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3-Azabicyclo[3.3.1]nonane-2,4-dione–acetic acid (1/1)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.098
Data-to-parameter ratio = 11.8

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

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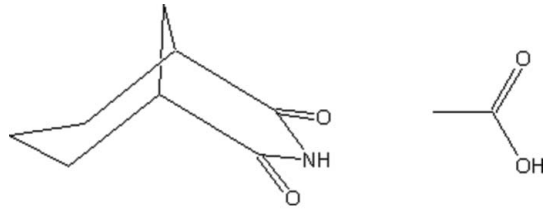
3-Azabicyclo[3.3.1]nonane-2,4-dione (cyclohexane-1,3-dicarboximide, $C_8H_{11}NO_2$) forms a 1:1 solvate with acetic acid ($C_2H_4O_2$). The crystal structure comprises hydrogen-bonded chains containing alternating cyclohexane-1,3-dicarboximide and acetic acid molecules.

Comment

The title solvate, (I), was first produced during an automated parallel crystallization screen on cyclohexane-1,3-dicarboximide. It was identified as a new crystal structure, different from the known unsolvated form (Howie & Skakle, 2001), by examination of its powder diffraction pattern, collected on a multi-sample X-ray powder diffractometer (Florence *et al.*, 2003). It was crystallized by crash cooling a subsaturated solution in glacial acetic acid from 383 to 288 K, and gave crystals of suitable size and quality for single-crystal X-ray diffraction.



(I)

The asymmetric unit of (I) contains one molecule of cyclohexane-1,3-dicarboximide and one molecule of acetic acid (Fig. 1). The structure exhibits a chain hydrogen-bonding motif [graph set $C_2^2(8)$], with cyclohexane-1,3-dicarboximide and acetic acid molecules alternating in the chain. The pair of hydrogen bonds (Table 1) to the acetic acid carboxyl group is in an *anti* configuration and only one of the carbonyl O atoms in the cyclohexane-1,3-dicarboximide molecule is used in the hydrogen bonding forming the chain (Fig. 2). There are no hydrogen bonds between different chains, but the chains stack upon one another, forming a column parallel to [001]. The alkyl substituents of the cyclohexane-1,3-dicarboximide molecules lie to the sides of the column, with the hydrogen-bonding substituents comprising the middle of the column (Fig. 3). Adjacent chains in the column have the cyclohexane-1,3-dicarboximide alkyl groups on alternating sides of the column.

The chain motif in this structure is closely related to the chain motif observed in both the anhydrous form of cyclohexane-1,3-dicarboximide and in the crystal structure of acetic acid. Fig. 4 shows overlays of the chain motif of (I) with the

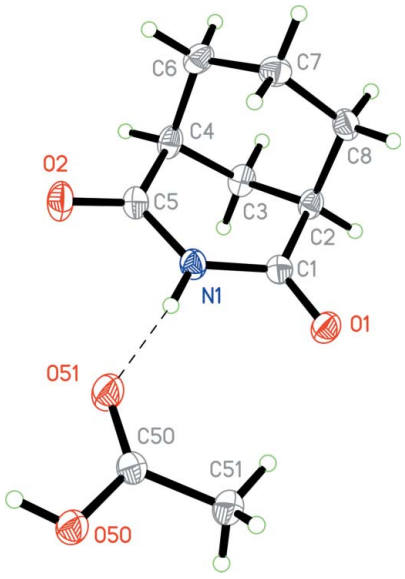


Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres. The dashed line indicates an N—H···O hydrogen bond.

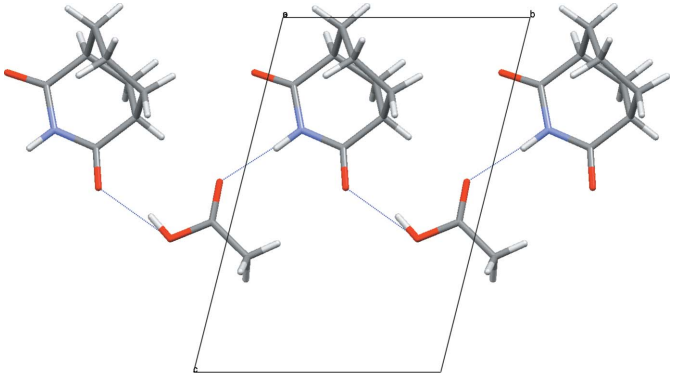


Figure 2
View perpendicular to the *bc* plane, showing the chain hydrogen-bonding motif present in (I). Dotted blue lines indicate hydrogen bonds.

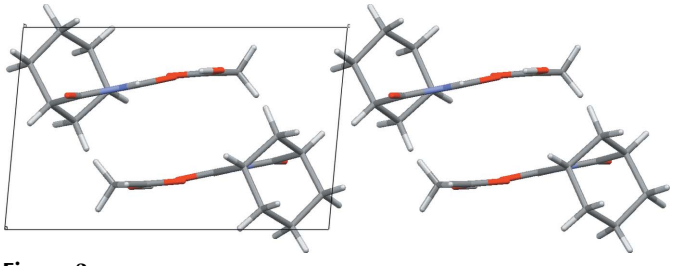


Figure 3
View perpendicular to the *ac* plane, showing the stacking of hydrogen-bonded chains.

chain from the unsolvated cyclohexane-1,3-dicarboximide structure (Howie & Skakle, 2001) and with the chain from the orthorhombic form of acetic acid (Boese *et al.*, 1999). From these overlays it can be seen that the basic hydrogen-bonded backbone is the same in each of these structures.

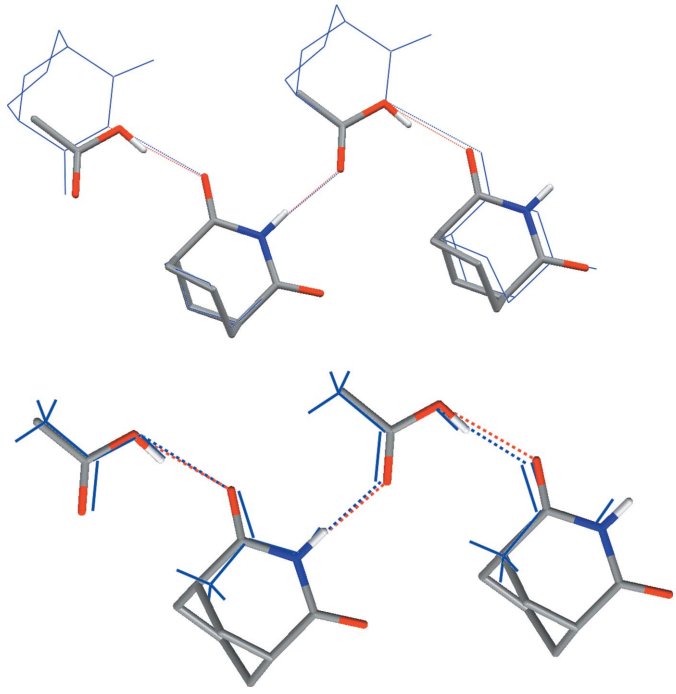


Figure 4
(*a*) Overlay of the chain present in (I) (normal colours) with the chain from unsolvated cyclohexane-1,3-dicarboxylic acid (blue). Dotted lines indicate hydrogen bonds; (*b*) overlay of the chain present in (I) with the chain from acetic acid (blue).

Experimental

3-Azabicyclo[3.3.1]nonane-2,4-dione (100 mg) was dissolved in glacial acetic acid (2 ml) at 383 K and crash cooled to 288 K to obtain single crystals of (I).

Crystal data

$C_8H_{11}NO_2\cdot C_2H_4O_2$
M_r = 213.23
Triclinic, $P\overline{1}$
a = 6.6224 (7) Å
b = 7.3580 (8) Å
c = 10.7995 (12) Å
α = 103.598 (2)°
β = 93.378 (2)°
γ = 97.272 (2)°
V = 505.22 (10) Å^3
Z = 2
D_x = 1.402 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2712 reflections
θ = 3.1–28.3°
μ = 0.11 mm^{-1}
T = 150 (2) K
Block, colourless
0.35 × 0.29 × 0.17 mm

Data collection

Bruker SMART APEX diffractometer
Narrow-frame ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.982
4424 measured reflections
2313 independent reflections
2121 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 28.3°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.04
2313 reflections
196 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.1277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O50—H50···O1^{i}	0.88 (2)	1.84 (2)	2.6849 (12)	160.2 (18)
N1—H1···O51	0.917 (16)	1.962 (16)	2.8752 (12)	174.0 (14)

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

All H atoms were located in a difference map and were refined isotropically; C—H bond lengths range from 0.94 (2) to 1.00 (2) Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Bruno *et al.*, 2002) and *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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