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Key indicators

Single-crystal X-ray study
 T = 150 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 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>.

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

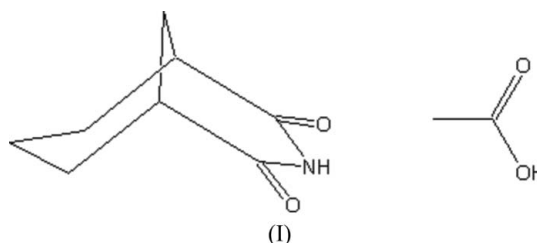
3-Azabicyclo[3.3.1]nonane-2,4-dione (cyclohexane-1,3-dicarboximide, $\text{C}_8\text{H}_{11}\text{NO}_2$) forms a 1:1 solvate with acetic acid ($\text{C}_2\text{H}_4\text{O}_2$). The crystal structure comprises hydrogen-bonded chains containing alternating cyclohexane-1,3-dicarboximide and acetic acid molecules.

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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.



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 $\text{C}_2^2(8)$], 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

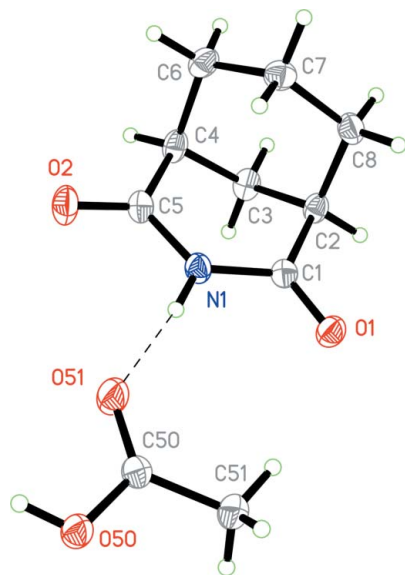


Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres. The dashed line indicates an N—H...O hydrogen bond.

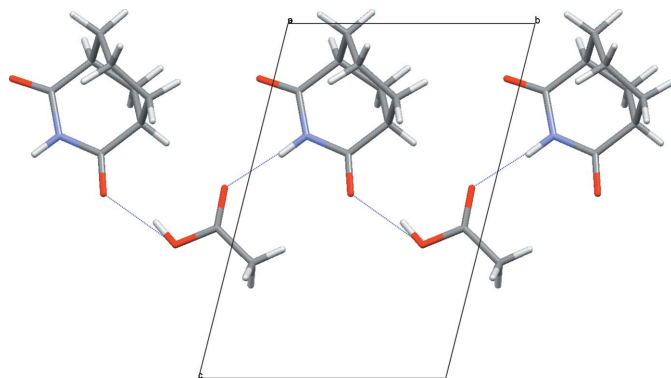


Figure 2
View perpendicular to the *bc* plane, showing the chain hydrogen-bonding motif present in (I). Dotted blue lines indicate hydrogen bonds.

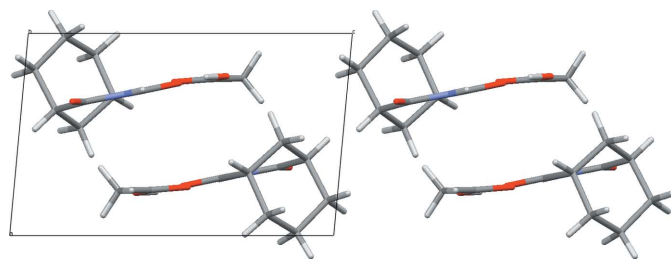


Figure 3
View perpendicular to the *ac* plane, showing the stacking of hydrogen-bonded chains.

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.

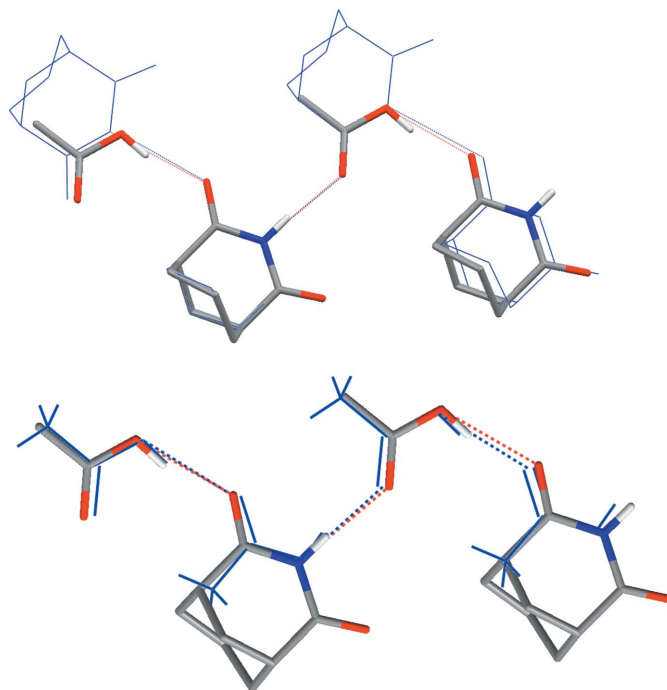


Figure 4
(a) Overlay of the chain present in (I) (normal colours) with the chain from unsolvated cyclohexane-1,3-dicarboxylic acid (blue). Dotted lines indicate hydrogen bonds; (b) overlay of the chain present in (I) with the chain from acetic acid (blue).

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, $P\bar{1}$
 $a = 6.6224$ (7) Å
 $b = 7.3580$ (8) Å
 $c = 10.7995$ (12) Å
 $\alpha = 103.598$ (2)°
 $\beta = 93.378$ (2)°
 $\gamma = 97.272$ (2)°
 $V = 505.22$ (10) Å³

$Z = 2$
 $D_x = 1.402$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2712 reflections
 $\theta = 3.1$ – 28.3 °
 $\mu = 0.11$ mm⁻¹
 $T = 150$ (2) K
 Block, colourless
 $0.35 \times 0.29 \times 0.17$ 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 $I > 2\sigma(I)$
 $R_{int} = 0.013$
 $\theta_{max} = 28.3$ °
 $h = -8 \rightarrow 8$
 $k = -9 \rightarrow 9$
 $l = -14 \rightarrow 13$

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, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O50—H50 \cdots O1 ⁱ	0.88 (2)	1.84 (2)	2.6849 (12)	160.2 (18)
N1—H1 \cdots 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: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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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