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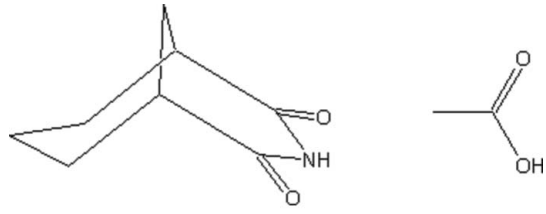
3-Azabicyclo[3.3.1]nonane-2,4-dione (cyclohexane-1,3-dicarboximide, $C_8H_{11}NO_2$) forms a 1:1 solvate with acetic acid ($C_2H_4O_2$). The crystal structure comprises hydrogen-bonded chains containing alternating cyclohexane-1,3-dicarboximide and acetic acid molecules.

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

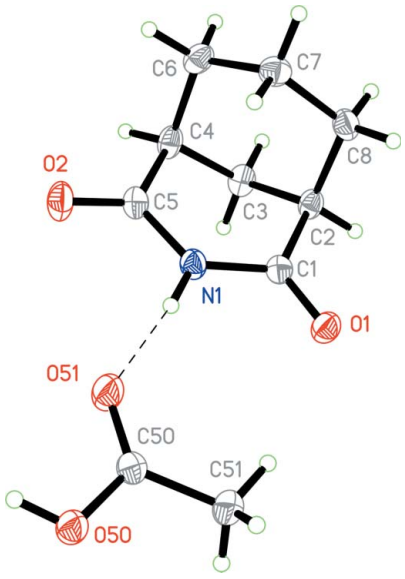


Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres. The dashed line indicates an N—H···O hydrogen bond.

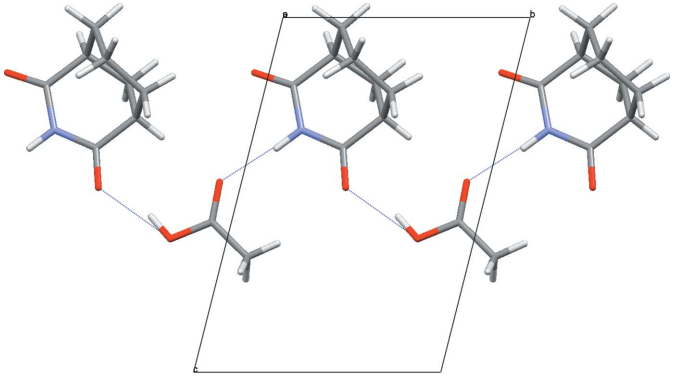


Figure 2
View perpendicular to the *bc* plane, showing the chain hydrogen-bonding motif present in (I). Dotted blue lines indicate hydrogen bonds.

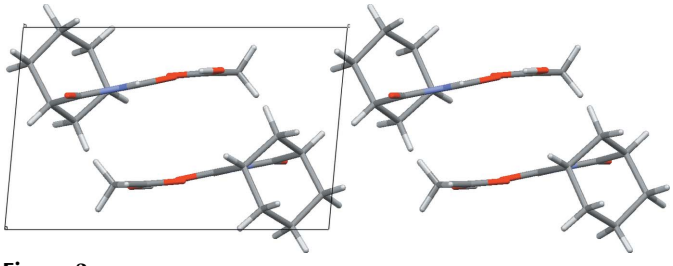


Figure 3
View perpendicular to the *ac* plane, showing the stacking of hydrogen-bonded chains.

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.

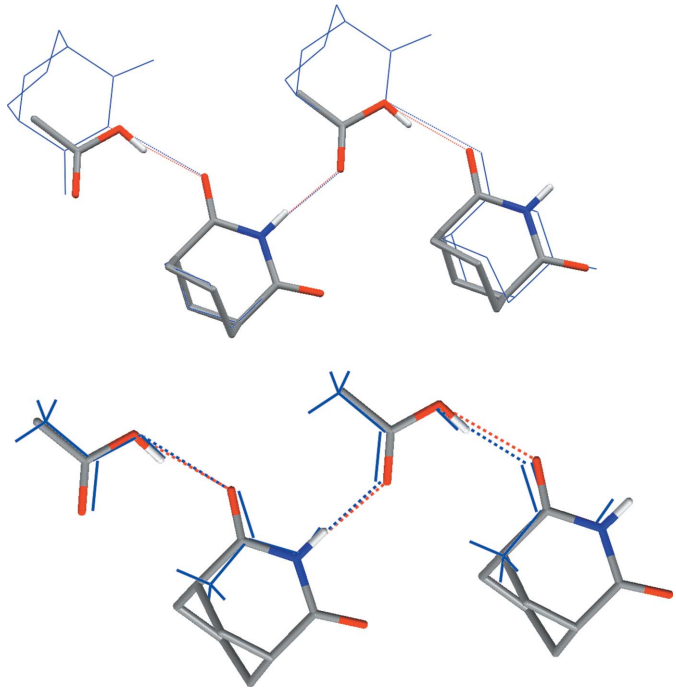


Figure 4
(*a*) Overlay of the chain present in (I) (normal colours) with the chain from unsolvated cyclohexane-1,3-dicarboxylic acid (blue). Dotted lines indicate hydrogen bonds; (*b*) overlay of the chain present in (I) with the chain from acetic acid (blue).

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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supporting information

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$\alpha = 103.598$ (2)°
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$\gamma = 97.272$ (2)°
$V = 505.22$ (10) Å^3
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$F(000) = 228$
$D_x = 1.402$ Mg m^{-3}
Melting point = 462–467 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2712 reflections
$\theta = 3.1$–28.3°
$\mu = 0.11$ mm^{-1}
$T = 150$ K
Block, colourless
0.35 × 0.29 × 0.17 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω rotation with narrow frames scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.963$, $T_{max} = 0.982$
4424 measured reflections
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2121 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.0°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.098$
$S = 1.04$
2313 reflections
196 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O51	0.24026 (16)	−0.08875 (12)	0.46398 (8)	0.0339 (2)
O50	0.21208 (13)	−0.22994 (11)	0.62416 (8)	0.0249 (2)
C51	0.2222 (2)	0.10010 (16)	0.67523 (11)	0.0274 (3)
H51A	0.102 (3)	0.087 (3)	0.7168 (18)	0.049 (5)*
H51B	0.333 (3)	0.116 (3)	0.7374 (18)	0.052 (5)*
H51C	0.223 (2)	0.207 (2)	0.6374 (16)	0.039 (4)*
C50	0.22593 (16)	−0.07895 (15)	0.57639 (10)	0.0206 (2)
H50	0.216 (3)	−0.330 (3)	0.5621 (18)	0.047 (5)*
O2	0.33694 (14)	−0.05870 (11)	0.15566 (8)	0.0301 (2)
O1	0.24571 (12)	0.42653 (11)	0.47939 (7)	0.02418 (19)
N1	0.29922 (14)	0.18908 (12)	0.31657 (9)	0.0207 (2)
H1	0.280 (2)	0.107 (2)	0.3685 (15)	0.034 (4)*
C8	0.10302 (17)	0.51372 (15)	0.21090 (10)	0.0231 (2)
H8A	0.006 (2)	0.551 (2)	0.2742 (14)	0.030 (4)*
H8B	0.127 (2)	0.613 (2)	0.1658 (14)	0.026 (3)*
C7	0.01654 (17)	0.32663 (16)	0.11624 (10)	0.0238 (2)
H7A	−0.032 (2)	0.233 (2)	0.1632 (13)	0.026 (3)*
H7B	−0.102 (2)	0.344 (2)	0.0648 (14)	0.032 (4)*
C6	0.17583 (17)	0.24951 (16)	0.02883 (10)	0.0237 (2)
H6A	0.207 (2)	0.329 (2)	−0.0312 (14)	0.029 (4)*
H6B	0.120 (2)	0.121 (2)	−0.0245 (14)	0.030 (4)*
C5	0.33876 (16)	0.11110 (15)	0.19154 (10)	0.0216 (2)
C4	0.37781 (16)	0.24459 (15)	0.10551 (10)	0.0219 (2)
H4	0.473 (2)	0.194 (2)	0.0481 (14)	0.026 (3)*
C3	0.46373 (17)	0.44298 (15)	0.18455 (11)	0.0226 (2)
H3A	0.599 (2)	0.445 (2)	0.2288 (14)	0.029 (3)*
H3B	0.481 (2)	0.529 (2)	0.1269 (13)	0.024 (3)*
C2	0.31151 (16)	0.50773 (14)	0.28069 (10)	0.0199 (2)
H2	0.361 (2)	0.629 (2)	0.3366 (13)	0.023 (3)*
C1	0.28424 (15)	0.37591 (14)	0.36779 (10)	0.0189 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O51	0.0602 (6)	0.0212 (4)	0.0231 (4)	0.0102 (4)	0.0106 (4)	0.0071 (3)
O50	0.0346 (4)	0.0192 (4)	0.0213 (4)	0.0040 (3)	0.0055 (3)	0.0052 (3)
C51	0.0358 (6)	0.0203 (5)	0.0246 (6)	0.0054 (4)	0.0021 (5)	0.0019 (4)
C50	0.0201 (5)	0.0191 (5)	0.0228 (5)	0.0028 (4)	0.0022 (4)	0.0053 (4)
O2	0.0410 (5)	0.0172 (4)	0.0324 (4)	0.0074 (3)	0.0093 (4)	0.0035 (3)
O1	0.0325 (4)	0.0207 (4)	0.0198 (4)	0.0045 (3)	0.0057 (3)	0.0046 (3)

N1	0.0255 (5)	0.0165 (4)	0.0215 (4)	0.0045 (3)	0.0051 (3)	0.0063 (3)
C8	0.0276 (5)	0.0207 (5)	0.0240 (5)	0.0084 (4)	0.0064 (4)	0.0080 (4)
C7	0.0235 (5)	0.0266 (6)	0.0221 (5)	0.0034 (4)	0.0023 (4)	0.0078 (4)
C6	0.0299 (6)	0.0214 (5)	0.0186 (5)	0.0006 (4)	0.0045 (4)	0.0039 (4)
C5	0.0209 (5)	0.0186 (5)	0.0251 (5)	0.0047 (4)	0.0048 (4)	0.0036 (4)
C4	0.0246 (5)	0.0188 (5)	0.0224 (5)	0.0034 (4)	0.0101 (4)	0.0031 (4)
C3	0.0230 (5)	0.0193 (5)	0.0253 (5)	0.0002 (4)	0.0082 (4)	0.0053 (4)
C2	0.0242 (5)	0.0145 (5)	0.0206 (5)	0.0011 (4)	0.0047 (4)	0.0033 (4)
C1	0.0174 (5)	0.0177 (5)	0.0209 (5)	0.0022 (4)	0.0018 (4)	0.0037 (4)

Geometric parameters (Å, º)

O51—C50	1.2092 (14)	C8—H8B	0.971 (15)
O50—C50	1.3259 (13)	C7—C6	1.5306 (15)
O50—H50	0.88 (2)	C7—H7A	0.982 (14)
C51—C50	1.4919 (15)	C7—H7B	0.971 (15)
C51—H51A	0.944 (19)	C6—C4	1.5402 (16)
C51—H51B	0.943 (19)	C6—H6A	0.984 (15)
C51—H51C	0.970 (17)	C6—H6B	0.995 (15)
O2—C5	1.2163 (14)	C5—C4	1.5126 (15)
O1—C1	1.2271 (13)	C4—C3	1.5264 (15)
N1—C1	1.3744 (13)	C4—H4	0.960 (14)
N1—C5	1.3916 (14)	C3—C2	1.5275 (14)
N1—H1	0.917 (16)	C3—H3A	0.989 (15)
C8—C7	1.5290 (16)	C3—H3B	0.989 (14)
C8—C2	1.5443 (15)	C2—C1	1.5043 (14)
C8—H8A	0.982 (15)	C2—H2	0.960 (14)
C50—O50—H50	108.9 (12)	C7—C6—H6B	110.0 (8)
C50—C51—H51A	108.4 (11)	C4—C6—H6B	110.4 (9)
C50—C51—H51B	108.5 (12)	H6A—C6—H6B	106.3 (12)
H51A—C51—H51B	107.1 (16)	O2—C5—N1	119.51 (10)
C50—C51—H51C	111.6 (10)	O2—C5—C4	123.15 (10)
H51A—C51—H51C	108.6 (14)	N1—C5—C4	117.33 (9)
H51B—C51—H51C	112.4 (15)	C5—C4—C3	110.52 (9)
O51—C50—O50	122.45 (10)	C5—C4—C6	109.11 (9)
O51—C50—C51	124.54 (10)	C3—C4—C6	109.86 (9)
O50—C50—C51	113.01 (9)	C5—C4—H4	106.4 (9)
C1—N1—C5	125.83 (9)	C3—C4—H4	111.2 (8)
C1—N1—H1	117.6 (10)	C6—C4—H4	109.6 (8)
C5—N1—H1	116.5 (10)	C4—C3—C2	108.09 (8)
C7—C8—C2	112.96 (9)	C4—C3—H3A	111.1 (8)
C7—C8—H8A	110.6 (9)	C2—C3—H3A	110.9 (8)
C2—C8—H8A	109.4 (8)	C4—C3—H3B	109.1 (8)
C7—C8—H8B	110.0 (8)	C2—C3—H3B	109.9 (8)
C2—C8—H8B	105.8 (8)	H3A—C3—H3B	107.8 (12)
H8A—C8—H8B	107.9 (12)	C1—C2—C3	109.90 (9)
C8—C7—C6	111.94 (9)	C1—C2—C8	109.72 (8)

C8—C7—H7A	109.7 (8)	C3—C2—C8	110.67 (9)
C6—C7—H7A	109.1 (8)	C1—C2—H2	104.9 (8)
C8—C7—H7B	109.7 (9)	C3—C2—H2	111.7 (8)
C6—C7—H7B	109.7 (9)	C8—C2—H2	109.8 (8)
H7A—C7—H7B	106.6 (12)	O1—C1—N1	119.50 (10)
C7—C6—C4	111.82 (9)	O1—C1—C2	123.37 (9)
C7—C6—H6A	110.7 (9)	N1—C1—C2	117.12 (9)
C4—C6—H6A	107.5 (9)		

C2—C8—C7—C6	−48.40 (12)	C6—C4—C3—C2	62.97 (11)
C8—C7—C6—C4	50.44 (12)	C4—C3—C2—C1	60.65 (11)
C1—N1—C5—O2	−175.89 (10)	C4—C3—C2—C8	−60.69 (11)
C1—N1—C5—C4	2.79 (16)	C7—C8—C2—C1	−67.27 (11)
O2—C5—C4—C3	−154.70 (11)	C7—C8—C2—C3	54.18 (11)
N1—C5—C4—C3	26.67 (13)	C5—N1—C1—O1	178.96 (10)
O2—C5—C4—C6	84.40 (13)	C5—N1—C1—C2	0.47 (15)
N1—C5—C4—C6	−94.23 (11)	C3—C2—C1—O1	148.81 (10)
C7—C6—C4—C5	62.82 (11)	C8—C2—C1—O1	−89.28 (12)
C7—C6—C4—C3	−58.48 (11)	C3—C2—C1—N1	−32.76 (12)
C5—C4—C3—C2	−57.48 (12)	C8—C2—C1—N1	89.15 (11)

Hydrogen-bond geometry (Å, º)

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*.