ALIPHATIC COMPOUNDS

Acetal (acetaldehyde diethylacetal) *[105-57-7]* **M 118.2, b 103.7-104°, d_4^{20} 0.831, n_D^{25} 1.38054, n^{25} 1.3682.** Dry acetal over Na to remove alcohols and H_2O, and to polymerise aldehydes, then fractionally distil. Or, treat it with alkaline H_2O_2 at 40-45° to remove aldehydes, then saturate with NaCl, separate, dry with K_2CO_3 and distil it from Na [Vogel *J Chem Soc* 616 *1948*]. [*Beilstein* **1** IV 3103.]

Acetaldehyde *[75-07-0]* **M 44.1, b 20.2°, d_4^{20} 0.788, n_D^{25} 1.33113, pK^{25} 13.57 (hydrate).** Acetaldehyde is usually purified by fractional distillation in a glass helices-packed column under dry N_2, discarding the first portion of distillate. Or, it is shaken for 30minutes with $NaHCO_3$, dried with $CaSO_4$ and fractionally distilled at 760mm through a 70cm Vigreux column (p 11). The middle fraction is collected and further purified by standing for 2hours at 0° with a small amount of hydroquinone (free radical inhibitor), followed by distillation [Longfield & Walters *J Am Chem Soc* **77** 810 *1955*]. [*Beilstein* **1** IV 3094.]

Acetaldehyde dimethyl acetal (1,1-dimethoxyethane) *[534-15-6]* **M 90.1, b 63-65°, d_4^{20} 0.852, n_D^{25} 1.36678.** Distil the dimethyl acetal through a fractionating column and fraction boiling at 63.8°/751mm is collected. It forms an azeotrope with MeOH. Alternatively purify it as for *acetal* above. It has been purified by GLC. [*Beilstein* **1** IV 3103.]

Acetamide *[60-35-5]* **M 59.1, m 81°, pK_1^{25} -1.4, pK_2^{25} +0.37.** Acetamide is crystallised by dissolving in hot MeOH (0.8mL/g), diluting with Et_2O and allowing to stand [Wagner *J Chem Edu* **7** 1135 *1930*]. Alternate crystallisation solvents are acetone, *benzene, chloroform, dioxane, methyl acetate or *benzene/ethyl acetate mixtures (3:1 and 1:1). It has also been recrystallised from hot water after treating with HCl-washed activated charcoal (which had been repeatedly washed with water until free from chloride ions), then crystallised again from hot 50% aqueous EtOH and finally twice from hot 95% EtOH [Christoffers & Kegeles *J Am Chem Soc* **85** 2562 *1963*]. Finally it is dried in a vacuum desiccator over P_2O_5. Acetamide is also purified by distillation (**b** 221-223°) or by sublimation *in vacuo*. It has also been purified by two recrystallisations from cyclohexane containing 5% (v/v) of *benzene. Needle-like crystals separate and are filtered, washed with a small volume of distilled H_2O and dried with a flow of dry N_2. [Slebocka-Tilk et al. *J Am Chem Soc* **109** *4620 1987*, *Beilstein* **2** H 175, **2** I 80, **2** II 177, **2** III 384, **2** IV 399.]

Acetamidine hydrochloride *[124-42-5]* **M 94.5, m 164-166°, 165-170°(dec), 174°, pK^{25} 12.40.** The hydrochlorde can be recrystallised from small volumes of EtOH. Alternatively it is dissolved in EtOH, filtered, Et_2O is added; filter the crystalline salt off under N_2 and dry it in a vacuum desiccator over H_2SO_4. The salt is deliquescent and should be stored in a tightly stoppered container. Its solubility in H_2O is 10% at room temperature and it is 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 & Parsons *Chem Ind (London)* 628 *1958*, Barnes et al. *J Am Chem Soc* **62** 1286 *1940* give **m** 177-178°, *Beilstein* **2** H 185, **2** I 85, **2** II 183, **2** III 416, **2** IV 428.]

***N*-(2-Acetamido)-2-aminoethanesulfonic acid (ACES)** *[7365-82-4]* **M 182.2, m > 220°(dec), pK_{Est} ~1.5, pK_2 6.9.** Recrystallise ACES from hot aqueous EtOH. [Perrin & Dempsey *Buffers for pH and Metal Ion Control* Chapman & Hall, London *1974*, *Beilstein* **4** III 1707.]

***N*-(2-Acetamido)iminodiacetic acid (ADA)** *[26239-55-4]* **M 190.2, m 219°(dec), pK_1 ~2.3, pK_2 6.6.** Dissolve ADA in water, add one equivalent of NaOH solution (to final pH of 8-9), then acidify with HCl to precipitate the free acid. This is filtered off, washed with water and dried *in vacuo*. [*Beilstein* **4** IV 2441.]

Acetamidomethanol *[625-51-4]* **M 89.1, m 47-50°, 54-56°, 55°.** Recrystallise it from freshly distilled Me_2CO, wash the crystals with dry Et_2O and dry them 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 $AgNO_3$. It also crystallises in needles from EtOAc containing a few drops of Me_2CO. It is *hygroscopic* and should be stored under dry conditions. [Bachmann et al. *J Am Chem Soc* **73** 2775 *1951*, Walter et al. *Chem Ber* **99** 3204 *1966*, Einhorn & Ladisch *Justus Liebigs Ann Chem* **343** 265 *1905*, *Beilstein* **2** IV 405.]