Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3-Aminocyclopent-2-en-1-one

Ray J. Butcher,[a]* Yousef M. Hijji[b] and Ellis Benjamin[b]

[a]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, and [b]Chemistry Department, Morgan State University, 1700 East Cold Spring Lane, Baltimore, MD, USA

Correspondence e-mail:
raymond.butcher@nrl.navy.mil

Received 12 December 2006
Accepted 5 January 2007

Key indicators

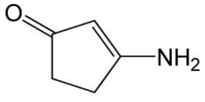
Single-crystal X-ray study
T = 103 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.145
Data-to-parameter ratio = 16.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, C_5H_7NO, was synthesized by the reaction of 1,3-cyclopentanedione with ammonium acetate under microwave conditions in 81% yield. The crystal packing is determined by intermolecular $N—H\cdots O$ and $C—H\cdots O$ hydrogen bonds.

Comment

3-Amino-2-cyclopent-2-en-1-one, (I), and its analogs are used as starting materials for the synthesis of potassium ATP (K_{ATP}) channel openers (Carroll *et al.*, 2004), anticonvulsants (Foster *et al.*, 1999), and necrosis factor-κB inhibitors (Fernandes *et al.*, 2004). Previous conventional syntheses of enaminones used the reaction of 1,3 cyclic diketones with NH_4OAc (Putkonen *et al.*, 2003), or 1,3 cyclic diketones with ammonia (Iida *et al.*, 1982). Enaminones have also been synthesized under microwave conditions using 1,3 cyclic diketones with NH_4OAc and with a montmorillonite solid support (Braibante *et al.*, 2003). Specifically, 3-amino-2-cyclopenten-1-one (I) has been synthesized using 1,3-cyclopentadione with ethanol and *p*-toluenesulfonic acid then amination with liquid ammonia, (Kikani *et al.*, 1991; Ruangsiyanand *et al.*, 1970).



(I)

We report here the synthesis of (I) under microwave conditions and give its X-ray structural data. The five-

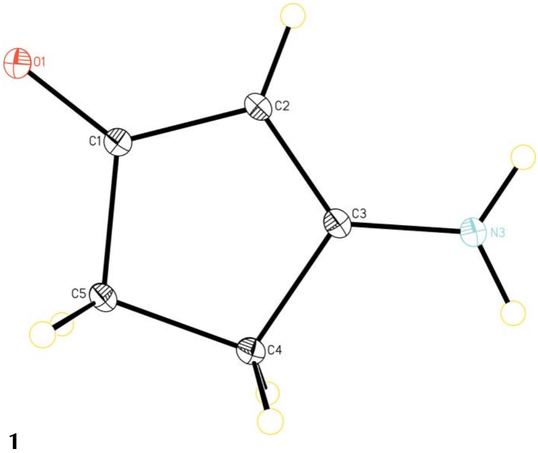


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 20% probability level. H atoms are represented by circles of arbitrary size.

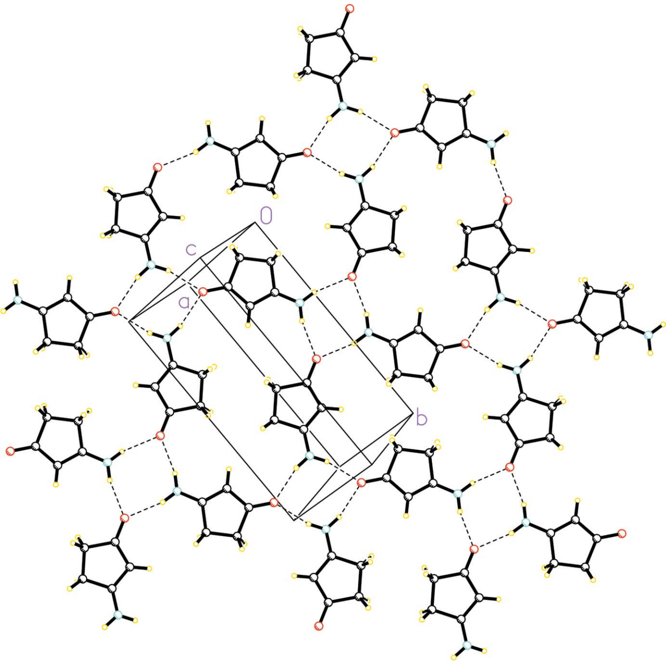


Figure 2
The molecular packing, showing the sheets linked by hydrogen-bonding interactions (dashed lines).

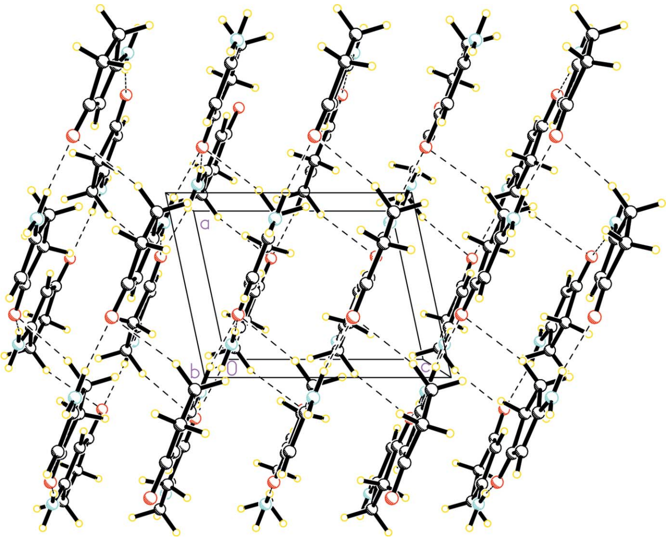


Figure 3
Molecular packing, viewed down the *b* axis, showing the weak out-of-plane C—H···O interactions (dashed lines).

membered cyclopentene ring is planar, with a mean deviation of 0.0147 (8) Å, the sp^3 C4 and C5 atoms showing the largest deviations [0.0187 (8) and 0.0197 (8) Å, respectively] as expected. The C—C bond lengths in the five-membered ring show that there is some delocalization over the atoms O1, C1, C2, and C3. The C1—C2 and C2—C3 bond lengths [1.4121 (18) and 1.3745 (19) Å, respectively] are shorter than normal single bonds but longer that double bonds (CSD, Version 5.27; Allen, 2002).

The amine H atoms participate in bifurcated hydrogen bonds to the O atoms of neighboring molecules, forming sheets in the (110) plane (Fig. 2). These sheets are further linked by weak out-of-plane C4—H···O1 intermolecular interactions in the [101] direction (Fig. 3). A search of the literature for derivatives of 3-amino-2-cyclopenten-1-one surprisingly gave only one example (Huang *et al.*, 1997), namely 3-(4-nitroanilino)-2-cyclopenten-1-one. This also contains a planar ring with similar metrical parameters.

Experimental

1,3-Cyclopentadione (0.1 g, 1.02 mmol) and NH_4OAc (0.08 g, 1.04 mmol) were thoroughly mixed in a CEM vial with a stirrer. The vial was capped and heated in a CEM Discover microwave for 5 min at 423 K and 150 W. The sample was cooled to 313 K, yielding a dark-brown solid. This was dissolved in methanol and flashed down a silica column (*ca* 40 g) using ethyl acetate (0.08 g, 81%). Brown crystals were formed on slow evaporation of a methanol solution.

Crystal data

C_5H_7NO
$M_r = 97.12$
Monoclinic, $P2_1/c$
$a = 5.6444$ (17) Å
$b = 12.085$ (4) Å
$c = 7.356$ (2) Å
$\beta = 102.026$ (4)°
$V = 490.8$ (3) Å^3
$Z = 4$
$D_x = 1.314$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 103$ (2) K
Chunk, colorless
0.44 × 0.42 × 0.35 mm

Data collection

Bruker APEX-2 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.960$, $T_{\max} = 0.968$
4315 measured reflections
1084 independent reflections
998 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 27.1°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.145$
$S = 1.10$
1084 reflections
64 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2 + 0.1689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.2487 (16)	C2—C3	1.3745 (19)
C1—C2	1.4121 (18)	C3—N3	1.3253 (17)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1^{i}	0.88	1.98	2.8458 (16)	166
N3—H3*B*···O1^{ii}	0.88	2.00	2.8567 (16)	165
C4—H4*B*···O1^{iii}	0.99	2.58	3.4979 (19)	155

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x+1, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x+1, y, z$.

Acta Cryst. (2007). E**63**, o610–o612 Butcher *et al.* • C_5H_7NO **o611**

All H atoms were initially located in a difference Fourier map. The H atoms were then placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 and 0.98 Å for CH and CH_2, respectively, and $U_{iso}(H) = 1.2U_{eq}(C)$. The N—H hydrogen was constrained to 0.86 Å, with $U_{iso}(H) = 1.5U_{eq}(N)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

Support to EB was provided by (RCMI) NIH grant No. RR17581.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Braibante, H. T. S., Braibante, M. E. F., Rosso, G. B. & Oriques, D. A. (2003). *J. Braz. Chem. Soc.* **14**, 994–997.

Bruker (2000). *SHELXTL* (Version 6.14) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2001). *SMART*. Version 5.631. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2002). *SAINT*. Version 6.45A. Bruker AXS Inc., Madison, Wisconsin, USA.

Carroll, W. A., *et al.* (2004). *J. Med. Chem.* **47**, 3163–3179.

Fernandes, P. A., *et al.* (2004). *Theochem*, **685**, 73–82.

Foster, J. E., Nicholson, J. M., Butcher, R., Stables, J. P., Edafiogho, I. O., Goodwin, A. M., Henson, M. C., Smith, C. A. & Scott, K. R. (1999). *Bioorg. Med. Chem.* **7**, 2415–2425.

Huang, K.-S., Stowell, J. G. & Byrn, S. R. (1997). *Acta Cryst.* C**53**, 1717–1719.

Iida, H., Yuasa, Y. & Kibayashi, C. (1982). *Synthesis*, pp. 471–472.

Kikani, B. B., Mckee, J. R. & Zanger, M. (1991). *Synthesis*, pp. 176.

Putkonen, T., Tolvanen, A., Jokela, R., Caccamese, S. & Parrinello, N. (2003). *Tetrahedron*, **59**, 8589–8595.

Ruangsiyanand, C., Rimek, H. J. & Zymalkowski, F. (1970). *Chem. Ber.* **103**, 2403–2410.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

o612 Butcher *et al.* • C_5H_7NO *Acta Cryst.* (2007). E**63**, o610–o612