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3-Azabicyclo[3.3.1]nonane-2,4-dione: CSP2001 structure prediction test case No. 1

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CSP2001 reference: Motherwell (2001).

Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.123
Data-to-parameter ratio = 19.0

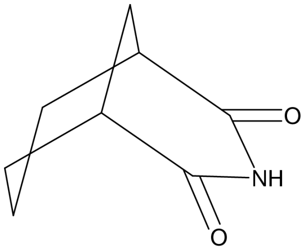
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Features of the structure of the title compound, $C_8H_{11}NO_2$, are the near planar arrangement of the dicarboximide fragment and the C atoms α to it and the molecular non-crystallographic C_s mirror symmetry. N—H···O hydrogen bonds link the molecules to form zigzag chains propagated in the a direction and sheets parallel to (001) are formed by replication of the chains by cell translation in the b direction. The sheets, related to one another by the operation of crystallographic centres of symmetry, are stacked in the c direction.

Comment

The structure of the title compound, (I), was undertaken initially simply because suitable crystals were available. It is gratifying that the compound was later found suitable for use as a case study for structure prediction.

Fig. 1 is a general view of the molecule of (I) showing the atom labels used in the X-ray structure analysis. Noted here is the near planar arrangement of the dicarboximide fragment of the molecule (N1, C1, O1, C5 and O2 including the α-C atoms, C2 and C4). The greatest displacement from the least squares plane so defined is that of O1 at 0.0391 (7) Å, while that of H1 (attached to N1) is only 0.005 (13) Å. Fig. 2 is another view of the molecule in which its non-crystallographic C_s mirror symmetry is clearly evident and seen to extend even as far as the H atoms. Bond distances and angles in various categories are summarized as ranges in Table 2. They are more or less as anticipated for a molecule of this kind and are not discussed further.



(I)

The arrangement of the molecules in the cell, particularly in the context of structure prediction, is worthy of discussion. The molecules are arranged in layers parallel to (001). One such layer, with the centroids of the molecules close to $z = \frac{1}{4}$, is shown in Fig. 3. Here intermolecular hydrogen bonds of the form N1—H1···O1 (Table 1) interconnect the molecules to

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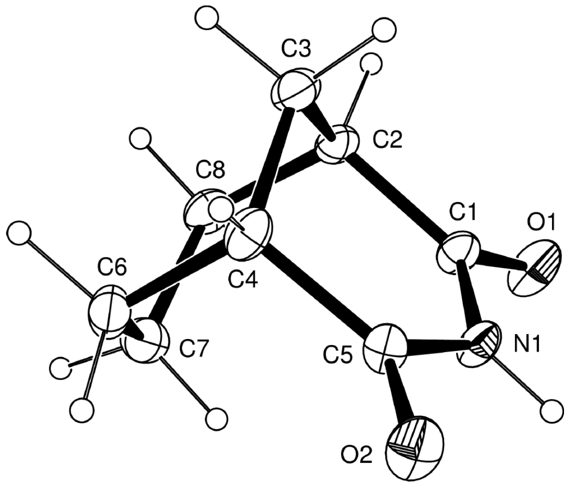

Figure 1
The molecule of (I) showing the atom labels. Non-H atoms are shown as 20% probability ellipsoids and H atoms are shown as small circles.

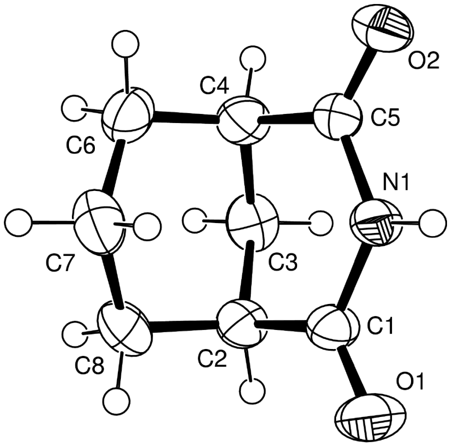

Figure 2
The molecule of (I) viewed edge-on to the plane defined by N1, C3 and C7 with C3 directed down into the page. Non-H atoms are now shown as 50% probability ellipsoids.

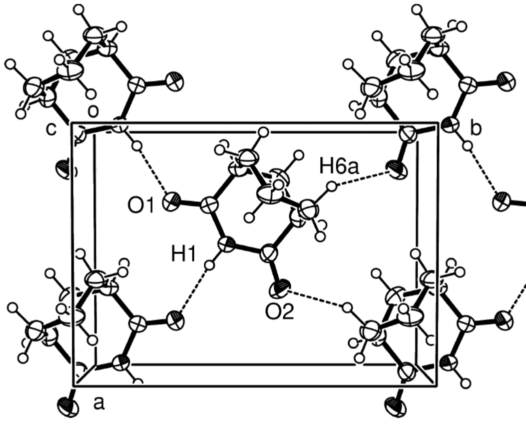

Figure 3
The view down *c* of a portion of a single layer of molecules of (I) whose centroids are close to $z = \frac{1}{4}$. The representation is the same as in Fig. 2, except that only selected atoms are now labelled in order to identify intermolecular contacts (dashed lines) which are discussed in more detail in the text.

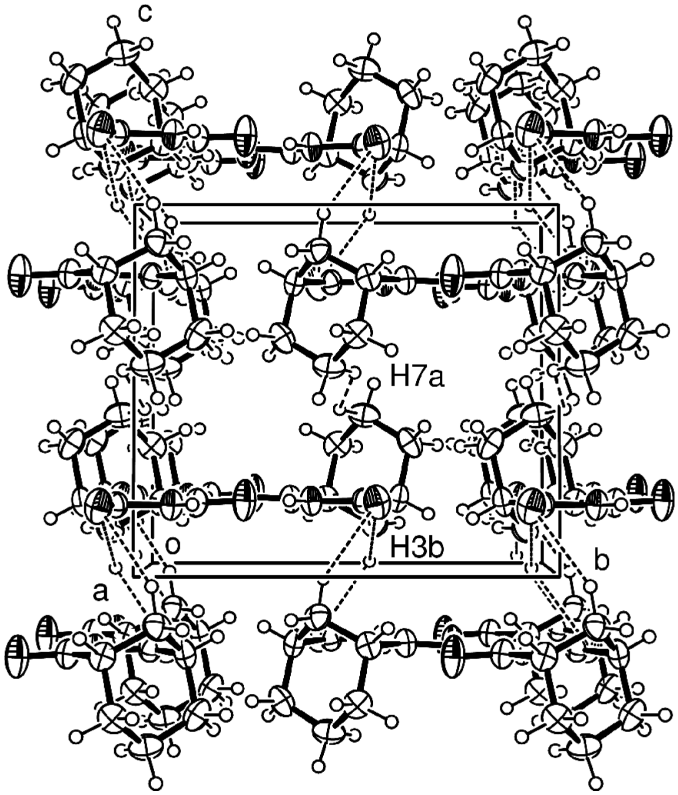

Figure 4
The unit cell of (I) viewed along *a* showing the stacking of the layers exemplified in Fig. 3 and in the same representation.

form zigzag chains propagated in the *a* direction in which each molecule is related to its neighbours by the operation of the *a*-glide of the space group $P2_1/a$. The chains are then related one to another by cell translation in the *b* direction creating in the process further intermolecular contacts of the form C6—H6*A*···O2 (Table 1).

These layers are then stacked in the *c* direction as shown in Fig. 4 where they are viewed along *a* and therefore seen edge-on. The layers are now related to one another by the operation of crystallographic centres of symmetry resulting in displacement of the molecules in the *ab* plane from one layer to the next and creating two cases. First, because of the choice of origin used in the structure analysis, the dicarboximide 'fronts' of the molecules are opposed to one another across interfaces at $z = 0$ and $z = 1$ creating intermolecular contacts of the form C3—H3*B*···O2 (Table 1). Second, in a similar manner, the alkyl 'backs' of the molecules are juxtaposed at $z = \frac{1}{2}$ at van der Waals distances of which H7···H7^{i} [symmetry code: (i) $1 - x$, $1 - y$, $1 - z$] at 2.28 (2) Å is the shortest.

Experimental

The material from which the sample crystal was taken was kindly supplied by P. J. Cox and so its provenance is limited. The synthesis of (I) has, however, been described by Hall (1958) and more recently by Poloński *et al.* (1996).

Crystal data

$C_8H_{11}NO_2$
$M_r = 153.18$
Monoclinic, $P2_1/a$
$a = 7.7046$ (5) Å
$b = 10.6062$ (7) Å
$c = 9.3384$ (6) Å
$\beta = 95.033$ (2)°
$V = 760.16$ (9) Å^3
$Z = 4$

$D_x = 1.338$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2072 reflections
$\theta = 2.2$–30.3°
$\mu = 0.10$ mm^{-1}
$T = 297$ (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ–ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{\min} = 0.972$, $T_{\max} = 0.981$
7616 measured reflections

2736 independent reflections
1584 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\max} = 32.5°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 7$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.123$
$S = 0.90$
2736 reflections
144 parameters

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0713P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1$^{\rm i}$	0.873 (15)	2.107 (15)	2.9739 (12)	172.2 (14)
C3—H3*B*···O2$^{\rm ii}$	1.004 (14)	2.594 (15)	3.5088 (16)	151.5 (11)
C6—H6*A*···O2$^{\rm iii}$	1.038 (16)	2.601 (17)	3.4313 (17)	136.7 (11)

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

Table 2
Selected bond lengths and angles (Å,°) for (I) summarized as ranges of values..

	Min.	Max.
C—N	1.3843 (13)	1.3866 (14)
C—O	1.2133 (13)	1.2199 (14)
C—C^a	1.5025 (15)	1.5042 (16)
C—C^b	1.5151 (18)	1.5379 (17)
C—N—C	125.86 (9)	
O—C—N	119.32 (10)	119.35 (10)
O—C—C	123.22 (10)	124.25 (11)
C—C—N	116.42 (9)	117.41 (10)
C—C—C	108.16 (10)	112.86 (10)

Notes: (*a*) $C_{\rm alkyl}$—$C_{\rm carbonyl}$; (*b*) alkyl C—C.

In the later stages of refinement, H atoms were found in a difference map and refined freely.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

The authors are deeply indebted to Philip J. Cox (Department of Chemistry, The Robert Gordon University, Aberdeen, Scotland) for providing the material from which the sample crystal was selected.

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