CHAPTER 4

PURIFICATION OF ORGANIC CHEMICALS

The general principles, techniques and methods of purification in Chapters 1 and 2 are applicable to this chapter. Most organic liquids and a number of solids can readily be purified by fractional distillation, usually at atmospheric pressure. Sometimes, particularly with high boiling or sensitive liquids, or when in doubt about stability, distillation or fractionation under reduced pressure should be carried out. To save space, the present chapter omits many substances for which the published purification methods involve simple distillation. Where boiling points are given, purification by distillation is another means of removing impurities. Literature references are omitted for methods which require simple recrystallisation from solution if the correct solvent can be guessed readily, and where no further information is given, e.g. spectra. Substances are listed alphabetically, usually with some criteria of purity, giving brief details of how they can be purified. Also noted are the molecular weights (to the first decimal place), melting points and/or boiling points together with the respective densities and refractive indexes for liquids, and optical rotations when the compounds are chiral. When the temperatures and/or the wavelengths are not given for the last three named properties then they should be assumed to be 20°C and the average of the wavelengths of the sodium D lines repectively; and densities are relative to water at 4°.

The present chapter includes commercially available organic chemicals. Most of the inorganic, metalorganic, organo- bismuth, boron, phosphorus, selenium, silicon and alkali metal compounds and metal ion salts of organic acids are in Chapter 5. Naturally occurring commercially available organic compounds for use in biochemistry, molecular biology and biology are in Chapter 6. Commercially available polymer supported reagents are indicated with § under the appropriate reagent.

Rapid purification procedures are noted for commonly used solvents and reagents which make them suitable for general use in synthetic chamistry.

Abbreviations of titles of periodicals are defined as in the Chemical Abstracts Service Source Index (CASSI). Other abbreviations are self evident (see Chapter 1, p. 30).

Ionisation constants of ionisable compounds are give as **pK** values (published from the literature) and refer to the **pKa** values at room temperature (~ 15°C to 25°C). The values at other temperatures are given as superscripts, e.g. $\mathbf{pK}^{25}$ for 25°C. Estimated values are entered as $\mathbf{pK}_{\mathbf{Est.}}$ (see Chapter 1, p. 7 for further information).

As a good general rule, all low boiling (<100°) organic liquids should be treated as highly flammable and toxic (because they can be inhaled in large quantities) and the necessary precautions should be taken.

Benzene, which has been used as a solvent successfully and extensively in the past for reactions and purification by chromatography and crystallisation is now considered a **very dangerous substance** so it hasto be used with extreme care. We emphasise that an alternative solvent system to benzene (e.g. toluene, toluene-petroleum ether, or a petroleum ether to name a few) should be used first. However, if no other solvent system can be found then all operations involving benzene have to be performed in an efficient fumehood and precautions must be taken to avoid inhalation and contact with skin and eyes. Whenever benzene is mentioned in the text an asterisk e.g. $^*C_6H_6$ or *benzene, is inserted to remind the user that special precaution should be adopted.

Abietic acid *[514-10-3]* **M 302.5, m 172-175°, $[\alpha]_D^{25}$ -116° (-106°)(c 1, EtOH), pK 5.27.** Crystd by dissolving 100g of acid in 95% EtOH (700mL), adding to H_2O (600mL) and cooling. Filter, dry in a vacuum (over KOH or $CaSO_4$) store in an O_2-free atmosphere. λ in EtOH nm(log ε): 2343(4.3), 241(4.4), 2505(4.2), 235(4.34) and 240(4.36). [*Org Synth* **23** 1 *1952* ; *J Am Chem Soc* **35** 3736 *1949*; *Monatsh Chem* **116** 1345 *1985*.]

***S*-Abscisic acid** *[21293-29-8]* **M 264.3, m 160-161°, 161-163° (sublimation), $[\alpha]_{287}$ + 24,000°, $[\alpha]_{245}$ -69,000° (c 1-50μg/mL in acidified MeOH or EtOH), pK_{Est} ~3.9.** Crystd from CCl_4-pet.ether, EtOH + hexane and sublimes at 120°.

Acenaphthalene *[208-96-8]* **M 152.2, m 92-93°.** Dissolved in warm redistd MeOH, filtered through a sintered glass funnel and cooled to -78° to ppte the material as yellow plates [Dainton, Ivin and Walmsley *Trans Faraday Soc* **56** 1784 *1960*]. Alternatively can be sublimed *in vacuo.*

Acenaphthaquinone *[82-86-0]* **M 182.2, m 260-261°.** Extracted with, then recrystd twice from *C_6H_6. [LeFevre, Sundaram and Sundaram *J Chem Soc* 974 *1963*].

Acenaphthene *[83-32-9]* **M 154.2, m 94.0°.** Crystd from EtOH. Purified by chromatography from CCl_4 on alumina with *benzene as eluent [McLaughlin and Zainal *J Chem Soc* 2485 *1960*].

RS*-Acenaphthenol** *[6306-07-6]* **M 170.2, m 144.5-145.5°, 146°, 148°.** If highly coloured (yellow), dissolve in boiling *benzene (14g in 200mL), add charcoal (0.5g), filter through a heated funnel, concentrate to 100mL and cool to give almost colourless needles. **Benzene vapour is* **TOXIC** *use an efficient fume cupboard.* The *acetate* has **b** 166-168°/5mm (bath temp 180-185°). [*Org Synth* Col.Vol. III 3 *1955*.] It can also be recrystd from *C_6H_6 or EtOH [Fieser and Cason *J Am Chem Soc* **62** 432 *1940*]. It forms a brick-red crystalline ***complex with 2,4,5,7-tetranitrofluoren-9-one which is recrystd from AcOH and dried in a vacuum over KOH and P_2O_5 at room temp, **m** 170-172° [Newman and Lutz *J Am Chem Soc* **78** 2469 *1956*].

Acetal (acetaldehyde diethylacetal) *[105-57-7]* **M 118.2, b 103.7-104°, d 0.831, n 1.38054, n^{25} 1.3682.** Dried over Na to remove alcohols and water, and to polymerise aldehydes, then fractionally distd. Or, treat with alkaline H_2O_2 soln at 40-45° to remove aldehydes, then the soln is saturated with NaCl, separated, dried with K_2CO_3 and distd from Na [Vogel *J Chem Soc* 616 *1948*].

Acetaldehyde *[75-07-0]* **M 44.1, b 20.2°, d 0.788, n 1.33113, pK^{25} 13.57 (hydrate).** Usually purified by fractional distn in a glass helices-packed column under dry N_2, discarding the first portion of distillate. Or, shaken for 30min with $NaHCO_3$, dried with $CaSO_4$ and fractionally distd at 760mm through a 70cm Vigreux column. The middle fraction was taken and further purified by standing for 2h at 0° with a small amount of hydroquinone, followed by distn [Longfield and Walters *J Am Chem Soc* **77** 810 *1955*].

Acetaldehyde ammonia trimer (hexahydro-2,4,6-trimethyl-1,3,5-triazine trihydrate) *[76231-37-3]* **M 183.3, m 94-96°, 95-97°, 97°, b 110°(partly dec).** Crystd from EtOH-Et_2O. When prepared it separates as the *trihydrate* which can be dried in a vacuum over $CaCl_2$ at room temp to give the anhydrous compound with the same melting point. The *dihydrate* melts at 25-28° then resolidifies and melts again at 94-95°. *IRRITATES THE EYES AND MUCOUS MEMBRANES.* [*J Org Chem* **38** 3288 *1973*.]

Acetaldehyde dimethyl acetal *[534-15-6]* **M 90.1, b 63-65°, d_4^{20} 0.852, n_D^{25} 1.36678.** Distd through a fractionating column and fraction boiling at 63.8°/751mm is collected. It forms an azeotrope with MeOH. It has been purified by GLC.

Acetamide *[60-35-5]* **M 59.1, m 81°, pK_1^{25} -1.4, pK_2^{25} +0.37.** Crystd by soln in hot MeOH (0.8mL/g), diltd with Et_2O and allowed to stand [Wagner *J Chem Educ* 7 1135 *1930*]. Alternate crystns are from acetone, *benzene, chloroform, dioxane, methyl acetate or from *benzene-ethyl acetate mixture (3:1 and 1:1). It has also been recrystd from hot water after treating with HCl-washed activated charcoal (which had been

repeatedly washed with water until free from chloride ions), then crystd again from hot 50% aq. EtOH and finally twice from hot 95% EtOH [Christoffers and Kegeles *J Am Chem Soc* **85** 2562 *1963*]. Final drying is in a vacuum desiccator over P_2O_5. Acetamide is also purified by distn (**b** 221-223°) or by sublimation *in vacuo*. Also purified by recrystn twice from cyclohexane containing 5% (v/v) of *benzene. Needle-like crystals separated by filtn, washed with a small volume of distd H_2O and dried with a flow of dry N_2. [Slebocka-Tilk et al. *J Am Chem Soc* **109** *4620 1987*.]

Acetamidine hydrochloride *[124-42-5]* **M 94.5, m 164-166°, 165-170° (dec), 174°, pK^{25} 12.40.** It can be recrystd from small volumes of EtOH. Alternatively dissolve in EtOH, filter, add Et_2O, filter the crystalline salt off under N_2, dry in a vacuum desiccator over H_2SO_4. The salt is deliquescent and should be stored in a tightly stoppered container. Solubility in H_2O is 10% at room temperature, soluble in Me_2CO. The *free base* reacts strongly alkaline in H_2O. It has λ_{max} 224nm (ε 4000) in H_2O. The *picrate* has **m** 252° (sintering at ~245°). [Dox *Org Synth* Coll Vol I 5 *1941*; Davies and Parsons *Chem Ind (London)* 628 *1958*; Barnes et al. *J Am Chem Soc* **62** 1286 *1940* give **m** 177-178°.]

***N*-(2-Acetamido)-2-aminoethanesulfonic acid (ACES)** *[7365-82-4]* **M 182.2, m > 220°(dec), pK_{Est} ~1.5, pK_2 6.9.** Recrystd from hot aqueous EtOH.

4-Acetamidobenzaldehyde *[122-85-0]* **M 163.2, m 156°.** Recrystd from water.

***p*-Acetamidobenzenesulfonyl chloride (*N*-acetylsulfanilyl chloride)** *[121-60-8]* **M 233.7, m 149°(dec).** Crystd from toluene, $CHCl_3$, or ethylene dichloride.

α-Acetamidocinnamic acid *[5469-45-4]* **M 205.2, m 185-186° ($2H_2O$), 190-191°(anhydr), 193-195°, pK_{Est} ~3.2.** Recrystd from H_2O as the dihydrate and on drying at 100° it forms the anhydrous compound which is *hygroscopic*. Alkaline hydrolysis yields NH_3 and phenylpyruvic acid. [Erlenmeyer and Früstück *Justus Liebigs Ann Chem* **284** 47 *1895*.]

Z-*O*-(2-Acetamido-2-deoxy-D-glycopyranosylideneamino)*N*-phenylcarbamate (PUGNAC) *[132063-05-9]* **M 335.3, m 171-174°(dec), 174-180°(dec), $[\alpha]_D^{20}$ +67.5° (c 0.2, MeOH).** Purified by flash chromatography (silica gel and eluted with AcOEt-hexane 3:2) evaporated, and the foam recrystallised from AcOEt-MeOH. TLC on Merck SiO_2 gel 60 F_{254} and detected by spraying with 0.025M I_2 in 10% aqueous H_2SO_4 and heat at 200° gave R_F 0.21. The acetate is hydrolysed with NH_3-MeOH. [*Helv Chim Acta* **68** 2254 *1985*; **73** 1918 *1990*.]

2-Acetamidofluorene *[53-96-3]* **M 223.3, m 194°, 196-198°.** Recrystd from toluene (1.3mg in 100mL). Solubility in H_2O is 1.3mg/L; UV λ_{max} nm(log ε) : 288(4.43), 313(4.13). [*J Org Chem* **21** 271 *1956*.] It can also be recrystd from 50% AcOH and sol in H_2O is 1.3mg/100mL at 25° [*Chem Ber* **35** 3285 *1902*]. 9-^{14}C and ω-^{14}C 2-acetamidofluorene were recrystd from aqueous EtOH and had **m** 194-195° and 194° respectively. *Potent* **CARCINOGEN.** [*Cancer Res* **10** 616 *1950*; Sadin et al. *J Am Chem Soc* **74** 5073 *1952*.]

***N*-(2-Acetamido)iminodiacetic acid (ADA)** *[26239-55-4]* **M 190.2, m 219° (dec), pK_1 ~2.3, pK_2 6.6.** Dissolved in water by adding one equivalent of NaOH soln (to final pH of 8-9), then acidified with HCl to ppte the free acid. Filtered and washed with water.

Acetamidomethanol *[625-51-4]* **M 89.1, m 47-50°, 54-56°, 55°.** Recryst from freshly distd Me_2CO, wash the crystals with dry Et_2O and dry in a vacuum desiccator over P_2O_5. R_F 0.4 on paper chromatography with $CHCl_3$/EtOH (2:8) as solvent and developed with ammoniacal $AgNO_3$. Also crystallises in needles from EtOAc containing a few drops of Me_2CO. It is *hygroscopic* and should be stored under dry conditions. [*J Am Chem Soc* **73** 2775 *1951*; *Chem Ber* **99** 3204 *1966*; *Justus Liebigs Ann Chem* **343** 265 *1905*.]

2-Acetamido-5-nitrothiazole *[140-40-9]* **M 187.2, m 264-265°.** Recrystd from EtOH or glacial acetic acid.

2-Acetamidophenol *[614-80-2]* **M 151.2, m. 209°, pK_{Est} ~9.4.** Recrystd from water or aqueous EtOH.

3-Acetamidophenol *[621-42-1]* **M 151.2, m 148-149°, pK^{25} ~9.59.** Recrystd from water.

4-Acetamidophenol *[103-90-2]* **M 151.2, m 169-170.5°, pK_{Est} ~10.0.** Recrystd from water or EtOH.

4-Acetamido-2,2,6,6-tetramethylpiperidine-1-oxyl (acetamidoTEMPO) *[14691-89-5]* **M 213.3, m 144-146°, 146-147°.** Dissolve in CH_2Cl_2, wash with saturated K_2CO_3, then saturated aqueous NaCl, dry (Na_2SO_4), filter and evaporate. The red solid is recrystd from aqueous MeOH, **m** 147.5°. [*J Org Chem* **56** 6110 *1991*; *Bull Acad Sci USSR, Div Chem Sci* **15** 1422 *1966*.]

5-Acetamido-1,3,4-thiadiazole-2-sulfonamide *[59-66-5]* **M 222.3, m 256-259° (dec).** Recrystd from water.

Acetanilide *[103-84-4]* **M 135.2, m 114°, pK^{25} 0.5.** Recrystd from water, aqueous EtOH, *benzene or toluene.

Acetic acid (glacial) *[64-19-7]* **M 60.1, m 16.6°, b 118°, d 1.049, n 1.37171, n^{25} 1.36995, pK^{25} 4.76.** Usual impurities are traces of acetaldehyde and other oxidisable substances and water. (Glacial acetic acid is very *hygroscopic*. The presence of 0.1% water lowers its **m** by 0.2°.) Purified by adding some acetic anhydride to react with water present, heating for 1h to just below boiling in the presence of 2g CrO_3 per 100mL and then fractionally distilling [Orton and Bradfield *J Chem Soc* 960 *1924*, 983 *1927*]. Instead of CrO_3, 2-5% (w/w) of $KMnO_4$, with boiling under reflux for 2-6h, has been used.
Traces of water have been removed by refluxing with tetraacetyl diborate (prepared by warming 1 part of boric acid with 5 parts (w/w) of acetic anhydride at 60°, cooling, and filtering off), followed by distn [Eichelberger and La Mer *J Am Chem Soc* **55** 3633 *1933*].
Refluxing with acetic anhydride in the presence of 0.2g % of 2-naphthalenesulfonic acid as catalyst has also been used [Orton and Bradfield *J Chem Soc* 983 *1927*]. Other suitable drying agents include $CuSO_4$ and chromium triacetate: P_2O_5 converts some acetic acid to the anhydride. Azeotropic removal of water by distn with thiophene-free *benzene or with butyl acetate has been used [Birdwhistell and Griswold *J Am Chem Soc* **77** 873 *1955*]. An alternative purification uses fractional freezing.
Rapid procedure: Add 5% acetic anhydride, and 2% of CrO_3. Reflux and fractionally distil.

Acetic anhydride *[108-24-7]* **M 102.1, b 138°, d 1.082, n 1.3904.** Adequate purification can usually be obtained by fractional distn through an efficient column. Acetic acid can be removed by prior refluxing with CaC_2 or with coarse Mg filings at 80-90° for 5days, or by distn from a large excess of quinoline (1% AcOH in quinoline) at 75mm pressure. Acetic anhydride can also be dried by standing with Na wire for up to a week, removing the Na and distilling from it under vacuum. (Na reacts vigorously with acetic anhydride at 65-70°). Dippy and Evans [*J Org Chem* **15** 451 *1950*] let the anhydride (500g) stand over P_2O_5 (50g) for 3h, then decanted it and stood it with ignited K_2CO_3 for a further 3h. The supernatant liquid was distd and the fraction **b** 136-138°, was further dried with P_2O_5 for 12h, followed by shaking with ignited K_2CO_3, before two further distns through a five-section Young and Thomas fractionating column. The final material distd at 137.8-138.0°. Can also be purified by azeotropic distn with toluene: the azeotrope boils at 100.6°. After removal of the remaining toluene, the anhydride is distd [sample had a specific conductivity of 5 x 10^{-9} $ohm^{-1}cm^{-1}$].
Rapid procedure: Shake with P_2O_5, separate, shake with dry K_2CO_3 and fractionally distil.

Acetic hydrazide *[1068-57-1]* **M 74.1, m 67°, b 127°/18mm.** Cryst as needles from EtOH. Reduces $NH_3/AgNO_3$.

Acetoacetamide *[5977-14-0]* **M 101.1, m 54-55°, 54-56°.** Recrystallise from $CHCl_3$, or Me_2CO/pet ether. Crystallises from pyridine with 4mol of solvent. Slightly soluble in H_2O, EtOH and AcOH but

insoluble in Et_2O. *Phenylhydrazone* has **m** 128°. [*Beilstein* **3, 4th Suppl**, p 1545; Kato *Chem Pharm Bull Jpn* **15** 921,923 *1967*; *Chem Ber* **35** 583 *1902*.]

Acetoacetanilide *[102-01-2]* **M 177.2, m 86°, pK 10.68**. Crystd from H_2O, aqueous EtOH or pet ether (b 60-80°).

Acetoacetylpiperidide *[1128-87-6]* **M 169.2, b 88.9°/0.1mm, n^{52} 1.4983.** Dissolved in *benzene, extracted with 0.5M HCl to remove basic impurities, washed with water, dried, and distd at 0.1mm [Wilson *J Org Chem* **28** 314 *1963*].

α-Acetobromoglucose (2,3,4,6-tetraacetyl-α-D-glucopyranosyl bromide) *[572-09-8]* **M 411.2, m 88-89°, $[\alpha]_D^{25}$ +199.3° (c 3, $CHCl_3$).** Crystd from isopropyl ether or pet ether (b 40-60°) [*Org Synth* **65** 236 *1897*].

Acetone *[67-64-1]* **M 58.1, b 56.2°, d 0.791, n 1.35880, pK_1^{25} -6.1 (basic, monoprotonated), pK_2^{25} 20.0 (acidic)** The commercial preparation of acetone by catalytic dehydrogenation of isopropyl alcohol gives relatively pure material. Analytical reagent quality generally contains less than 1% organic impurities but may have up to about 1% H_2O. Dry acetone is appreciably *hygroscopic*. The main organic impurity in acetone is mesityl oxide, formed by the aldol condensation. It can be dried with anhydrous $CaSO_4$, K_2CO_3 or type 4A Linde molecular sieves, and then distd. Silica gel and alumina, or mildly acidic or basic desiccants cause acetone to undergo the aldol condensation, so that its water content is increased by passage through these reagents. This also occurs to some extent when P_2O_5 or sodium amalgam is used. Anhydrous $MgSO_4$ is an inefficient drying agent, and $CaCl_2$ forms an addition compound. Drierite (anhydrous $CaSO_4$) offers the minimum acid and base catalysis of aldol formation and is the recommended drying agent for this solvent [Coetzee and Siao *Inorg Chem* **14v** 2 *1987*; Riddick and Bunger *Organic Solvents* Wiley-Interscience, N.Y., 3rd edn, 1970]. Acetone was shaken with Drierite (25g/L) for several hours before it was decanted and distd from fresh Drierite (10g/L) through an efficient column, maintaining atmospheric contact through a Drierite drying tube. The equilibrium water content is about 10^{-2}M. Anhydrous $Mg(ClO_4)_2$ **should not be used as drying agent because of the risk of EXPLOSION with acetone vapour.**
Organic impurities have been removed from acetone by adding 4g of $AgNO_3$ in 30mL of water to 1L of acetone, followed by 10mL of M NaOH, shaking for 10min, filtering, drying with anhydrous $CaSO_4$ and distilling [Werner *Analyst (London)* **58** 335 *1933*]. Alternatively, successive small portions of $KMnO_4$ have been added to acetone at reflux, until the violet colour persists, followed by drying and distn. Refluxing with chromium trioxide (CrO_3) has also been used. Methanol has been removed from acetone by azeotropic distn (at 35°) with methyl bromide, and treatment with acetyl chloride.
Small amounts of acetone can be purified as the NaI addition compound, by dissolving 100g of finely powdered NaI in 400g of boiling acetone, then cooling in ice and salt to -8°. Crystals of $NaI.3Me_2CO$ are filtered off and, on warming in a flask, acetone distils off readily. [This method is more convenient than the one using the bisulfite addition compound.] Also purified by gas chromatography on a 20% free fatty acid phthalate (on Chromosorb P) column at 100°.
For efficiency of desiccants in drying acetone see Burfield and Smithers [*J Org Chem* **43** 3966 *1978*]. The water content of acetone can be determined by a modified Karl Fischer titration [Koupparis and Malmstadt *Anal Chem* **54** 1914 *1982*].
Rapid procedure: Dry over anhydrous $CaSO_4$ and distil.

Acetone cyanohydrin *[75-86-5]* **M 85.1, b 48°/2.5mm, 68-70°/11mm, 78-82°/15mm, d_4^{20} 0.93.** Dry with Na_2SO_4, and distil as rapidly as possible under vacuum to avoid decomposition. Discard fractions boiling below 78-82°/15mm. Store in the dark. **USE AN EFFICIENT FUME HOOD as HCN (POISONOUS) is always present.** [*Org Synth* Col.Vol. II 7 *1940*.]

Acetonedicarboxylic acid *[542-05-2]* **M 146.1, m 138° (dec), pK^{25} 3.10.** Crystd from ethyl acetate and stored over P_2O_5. Decarboxylates in hot water.

Acetone semicarbazone *[110-20-3]* **M 115.1, m 187°, pK^{25} 1.33.** Crystd from water or from aqueous EtOH.

Acetonitrile (methyl cyanide) *[75-05-8]* **M 41.1, b 81.6°, d^{25} 0.77683, n 1.3441, n^{25} 1.34163.** Commercial acetonitrile is a byproduct of the reaction of propylene and ammonia to acrylonitrile. The following procedure that significantly reduces the levels of acrylonitrile, allyl alcohol, acetone and *benzene was used by Kiesel [*Anal Chem* **52** 2230 *1988*]. Methanol (300mL) is added to 3L of acetonitrile fractionated at high reflux ratio until the boiling temperature rises from 64° to 80°, and the distillate becomes optically clear down to λ = 240nm. Add sodium hydride (1g) free from paraffin, to the liquid, reflux for 10min, and then distil rapidly until about 100mL of residue remains. Immediately pass the distillate through a column of acidic alumina, discarding the first 150mL of percolate. Add 5g of CaH_2 and distil the first 50mL at a high reflux ratio. Discard this fraction, and collect the following main fraction. The best way of detecting impurities is by gas chromatography.

Usual contaminants in commercial acetonitrile include H_2O, acetamide, NH_4OAc and NH_3. Anhydrous $CaSO_4$ and $CaCl_2$ are inefficient drying agents. Preliminary treatment of acetonitrile with cold, satd aq KOH is undesirable because of base-catalysed hydrolysis and the introduction of water. Drying by shaking with silica gel or Linde 4A molecular sieves removes most of the water in acetonitrile. Subsequent stirring with CaH_2 until no further hydrogen is evolved leaves only traces of water and removes acetic acid. The acetonitrile is then fractionally distd at high reflux, taking precaution to exclude moisture by refluxing over CaH_2 [Coetzee *Pure Appl Chem* **13** 429 *1966*]. Alternatively, 0.5-1% (w/v) P_2O_5 is often added to the distilling flask to remove most of the remaining water. Excess P_2O_5 should be avoided because it leads to the formation of an orange polymer. Traces of P_2O_5 can be removed by distilling from anhydrous K_2CO_3.

Kolthoff, Bruckenstein and Chantooni [*J Am Chem Soc* **83** 3297 *1961*] removed acetic acid from 3L of acetonitrile by shaking for 24h with 200g of freshly activated alumina (which had been reactivated by heating at 250° for 4h). The decanted solvent was again shaken with activated alumina, followed by five batches of 100-150g of anhydrous $CaCl_2$. (Water content of the solvent was then less than 0.2%). It was shaken for 1h with 10g of P_2O_5, twice, and distd in a 1m x 2cm column, packed with stainless steel wool and protected from atmospheric moisture by $CaCl_2$ tubes. The middle fraction had a water content of 0.7 to 2mM.

Traces of unsaturated nitriles can be removed by an initial refluxing with a small amount of aq KOH (1mL of 1% solution per L). Acetonitrile can be dried by azeotropic distn with dichloromethane, *benzene or trichloroethylene. Isonitrile impurities can be removed by treatment with conc HCl until the odour of isonitrile has gone, followed by drying with K_2CO_3 and distn.

Acetonitrile was refluxed with, and distd from alkaline $KMnO_4$ and $KHSO_4$, followed by fractional distn from CaH_2. (This was better than fractionation from molecular sieves or passage through a type H activated alumina column, or refluxing with KBH_4 for 24h and fractional distn)[Bell, Rodgers and Burrows *J Chem Soc, Faraday Trans 1* **73** 315 *1977*; Moore et al. *J Am Chem Soc* **108** 2257 *1986*].

Material suitable for polarography was obtained by refluxing over anhydrous $AlCl_3$ (15g/L) for 1h, distilling, refluxing over Li_2CO_3 (10g/L) for 1h and redistg. It was then refluxed over CaH_2 (2g/L) for 1h and fractionally distd, retaining the middle portion. The product was not suitable for UV spectroscopy use. A better purification procedure used refluxing over anhydrous $AlCl_3$ (15g/L) for 1h, distg, refluxing over alkaline $KMnO_4$ (10g $KMnO_4$, 10g Li_2CO_3/L) for 15min, and distg. A further reflux for 1h over $KHSO_4$ (15g/L), then distn, was followed by refluxing over CaH_2 (2g/L) for 1h, and fractional distn. The product was protected from atmospheric moisture and stored under nitrogen [Walter and Ramalay *Anal Chem* **45** 165 *1973*]. Purificaton of "General Purity Reagent" for this purpose is not usually satisfactory because very large losses occur at the $KMnO_4$, $LiCO_3$ step. For electrochemical work involving high oxidation fluorides, further reflux over P_2O_5 (1g/mL for 0.5h) and distilling (discarding 3% of first and last fractions) and repeating this step is necessary. The distillate is kept over molecular sieves in *vac* after degassing, for 24h and vac distd onto freshly activated 3A molecular sieves. The MeCN should have absorption at 200nm of <0.05 (H_2O reference) and UV cutoff at *ca* 175nm. Also the working potential range of purified Et_4N^+ BF_4^- (0.1mol.dcm^{-3} in the MeCN) should be +3.0 to -2.7V *vs* Ag^+/Ag^0. If these criteria are not realised then further impurities can be removed by treatment with activated neutral alumina (60 mesh) *in vacuo* before final molecular sieves treatment [Winfield *J Fluorine Chem* **25** 91 *1984*].

Acetonitrile has been distd from $AgNO_3$, collecting the middle fraction over freshly activated Al_2O_3. After standing for two days, the liquid was distd from the activated Al_2O_3. Specific conductivity 0.8-1.0 x 10^{-8} mhos [Harkness and Daggett *Can J Chem* **43** 1215 *1965*]. *Acetonitrile* ^{14}C was purified by gas chromatography and is water free and distd at 81°. [*J Mol Biol* **87** 541 *1974*.]

Rapid procedure: Dry over anhydrous K_2CO_3 for 24h, followed by further drying for 24h over 3A molecular sieves or boric anhydride, followed by distn. Alternatively, stir over P_2O_5 (5% w/v) for 24h then distil. However this last method is not suitable for use in reactions with very acid sensitive compounds.

Acetonylacetone (2,5-hexanedione) *[110-13-4]* **M 114.2, m -9°, b 76-78°/13mm, 88°/25mm, 137°/150mm, 188°/atm, d_4^{20} 0.9440, n_D^{20} 1.423, pK 18.7.** Purified by dissolving in Et_2O, stirred with K_2CO_3 (a quarter of the wt of dione), filtered, dried over anhydrous Na_2SO_4 (**not $CaCl_2$**), filtered, evapd and distd in a vacuum. It is then redistd through a 30cm Vigreux column (oil bath temp 150°). It is miscible with H_2O and EtOH. The *dioxime* has **m** 137° (plates from *C_6H_6), *mono-oxime* has **b** 130°/11mm, and the *2,4-dinitrophenylhydrazone* has **m** 210-212° (red needles from EtOH). [*Chem Ber* **22** 2100 *1989*; for enol content see *J Org Chem* **19** 1960 *1954*.]

4-Acetophenetidine (phenacetin) *[62-44-2]* **M 179.2, m 136°.** Crystd from H_2O or purified by soln in cold dilute alkali and reppted by addn of acid to neutralisation point. Air-dried.

Acetophenone *[98-86-2]* **M 120.2, m 19.6°, b 54°/2.5mm, 202°/760mm, d^{25} 1.0238, n^{25} 1.5322, pK 19.2.** Dried by fractional distn or by standing with anhydrous $CaSO_4$ or $CaCl_2$ for several days, followed by fractional distn under reduced pressure (from P_2O_5, optional), and careful, slow and repeated partial crystns from the liquid at 0° excluding light and moisture. It can also be crystd at low temperatures from isopentane. Distn can be followed by purification using gas-liquid chromatography [Earls and Jones *J Chem Soc, Faraday Trans 1* **71** 2186 *1975*.]
§ A commercial polystyrene supported version is available — scavanger resin (for diol substrate).

Aceto-*o*-toluidide *[120-66-1]* **M 149.2, m 110°, b 296°/760mm.** Crystd from H_2O, EtOH or aqueous EtOH.

Aceto-*m*-toluidide *[537-92-8]* **M 149.2, m 65.5°, b 182-183°/14mm, 303°/760mm.** Crystd from H_2O, EtOH or aqueous EtOH.

Aceto-*p*-toluidide *[103-89-9]* **M 149.2, m 146°, b 307°/760mm.** Crystd from aqueous EtOH.

Acetoxime (acetone oxime) *[127-06-0]* **M 73.1, m 63°, b 135°/760mm, pK^{40} 0.99.** Crystd from pet ether (b 40-60°). Can be sublimed.

Acetoxyacetone (acetol acetone) *[592-20-1]* **M 116.1, b 65°/11mm, 73-75°/17mm, 174-176°/atm, d_4^{20} 1.0757, n_D^{20} 1.4141.** Distil under reduced pressure, then redistil at atm pressure. It is miscible with H_2O but is slowly decomposed by it. Store in dry atmosphere. The *2,4-dinitrophenylhydrazone* has **m** 115-115.5° (from $CHCl_3$/hexane). [*J Chem Soc* **59** 789 *1891*; *J Org Chem* **21** 68 *1956*; *Justus Liebigs Ann Chem* **335** 260 *1904*.]

4-Acetoxy-2-azetidinone *[28562-53-0]* **M 129.1, m 38-41°.** Dissolve in $CHCl_3$, dry ($MgSO_4$) concentrate at 40°/70mm, or better at room temperature to avoid decomposition. Wash and stir the residual oil with hexane by decantation and discard wash. Dry the oil at high vacuum when it should solidify, **m** 34°. It can be distd at high vacuum, 80-82°/10^{-3}mm, but this results in extensive losses. The purity can be checked by TLC using Merck Silica Gel F_{254} and eluting with EtOAc. The azetidinone has R_F 0.38 (typical impurities have R_F 0.67). The spots can be detected by the TDM spray. This is prepared from (A) 2.5g 4,4'-tetramethyldiaminodiphenylmethane (TDM) in 10mL AcOH and diluted with 50mL of H_2O, (B) 5g KI in 100mL of H_2O and (C) 0.3g ninhydrin in 10mL of AcOH and 90mL of H_2O. The spray is prepared by mixing (A) and (B) with 1.5mL of (C) and stored in a brown bottle. [*Justus Liebigs Ann Chem* 539 *1974*; *Org Synth* **65** 135 *1987*.]

1-Acetoxy-1,3-butadiene (1,3-butadienyl acetate) ***cis-trans*** mixture *[1515-76-0]* **M 112.1, b 42-43°/16mm, 51-52°/20mm, 60-61°/40mm, d $_4^{20}$ 0.9466, n $_D^{20}$ 1.4622.** The commercial sample is stabilised with 0.1% of *p-tert*-butylcatechol. If the material contains crotonaldehyde (by IR, used in its synthesis) it should be dissolved in Et_2O, shaken with 40% aqueous sodium bisulfite, then 5% aqueous

Na_2CO_3, water, dried (Na_2SO_4) and distilled several times in a vac through a Widmer (*Helv Chim Acta* **7** 59 *1924*) or Vigreux column [Wicterle and Hudlicky *Collect Czech Chem Commun* **12** 564 *1947*; Hagemeyer and Hull *Ind Eng Chem* **41** 2920 *1949*].

1-Acetoxy-2-butoxyethane *[112-07-2]* **M 160.2, b 61-62°/0.2mm, 75-76°/12mm, 185.5°/740mm, 188-192°/atm, d_4^{20} 0.9425, n_D^{20} 1.4121.** Shake with anhydrous Na_2CO_3, filter and distil in a vacuum. Redistn can be then be carried out at atmospheric pressure. [*J Org Chem* **21** 1041 *1956*.]

3*R*,4*R*,1'*R*-4-Acetoxy-3-[1-(*tert*-butylmethylsilyloxy)ethyl]-2-azetinone see Chapter 5.

2-Acetoxyethanol *[542-59-6]* **M 104.1, b 187°/761mm, 187-189°/atm, d_4^{20} 1.108, n_D^{20} 1.42.** Dry over K_2CO_3 (**not $CaCl_2$**), and distil. [*J Chem Soc* 3061 1950; rate of hydrolysis: *J Chem Soc* 2706 *1951*.]

1-Acetoxy-2-ethoxyethane *[111-15-9]* **M 132.2, b 156-159°, d_4^{20} 0.97, n_D^{20} 1.406.** Shake with anhydr Na_2CO_3, filter and distil in vac. Redistn can then be carried out at atm pressure. [*J Org Chem* **21** 1041 *1956*.]

1-Acetoxy-2-methoxyethane *[110-49-6]* **M 118.1, b 141°/732mm, 140144°/atm, d_4^{20} 1.009, n_D^{20} 1.4011.** Shake with anhydrous Na_2CO_3, filter and distil in a vacuum. Redistn can be then be carried out at atmospheric pressure. [*J Org Chem* **21** 1041 *1956*.]

***R*-(-)-α-Acetoxyphenylacetic (acetyl mandelic) acid** *[51019-43-3]* **M 194.2, m 96-98°, $[\alpha]_D^{20}$ -153.7° (c 2.06, Me_2CO), $[\alpha]_{546}^{20}$ -194° (c 2.4, Me_2CO), pK_{Est} ~2.9** Recrysts from H_2O with 1mol of solvent which is removed on drying, or from solvents as for the *S*-isomer. [*J Chem Soc* 227 *1943*.]

***S*-(+)-α-Acetoxyphenylacetic (acetyl mandelic) acid** *[7322-88-5]* **M 194.2, m 80-81°, 95-97.5°, $[\alpha]_D^{27}$ +158° (c 1.78, Me_2CO), $[\alpha]_{546}^{20}$ +186° (c 2, Me_2CO).** Recryst from *benzene-hexane or toluene and has characteristic NMR and IR spectra. [*Justus Liebigs Ann Chem* **622** 10 *1959*; *J Org Chem* **39** 1311 *1974*.]

21-Acetoxypregnenolone *[566-78-9]* **M 374.5, m 184-185°.** Crystd from Me_2CO.

***S*-(-)-2-Acetoxypropionyl chloride** *[36394-75-9]* **M 150.6, b 51-53°/11mm, d_4^{20} 1.19, n_D^{20} 1.423, $[\alpha]_D^{27}$ -33°, (c 4, $CHCl_3$), $[\alpha]_{546}^{20}$ -38° (c 4, $CHCl_3$).** It is moisture sensitive and is hydrolysed to the corresponding acid. Check the IR spectrum. If the OH band above 3000cm $^{-1}$ is too large and broad then the mixture should be refluxed with pure acetyl chloride for 1h, evapd and distd under reduced pressure.

***S*-Acetoxysuccinic anhydride** *[59025-03-5]* **M 158.1, m 58° (*RS* 81.5-82.5°, 86-87°), $[\alpha]_D^{20}$ -26.0° (c 19, Me_2CO), $[\alpha]_D^{20}$ -28.4° (c 13, Ac_2O).** Recrystd from Ac_2O and dry in a vacuum over KOH, or by washing with dry Et_2O due to its deliquescent nature. [*J Chem Soc* 788 *1933; Synth Commun* **16** 183 *1986*; *J Org Chem* **52** 1040 *1988*; *RS* : *J Am Chem Soc* **88** 5306 *1966*.]

Acetylacetone (2,4-pentanedione) *[123-54-6]* **M 100.1, b 45°/30mm, $d^{30.2}$ 0.9630, $n^{18.5}$ 1.45178, pK_1^{25} -5.0 (enol), -6.6 (keto), pK_2^{25} 8.95** Small amounts of acetic acid were removed by shaking with small portions of 2M NaOH until the aqueous phase remained faintly alkaline. The sample, after washing with water, was dried with anhydrous Na_2SO_4, and distd through a modified Vigreux column [Cartledge *J Am Chem Soc* **73** 4416 *1951*]. An additional purification step is fractional crystn from the liquid. Alternatively, there is less loss of acetylacetone if it is dissolved in four volumes of *benzene and the soln is shaken three times with an equal volume of distd water (to extract acetic acid): the *benzene is then removed by distn at 43-53° and 20-30mm through a helices-packed column. It is then refluxed over P_2O_5 (10g/L) and fractionally distd under reduced pressure. The distillate (sp conductivity 4 x 10^{-8} $ohm^{-1}cm^{-1}$) was suitable for polarography [Fujinaga and Lee *Talanta* **24** 395 *1977*]. To recover used acetylacetone, metal ions were stripped from the soln at pH 1 (using 100mL 0.1M H_2SO_4/L of acetylacetone). The acetylacetone was washed with

(1:10) ammonia soln (100mL/L) and with distd water (100mL/L, twice), then treated as above. It complexes with Al, Be, Ca, Cd, Ce , Cu, Fe^{2+}, Fe^{3+}, Mn, Mg, Ni, Pb and Zn.

***N*-Acetyl-L-alaninamide** *[15962-47-7]* **M 130.2, m 162°.** Crystd repeatedly from EtOH-diethyl ether.

***N*-Acetyl-ß-alanine** *[3025-95-4]* **M 127.2, m 78.3-80.3°, pK^{25} 4.45.** Crystd from acetone.

***N*-Acetyl-L-alanyl-L-alaninamide** *[30802-37-0]* **M 201.2, m 250-251°.** Crystd repeatedly from EtOH/diethyl ether.

***N*-Acetyl-L-alanyl-L-alanyl-L-alaninamide** *[29428-34-0]* **M 272.3, m 295-300°.** Crystd from MeOH/diethyl ether.

***N*-Acetyl-L-alanylglycinamide** *[76571-64-7]* **M 187.2, m 148-149°.** Crystd repeatedly from EtOH/diethyl ether.

Acetyl-α-amino-*n*-butyric acid *[34271-24-4]* **M 145.2, pK^{25} 3.72.** Crystd twice from water (charcoal) and air dried [King and King *J Am Chem Soc* **78** 1089 *1956*].

9-Acetylanthracene *[784-04-3]* **M 220.3, m 75-76°.** Crystd from EtOH. [Masnori et al. *J Am Chem Soc* **108** 1126 *1986*.]

***N*-Acetylanthranilic acid** *[89-52-1]* **M 179.1, m 182-184°, 185-186°, 190°(dec), pK^{20} 3.61.** Wash with distilled H_2O and recrystallise from aqueous AcOH, dry and recrystallise again from EtOAc. Also recryst from water or EtOH. [*J Chem Soc* 2495 *1931*; *J Am Chem Soc* **77** 6698 *1955*.]

2-Acetylbenzoic acid *[577-56-0]* **M 164.2, m 115-116°, 116-118°, pK^{25} 4.10.** Recrystallises from $*C_6H_6$ and H_2O (15g/100mL). The *oxime* has **m** 156-157°, and the *2,4-dinitrophenylhydrazone* has **m** 185-186°(needles from EtOH). [*J Am Chem Soc* **69** 1547 *1947*.]

4-Acetylbenzoic acid *[586-89-0]* **M 164.2, m 207.5-209.5°, 208.6-209.4°, pK^{25} 3.70, 5.10 (EtOH).** Dissolve in 5% aqueous NaOH, extract with Et_2O, and acidify the aqueous soln. Collect the ppte, and recrystallise from boiling H_2O (100 parts) using decolorising charcoal [*J Org Chem* **24** 504 *1959*; *J Chem Soc* 265 *1957*; *J Am Chem Soc* **72** 2882 *1050*, **74** 1058 *1952*].

Acetylbenzonitrile *[1443-80-7]* **M 145.2, m 57-58°.** Recrystd from EtOH [Wagner et al. *J Am Chem Soc* **108** 7727 *1986*].

4-Acetylbiphenyl *[92-91-1]* **M 196.3, m 120-121°, b 325-327°/760mm.** See 4'-phenylacetophenone on p. 327.

Acetyl-5-bromosalicylic acid *[1503-53-3]* **M 259.1, m 168-169°, pK_{Est} ~3.0.** Crystd from EtOH.

2-Acetylbutyrolactone *[517-23-7]* **M 128.1, b 105°/5mm, 120-123°/11mm, 142-143°/30mm, d_4^{20} 1.1846, n_D^{20} 1.459.** Purified by distillation, which will convert any free acid to the lactone, alternatively dissolve in Et_2O, wash well with 0.5N HCl, dry the organic layer and distil. The solubility in H_2O is 20% v/v. The *2,4-dinitrophenylhydrazone* forms orange needles from MeOH, **m** 146°. The lactone hydrolyses in mineral acid to 2-acetyl-4-hydroxybutyric acid which can be converted to the *di-n-propylamine salt* with **m** 68-70°. The lactone is a **SKIN IRRITANT**. [*Yakugaku Zasshi (J Pharm Soc Japan)* **62** 417(439) *1942*; *Helv Chim Acta* **35** 2401 *1952*.]

Acetyl chloride *[75-36-5]* **M 78.5, b 52°, d 1.1051, n 1.38976.** Refluxed with PCl_5 for several hours to remove traces of acetic acid, then distd. Redistd from one-tenth volume of dimethylaniline or quinoline to remove free HCl. A.R. quality is freed from HCl by pumping it for 1h at -78° and distg into a trap at -196°.

Acetyl bromide *[506-96-7]* **M 123.0, b 76-77°, d 1.65.** Boiled with PBr_3/Ac_2O for 1h then distd off and redistd. Store dry. [Burton and Degering *J Am Chem Soc* **62** 227 *1940.*] **LACHRYMATORY.**

Acetylcyclohexane (cyclohexyl methylketone) *[823-76-7]* **M 126.2, b 64°/11mm, 76.2-77°/25mm, d_4^{20} 0.9178, n_D^{20} 1.4519.** Dissolve in Et_2O, shake with H_2O, dry, evaporate and fractionate under reduced pressure. [UV: *J Am Chem Soc* **74** 518 *1952*; enol content: *J Org Chem* **19** 1960 *1954.*] The *semicarbazone* has **m** 174°; the *2,4-dinitrophenylhydrazone* has **m** 139-140° [*Helv Chim Acta* **39** 1290 *1956*].

2-Acetylcyclohexanone *[874-23-7]* **M 140.2, m -11°, b 62-64°/2.5mm, 95-98°/10mm, 111-112°/18mm, d_4^{20} 1.08, n_D^{20} 1.51.** Dissolve in ligroin (b 30-60°), wash with saturated aqueous $NaHCO_3$ dry over Drierite and fractionate in a vacuum. [*J Am Chem Soc* **75** 626, 5030 *1953*; *Chem Ber* **87** 108 *1954.*] It forms a *Cu salt* which crystallises in green leaflets from EtOH, **m** 162-163° [UV: *J Chem Soc* 4419 *1957*].

2-Acetylcyclopentanone *[1670-46-8]* **M 126.2, b. 72-75°/8mm, 82-86°/12mm, 88°/18mm, d_4^{20} 1.043, n_D^{20} 1.490.** Dissolve in pet ether (b 30-60°), wash with satd aq $NaHCO_3$, dry over Drierite and fractionate in a vacuum. It gives a violet colour with ethanolic $FeCl_3$ and is only slowly hydrolysed by 10% aq KOH but rapidly on boiling to yield 6-oxoheptanoic acid. [*J Am Chem Soc* **75** 5030 *1953*; *J Chem Soc* 4232 *1956*; UV: *J Am Chem Soc* **81** 2342 *1959* .] It gives a gray green *Cu salt* from Et_2O-pentane, **m** 237-238° [*J Am Chem Soc* **79** 1488 *1957*].

Acetyldigitoxin-α *[25395-32-8]* **M 807.0, m 217-221°, $[\alpha]_D^{20}$+5.0 (c 0.7, pyridine).** Crystd from MeOH as plates.

Acetylene *[74-86-2]* **M 26.0, m -80.8°, b -84°, pK ~25.** If very impure it should be purified by successive passage through spiral wash bottles containing, in this order, satd aq $NaHSO_4$, H_2O, 0.2M iodine in aq KI (two bottles), sodium thiosulfate soln (two bottles), alkaline sodium hydrosulfite with sodium anthraquinone-2-sulfonate as indicator (two bottles), and 10% aqueous KOH soln (two bottles). The gas was then passed through a Dry-ice trap and two drying tubes, the first containing $CaCl_2$, and the second, Dehydrite $[Mg(ClO_4)_2]$ [Conn, Kistiakowsky and Smith *J Am Chem Soc* **61** 1868 *1939*]. Acetone vapour can be removed from acetylene by passage through H_2O, then concd H_2SO_4, or by passage through two gas traps at -65° and -80°, concd H_2SO_4 and a soda lime tower, a tower of 1-mesh Al_2O_3 then into H_2SO_4 [*Org Synth* Coll Vol **1** 229 *1941*, **3** 853 *1955*; **4** 793 *1963*]. Sometimes it contains acetone and air. These can be removed by a series of bulb-to-bulb distns, e.g. a train consisting of a conc H_2SO_4 trap and a cold EtOH trap (-73°), or passage through H_2O and H_2SO_4, then over KOH and $CaCl_2$. [See Brandsma *Preparative Acetylenic Chemistry*, 1st Edn Elsevier 1971, for pK p15, ISBN 0444409475; 2nd Edn Elsevier 1988, ISBN 0444429603, and Chapter 5 for sodium acetylide.] It is also available commercially as 10ppm in helium, and several concentrations in N_2 for instrument calibration.
Sodium acetylide *[1066-26-8]* M 48.0, was prepd by dissolving Na (23g) in liquid NH_3 (1L) and bubbling acetylene until the blue color was discharged (ca 30min) and evapd to dryness [Saunders *Org Synth* Coll Vol III 416 *1955*]; and is available commercially as a suspension in xylene/light mineral oil. [See entry in Chapter 5.]

Acetylenedicarboxamide *[543-21-5]* **M 112.1, m 294°(dec).** Crystd from MeOH.

Acetylenedicarboxylic acid *[142-45-0]* **M 114.1, m 179°(anhydrous), pK_1^{19} 1.04, pK_2^{19} 2.50.** Crystd from aqueous ether as dipicrate. For mono K salt see entry in Chapter 5.

***N*-Acetylethylenediamine** *[1001-53-2]* **M 102.1, m 50-51°, 51°, b 128°/3mm, 125-130°/5mm, 133-139°/27mm, pK^{25} 9.28.** It has been fractionated under reduced pressure and fraction **b** 125-130°/5mm was refractionated; fraction **b** 132-135°/4mm was collected and solidified. It is a low melting *hygroscopic* solid which can be recrystd from dioxane-Et_2O. It is soluble in H_2O, Et_2O and $*C_6H_6$. The *p-toluenesulfonate salt* can be recrystd from EtOH-EtOAc 1:8, has **m** 125-126° but the free base cannot be recovered from it by basifying and extracting with CH_2Cl_2.The *picrate* has **m** 175° (from EtOH) [*J Am Chem Soc* **63** 853 *1941*, **78** 2570 *1956*].

2-Acetylfluorene *[781-73-7]* **M 208.3, m 132°.** Crystd from EtOH.

Acetyl fluoride *[557-99-3]* **M 62.0, b 20.5°/760mm, d 1.032.** Purified by fractional distn.

***N*-Acetyl-D-galactosamine** *[14215-68-0]* **M 221.2, m 160-161°, $[\alpha]_{546}$ +102° (c 1, H_2O).** Crystd from MeOH/Et_2O.

***N*-Acetyl-D-glucosamine** *[7512-17-6]* **M 221.2, m *ca* 215°, $[\alpha]_{546}$ +49° after 2h (c 2, H_2O).** Crystd from MeOH/Et_2O.

***N*-Acetylglutamic acid** *[1188-37-0]* **M 189.2, m 185° (*RS*); 201° (*S*), $[\alpha]^{25}$ -16.6° (in H_2O), $pK_{Est\ (1)}$ ~3.4, $pK_{Est(2)}$ ~4.3.** Likely impurity is glutamic acid. Crystd from boiling water.

***N*-Acetylglycinamide** *[2620-63-5]* **M 116.1, m 139-139.5°.** Repeated crystn from EtOH/Et_2O. Dried in a vacuum desiccator over KOH.

***N*-Acetylglycine** *[543-24-8]* **M 117.1, m 206-208°, pK_1^{25} -1.92, pK_2^{25} 3.69.** Treated with acid-washed charcoal and recrystd three times from water or EtOH/Et_2O and dried *in vacuo* over KOH [King and King *J Am Chem Soc* **78** 1089 *1956*].

***N*-Acetylglycyl-L-alaninamide** *[34017-20-4]* **M 175.2.** Repeated crystn from EtOH/Et_2O. Dried in a vacuum desiccator over KOH.

***N*-Acetylglycylglycinamide** *[27440-00-2]* **M 173.2, m 207-208°.** Repeated crystn from EtOH/Et_2O. Dried in a vacuum desiccator over KOH.

***N*-Acetylglycylglycylglycinamide** *[35455-24-4]* **M 230.2, m 253-255°.** Repeated crystn from EtOH/Et_2O. Dried in a vacuum desiccator over KOH.

***N*-Acetylhistidine (H_2O)** *[39145-52-3]* **M 171.2, m 148° (*RS*); 169° (*S*) $[\alpha]^{25}$ +46.2° (H_2O).** Likely impurity is histidine. Crystd from water, then 4:1 acetone:water.

***N*-Acetyl-*RS*-homocysteine thiolactone (Citiolone)** *[1195-16-0] [17896-21-8 for ±]* **M 159.2, m 110°, 109-111°, 111.5-112.5°.** Dry in a vacuum desiccator and recrystallise from toluene as needles. It is a ninhydrin -ve substance which gives a "slow" nitroprusside test. λ_{max} 238nm (ε 4,400 $M^{-1}cm^{-1}$); ν (nujol) 1789s and 851ms cm^{-1}. [*J Am Chem Soc* **78** 1597 *1956*; *J Chem Soc* 2758 *1963*.]

***N*-Acetylimidazole** *[2466-76-4]* **M 110.1, m 101.5-102.5°, pK^{25} 3.6.** Crystd from isopropenyl acetate. Dried in a vacuum over P_2O_5.

3-Acetylindole *[703-80-0]* **M 159.2, m 188-190°, 191-193°, 194°, pK^{25} 12.99 (acidic).** Recrystd from MeOH or *C_6H_6 containing a little EtOH. The *phenylureido* derivative has **m** 154°. [*J Chem Soc* 461 *1946*.]

Acetyl iodide *[507-02-8]* **M 170.0, b 108°/760mm.** Purified by fractional distn.

***N*-Acetyl-L-leucinamide** *[28529-34-2]* **M 177.2, m 133-134°.** Recrystd from $CHCl_3$ and pet ether (b 40-60°).

3-(S-Acetylmercapto)isobutyric acid *[RS 33325-40-5]* **M 162.2, m 40-40.5°, b *c a* 120°/1.25mm, pK_{Est} ~4.0.** Distil under vacuum and recrystd from *C_6H_6. [*Chem Abstr* **38** 3616 *1944*.]

Acetyl methanesulfonate *[5539-53-7]* **M 170.2, b <120°/<0.01mm.** The main impurity is methanesulfonic acid. Reflux with redistd acetyl chloride for 6-10h, i.e. until no further HCl is absorbed in a trap, and exclude moisture. Dist off excess of AcCl and carefully dist below 0.001mm with the bath temp below 120° to give the anhydride as a pale yellow oil which solidifies below 0°. Below ~130° it decomp to the

disulfonic anhydride and above ~130° polymers are formed. It is used for cleaving ethers [Prep, IR, NMR: Karger and Mazur *J Org Chem* **36** 528, 532 *1971*].

***N*-Acetyl-L-methionine** *[65-82-7]* **M 191.3, m 104°, $[\alpha]_{546}$ -24.5° (c 1, in H_2O), pK_{Est} ~3.4.** Crystd from water or ethyl acetate. Dried in a vacuum over P_2O_5.

Acetylmethionine nitrile *[538-14-7]* **M 172.3, m 44-46°.** Crystd from diethyl ether.

5-Acetyl-2-methoxybenzaldehyde *[531-99-7]* **M 166.2 , m 144°.** Crystd from EtOH or Et_2O.

***N*-Acetyl-*N'*-methyl-L-alanimide** *[19701-83-8]* **M 144.2.** Crystd from EtOAc/Et_2O, then from EtOH and Et_2O.

4-Acetyl-1-methyl-1-cyclohexene *[6090-09-1]* **M 138.2, b 73-75°/7.5mm, 85-86°/13mm, 94-94.7°/20mm, 204.5-206°/747mm, d_4^{20} 1.0238, n_D^{20} 1.469.** Purified by fractionation under reduced pressure *in vacuo*, and when almost pure it can be fractionated at atmospheric pressure, preferably in an inert atm. Forms two *semicarbazones* one of which is more soluble in $*C_6H_6$, and both can be recryst from EtOH, more soluble has **m** 149°(151°), and the less soluble has **m** 172-175°(191°). *4-Nitrophenylhydrazone* has **m** 166-167° and the *2,4-dinitrophenylhydrazone* has **m** 114-115°. [*Helv Chim Acta* **17** 129, 140 *1934*; *Justus Liebigs Ann Chem* **564** 109 *1949*.]

***N*-Acetyl-6*N'*-methylglycinamide** *[7606-79-3]* **M 130.2.** Recrystd from EtOH/Et_2O mixture.

***N*-Acetyl-6*N'*-methyl-L-leucine amide** *[32483-15-1]* **M 186.3.** Recrystd from EtOH/hexane mixture.

4-Acetylmorpholine *[1696-20-4]* **M 129.2, m 13.8-14°, 14°, 14.5°, b 96-97°/6mm, 113-128°/22mm, 242-247°/760mm, d_4^{20} 1.0963, n_D^{20} 1.4830.** Distd through an 8inch Fenske (glass helices packing) column with a manual take-off head. Purified by fractional distn. The *hydrobromide* has **m** 172-175°. [*J Am Chem Soc* **75** 357 *1953*, *J Org Chem* **21** 1072 *1956*.]

1-Acetylnaphthalene *[941-98-0]* **M 170.1, m 10.5°, b 93-95°/0.1mm, 167°/12mm, 302°/atm, d_4^{20} 1.12, pK -6.22 (H_0 scale, aq H_2SO_4).** If the NMR spectrum indicates the presence of impurities, probably 2-acetylnaphthalene, convert the substance to its picrate by dissolving in *benzene or EtOH and adding excess of satd picric acid in these solvents until separation of picrates is complete. Recryst the picrate till **m** is 118°. Decompose the picrate with dil NaOH and extract with Et_2O. Dry the extract (Na_2SO_4), filter, evap and dist. The *2,4-dinitrophenylhydrazone* crysts from EtOH and has **m** 259°. [*Justus Liebigs Ann Chem* **380** 95 *1911*; *J Am Chem Soc* **61** 3438 *1939*.]

2-Acetylnaphthalene (2-acetonaphthenone, ß-Acetonaphthone, 2-acetonaphthalene, methyl-2-naphthylketone) *[93-08-3]* **M 170.2, m 52-53°, 55°, 55.8°, b 164-166°/8mm, 171-173°/17mm, 301-303°/atm, pK -6.16 (H_0 scale, aq H_2SO_4).** Separated from the 1-isomer by fractional crystn of the picrate in EtOH (see entry for the 1-isomer above) **m** 82°. Decomposition of the picrate with dil NaOH and extraction with Et_2O then evaporation gives purer 2-acetylnaphthalene. If this residue solidifies it can be recrystd from pet ether, EtOH or acetic acid; otherwise it should be distild in a vac and the solid distillate is recrystd [Gorman and Rodgers *J Am Chem Soc* **108** 5074 *1986*; Levanon et al. *J Phys Chem* **91** 14 *1987*]. Purity should be checked by high field NMR spectroscopy. *Oxime* has **m** 145° decomp, and the *semicarbazone* has **m** 235°. [*Justus Liebigs Ann Chem* **380** 95 *1911*; *J Am Chem Soc* **72** 753 and 5626 *1950*, *J Org Chem* **5** 512 *1940*.]

***N*-Acetyl-D-penicillamine** *[15537-71-0]* **M 191.3, m 189-190° (dec), $[\alpha]_D$ +18° (c 1, in 50% EtOH).** See *N*-acetyl penicillamine on p. 507 in Chapter 6.

***N*-Acetyl-L-phenylalanine** *[2018-61-3]* **M 207.2, m 170-171°, $[\alpha]_D$ +41° (c 1, EtOH), (DL) m 152.5-153°, pK_{Est} 3.5.** Crystd from $CHCl_3$ and stored at 4°. (DL)-isomer crystd from water or acetone.

***N*-Acetyl-L-phenylalanine ethyl ester** *[2361-96-8]* **M 235.3, m 93-94°.** Crystd from aq EtOH or H_2O. [Izumiya and Fruton *J Biol Chem* **218** 59 *1956*.]

1-Acetyl-2-phenylhydrazine *[114-83-0]* **M 150.2, m 128.5°, pK^{25} 1.3.** Crystd from aq EtOH.

1-Acetylpiperazine *[13889-98-0]* **M 128.2, m 32-34°, 52°, pK^{25} 7.94.** Purified by recrystn from 40% aqueous EtOH or from EtOH-Et_2O. It is an **irritant,** and is *hygroscopic*. The *hydrochloride* has **m** 191° (from EtOH), and the *tosylate* has **m** 148-149° (from EtOH-EtOAc, 1:16). The free base, however, cannot be isolated by basifying the tosylate salt and extractn with CH_2Cl_2. [*Chem Ber* **66** 113 *1933*; *J Am Chem Soc* **75** 4949 *1953*, 2570 **78** *1956*.]

1-Acetyl-4-piperidone *[32161-06-1]* **M 141.2, b 124-128°/0.2mm, 218°/760mm, d_4^{25} 1.1444, n_D^{25} 1.5023.** Purified by fractional distn through a short Vigreux column (15mm). The *2,4-dinitrophenylhydrazone* has **m** 212-213° (from EtOH). It is freely soluble in H_2O but insoluble in Et_2O. [*J Am Chem Soc* 901 **71** *1949*.]

3-Acetylpyridine *[350-03-8]* **M 121.1, m 13-14°, b 65-66°/1mm, 92-95°/8-9mm, 105°(113°)/16mm, 219-221°/760mm, d_4^{20} 1.1065, n_D^{20} 1.1065, pK^{25} 3.18.** It is purified by dissolving in HCl, extracting with Et_2O to remove the possible impurity of nicotinic acid, basified with NaOH and extracted with Et_2O. The dried extract is filtered, evaporated and the residual oil distd. If the NMR spectrum indicates further impurities then convert to the *phenylhydrazone* (**m** 137°, yellow needles from EtOH). This is hydrolysed with HCl [*Chem Ber* **22** 597 *1889*], the phenylhydrazine HCl is removed by filtration, $NaNO_2$ is added, the soln is basified with aq NaOH and extracted with Et_2O as before and distd at atmospheric pressure to give 3-acetylpyridine as a colourless oil. Purification can be achieved by shaking with 50% aq KOH, extracting with Et_2O, drying the extract and distilling at atmospheric pressure or in a vacuum. [*J Am Chem Soc* **79** 4226 *1957*]. The *hydrochloride* has **m** 180-181° (from MeOH-EtOH), the *picrate* has **m** 133.8-134.8° (from H_2O), and the *phenylhydrazone* has **m** 137° (129-130)° (from EtOH) [*J Am Chem Soc* **71** 2285 1949]. The *ketoxime* has **m** 112° (from EtOH or $*C_6H_6$. [*J Am Chem Soc* **55** 816 *1933*, **63** 490 *1941*, **67** 1468 *1945*, **79** 4226 *1957*.]

Acetylsalicylic acid (Aspirin) *[50-78-2]* **M 180.2, m 133.5-135°, pK^{25} 3.38, (pK^{17} 4.56).** Crystd twice from toluene, washed with cyclohexane and dried at 60° under vacuum for several hours [Davis and Hetzer *J Res Nat Bur Stand* **60** 569 *1958*]. Has also been recrystd from isopropanol and from diethyl ether/pet ether (b 40-60°).

***O*-Acetylsalicyloyl chloride** *[5538-51-2]* **M 198.6, m 45°, 46-49°, 48-52°, b 107-110°/0.1mm, 135°/12mm, n_D^{20} 1.536.** Check first the IR to see if an OH frequency is present. If so then some free acid is present. Then reflux with acetyl chloride for 2-3h and fractionate at high vac. The distillate should crystallise. It can be recryst from hexane. [*J Chem Soc* **89** 1318 *1906*.]

***O*-Acetylsalicylsalicylic acid** *[530-75-6]* **M 300.3, m 159°.** Crystd from dilute acetic acid.

***N*-(4)-Acetylsulfanilamide** *[144-80-9]* **M 214.2, m 216°.** Crystd from aqueous EtOH.

2-Acetylthiazole *[24295-03-2]* **M 127.2, b 89-91° (90-95°)/12mm, 95-105°/15mm, d_4^{20} 1.23, n_D^{20} 1.55.** Check NMR spectrum, if not too bad, distil through an efficient column in a vacuum. The *oxime* sublimes at 140-145°, **m** 159° (cryst from H_2O) has **m** 163-165.5° [*Helv Chim Acta* **31** 1142 *1948*; *J Am Chem Soc* **79** 4524 *1957*; *Helv Chim Acta* ***40*** 554 *1957*].

2-Acetylthiophene (methyl 2-thienyl ketone) *[88-15-3]* **M 126.2, m 9.2-10.5°, 10.45°, 10-11°, b 77°/4mm, 89-91°/9mm, 94.5-96.5°/13mm, 213-214°/atm, d_4^{20} 1.17, n_D^{20} 1.5666.** Fractionally distd through a 12 plate column and fraction **b** 77°/4mm was collected. Also wet the acetylthiophene in order to remove and free thiophene which forms an azeotrope with H_2O, **b** 68°, Store in a brown bottle and the clear colourless liquid remains thus for extended periods. [*Org Synth* **28** 1 *1948*; *J Am Chem Soc* **69** 3093 *1947*.] The red *4-nitrophenylhydrazone* crysts from EtOH, **m** 181-182°.

3-Acetylthiophene (methyl 3-thienyl ketone) *[1468-83-3]* **M 126.2, m 57°, 60-63°, b 106-107°/25mm, 208-210°/748mm.** Recrystd from pet ether (b 30-60°) or EtOH. *2,4-dinitrophenylhydrazone* crystallises from $CHCl_3$, **m** 265°, and the *semicarbazone* crystallises from EtOH, **m** 174-175°. [*J Am Chem Soc* **70** 1555 *1948*.]

***N*-Acetylthiourea** *[591-08-2]* **M 118.2, m 164-165°, 165-168°.** Recrystd from AcOH, the solid is washed with Et_2O and dried in air then at 100°. [*Collect Czech Chem Commun* **24** 3678 *1959*.]

Acetyl *p*-toluenesulfonate *[26908-82-7]* **M 214.2, m 54-56°.** The most likely impurity is *p*-toluenesulfonic acid (could be up to 10%). This can be removed by dissolving in dry Et_2O and cooling until the anhydride crystallises out. It decomp on heating; below ~130° it gives the disulfonic anhydride and above ~130° polymers are formed. It is used for cleaving ethers [Prep, IR, NMR: Karger and Mazur *J Org Chem* **36** 528, 532 *1971*].

1-*O*-Acetyl-2,3,5-tri-*O*-benzoyl-β-D-ribofuranose *[6974-32-9]* **M 504.5, m 128-130°, 130-131°, 131-132°, $[\alpha]_D^{20}$ +44.2° (c 1, $CHCl_3$).** Recrystd from EtOH or isoPrOH. [*Helv Chim Acta* **42** 1171 *1959*; NMR: *J Org Chem* **33** 1799 *1968*; IR: *Chem Pharm Bull Jpn* **11** 188 *1963*.]

***N*-Acetyltryptophan M 246.3,** *[87-32-1]* **m 206° (RS), pK_{Est} ~3.8;** *[1218-34-4]* **m 188° (S), $[\alpha]^{25}$ +30.1° (aq NaOH).** Likely impurity is tryptophan. Crystd from EtOH by adding water.

***N*-Acetyl-L-valine amide** *[37933-88-3]* **M 158.2, m 275°.** Recrystd from CH_3OH/Et_2O.

***cis*-Aconitic acid** *[585-84-2]* **M 174.1, m 126-129°(dec).** Crystd from water by cooling (sol: 1g in 2mL of water at 25°). Dried in a vacuum desiccator.

***trans*-Aconitic acid (1,2,3-propenetriscarboxylic acid)** *[4023-65-8]* **M 174.1, m 195°(dec), m 198-199°(dec), 204-205°(dec), pK_1^{25} 2.81, pK_2^{25} 4.46.** Purified by dissolving in AcOH (77g/150mL), filtering and cooling. The acid separates (55g) as colourless needles. A further quantity (10g) can be obtained by reducing the vol of the filtrate. The acid is dried in air then in a vacuum desiccator over NaOH. The acid can be recrystd from Me_2CO-$CHCl_3$. The highest **m** is obtained with the very dry acid. The **m** (209°) is obtained on a Dennis bar [*J Am Chem Soc* **52** 3128 *1930*, *Org Synth* Coll Vol II 12 *1943*].

***cis*-Aconitic anhydride** *[6318-55-4]* **M 156.1, m 75°, 76-78°, 78-78.5°.** Reflux in xylene (7.5 parts) for 1h, then evaporate and recrystallise the residue from *C_6H_6. Alternatively, reflux in Ac_2O, evaporate and recrystallise from *C_6H_6. It is sensitive to moisture. [IR: *Acta Chem Scand 21* 291 *1967*, *Chem Ber* **61** 2523 *1928*; NMR: *Biochemistry* **5** 2335 *1966*.]

Aconitine *[302-27-2]* **M 645.8, m 204°, $[\alpha]_{546}$ +20° (c 1, $CHCl_3$), pK^{15} 8.35.** Crystd from EtOH, $CHCl_3$ or toluene.

Aconitine hydrobromide *[6034-57-7]* **M 726.7, m 207°.** Crystd from water or EtOH/ether.

Acridine (2,3-benzoquinoline) *[260-94-6]* **M 179.2, m 111° (sublimes), b 346°, pK 5.58 (pK^{25} of excited state 10.65).** Crystd twice from *benzene/cyclohexane, or from aqueous EtOH, then sublimed, removing and discarding the first 25% of the sublimate. The remainder was again crystd and sublimed, discarding the first 10-15% [Wolf and Anderson *J Am Chem Soc* **77** 1608 *1955*].
Acridine can also be purified by crystn from *n*-heptane and then from ethanol/water after pre-treatment with activated charcoal, or by chromatography on alumina with pet ether in a darkened room. Alternatively, acridine can be ppted as the hydrochloride from *benzene soln by adding HCl, after which the base is regenerated, dried at 110°/50mm, and crystd to constant melting point from pet ether [Cumper, Ginman and Vogel *J Chem Soc* 4518 *1962*]. The regenerated free base may be recrystd, chromatographed on basic alumina, then vac-sublimed and zone-refined. [Williams and Clarke, *J Chem Soc, Faraday Trans 1* **73** 514 *1977*; Albert, *The Acridines*

Arnold Press *1966*]. It can exists in five crystalline forms and is steam volatile. It is a strong IRRITANT to skin and mucous membranes and can become a chronic irritant— handle with CARE.

Acridine Orange *[494-38-2]* **M 349.94, m 181-182° (free base).** The double salt with $ZnCl_2$ (6g) was dissolved in water (200mL) and stirred with four successive portions (12g each) of Dowex-50 ion-exchange resin (K^+ form) to remove the zinc. The soln was then concentrated in vacuum to 20mL, and 100mL of ethanol was added to ppte KCl which was removed. Ether (160mL) was added to the soln from which, on chilling, the dye crystallises as its chloride. It was separated by centrifuging, washed with chilled ethanol and ether, and dried under vac, before being recryst from ethanol (100mL) by adding ether (50mL), and chilling. Yield 1g. [Pal and Schubert *J Am Chem Soc* **84** 4384 *1962*].
It was recrystd twice as the free base from ethanol or methanol/water by dropwise addition of NaOH (less than 0.1M). The ppte was washed with water and dried under vacuum. It was dissolved in $CHCl_3$ and chromatographed on alumina: the main sharp band was collected, concentrated and cooled to -20°. The ppte was filtered, dried in air, then dried for 2h under vacuum at 70°. [Stone and Bradley *J Am Chem Soc* **83** 3627 *1961*; Blauer and Linschitz *J Phys Chem* **66** 453 *1962*.]

Acridine Yellow G *[135-49-9]* **M 273.8, m 325°, CI 46025.** Crystd from 1:1 *benzene/methanol.

Acridone *[578-95-0]* **M 195.2, m >300°, pK_1 -0.32 (basic), pK_2 14 (acidic).** Dissolve ~1g in *ca* 1% NaOH (100mL), add 3M HCl to pH 4 when acridone separates as a pale yellow solid with **m** just above 350° (sharp). It can be recrystd from large vols of H_2O to give a few mg. It is soluble in 160 parts of boiling EtOH (540 parts at 22°) [*J Chem Soc* 1294 *1956*]. A few decigms are best crystallised as the *hydrochloride* from 400 parts of 10N HCl (90% recovery) from which the free base is obtained by washing the salt with H_2O. A small quantity can be recrystd (as the neutral species) from boiling AcOH. Larger quantities are best recrystallised from a mixture of 5 parts of freshly distd aniline and 12.5 parts of glacial acetic acid. Acridone distils unchanged at atmospheric pressure, but the boiling point was not recorded, and some sublimation occurs below 350°. It has UV: λ_{max} 399nm. [see Albert, *The Acridines* Arnold Press pp. 201, 372 *1966*.]

***N*-(9-Acridinyl)maleimide (NAM)** *[49759-20-8]* **M 274.3, m 248°, 255-258°.** Purified by chromatography on silica gel using CH_2Cl_2 as eluant. Evaporation of pooled fractions that gave the correct NMR spectra gave a solid which was recrystd from Me_2CO as pale yellow prisms. IR ν (nujol): 1710 (imide); UV (MeOH): λ_{max} (nm), (ε $M^{-1}cm^{-1}$): 251 (159 500), 343 shoulder (7 700), 360 (12 400) and 382shoulder (47 000). [*Chem Pharm Bull Jpn* **26** 596 *1978*; *Eur J Biochem* **25** 64 *1972*.]

Acriflavine *[8048-52-0]* **M 196.2, pK >12.** Treated twice with freshly ppted AgOH to remove proflavine, then recrystd from absolute methanol [Wen and Hsu *J Phys Chem* **66** 1353 *1962*].

Acriflavin Mixture (Euflavin, 3,6-diamino-10-methylacridinium chloride) *[8063-24-9]* **M 259.7, m 179-181°.** Purified by dissolving in 50 parts of H_2O, shake with a small excess of freshly ppted and washed Ag_2O. The mixture is set aside overnight at 0° and filtered. The cake is not washed. The pH of the filtrate is adjusted to 7.0 with HCl and evaporated to dryness. The residue is then crystd twice from MeOH, twice from H_2O and dried at 120°. λ_{max} at 452nm has a logε value of 4.67. It is a red powder which readily absorbs H_2O. The solubility is increased in the presence of proflavin. The *dihydrochloride* is a deep red crystn powder. It is available as a mixture of 3,6-diaminoacridinium chloride (35%) and its 10-metho-chloride (65%). [see Albert, *The Acridines* Arnold Press p. 346 *1966*; *Chem Ber* **45** 1787 *1912*].

Acrolein (acraldehyde) *[107-02-8]* **M 56.1, b 52.1°, n 1.3992, d 0.839.** Purified by fractional distn. under nitrogen, drying with anhydrous $CaSO_4$ and then distilling under vac. Blacet, Young and Roof [*J Am Chem Soc* **59** 608 *1937*] distd under nitrogen through a 90cm column packed with glass rings. To avoid formation of diacryl, the vapour was passed through an ice-cooled condenser into a receiver cooled in an ice-salt mixture and containing 0.5g catechol. The acrolein was then distd twice from anhydrous $CuSO_4$ at low pressure, catechol being placed in the distilling flask and the receiver to avoid polymerization. [Alternatively, hydroquinone (1% of the final soln) can be used.]

Acrolein diacetyl acetal (1,1-diacetoxy-2-propene). *[869-29-4]* **M 158.2, b 75°/10mm, 184°/atm, d_4^{20} 1.08, n_D^{20} 1.4203.** Check the NMR spectrum. If it is not satisfactory then add Ac_2O and a drop of conc H_2SO_4 and heat at 50° for 10min. Then add anhydrous NaOAc (*ca* 3g/ 100g of liquid) and fractionate. Note that it forms an azeotrope with H_2O, so do not add H_2O at any time. It is a **highly flammable and TOXIC** liquid, keep away from the skin. [*J Am Chem Soc* **73** 5282 *1951*.]

Acrolein diethyl acetal *[3054-95-3]* **M 130.2, b 120-125°/atm, n_4^{20} 1.398-1.407.** Add Na_2CO_3 (*ca* 3.5%) and distil using an efficient column, or better a spinning band column. [*Org Synth* **25** 1 *1945*.]

Acrolein dimethyl acetal (1,1-dimethoxy-2-propene) *[6044-68-4]* **M 102.1, b 87.5-88°/750mm, 89-90°/760mm, d_4^{20} 0.86, n_D^{20} 1.3962.** Fractionally distil (after adding 0.5g of hydroquinone) under reduced press through an all glass column (40cm x 2.5 cm) packed with glass helices and provided with a heated jacket and a total reflux variable take-off head. Stainless steel Lessing rings (1/8 x 1/8 in) or gauze have been used as packing. It is a **highly flammable and TOXIC** liquid, keep away from the skin. [*J Chem Soc* 2657 *1955*.]

Acrolein semicarbazone *[6055-71-6]* **M 113.1, m 171°.** Crystd from water.

Acrylamide *[79-06-1]* **M 71.1, m 84°, b 125°/25mm.** Crystd from acetone, chloroform, ethyl acetate, methanol or *benzene/chloroform mixture, then vac dried and kept in the dark under vac. Recryst from $CHCl_3$ (200g dissolved in 1L heated to boiling and filtered without suction in a warmed funnel through Whatman 541 filter paper. Allowed to cool to room temp and kept at -15° overnight). Crystals were collected with suction in a cooled funnel and washed with 300mL of cold MeOH. Crystals were air-dried in a warm oven. [Dawson et al. *Data for Biochemical Research*, Oxford Press 1986 p. 449.]
CAUTION: *Acrylamide is extremely* **TOXIC** *and precautions must be taken to avoid skin contact or inhalation. Use gloves and handle in a well ventilated fume cupboard.*

Acrylic acid *[79-10-7]* **M 72.1, m 13°, b 30°/3mm, d 1.051, pK^{25} 4.25.** Can be purified by steam distn, or vacuum distn through a column packed with copper gauze to inhibit polymerisation. (This treatment also removes inhibitors such as methylene blue that may be present.) Azeotropic distn of the water with *benzene converts aqueous acrylic acid to the anhydrous material.

Acrylonitrile *[107-13-1]* **M 53.1, b 78°, d 0.806, n^{25} 1.3886.** Washed with dilute H_2SO_4 or dilute H_3PO_4, then with dilute Na_2CO_3 and water. Dried with Na_2SO_4, $CaCl_2$ or (better) by shaking with molecular sieves. Fractionally distd under nitrogen. Can be stabilised by adding 10ppm *tert*-butyl catechol. Immediately before use, the stabilizer can be removed by passage through a column of activated alumina (or by washing with 1% NaOH soln if traces of water are permissible in the final material), followed by distn. Alternatively, shaken with 10% (w/v) NaOH to extract inhibitor, and then washed in turn with 10% H_2SO_4, 20% Na_2CO_3 and distd water. Dried for 24h over $CaCl_2$ and fractionally distd under N_2 taking the fraction boiling at 75.0 to 75.5°C (at 734mm Hg). Stored with 10ppm *tert*-butyl catechol. Acrylonitrile is distilled off as required. [Burton *et al, J Chem Soc, Faraday Trans 1* **75** 1050 *1979*.]

Acryloyl chloride *[814-68-6]* **M 90.5, b 72-74°/740mm, 74°/760mm, d_4^{20} 1.1127, n_D^{20} 1.4337.** Distil rapidly through an efficient 25cm column after adding 0.5g of hydroquinone/200g of chloride, and then redistil carefully at atmospheric pressure preferably in a stream of dry N_2. [*J Am Chem Soc* **72** 72, 2299 *1950*.] **The liquid is an irritant and is TOXIC.**

Actarit (*p*-acetamidophenylacetic acid) *[18699-02-0]* **M 193.2, m 174-175°.** Crystd from MeOH + Me_2CO or aq EtOH.

Adamantane *[281-23-2]* **M 136.2, m 269.6-270.8° (sublimes).** Crystd from acetone or cyclohexane, sublimed in a vacuum below its melting point. [Butler et al. *J Chem Soc, Faraday Trans 1* **82** 535 *1986*.] Adamantane was also purified by dissolving in *n*-heptane (*ca* 10mL/g of adamantane) on a hot plate, adding activated charcoal (2g/100g of adamantane), and boiling for 30min, filtering the hot soln through a filter paper, concentrating the filtrate until crystn just starts, adding one quarter of the original volume *n*-heptane and

allowing to cool slowly over a period of hours. The supernatant was decanted off and the crystals were dried on a vacuum line at room temperature. [Walter et al. *J Am Chem Soc* **107** 793 *1985.*]

1-Adamantane acetic acid *[4942-47-6]* **M 194.3, m 136°, pK_{Est} ~4.8.** Dissolve in hot N NaOH, treat with charcoal, filter and acidify. Collect solid, wash with H_2O, dry and recryst from MeOH. [*Chem Ber* **92** 1629 *1959.*]

1-Adamantane carboxylic acid *[828-51-3]* **M 180.3, m 175-176.5°, 177°, pK_{Est} ~4.9.** Possible impurities are trimethylacetic acid and C9 and C13 acids. Dissolve 15g of acid in CCl_4 (300mL) and shake with 110mL of 15N aqueous NH_3 and the ammonium salt separates and is collected. Acid impurities form soluble ammonium salts. The salt is washed with cold Me_2CO (20mL) and suspended in H_2O (250mL). This is treated with 12N HCl and extracted with $CHCl_3$ (100mL). The dried (Na_2SO_4) is evaporated and the residue recrystd from a mixture of MeOH (30mL) and H_2O (*ca* 10mL) to give the pure acid (10-11g). [*Org Synth* Coll Vol.V 20 *1973.*] Also recrystd from absolute EtOH and dried under vacuum at 100°.
Alternatively, the acid (5g) is refluxed for 2h with 15mL of MeOH and 2mL of 98% H_2SO_4 (cool when mixing this soln). Pour into 10 volumes of H_2O and extract with the minimum volume of $CHCl_3$ to give clear separation of phases. The extract is washed with H_2O and dried ($CaCl_2$) and distd. The methyl ester is collected at 77-79°/1mm, **m** 38-39°. The ester is hydrolysed with the calculated amount of N KOH and refluxed until clear. Acidification with HCl provides the pure acid with 90% recovery. [*Org Synth* **4** 1 *1964.*] The *amide* crysts from cyclohexane, **m** 189°. [*Chem Ber* **62** 1629 *1959.*]

1,3-Adamantane diamine dihydrochloride *[26562-81-2]* **M 239.2, m >310°, $pK_{Est(1)}$ ~8.1, $pK_{Est(2)}$ ~10.1.** Dissolve in boiling conc HCl (400mg in 15mL) and evaporate to dryness. Dissolve in absolute EtOH and add dry Et_2O to crystallise the *dihydrochloride*. [*Chem Ber* **93** 1366 *1960.*]

1,3-Adamantane dicarboxylic acid *[39269-10-8]* **M 224.3, m 276°, 276-278°, 279°, $pK_{Est(1)}$ ~4.9. $pK_{Est(2)}$ 5.9.** Dissolve in aq NaOH, treat with charcoal, filter and acidify with dilute HCl. Recryst from MeOH. [*Chem Ber* **93** 1366 *1960.*]

1-Adamantane methylamine *[17768-41-1]* **M 165.3, b 83-85°/0.3mm, d_4^{20} 0.93, pK_{Est} ~10.2.** Dissolve in Et_2O, dry over KOH and distil. The *N-Tosyl* derivative has **m** 134-135° (from EtOH). [*Chem Ber* **96** 550 *1963.*]

1-Adamantanol (1-hydroxyadamantane) *[768-95-6]* **M 152.4, m 288.5-290°.** If 2-adamantanol is a suspected impurity then dissolve substance (10g) in acetone (100mL) and Jones's reagent {CrO_3 (10.3g) in H_2O (30mL)} and conc H_2SO_4 (8.7mL) is added dropwise (turns green in colour) until excess reagent is present (slight red colour). Allow to stir overnight, decant the acetone soln from the Cr salts and adamantan-2-one, and dry (Na_2SO_4) and evaporate to dryness. The residue (*ca* 7g) is chromatographed through Al_2O_3 (250g) and washed with 50% *benzene-pet ether (b 40-60°), then 100% Et_2O (to remove any adamantan-2-one present) and the 1-adamantanol is then eluted with 5% MeOH in Et_2O. The eluate is evaporated, and the residue is recrystd from pet ether (b 30-60°) at -70°, **m** 287.2-288.5°. It has characteristic IR, ν 3640, 1114, 1086, 982 and 930cm^{-1}. [*J Am Chem Soc* **83** 182 *1961.*]
Alternatively, if free from the 2-isomer, dissolve in tetrahydrofuran, dilute with H_2O to ppte the alcohol. Collect, dry and sublime in a vacuum at 130°. [*Chem Ber* **92** 1629 *1959.*]

2-Adamantanol (2-hydroxyadamantane) *[700-57-2]* **M 152.4, m 296.2-297.7°.** Can be purified by chromatography as for the 1-isomer. It crystallises from cyclohexane and has characteristic IR, ν 3600, 1053, 1029 and 992cm^{-1} [*J Am Chem Soc* **8** 182 *1961*].

2-Adamantanone *[700-58-3]* **M 150.2, m 256-258°(sublimes).** Purified by repeated sublimation *in vacuo.* [Butler et al. *J Chem Soc, Faraday Trans 1* **82** 535 *1986.*]

***N*-(1-Adamantyl)acetamide** *[880-52-4]* **M 193.3, m 149°.** Wash well with H_2O, dry and recrystallise from cyclohexane. *It is an* **irritant.** [*Chem Ber* **92** 1629 *1959.*]

1-Adamantylamine *[768-94-5]* **M 151.2, m 160-190°, 208-210°, pK^{25} 10.58.** Dissolve in Et_2O, dry over KOH, evaporate and sublime in a vacuum. [*Chem Ber* **93** 226 *1960.*]

1-Adamantylamine hydrochloride *[665-66-7]* **M 187.7, m 360° (dec).** Dissolve in dry EtOH, add a few drops of dry EtOH saturated with HCl gas, followed by dry Et_2O to crystallise the hydrochloride. Dry the salt in vacuum. [*Chem Ber* **93** 226 *1960.*]

2-Adamantylamine hydrochloride *[10523-68-9]* **M 187.7, m >300°, pK$_{Est}$ ~10.4.** The free amine in Et_2O, liberated by the action of alkali in H_2O, is dried over KOH, filtered, evap and sublimed at 110°/12Torr, **m** 230-236°. The base is dissolved in EtOH and crystd by the addition of Et_2O, and dried in vac. [*Justus Liebigs Ann Chem* **658** 151 *1962*].

1-Adamantyl bromide *[768-90-1]* **M 215.1, m 117-119°, 118°, 119.5-120°.** If coloured, dissolve in CCl_4, wash with H_2O, treat with charcoal, dry ($CaCl_2$), filter, evap to dryness. Dissolve in a small volume of MeOH and cool in a CO_2/trichloroethylene bath and collect the crystals. Sublime at 90-100°/water pump vacuum. [*Chem Ber* **92** 1629 *1959*; *J Am Chem Soc* **83** 2700 *1961.*]

1-Adamantyl bromomethylketone *[5122-82-7]* **M 257.2, m 76-79°, 78-79°.** Dissolve in Et_2O, wash with H_2O, dry ($MgSO_4$), evaporate and crystallise residue from small volumes of MeOH. **LACHRYMATORY.** [*Chem Ber* **93** 2054 *1960.*]

1-Adamantyl chloride *[935-56-8]* **M 170.7, m 164.3-165.6°.** Crystd from aqueous MeOH and sublimed at 100°/12Torr. Also crystd from MeOH at -70°. [*Chem Ber* **92** 1629 *1959*; *J Am Chem Soc* **83** 2700 *1961.*]

1-Adamantyl fluoride (1-fluoroadamantane) *[768-92-3]* **M 154.2, m 210-212° (dec), 259-260° (dec).** Dissolve in Et_2O, dry over Na_2SO_4, evaporate to dryness and sublime the residue at 90-100°/12mm. Recryst sublimate from MeOH, **m** 259-260°. [*Zh Org Khim* **30** 1609 *1965.*] To remove 1-hydroxyadamantane impurity, dissolve in cyclohexane cool for many hours, filter off the hydroxyadamantane, and evaporate to dryness. Recrystallise the residue from pet ether at -77° and sublime in vacuum, **m** 210-212° dec (sealed tube). [*J Org Chem* **30** 789 *1965.*]

1-Adamantyl fluoroformate *[62087-82-5]* **M 198.2, m 31-32°.** Dissolve in *n*-hexane (*ca* 10g in 150 mL) and keep at 0° for 24h. Any 1-adamantanol present will separate. Filter and evaporate to dryness. Crystalline residue has **m** 31-32° (ν 1242, 1824 and 2340 cm^{-1}). There should be no OH str band above 2500 cm^{-1}. [*Z Phys Chem* **357** 1647 *1976*; Haas et al. *J Am Chem Soc* **88** 1988 *1966.*]

1-Adamantyl iodide (1-iodoadamantane) *[768-93-4]* **M 262.1, m 75.3-76.4°.** Dissolve in Et_2O, shake with aqueous $NaHSO_3$, aqueous K_2CO_3, and H_2O, dry (Na_2SO_4), evaporate and recrystallise from MeOH at -70° (to avoid alcoholysis) giving white crystals. [*J Am Chem Soc* **83** 2700 *1961*; lit **m** of 151-152.5° is incorrect.] Also purified by recrystn from pet ether (40-60°C) followed by rigorous drying and repeated sublimation.

1-Adamantyl isocyanate *[4411-25-0]* **M 177.3, m 144-145°.** Recryst from *n*-hexane and sublime. **Irritant.** [*Chem Ber* **95** 2302 *1962.*]

1-Adamantyl isothiocyanate *[4411-26-1]* **M 193.3, m 168-169°.** Dissolve in Et_2O, wash with H_2O, dry (Na_2SO_4), evaporate and sublime the residue in a vacuum at 140°, and recryst from MeOH. **Irritant.** [*Chem Ber* **95** 2302 *1962.*]

***N*-(1-Adamantyl)urea** *[13072-69-0]* **M 194.2, m >250° (dec), 268-272° (dec).** Wash with H_2O and dioxane and recryst from EtOH. [*Chem Ber* **95** 2302 *1962.*]

Adenine *[73-24-5]* **M 135.1, m 360-365° (dec rapid heating), pK$_1^{25}$ 4.12, pK$_2^{25}$ 9.83.** Crystd from distd water.

Adenosine *[58-61-7]* **M 267.3, m 234-236°,** $[\alpha]_{546}$ **-85° (c 2, 5% NaOH),** pK_1^{25} **3.48,** pK_2^{25} **12.5.** Crystd from distilled water.

Adipic acid *[124-04-9]* **M 146.1, m 154°,** pK_1^{25} **4.44,** pK_2^{25} **5.45.** For use as a volumetric standard, adipic acid was crystd once from hot water with the addition of a little animal charcoal, dried at 120° for 2h, then recrystd from acetone and again dried at 120° for 2h. Other purification procedures include crystn from ethyl acetate and from acetone/petroleum ether, fusion followed by filtration and crystn from the melt, and preliminary distn under vac.

Adiponitrile (1,4-dicyanobutane) *[111-69-3]* **M 108.14, m 2.4°, b 123°/0.5mm, 153°/6mm, 175°/26mm, 184°/30mm, 295°/atm,** d_4^{20} **0.9396,** n_D^{20} **1.4371.** Reflux over P_2O_5 and $POCl_3$, and fractionally distil, then fractionate through an efficient column. **The liquid is TOXIC and is an IRRITANT.** [*Chem Ber* **67** 1770 *1934*; *Justus Liebigs Ann Chem* **596** 127 *1955*; *Can J Chem* **34** 1662 *1956*; *J Am Chem Soc* **62** 228 *1940*.]

Adonitol (Ribitol) *[488-81-3]* **M 152.2, m 102°.** Crystallise from EtOH by addition of diethyl ether.

Adrenalin see **epinephrine.**

Adrenochrome *[54-06-8]* **M 179.2, m 125-130°.** Crystd from MeOH/formic acid, as hemihydrate, and stored in a vacuum desiccator.

Adrenosterone (Reichstein's G) *[382-45-6]* **M 300.4, m 220-224°.** Crystd from EtOH. Can be sublimed under high vacuum.

Agaricic acid [1-(*n*-hexadecyl)citric acid] *[666-99-9]* **M 416.6, m 142°(dec),** $[\alpha]_D$ **-9.8° (in NaOH),** $pK_{Est(1)}$ **~2.7,** $pK_{Est(2)}$ **~4.2,** $pK_{Est(3)}$ **~5.5.** Crystd from EtOH.

Agmatine sulfate [5-guanidinopent-1-ylamine sulfate] *[2482-00-0]* **M 228.3, m 231°,** $pK_{Est(1)}$ **~9.1,** $pK_{Est(2)}$ **~13.0.** Crystd from aqueous MeOH.

Agroclavin *[548-42-5]* **M 238.3, m 198-203°(dec), 205-206°,** $[\alpha]_D^{20}$ **-155° (c 1,** $CHCl_3$**),** pK_{Est} **~8.0.** Crystd from diethyl ether.

Ajmalicine *[483-04-5]* **M 352.4, m 250-252°(dec),** $[\alpha]_{546}$ **-76° (c 0.5,** $CHCl_3$**),** pK_{Est} **~7.4.** Crystd from MeOH.

Ajmalicine hydrochloride *[4373-34-6]* **M 388.9, m 290°(dec),** $[\alpha]_D$ **-17° (c 0.5, MeOH).** Crystd from EtOH.

Ajmaline [γ-yohimbine] *[4360-12-7]* **M 326.4, m 160° (MeOH), 205-206° (anhyd),** $[\alpha]_D^{20}$ **+144° (c 0.8,** $CHCl_3$**),** pK_{Est} **~7.5.** Crystd from MeOH.

Ajmaline hydrochloride *[4410-48-4]* **M 388.9, m 140°.** Crystd from water.

Alanine (*RS*) *[302-72-7]* **M 89.1, m 295-296°,** (*S*) *[56-41-7]* **m 297°(dec),** $[\alpha]_D^{15}$ **+14.7° (in 1M HCl),** pK_1^{25} **2.34,** pK_2^{25} **9.87.** Crystd from water or aqueous EtOH, e.g. crystd from 25% EtOH in water, recrystd from 62.5% EtOH, washed with EtOH and dried to constant weight in a vacuum desiccator over P_2O_5. [Gutter and Kegeles *J Am Chem Soc* **75** 3893 *1953*.] 2,2'-Iminodipropionic acid is a likely impurity.

ß-Alanine *[107-95-9]* **M 89.1, m 205°(dec),** pK_1^{25} **3.55,** pK_2^{25} **10.24.** Crystd from filtered hot saturated aqueous soln by adding four volumes of absolute EtOH and cooling in an ice-bath. Recrystd in the same way and then finally, crystd from a warm saturated soln in 50% EtOH by adding four volumes of absolute

EtOH cooled in an ice bath. Crystals were dried in a vacuum desiccator over P_2O_5. [Donovan and Kegeles *J Am Chem Soc* **83** 255 *1961*.]

***S*-Alaninol [*S*-2-Aminopropan-1-ol]** *[2749-11-3]* **M 75.1, b 167-169°/760mm, d_4^{20} 0.961, n_D^{20} 1.456, $[\alpha]_{546}$ +26.0° (c 2, EtOH), pK^{25} 9.43.** Purification as for *S*-2-amino-3-methylbutan-1-ol

Aldol (3-hydroxybutanal) *[107-89-1]* **M 88.1, b 80-81°/20mm.** An ethereal soln was washed with a saturated aqueous soln of $NaHCO_3$, then with water. The non-aqueous layer was dried with anhydrous $CaCl_2$ and distd immediately before use. The fraction, **b** 80-81°/20mm, was collected, [Mason, Wade and Pouncy *J Am Chem Soc* **76** 2255 *1954*].

Aldosterone *[52-39-1]* **M 360.5, m 108-112°(hydrate), 164°(anhydr), $[\alpha]_D^{25}$ +161° (c 1, $CHCl_3$).** Crystd from aqueous acetone. *Acetate*, cryst from Me_2CO + Et_2O, has **m** 198-199°, $[\alpha]_D^{24}$ +121.7° (c 0.7, $CHCl_3$)

Aldrin *[309-00-2]* **M 354.9, m 103-104.5°.** Crystd from MeOH. **POISONOUS**

Aleuritic acid [*RS-erythro*-9,10,16-trihydroxyhexadecanoic acid] *[533-87-9]* **M 304.4, m 100-101°.** Crystd from aqueous EtOH. *Hydrazide* cryst from EtOH has **m** 139-140°.

Alginic acid *[9005-32-7]* **M 48,000-186000**. To 5g in 550mL water containing 2.8g $KHCO_3$, were added 0.3mL acetic acid and 5g potassium acetate. EtOH to make the soln 25% (v/v) in EtOH was added and any insoluble material was discarded. Further addition of EtOH, to 37% (v/v), ppted alginic acid. [Pal and Schubert *J Am Chem Soc* **84** 4384 *1962*.]

Aliquat 336 (methyltricaprylylammonium chloride, tri-*n*-octylmethylammonium chloride) *[5137-55-3]* **M 404.2, d 0.884.** A 30% (v/v) soln in *benzene was washed twice with an equal volume of 1.5M HBr. [Petrow and Allen, *Anal Chem* **33** 1303 *1961*.] Purified by dissolving 50g in $CHCl_3$ (100mL) and shaking with 20% NaOH soln (200mL) for 10min, and then with 20% NaCl (200mL) for 10min. Washed with small amount of H_2O and filtered through a dry filter paper [Adam and Pribil *Talanta* **18** 733 *1971*].

Alizarin (1,2-dihydroxyanthraquinone) *[72-48-0]* **M 240.2, m 290°, d 0.884, pK_1^{25} 7.45, pK_2^{25} 11.80.** Crystd from glacial acetic acid or 95% EtOH. Can also be sublimed at 110°/2mm.

Alizarin-3-methyliminodiacetic acid (Alizarin Complexone) ($2H_2O$) *[3952-78-1]* **M 421.4, m 189°(dec), $pK_{Est(1)}$~4.9, $pK_{Est(2)}$~7.5.** Purified by suspending in 0.1M NaOH (1g in 50mL), filtering the solution and extracting alizarin with 5 successive portions of CH_2Cl_2. Then add HCl dropwise to precipitate the reagent, stirring the solution in a bath. Filter ppte on glass filter, wash with cold water and dry in a vacuum desiccator over KOH [Ingman *Talanta* **20** 135 *1973*].

Alizarin Yellow R [5-(4-nitrophenylazosalicylic acid), Mordant Orange I] *[2243-76-7]* **M 287-2, m 253-254°(dec), >300°, pK^{25} 11.17.** The free acid is ppted by adding HCl to an aq soln of the Na salt. After 2 recrystns from aq AcOH, it has **m** 255°(dec); [m 253-254° dec was reported *J Chem Soc* **79** 49 *1901*]. The free acid can be recrystd from dilute AcOH as orange brown needles. The Na salt changes colour from yellow to red when the pH is increased from 10.2 to 12.0. [*J Am Chem Soc* **75** 5838 *1953*.]

***n*-Alkylammonium chloride n=2,4,6**. Recrystd from EtOH or an EtOH/Et_2O mixture. [Hashimoto and Thomas *J Am Chem Soc* **107** 4655 *1985;* Chu and Thomas *J Am Chem Soc* **108** 6270 *1986*.]

***n*-Alkyltrimethylammonium bromide n=10,12,16**. Recrystd from an EtOH/Et_2O mixture. [Hashimoto and Thomas *J Am Chem Soc* **107** 4655 *1985*.]

Allantoin *[97-59-6]* **M 158.1, m 238°(dec).** Crystd from water or EtOH.

Next Page