An Efficient Synthesis of 3-Aminocyclopent-2-en-1-one

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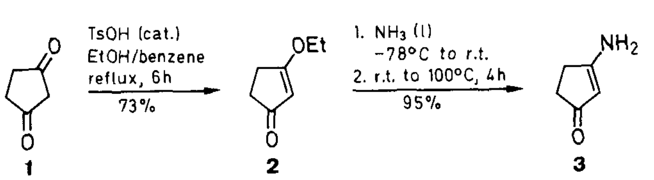
A two step, efficient synthesis of the key intermediate 3-aminocyclopent-2-en-1-one starting from 1,3-cyclopentanedione is described. This involves the reaction of ammonia gas with 3-ethoxycyclopent-2-en-1-one.

In the course of our investigations into synthesis of natural products,[1] we needed a convenient synthesis of 3-aminocyclopent-2-en-1-one (**3**). The literature method[2] is inconvenient as it involves extremely high pressure.

We have developed a simple and convenient synthesis of **3** starting from 1,3-cyclopentanedione (**1**) in high yield. Compound **1** was converted to the vinyl ether **2** by refluxing with ethanol and a catalytic amount of *p*-toluenesulfonic acid in benzene. Compound **2** was then introduced into a Parr pressure vessel with liquid ammonia. Subsequent heating of the mixture from − 78 °C to 100 °C gave compound **3** in 95 % yield.



3-Ethoxycyclopent-2-en-1-one (2):

Compound **1** (9.8 g, 0.1 mol), anhydrous (EtOH (15 mL) and TsOH (1 mg, catalytic) are dissolved in benzene (100 mL). The solution is stirred magnetically and refluxed for 6 h. Benzene and EtOH are evaporated under reduced pressure and the mixture is subjected to column chromatography over silica gel. Elution with acetone provides compound **2**; yield: 9.2 g (73 %); bp 155–158 °C/1 Torr (Lit.[2] bp 180–182 °C/10 Torr).

IR: ν = 2975, 1707 cm^{-1}.

^{1}H-NMR (80 MHz; DMSO-d_6): δ = 1.37 (t, 3 H), 2.43 (br, 4 H), 4.06 (q, 2 H), 5.24 (s, 1 H).

3-Aminocyclopent-2-en-1-one (3):

The vinyl ether **2** (1 g, 0.01 mol) is introduced into a Parr pressure vessel which is then cooled to − 78 °C in a dry ice/acetone bath. Liquid NH_3 (1 mL) is introduced into the vessel which is subsequently secured and carefully allowed to warm to r.t. Then, gradual heating is initiated until 100 °C. After 4 h, the vessel is cooled to − 78 °C and opened carefully to afford **3**; yield: 0.92 g (95 %). Compound **3** is pure enough for most of our purposes but it can be further purified by recrystallization (MeOH); mp 211–212.5 °C (Lit.[2] mp 212–214 °C).

IR: ν = 3307, 2980, 1710 cm^{-1}.

^{1}H-NMR (80 MHz; DMSO-d_6): δ = 2.98 (d, 2 H), 3.25 (d, 2 H), 5.51 (s, 1 H), 8.02 (br 2 H).

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(2) Rungsiyanand, Ch.; Rimek, H.J.; Zymalkowski, F. *Chem. Ber.* **1970**, *103(8)*, 2403.