m** 159°, pptd from MeOH soln, filtered, washed with MeOH, then dried at 55°) or the picrate (**m** 201°) which, after crystn were reconverted to the amine. The method using the picrate [Packer, Vaughan and Wong *J Am Chem Soc* **80** 905 *1958*] is as follows: quinoline is added to picric acid dissolved in the minimum volume of 95% EtOH, giving yellow crystals which were washed with EtOH, air-dried and crystd from acetonitrile. These were dissolved in dimethyl sulfoxide (previously dried over 4A molecular sieves) and passed through basic alumina, on which the picric acid is adsorbed. The free base in the effluent is extracted with *n*-pentane and distd under vacuum. Traces of solvent can be removed by vapour-phase chromatography. [Moonaw and Anton *J Phys Chem* **80** 2243 *1976*.] The $ZnCl_2$ and dichromate complexes have also been used. [Cumper, Redford and Vogel *J Chem Soc* **1176** *1962*.]

2-Quinolinealdehyde *[5470-96-2]* **M 157.2, m 71°, pK_{Est} ~3.3.** Steam distd. Crystd from H_2O. Protected from light.

8-Quinolinecarboxylic acid *[86-59-9]* **M 173.2, m 186-187.5°, pK_1^{25} 1.82, pK_2^{25} 6.87.** Crystd from water.

Quinoline ethiodide (1-ethylquinolinium iodide) *[634-35-5]* **M 285.1, m 158-159°.** Crystd from aqueous EtOH.

Quinoxaline *[91-19-0]* **M 130.2, m 28° (anhydr), 37°(H_2O), b 108-110°/0.1mm, 140°/40mm, pK_1^{20}-5.52 (-5.8, dication), pK_2^{25} 2.08 (monocation).** Crystd from pet ether. Crystallises as the monohydrate on addition of water to a pet ether soln.

Quinoxaline-2,3-dithiol *[1199-03-7]* **M 194.1, m 345°(dec), pK_1 6.9, pK_2 9.9.** Purified by repeated dissolution in alkali and re-pptn by acetic acid.

***p*-Quinquephenyl (*p*-pentaphenyl)** *[61537-20-0]* **M 382.5, m 388.5°.** Recrystd from boiling dimethyl sulfoxide (**b** 189°, lowered to 110°). The solid obtained on cooling was filtered off and washed repeatedly with toluene, then with conc HCl. The final material was washed repeatedly with hot EtOH. It was also recrystd from pyridine, then sublimed *in vacuo*.

Quinuclidine (1-azabicyclo[2.2.2]octane) *[100-76-5]* **M 111.2, m 158°(sublimes), pK^{25} 10.95.** Crystd from diethyl ether.

D-Raffinose ($5H_2O$) *[17629-30-0 ($5H_2O$); 512-69-6 (anhydr)]* **M 594.5, m 80°, $[\alpha]_{546}^{20}$ +124° (c 10, H_2O).** Crystd from aqueous EtOH.

Rauwolscine hydrochloride *[6211-32-1]* **M 390.0, m 278-280°.** Crystd from water.

Reductic acid (1,2-dihydroxycyclopent-1,2-en-3-one) *[80-72-8]* **M 114.1, m 213°, pK^{20} 4.72.** Crystd from ethyl acetate.

Rescinnamine *[24815-24-5]* **M 634.7, m 238-239°(vac), $[\alpha]_D^{20}$ -97° (c 1, $CHCl_3$), $pK_{Est(1)}$~<0 (carbazole N), $pK_{Est(2)}$~7.0 (quinolizidine N).** Crystd from *benzene or MeOH.

Reserpic acid *[83-60-3]* **M 400.5, m 241-243°, $pK_{Est(1)}$~<0 (carbazole N), $pK_{Est(2)}$~4.0 (CO_2H), $pK_{Est(3)}$~7.4 (quinolizidine N).** Crystd from MeOH. The *hydrochloride $0.5H_2O$* has **m** 257-259°, $[\alpha]_D^{23}$ -81° (H_2O).

Reserpine *[50-55-5]* **M 608.7, m 262-263°, $[\alpha]_{546}^{20}$ -148° (c 1, $CHCl_3$), $pK_{Est(1)}$~<0 (carbazole N), pK_2 6.6 (7.4)(quinolizidine N).** Crystd from aq acetone.

Resorcinol *[108-46-3]* **M 110.1, m 111.2-111.6°, pK_1^{25} 9.23, pK_2^{25} 13.05.** Crystd from *benzene, toluene or *benzene/diethyl ether.

Retene *[483-65-8]* **M 234.3, m 99°.** Crystd from EtOH.

Retinal (vitamin A aldehyde), Retinoic acid (vitamin A acid), Retinol (vitamin A alcohol) see entries in Chapter 6.

Retinyl acetate *[127-47-9]* **M 328.5, m 57°.** Separated from retinol by column chromatography, then crystd from MeOH. See Kofler and Rubin [*Vitamins and Hormones (NY)* **18** 315 *1960*] for review of purification methods. Stored in the dark, under N_2 or Ar, at 0°. See Vitamin A acetate p. 574 in Chapter 6.

Retinyl palmitate *[79-81-2]* **M 524.9, m 28-29°, $\varepsilon_{1cm}^{1\%}$ (*all-trans*) 1000 (325 nm) in EtOH.** Separated from retinol by column chromatography on water-deactivated alumina with hexane containing a very small percentage of acetone. Also chromatographed on TLC silica gel G, using pet ether/isopropyl ether/acetic acid/water (180:20:2:5) or pet ether/acetonitrile/acetic acid/water (190:10:1:15) to develop the chromatogram. Then recrystd from propylene at low temperature.

Rhamnetin (3,3'-4',5-tetrahydroxy-7-methoxy flavone, 7-methyl quercitin) *[90-19-7]* **M 316.3, m >300°(dec), several phenolic pKs ~7-10.5.** Crystd from EtOH.

L-α-Rhamnose (H_2O) *[10030-85-0 (H_2O); 3615-41-6 (anhydr)]* **M 182.2, m 105°, $[\alpha]_D^{15}$ +9.1° (c 5, H_2O).** Crystd from water or EtOH.

Rhodamine B chloride [3,5-bis-(diethylamino)-9-(2-carboxyphenyl)xanthylium chloride] *[81-88-9]* **M 479.0, m 210-211°(dec), CI 45170, λmax 543nm, {Free base** *[509-34-2]* **CI 749}, pK 5.53.** Major impurities are partially dealkylated compounds not removed by crystn. Purified by chromatography, using ethyl acetate/isopropanol/ammonia (conc)(9:7:4, R_F 0.75 on Kieselgel G). Also crystd from conc soln in MeOH by slow addition of dry diethyl ether; or from EtOH containing a drop of conc HCl by slow addition of ten volumes of dry diethyl ether. The solid was washed with ether and air dried. The dried material has also been extracted with *benzene to remove oil-soluble material prior to recrystn. Store in the dark.

Rhodamine 6G [Basic Red 1, 3,5-bis-(ethylamino)-9-(2-ethoxycarbonylphenyl)-2,7-dimethylxanthylium chloride] *[989-38-8]* **M 479.3, CI 45160, λmax 524nm, pK 5.58.** Crystd from MeOH or EtOH, and dried in a vac oven.

Rhodanine (2-mercaptothiazolidin-4-one) *[141-84-4]* **M 133.2, m 168.5° (capillary), pK^{20} 5.18.** Crystd from glacial acetic acid or water.

Riboflavin, riboflavin-5'-phosphate (Na salt, $2H_2O$) and **ribonucleic acid** see entries in Chapter 6.

α-D-Ribose *[50-69-1]* **M 150.1, m 90°, $[\alpha]_{546}^{20}$ -24° (after 24h, c 10, H_2O), pK^{25} 12.22.** Crystd from aqueous 80% EtOH, dried under vacuum at 60° over P_2O_5 and stored in a vacuum desiccator.

Ricinoleic acid (*dl* 12-hydroxyoleic acid) *[141-22-0]* **M 298.5, m 7-8° (α-form), 5.0° (γ-form), n 1.4717, pK_{Est} ~4.5.** Purified as methyl acetylricinoleate [Rider *J Am Chem Soc* **53** 4130 *1931*], fractionally distilling at 180-185°/0.3mm, then 87g of this ester was refluxed with KOH (56g), water (25mL), and MeOH (250mL) for 10min. The free acid was separated, crystd from acetone at -50°, and distd in small batches, **b** 180°/0.005mm. [Bailey et al. *J Chem Soc* 3027 *1957*.]

Rosaniline HCl (Magenta I, Fuschin) *[632-99-5]* **M 337.9, m >200°(dec).** Purified by dissolving in EtOH, filtering and adding H_2O. Filter or centrifuge and wash the ppte with Et_2O and dry in air. Could be crystd from H_2O. Also recrystd from water and dried *in vacuo* at 40°. Crystals have a metallic green lustre. UV max in EtOH is at 543nm (ε 93,000). Solubility in H_2O is 0.26%. A carmine red colour is produced in EtOH. [Scalan *J Am Chem Soc* **57** 887 *1937*.]

***p*-Rosolic acid (4-[bis-{4-hydroxyphenyl}methylene]-2,5-cyclohexadien-one, 4',4"-di-hydroxy-fuschson, aurin, corallin)** *[603-45-2]* **M 290.3, m 292°, 295-300° (dec with liberation of phenol), 308-310°(dec), pK_1 3.11, pK_2 8.62.** It forms green crystals with a metallic lustre but the colour depends on the solvent used. When recrystd from brine (satd aqueous NaCl) acidified with HCl it forms red needles, but when recrystd from EtOH-AcOH the crystals have a beetle iridescent green colour. It has been recrystd from Me_2CO (although it dissolves slowly), methyl ethyl ketone, 80-95% AcOH and from AcOH-*C_6H_6. An aq KOH soln is golden yellow and a 70% H_2SO_4 soln is deep red in colour. An alternative purification is to dissolve this triphenylmethane dye in 1.5% of aq NH_3, filter, and heat to 70-80°, then acidify with dilute AcOH by adding it slowly with vigorous stirring, whereby the aurin separates as a brick-red powder or as purplish crystals depending on the temperature and period of heating. Filter off the solid, wash with H_2O and a little dilute AcOH then H_2O again. Stir this solid with Et_2O to remove any ketones and allow to stand overnight in the Et_2O, then fiter and dry in air then in a vacuum. [Gomberg and Snow *J Am Chem Soc* 47 202 *1925*; Baines and Driver *J Chem Soc* 123 1216 *1923*; UV: Burawoy *Chem Ber* **64** 462 *1941*; Neuk and Schmid *J Prakt Chem* [2] **23** 549 *1881*.]

Rubijervine (slanid-5-ene-3β-12α-diol) *[79-58-3]* **M 413.6, m 240-246°, $[\alpha]_D^{20}$ +19° (EtOH), pK_{Est} ~7.0.** Crystd from EtOH. It has solvent of crystn.

Rubrene *[517-51-1]* **M 532.7, m >315°.** See 5,6,11,12-tetraphenylnaphthacene on p. 366.

(+)-Rutin (quercetin-3-rubinoside) *[153-18-4]* **M 610.5, m 188-189, $[\alpha]_{546}^{20}$ +13° (c 5, EtOH) (polyphenolic flavone pKs 7—10).** Crystd from MeOH or water/EtOH, air dried, then dried for several hours at 110°.

Saccharic acid (D-glucaric acid) *[87-73-0]* M 210.1, m 125-126°, $[\alpha]_D^{20}$ +6.9°→ +20.6° (H_2O), pK_1 3.01, pK_2 3.94 (D-isomer).

Crystd from 95% EtOH.

Safranine O *[477-73-6]* **M 350.9, λ_{max} 530nm, pK^{25} 6.4.** Crystd from *benzene/MeOH (1:1) or water. Dried under vacuum over H_2SO_4.

Safrole (5-allyl-1,3-benzodioxole, 4-allyl-1,2-methylenedioxybenzene) *[94-59-7]* **M 162.1, m~ 11°, b 69-70°/1.5mm, 104-105°/6mm, 231.5-232°/atm, 235-237°/atm, d_4^{20} 1.0993, n_D^{20} 1.53738.** It has been purified by fractional distn, although it has also been recrystd from low boiling pet ether at low temperatures. [IR: Briggs et al. *Anal Chem 29* **904** *1957*; UV: Patterson and Hibbert *J Am Chem Soc* **65** 1962 *1943*.] The *maleic anhydride adduct* forms yellow crystals from toluene **m** 257° [Hickey *J Org Chem* **13** 443 *1948*], and the *picrate* forms orange-red crystals from $CHCl_3$ [Baril and Magrdichian *J Am Chem Soc* **58** 1415 *1936*].

D(-)-Salicin *[138-52-3]* **M 286.3, m 204-208°, $[\alpha]_D^{25}$ -63.5° (c *ca* 3, H_2O).** Crystd from EtOH.

Salicylaldehyde (*o*-hydroxybenzaldehyde) *[90-02-8]* **M 122.1, b 93°/25mm, 195-197°/760mm, d 1.167, n 1.574, pK^{25} 8.37.** Ppted as the bisulfite addition compound by pouring the aldehyde slowly and with stirring into a 25% soln of $NaHSO_3$ in 30% EtOH, then standing for 30min. The ppte, after filtering at the pump, and washing with EtOH, was decomposed with aq 10% $NaHCO_3$, and the aldehyde was extracted into diethyl ether, dried with Na_2SO_4 or $MgSO_4$, and distd, under reduced pressure. Alternatively, salicylaldehyde can be pptd as its copper complex by adding it to warm, satd soln of copper acetate, shaking and then standing in ice. The ppte was filtered off, washed thoroughly with EtOH, then with diethyl ether, and decomposed with 10% H_2SO_4, the aldehyde was extracted into diethyl ether, dried and distd. It has also been purified by repeated vacuum distn, and by dry column chromatography on Kieselgel G [Nishiya et al. *J Am Chem Soc* **108** 3880 *1986*]. The *acetyl* derivative has **m** 38-39° (from pet ether or EtOH) and **b** 142°/18mm, 253°/atm.

Salicylaldoxime *[94-67-7]* **M 137.1, m 57°, pK_{Est} ~ 8.3.** Crystd from $CHCl_3$/pet ether (b 40-60°).

Salicylamide *[65-45-2]* **M 137.1, m 142-144°, pK^{20} 8.37.** Crystd from water or repeatedly from chloroform [Nishiya et al. *J Am Chem Soc* **108** 3880 *1986*].

Salicylanilide *[87-17-2]* **M 213.2, m 135°, pK_{Est} ~8.3.** Crystd from water.

Salicylhydroxamic acid *[89-73-6]* **M 153.1, m 179-180°(dec), pK_1^{30} 2.15, pK_2^{30} 7.46, pK_3^{30} 9.72.** Crystd from acetic acid.

Salicyclic acid (2-hydroxybenzoic acid) *[69-72-7]* **M 138.1, m 157-159°, 158-160°, 159.5°, 159-160°, 162°, b 211°/20mm, pK_1^{25} 3.01, pK_2^{25} 13.43 (13.01).** It has been purified by steam distn, by recrystn from H_2O (solubility is 0.22% at room temp and 6.7% at 100°), absolute MeOH, or cyclohexane and by sublimation in a vacuum at 76°. The *acid chloride* (needles) has **m** 19-19.5°, **b** 92°/15mm, *amide* **m** 133° (yellow needles from H_2O), and *anilide* (prisms fron H_2O) **m** 135°. The *O-acetyl* derivative has **m** 135° (rapid heating and the liquid resolidifies at 118°) and the *o-benzoyl* derivative has **m** 132° (aq EtOH). [IR: Hales et al. *J Chem Soc* 3145 *1954*; UV: Bergmann et al. *J Chem Soc* 2351 *1950*].

Sarcosine *[107-97-1]* **M 89.1, m 212-213°(dec), pK_1^{20} 2.12, pK_2^{20} 10.19.** Crystd from abs EtOH.

Scopoletin (7-hydroxy-6-methoxycoumarin) *[92-61-5]* **M 192.2, m 206°, 208-209°, pK 8.96 (70%aq EtOH).** Crystd from water, acetic acid or *C_6H_6/MeOH.

Sebacic acid *[111-20-6]* **M 202.3, m 134.5°, pK_1^{25} 4.58, pK_2^{25} 5.54.** Purified *via* the disodium salt which, after crystn from boiling water (charcoal), was again converted to the free acid. The free acid was crystd repeatedly from hot distd water or from Me_2CO+pet ether and dried under vacuum.

Sebacic acid monomethyl ester *[818-88-2]* **M 216.3, m 42-43°, b 169-171°/4mm.** Recrystd from Me_3CO+pet ether or pet ether at low temperature and distd in a vacuum.

Sebaconitrile (decanedinitrile) *[1871-96-1]* **M 164.3, m 8°, b 199-200°.** Mix with P_2O_5 (10% by wt) and distilled from it, then redistilled.

Secobarbital (5-allyl-5-1'-methylbutylbarbituric acid) *[76-73-3]* **M 260.3, m 100°, $pK_{Est(1)}$~3.5, $pK_{Est(2)}$~12.0.** A soln of the salt in 10% HCl was ppted and the acid form was extracted by the addition of ether. Then purified by repeated crystn from $CHCl_3$. [Buchet and Sandorfy *J Phys Chem* **88** 3274 *1984*.]

Semicarbazide hydrochloride (hydrazine carboxamide hydrochloride) *[563-41-7]* **M 111.5, m 173°(dec), 175°(dec), pK^{24} 3.66.** Crystd from aqueous 75% EtOH and dried under vacuum over $CaSO_4$. Also crystd from a mixture of 3.6 mole % MeOH and 6.4 mole % of water. [Kovach et al. *J Am Chem Soc* **107** 7360 *1985*.] IR: ν 700, 3500 cm^{-1} [*Org Synth* Coll Vol I 485 *1941*; Davison and Christie *J Chem Soc* 3389 *1955*; Thiele and Stange *Chem Ber* **27** 33 *1894*; pK: Bartlett *J Am Chem Soc* **54** 2853 *1923*]. The ***free base*** crystd as prisms from abs EtOH, **m** 96° [Curtius and Heidenreich *Chem Ber* **27** 55 *1894*]. **TOXIC ORALLY**, possible **CARCINOGEN** and **TERATOGEN**.

Sennoside A *[81-27-6]* **M 862.7, m 220-240°(dec), $[\alpha]_D^{20}$ -147° (c 5, Me_2CO/H_2O 7:1).** Crystd from aq acetone or large vols of H_2O.

Sennoside B *[128-57-4]* **M 962.7, m 182-190°(dec), $[\alpha]_D^{20}$ -100° (c 2, Me_2CO/H_2O 7:3).** Crystd from aq acetone or large vols of H_2O.

L-Serine *[56-45-1]* **M 105.1, m 228°(dec), $[\alpha]_D^{25}$ +14.5° (1M HCl), $[\alpha]_{546}^{20}$ +16° (c 5, 5M HCl), pK_1^{25} 2.15, pK_2^{25} 9.21.** Likely impurity is glycine. Crystd from water by adding 4 volumes of EtOH. Dried. Stored in a desiccator.

Serotonin creatinine sulfate (H_2O) *[971-74-4]* **M 405.4, m 220°(dec), pK_1 10.1, pK_2 11.1, pK_3 18.25 (NH) for serotonin, pK 4.9 for creatinine.** Crystd (as monohydrate) from water.

Shikimic acid *[138-59-0]* **M 174.2, m 183-184.5°, 190°, $[\alpha]_{546}^{20}$ -210° (c 2, H_2O), pK^{14} 4.15.** Crystd from water or MeOH/AcOEt and sublimes in a vac.

Sinomenine hydrochloride (7,8-didehydro-4-hydroxy-3,7-dimethoxy-17-methyl-9α,13α,14α-morphan-6-one HCl) *[6080-33-7]* **M 365.9, m 231°, $[\alpha]_D^{17}$ -83° (c 4, H_2O), $pK_{Est(1)}$~10.0 (N), $pK_{Est(2)}$~10.4 (OH).** Crystd from water.

β-Sitosterol *[83-46-5]* **M 414.7, m 136-137°, $[\alpha]_{546}^{20}$ -42° (c 2, $CHCl_3$).** Crystd from MeOH. Also purified by zone melting.

Skellysolve A is essentially *n*-pentane, **b** 28-30°,
Skellysolve A is essentially *n*-hexane, **b** 60-68°,
Skellysolve C is essentially *n*-heptane, **b** 90-100°,
Skellysolve D is mixed heptanes, **b** 75-115°,
Skellysolve E is mixed octanes, **b** 100-140°,
Skellysolve F is pet ether, **b** 30-60°,
Skellysolve G is pet ether, **b** 40-75°,
Skellysolve H is hexanes and heptanes, **b** 69-96°,
Skellysolve L is essentially octanes, **b** 95-127°. For methods of purification, see **petroleum ether**.

Smilagenin *[126-18-1]* **M 416.6, m 185°, $[\alpha]_D^{25}$ -69° (c 0.5, $CHCl_3$).** Crystd from acetone.

Solanidine *[80-78-4]* **M 397.6, m 218-219°, $[\alpha]_D^{20}$ -29° (c 0.5, $CHCl_3$), pK^{15} 6.66.** Crystd from $CHCl_3$/MeOH.

α-Solanine *[20562-02-1]* **M 868.1, m 286°(dec), $[\alpha]_D^{20}$ -58° (c 0.8, pyridine), pK^{15} 6.66.**
See α-solanine on p. 566 in Chapter 6.

Solanone [*S*(+)-*trans*-**2-methyl-5-isopropyl-1,3-nonan-8-one**] *[1937-54-8]* **M 194.3, b 60°/1mm, $[\alpha]_D^{20}$ +14° (neat).** Purified by high vacuum distillation and stored in sealed ampules [Kohda and Sato *J Chem Soc, Chem Commun* 951 *1981*]. It has UV (hexane) at λmax 230nm (ε 11,800).

Solasodine *[126-17-0]* **M 413.6, m 202°, $[\alpha]_D^{25}$ -100° (c 2, MeOH), pK 7.7.** Crystd (as monohydrate) from MeOH or aq 80% EtOH, and sublimes in a vac.

Solasonine (solasodine-3-O-mannoglucoside) *[19121-58-5]* **M 884.0, m 279°, $[\alpha]_D^{20}$ -75° (c 0.5, MeOH), pK_{Est} ~ 7.7.** Crystd from aq 80% dioxane or MeOH.

Solochrome Violet R [4-hydroxy-3-(2-hydroxynaphthyl-1-ylazo)benzenesulfonic acid] *[2092-55-9]* **M 367.3, CI 15670, λmax 501nm, pK_2^{25} 7.22 (OH), pK_3^{25} 13.39 (OH).** Converted to the monosodium salt by pptn with NaOAc/AcOH buffer of pH 4, then purified by pptn of the free acid from aq soln with conc HCl, washing and extracting with EtOH in a Soxhlet extractor. The acid ppted on evaporating the EtOH and was reconverted to the sodium salt.as described for *Chlorazole Sky Blue FF*. Dried at 110°. It is *hygroscopic*. [Coates and Rigg *Trans Faraday Soc* **57** 1088 *1961*.]

Sorbic acid (2,4-hexadienoic acid) *[110-44-1]* **M 112.1, m 134°, pK^{25} 4.76.** Crystd from water.

Sorbitol *[50-70-4]* **M 182.2, m 89-93° (hemihydrate), 110-111° (anhydrous), $[\alpha]_{546}^{20}$ -1.8° (c 10, H_2O), pK^{60} 13.00.** Crystd (as hemihydrate) several times from EtOH/water (1:1), then dried by fusing and storing over $MgSO_4$.

(-)-Sparteine sulfate pentahydrate *[6160-12-9]* **M 422.5, m loses H_2O at 100° and turns brown at 136° (dec), $[\alpha]_D^{20}$ -22° (c 5, H_2O), $[\alpha]_D^{21}$ -16° (c 10, EtOH for free base), pK_1^{20} 2.24, pK_2^{20} 9.46.** Recrystd from aq EtOH or H_2O although the solubility in the latter is high. The *free (-)-base* has **b** 173°/8mm and is steam volatile but resinifies in air. The *dipicrate* forms yellow needles from EtOH-Me_2CO, **m** 205-206° [Clemo et al. *J Chem Soc* 429 *1931*; see also Bolnmann and Schuman *The Alkaloids* (Ed Manske) Vol 9 175 *1967*]. The *free (±)-base* has **m** 71-72.5° [van Tamelen and Foltz *J Am Chem Soc* **82** 2400 *1960*].

Spermidine [*N*-**(3-aminopropyl)-1,4-diaminobutane**] *[124-20-9]* **M 145.3, m 23-25°, b 128-131°/15mm, d 0.918, n 1.482, pK_1^{25} 8.25, pK_2^{25} 9.64, pK_3^{25} 10.43.** It is a strong base with an alkylamine odour and absorbs CO_2 from the atmosphere. It is purified by shaking with solid K_2CO_3 or NaOH, decanting and distilling from K_2CO_3 in a vacuum. Store in the dark under N_2.

Spermidine trihydrochloride *[334-50-9]* **M 245.3, m ~250°(dec), 256-258°, for pKa see *free base* above.** Recrystd from dry 3% HCl in ethanol adding dry Et_2O if necessary. Filter rapidly and dry in a vac desiccator. Alternatively centrifuge the crystals off wash them with dry Et_2O and dry in a vacuum.

Spermine 4HCl (*N,N*-**bis(3-aminopropyl)-1,4-butanediamine 4HCl**) *[306-67-2]* **M 348.2, m 313-315°.** The pKs are similar to spermidine above. Purification as for spermidine trihydrochloride above.

Squalene (**all-*trans*- 2,6,10,15,19,23-hexamethyltetracosahexa-2,6,10,14,18,22-ene, spinacen**) *[111-02-4]* **M 410.7, m ~75°, b 203°/0.15mm.** See squalene on p. 567 in Chapter 6.

Squaric acid (**3,4-dihydroxy-3-cyclobutene-1,2-dione**) *[2892-51-5]* **M 114.1, m 293°(dec), 294°(dec), >300°, pK_1^{20} 1,50 , pK_2^{20} 2.93.** Purified by recryst from H_2O — this is quite simple because the acid is ~ 7% soluble in boiling H_2O and only 2% at room temperature. It is not soluble in Me_2CO or Et_2O hence it can be rinsed with these solvents and dried in air or a vacuum. It is not hygroscopic and gives an intense purple colour with $FeCl_3$. It has IR ν at 1820 (C=O) and 1640 (C=C) cm^{-1}; and UV λmax at 269.5nm (ε 37K $M^{-1}cm^{-1}$).) [Cohn et al. *J Am Chem Soc* **81** 3480 *1959*; Park et al. *J Am Chem Soc* **84** 2919 *1962*] See also **pKa** values of 0.59 ±0.09 and 3.48 ±0.023 [Scwartz and Howard *J Phys Chem* **74** 4374 *1970*].

Starch *[9005-84-9]* **M (162.1)n.** See entry on p. 567 in Chapter 6.

Stearic acid (octadecanoic acid) *[57-11-4]* **M 284.5, m 71.4°, 72°, b 144-145°/27mm, 383°/760mm, d 0.911, n 1.428, pK_{Est} ~5.0.** Crystd from acetone, acetonitrile, EtOH (5 times), aq MeOH, ethyl methyl ketone or pet ether (b 60-90°), or by fractional pptn by dissolving in hot 95% EtOH and pouring into distd water, with stirring. The ppte, after washing with distd water, was dried under vacuum over P_2O_5. It has also been purified by zone melting and partial freezing. [Tamai et al. *J Phys Chem* **91** 541 *1987*.]

Stigmasterol *[83-48-7]* **M 412.7, m 170°, $[\alpha]_D^{22}$ -51° ($CHCl_3$), $[\alpha]_{546}^{20}$ -59° (c 2, $CHCl_3$).** Crystd from hot EtOH. Dried in vacuum over P_2O_5 for 3h at 90°. Purity was checked by NMR.

***cis*-Stilbene** *[645-49-8]* **M 180.3, b 145°/12mm.** Purified by chromatography on alumina using hexane and distd under vacuum. (The final product contains *ca* 0.1% of the *trans*-isomer). [Lewis et al. *J Am Chem Soc* **107** 203 *1985*; Saltiel *J Phys Chem* **91** 2755 *1987*.]

***trans*-Stilbene** *[103-30-0]* **M 180.3, m 125.9°, b 305-307°/744mm, d 0.970.** Purified by vac distn. (The final product contains about 1% of the *cis* isomer). Crystd from EtOH. Purified by zone melting. [Lewis et al. *J Am Chem Soc* **107** 203 *1985*; Bollucci et al. *J Am Chem Soc* **109** 515 *1987*; Saltiel *J Phys Chem* **91** 2755 *1987*.]

(-)-Strychnine *[57-24-9]* **M 334.4, m 268°, $[\alpha]_{546}^{20}$ -139° (c 1, $CHCl_3$), pK_1^{20} 2,50, pK_2^{20} 8.2.** Crystd as the hydrochloride from water, then neutralised with ammonia.

Styphnic acid (2,4,6-trinitroresorcinol) *[82-71-3]* **M 245.1, m 177-178°, 179-180°, pK_1^{25} 0.06 (1.74), pK_2^{25} 4.23 (4.86).** Crystd from ethyl acetate or water containing HCl **[EXPLODES violently on rapid heating.]**

Styrene *[100-42-5]* **M 104.2, b 41-42°/18mm, 145.2°/760mm, d 0.907, n 1.5469, n^{25} 1.5441.** Styrene is difficult to purify and keep pure. Usually contains added inhibitors (such as a trace of hydroquinone). Washed with aqueous NaOH to remove inhibitors (e.g. *tert*-butanol), then with water, dried for several hours with $MgSO_4$ and distd at 25° under reduced pressure in the presence of an inhibitor (such as 0.005% *p-tert*-butylcatechol). It can be stored at -78°. It can also be stored and kept anhydrous with Linde type 5A molecular sieves, CaH_2, $CaSO_4$, BaO or sodium, being fractionally distd, and distd in a vacuum line just before use. Alternatively styrene (and its deuterated derivative) were passed through a neutral alumina column before use [Woon et al. *J Am Chem Soc* **108** 7990 *1986*; Collman *J Am Chem Soc* **108** 2588 *1986*].

(±)-Styrene glycol (±-1-phenyl-1,2-ethanediol) *[93-56-1]* **M 138.2, m 67-68°.** Crystd from pet ether.

Styrene oxide *[96-09-3]* **M 120.2, b 84-86°/16.5mm, d 1.053, n 1.535.** Fractional distn at reduced pressure does not remove phenylacetaldehyde. If this material is present, the styrene oxide is treated with hydrogen under 3 atmospheres pressure in the presence of platinum oxide. The aldehyde, but not the oxide, is reduced to ß-phenylethanol) and separation is now readily achieved by fractional distn. [Schenck and Kaizermen *J Am Chem Soc* **75** 1636 *1953*.]

Suberic acid (hexane-1,6-dicarboxylic acid) *[505-48-6]* **M 174.2, m 141-142°, pK_1^{25} 4.12, pK_2^{25} 5.40.** Crystd from acetone and sublimes at 300° without dec.

Succinamic acid (succinic acid amide) *[638-32-4]* **M 117.1, m 155°, 156-157°, pK^{25} 4.54.** Crystd from Me_2CO or H_2O and dried in vac. Not v sol in MeOH. Converted to succinimide above 200°.

Succinamide *[110-14-5]* **M 116.1, m 262-265°(dec).** Crystd from hot water.

Succinic acid *[110-15-6]* **M 118.1, m 185-185.5°, pK_1^{25} 4.21, pK_2^{25} 5.72.** Washed with diethyl ether. Crystd from acetone, distd water, or *tert*-butanol. Dried under vacuum over P_2O_5 or conc H_2SO_4. Also

purified by conversion to the disodium salt which, after crystn from boiling water (charcoal), is treated with mineral acid to regenerate the succinic acid. The acid is then recrystd and vacuum dried.

Succinic anhydride *[108-30-5]* **M 100.1, m 119-120°.** Crystd from redistd acetic anhydride or $CHCl_3$, then filtered, washed with diethyl ether and dried under vacuum.

Succinimide *[123-56-8]* **M 99.1, m 124-125°, pK^{25} 9.62.** Crystd from EtOH (1mL/g) or water.

Succinonitrile *[110-61-2]* **M 80.1, m 57.9°, b 108°/1mm, 267°/760mm.** Purified by vacuum sublimation, also crystd from acetone.

D(+)-Sucrose (β-D-fructofuranosyl-α-D-glucopyranoside) *[57-50-1]* **M 342.3, m 160-186°, 186-188°, $[\alpha]^{20}_{546}$ +78° (c 10, H_2O), $[\alpha]^{20}_{D}$ + 66° (c 26, H_2O), pK 12.62.** Crystd from water (solubility: 1g in 0.5mL H_2O at 20°, 1g in 0.2mL in boiling H_2O). Sol in EtOH (0.6%) and MeOH (1%). *Sucrose diacetate hexaisobutyrate* is purified by melting and, while molten, treated with $NaHCO_3$ and charcoal, then filtered.

D-Sucrose octaacetate *[126-14-7]* **M 678.6, m 83-85°, $[\alpha]^{20}_{546}$ +70° (c 1, $CHCl_3$).** Crystd from EtOH.

Sudan I (Solvent Yellow 14, 1-phenylazo-2-naphthol) *[824-07-9]* **M 248.3, m 135°, CI 12055, λmax 476nm, pK_{Est} ~9.0.** Crystd from EtOH.

Sudan III [Solvent Red 23, 1-(*p*-phenylazo-phenylazo)-2-naphthol] *[85-86-9]* **M 352.4, m 199°(dec), CI 26100, λ_{max} 354, 508 nm, pK_{Est} ~9.0.** Crystd from EtOH, EtOH/water or *benzene/abs EtOH (1:1).

Sudan IV [Solvent Red 24, 1-(4-*o*-tolylazo-*o*-tolylazo)-2-naphthol] *[85-83-6]* **M 380.5, m ~184°(dec), CI 26105, λmax 520nm, pK_{Est} ~9.0.** Crystd from EtOH/water or acetone/water.

Sulfaguanidine *[57-67-0]* **M 214.2, m 189-190°, pK_1 0.48, pK_2 2.75.** Crystd from hot water (7mL/g).

Sulfamethazine *[57-68-1]* **M 278.3, m 198-200°, pK_1 2.65, pK_2 7.4.** Crystd from dioxane.

Sulfanilamide (*p*-aminobenzenesulfonamide) *[63-74-1]* **M 172.2, m 166°, pK^{20}_1 2.30, pK^{20}_2 10.26.** Crystd from water or EtOH.

Sulfanilic acid (4-aminobenzenesulfonic acid) *[121-57-3]* **M 173.2, pK^{25}_1 <1, pK^{25}_2 3.23.** Crystd (as dihydrate) from boiling water. Dried at 105° for 2-3h, then over 90% H_2SO_4 in a vacuum desiccator.

Sulfapyridine *[144-83-2]* **M 349.2, m 193°, pK^{20} 8.64.** Crystd from 90% acetone and dried at 90°.

***o*-Sulfobenzoic acid (H_2O)** *[123333-68-6 (H_2O); 632-25-7]* **M 202.2, m 68-69°, $pK_{Est(1)}$<1, $pK_{Est(2)}$~3.1 (CO_2H).** Crystd from water.

***o*-Sulfobenzoic acid (monoammonium salt)** *[6939-89-5]* **M 219.5.** Crystd from water.

***o*-Sulfobenzoic anhydride** *[81-08-3]* **M 184.2, m 128°, b 184-186°/18mm.** See also 2,1-benzoxathiol-3-one-1,1-dioxide on p. 126.

Sulfolane (tetramethylenesulfone) *[126-33-0]* **M 120.2, m 28.5°, b 153-154°/18mm, 285°/760mm, d 1.263, n^{30} 1.4820.** Prepared commercially by Diels-Alder reaction of 1,3-butadiene and sulfur dioxide, followed by Raney nickel hydrogenation. The principle impurities are water, 3-sulfolene, 2-sulfolene and 2-isopropyl sulfolanyl ether. It is dried by passage through a column of molecular sieves. Distd

under reduced pressure through a column packed with stainless steel helices. Again dried with molecular sieves and distd. [Cram et al. *J Am Chem Soc* **83** 3678 *1961*; Coetzee *Pure Appl Chem* **49** 211 *1977*.]
Also, it was stirred at 50° and small portions of solid $KMnO_4$ were added until the colour persisted during 1h. Dropwise addition of MeOH then destroyed the excess $KMnO_4$, the soln was filtered, freed from potassium ions by passage through an ion-exchange column and dried under vacuum. It has also been vacuum distd from KOH pellets. It is *hygroscopic*. [See Sacco et al. *J Phys Chem* **80** 749 *1976; J Chem Soc, Faraday Trans 1* **73** 1936 *1977*; **74** 2070 *1978*; *Trans Faraday Soc* **62** 2738 *1966*.] Coetzee has reviewed the methods of purification of sulfolane, and also the removal of impurities. [Coetzee in *Recommended Methods of Purification of Solvents and Tests for Impurities*, Coetzee Ed. Pergamon Press, 1982.]

5-Sulfosalicylic acid *[5965-83-3]* **M 254.2, m 108-110°, pK_1^{25} <0, pK_2^{25} 2.67, pK_3^{25} 11.67.** Crystd from water. Alternatively, it was converted to the monosodium salt which was crystd from water and washed with a little water, EtOH and then diethyl ether. The free acid was recovered by acidification.

Syringaldehyde (3,5-dimethoxy-4-hydroxybenzaldehyde) *[134-96-3]* **M 182.2, m 113°, pK_{Est} ~8.** Crystd from pet ether.

Syringic acid (3,5-dimethoxy-4-hydroxybenzoic acid) *[530-57-4]* **M 198.2, m 204-205°, 206.5°, 206-209°, 209-210°, pK_1^{25} 4.34, pK_2^{25} 9.49.** Recrystd from H_2O using charcoal [Bogert and Coyne *J Am Chem Soc* **51** 571 *1929*; Anderson and Nabenhauer *J Am Chem Soc* **48** 3001 *1926*.] The *methyl ester* has **m** 107° (from MeOH), the *4-acetyl* derivative has **m** 190° and the *4-benzoyl* derivative has **m** 229-232°. [Hahn and Wassmuth *Chem Ber* **67** 2050 *1934*; UV: Lemon *J Am Chem Soc* **69** 2998 *1947* and Pearl and Beyer *J Am Chem Soc* **72** 1743 *1950*.]

D(-)-Tagatose *[87-81-0]* **M 180.2, m 134-135°, $[\alpha]_{546}$ -6.5° (c 1, H_2O).** Crystd from aqueous EtOH.

***d*-Tartaric acid** *[147-71-7]* **M 150.1, m 169.5-170° (2*S*,3*S*-form, natural) $[\alpha]_{546}^{20}$ -15° (c 10, H_2O); m 208° (2*RS*,3*RS*-form), pK_1^{25} 3.03, pK_2^{25} 4.46, pK_3^{25} 14.4.** Crystd from distilled H_2O or *benzene/diethyl ether containing 5% of pet ether (b 60-80°) (1:1). Soxhlet extraction with diethyl ether has been used to remove an impurity absorbing at 265nm. It has also been crystd from absolute EtOH/hexane, and dried in a vacuum for 18h [Kornblum and Wade *J Org Chem* **52** 5301 *1987*].

***meso*-Tartaric acid** *[147-73-9]* **M 150.1, m 139-141°, pK_1^{25} 3.17, pK_2^{25} 4.91.** Crystd from water, washed with cold MeOH and dried at 60° under vacuum.

Taurocholic acid *[81-24-3]* **M 515.6, m 125°(dec), $[\alpha]_D$ +38.8 (c 2, EtOH), pK 1.4.** Crystd from EtOH/diethyl ether.

Terephalaldehyde *[623-27-8]* **M 134.1, m 116°, b 245-248°/771mm.** Crystd from water.

Terephthalic acid (benzene-1,4-dicarboxylic acid) *[100-21-0]* **M 166.1, m sublimes >300° without melting, pK_1^{20} 3.4, pK_2^{20} 4.34.** Purified *via* the sodium salt which, after crystn from water, was reconverted to the acid by acidification with mineral acid.

Terephthaloyl chloride *[100-20-9]* **M 203.0, m 80-82°.** Crystd from dry hexane.

***o*-Terphenyl** *[84-15-1]* **M 230.3, m 57-58°.** Crystd from EtOH. Purified by chromatography of CCl_4 solns on alumina, with pet ether as eluent, followed by crystn from pet ether (b 40-60°) or pet ether/*benzene. They can also be distd under vacuum.

***m*-Terphenyl** *[92-06-8]* **M 230.3, m 88-89°.** Purification as for *o*-terphenyl above.

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