

insoluble in Et₂O. *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 H₂O, 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 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°, [α]_D²⁵ +199.3° (c 3, CHCl₃). 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*₁²⁵ -6.1 (basic, mono-protonated), *pK*₂²⁵ 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% H₂O. 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 CaSO₄, K₂CO₃ 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 P₂O₅ or sodium amalgam is used. Anhydrous MgSO₄ is an inefficient drying agent, and CaCl₂ forms an addition compound. Drierite (anhydrous CaSO₄) 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 10⁻²M. Anhydrous Mg(ClO₄)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 AgNO₃ in 30mL of water to 1L of acetone, followed by 10mL of M NaOH, shaking for 10min, filtering, drying with anhydrous CaSO₄ and distilling [Werner *Analyst (London)* 58 335 1933]. Alternatively, successive small portions of KMnO₄ have been added to acetone at reflux, until the violet colour persists, followed by drying and distn. Refluxing with chromium trioxide (CrO₃) 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.3Me₂CO 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 CaSO₄ and distil.

Acetone cyanohydrin [75-86-5] *M* 85.1, *b* 48°/2.5mm, 68-70°/11mm, 78-82°/15mm, *d*₄²⁰ 0.93. Dry with Na₂SO₄, 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 P₂O₅. 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.