Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Azaspiro[5.5]undecane-2,4-dione

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.087
Data-to-parameter ratio = 18.1

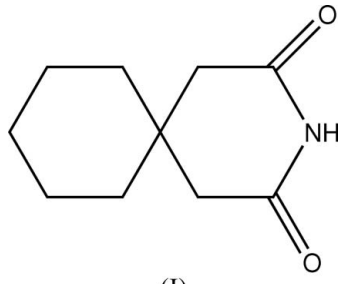
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 December 2005
Accepted 12 December 2005
Online 16 December 2005

In the title compound, $C_{10}H_{15}N_1O_2$, the cyclohexane ring adopts a chair conformation and the piperidine ring adopts an envelope conformation. In the crystal structure, hydrogen-bonded dimers are formed *via* N—H···O interactions, and the molecular packing is stabilized by van der Waals interactions.

Comment

As an efficient intermediate in the preparation of gabapentin, the title compound, (I), plays an important role in its organic synthesis (Ferrari *et al.*, 2004).



(I)

Bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). The cyclohexane ring has the expected chair conformation, atoms C3 and C8 having deviations of 0.648 (2) and −0.654 (2) Å, respectively, from the least-squares plane through the other four atoms. The piperidine ring adopts an envelope conformation, with atom C3 deviating by 0.654 (2) Å from the mean plane through the other five atoms. In the crystal structure, centrosymmetric $R_2^2(8)$ dimers (Etter, 1990) are formed through hydrogen-bonding interactions (Table 2) between the NH and carbonyl groups (Fig. 2). The molecular packing is further stabilized by van der Waals interactions.

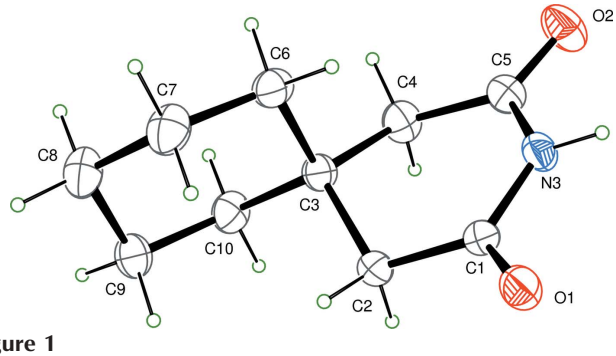


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 40% probability level. H atoms are drawn as spheres of arbitrary radius.

Experimental

A mixture of acetic anhydride (66.5 g, 0.65 mol), ammonium acetate (66.5 g, 0.86 mol) and 1,1-cyclohexanediacetic acid (100 g, 0.53 mol) was heated to 433–443 K for 8 h, eliminating by distillation the acetic acid that formed. Water (200 ml) and *sec*-butyl alcohol (100 g) were added after the mixture was cooled to 363–383 K. The pH was adjusted to 9 using 30% aqueous ammonia and the precipitate was collected, washed with water and recrystallized from methanol (300 ml) to give 84.2 g (yield 93.1%) of dry 3-azaspiro[5.5]undecane-2,4-dione (Ferrari *et al.*, 2004). This was recrystallized from a mixed solvent of ethanol and acetone (4:1 *v*/*v*), giving colorless crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_{10}H_{15}NO_2$	D_x = 1.273 Mg m^{-3}
M_r = 181.23	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7272 reflections
a = 11.402 (7) Å	θ = 3.1–27.5°
b = 6.231 (3) Å	μ = 0.09 mm^{-1}
c = 13.942 (5) Å	T = 296 (1) K
β = 107.324 (18)°	Block, colorless
V = 945.6 (8) Å^3	0.32 × 0.28 × 0.16 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	2152 independent reflections
ω scans	1530 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	R_{int} = 0.042
T_{min} = 0.968, T_{max} = 0.986	θ_{max} = 27.5°
8782 measured reflections	$h = -14 \rightarrow 14$
	$k = -8 \rightarrow 7$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[0.0002F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
2152 reflections	Extinction correction: Larson (1970)
119 parameters	Extinction coefficient: 76 (18)
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

O1—C1	1.2215 (14)	N3—C1	1.3754 (14)
O2—C5	1.2151 (14)	N3—C5	1.3817 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H301···O1^{i}	0.84	2.06	2.8885 (14)	171

Symmetry code: (i) $-x+1, -y+2, -z$.

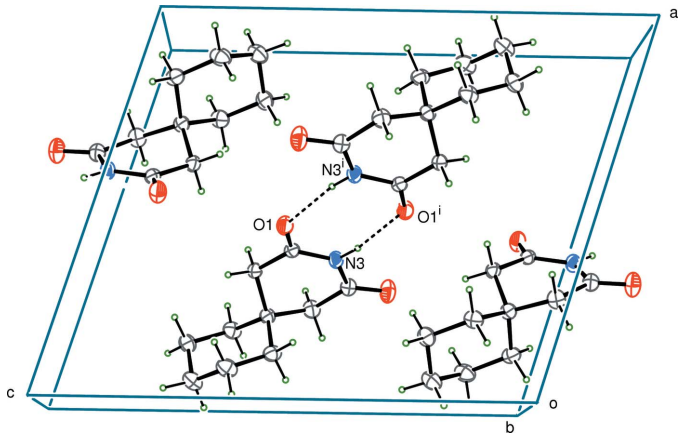


Figure 2
Partial packing digram for (I), showing the hydrogen-bonded (dashed lines) dimer [symmetry code: (i) $1-x, 2-y, -z$].

The H atoms of the amino group were located in difference Fourier maps and included in the refinement as riding, based on the as-found N—H bond lengths, but their isotropic displacement parameters were initially refined and then fixed in the final stage. All other H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the refinement in the riding model, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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