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Crystal structure of 2-cyano-*N*′-(cyclohexylidene)acetohydrazide

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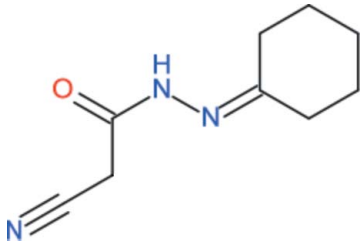
In the title compound, $C_9H_{13}N_3O$, the cyclohexylidene ring adopts a chair conformation and the bond-angle sum at the C atom linked to the N atom is 359.6°. The cyanoacetohydrazide grouping is close to planar (r.m.s. deviation for the non-H atoms = 0.031 Å) and subtends a dihedral angle of 64.08 (4)° with the four C atoms forming the seat of the chair. The C═O and N—H groups are in a *syn* conformation (O—C—N—H = −5°). In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds generate $R_2^2(8)$ loops; this dimer linkage is reinforced by a pair of C—H···O interactions, which generate $R_2^2(14)$ loops. The dimers are linked by C—H···N_c (c = cyanide) interactions into [100] ladders, which feature $C(4)$ chains and $R_4^4(20)$ loops.

Keywords: crystal structure; hydrazide; cyclohexylidene; inversion dimer.

CCDC reference: 1004279

1. Related literature

For background to the role of hydrazides as potential anticancer agents, see: Sechi *et al.* (2008); Manivel *et al.* (2009); Mohareb *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_9H_{13}N_3O$
M_r = 179.22
Triclinic, $P\overline{1}$
a = 4.8420 (2) Å
b = 9.7407 (7) Å
c = 10.7071 (8) Å
α = 73.917 (9)°
β = 82.819 (10)°
γ = 75.980 (9)°
V = 469.87 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.13 × 0.12 × 0.04 mm

2.2. Data collection

Rigaku Mercury CCD diffractometer
6176 measured reflections
2136 independent reflections
1789 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.101
S = 1.08
2136 reflections
121 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 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
N2—H1···O1^{i}	0.900 (14)	2.052 (15)	2.9399 (12)	168.4 (11)
C6—H6B···O1^{i}	0.99	2.32	3.2736 (13)	161
C8—H8B···N3^{ii}	0.99	2.41	3.3783 (14)	165

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x+1, y, z$.

Data collection: *CrystalClear* (Rigaku, 2012); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU0002).

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S1. Experimental

Cyclohexanone (0.98 g, 0.01 mol) was added to a solution of cyanoacetylhydrazine (0.99 g, 0.01 mol) in 1,4-dioxane (20 ml). The mixture was heated under reflux for 2 h and then poured into a beaker containing an ice/water mixture: the solid product was collected by filtration. Yellow slabs of the title compound were obtained by slow evaporation of an ethanol solution.

S2. Refinement

The N-bound H atom was located in a difference map and its position was freely refined. The C-bound H atoms were placed in idealized locations (C—H = 0.99 Å) and refined as riding atoms. The constraint U_{iso}(H) = 1.2U_{eq}(carrier) was applied in all cases.

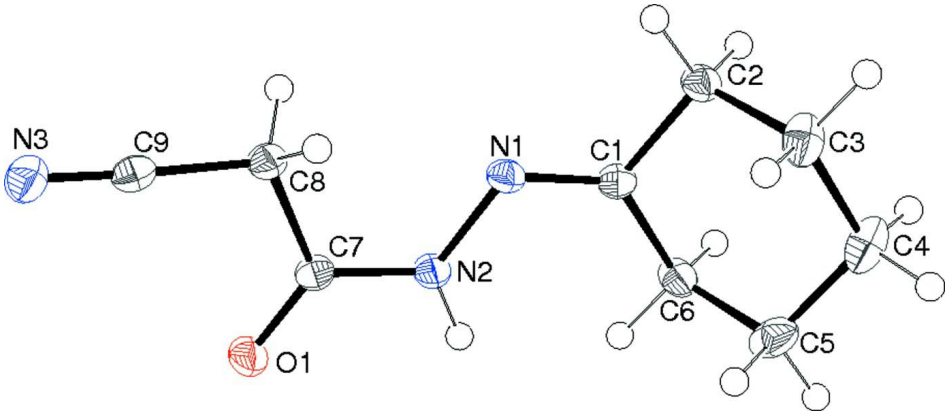

Figure 1

The molecular structure of the title compound showing 50% displacement ellipsoids.

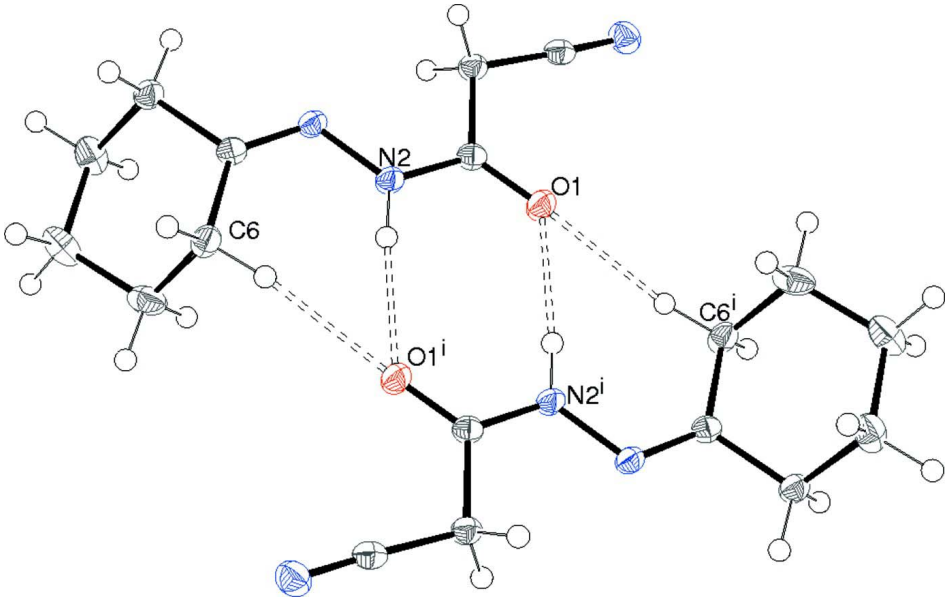

Figure 2

An inversion dimer in the crystal of the title compound, with N—H···O and C—H···O hydrogen bonds indicated by double-dashed lines. Symmetry code: (i) –x, 1–y, –z.

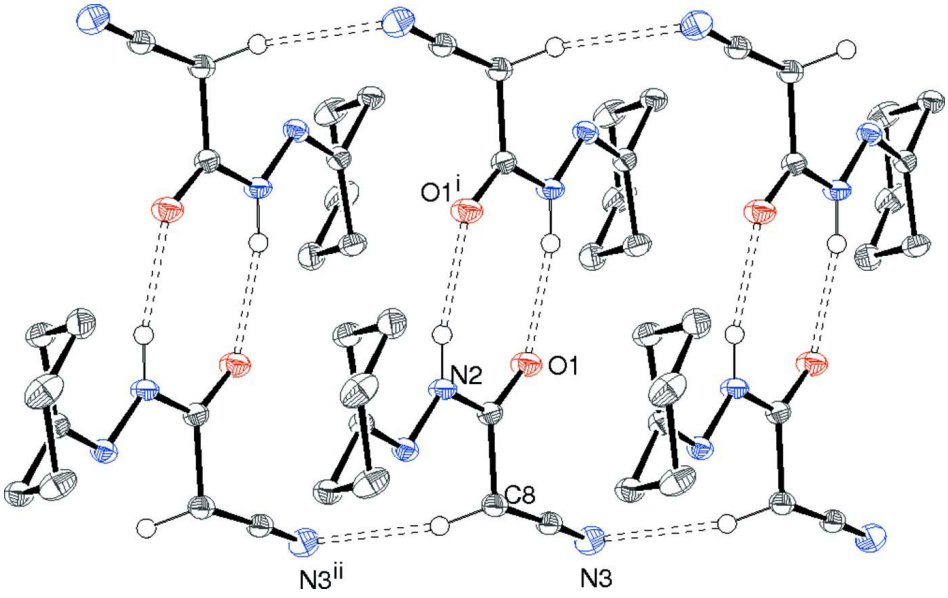

Figure 3

Part of a [100] double chain in the crystal of the title compound, with hydrogen bonds indicated by double-dashed lines. Symmetry codes: (i) –x, 1–y, –z; (ii) 1 + *x*, *y*, *z*.

2-Cyano-*N*′-(cyclohexylidene)acetohydrazide

Crystal data

$C_9H_{13}N_3O$
M_r = 179.22
Triclinic, $P\overline{1}$

Hall symbol: -P 1
a = 4.8420 (2) Å
b = 9.7407 (7) Å

c = 10.7071 (8) Å
α = 73.917 (9)°
β = 82.819 (10)°
γ = 75.980 (9)°
V = 469.87 (5) Å^3
Z = 2
F(000) = 192
D_x = 1.267 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5790 reflections
θ = 2.6–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Cut slab, yellow
0.13 × 0.12 × 0.04 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
6176 measured reflections
2136 independent reflections

1789 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 2.6°
$h = -6 \rightarrow 5$
$k = -12 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.101
S = 1.08
2136 reflections
121 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.0762P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.29 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.5886 (2)	0.25153 (11)	0.23432 (10)	0.0172 (2)
C2	0.7514 (2)	0.14590 (11)	0.34545 (10)	0.0201 (2)
H2A	0.6664	0.1673	0.4289	0.024*
H2B	0.9522	0.1560	0.3350	0.024*
C3	0.7394 (2)	−0.01067 (12)	0.34647 (11)	0.0264 (3)
H3A	0.8587	−0.0815	0.4150	0.032*
H3B	0.5406	−0.0233	0.3673	0.032*
C4	0.8456 (3)	−0.04206 (13)	0.21466 (12)	0.0290 (3)
H4A	0.8225	−0.1407	0.2159	0.035*

H4B	1.0513	−0.0415	0.1989	0.035*
C5	0.6822 (2)	0.07141 (13)	0.10418 (11)	0.0272 (3)
H5A	0.4801	0.0636	0.1148	0.033*
H5B	0.7631	0.0511	0.0199	0.033*
C6	0.6986 (2)	0.22686 (12)	0.10284 (10)	0.0217 (2)
H6A	0.8983	0.2385	0.0843	0.026*
H6B	0.5815	0.2992	0.0343	0.026*
C7	−0.0539 (2)	0.51593 (11)	0.19148 (10)	0.0173 (2)
C8	−0.1182 (2)	0.51675 (11)	0.33402 (10)	0.0188 (2)
H8A	−0.1150	0.4156	0.3873	0.023*
H8B	0.0320	0.5522	0.3629	0.023*
C9	−0.3962 (2)	0.61033 (11)	0.35514 (10)	0.0190 (2)
N1	0.36168 (17)	0.33947 (9)	0.26408 (8)	0.0176 (2)
N2	0.19288 (17)	0.42828 (9)	0.16325 (8)	0.0183 (2)
H1	0.227 (3)	0.4221 (14)	0.0801 (14)	0.022*
N3	−0.61224 (19)	0.68227 (10)	0.37675 (9)	0.0251 (2)
O1	−0.21717 (15)	0.59183 (8)	0.10701 (7)	0.0228 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0182 (4)	0.0185 (5)	0.0157 (5)	−0.0059 (4)	−0.0009 (4)	−0.0038 (4)
C2	0.0207 (5)	0.0225 (5)	0.0153 (5)	−0.0025 (4)	−0.0019 (4)	−0.0038 (4)
C3	0.0310 (6)	0.0197 (5)	0.0229 (6)	−0.0032 (4)	0.0056 (5)	−0.0016 (4)
C4	0.0347 (6)	0.0210 (5)	0.0309 (6)	−0.0078 (4)	0.0109 (5)	−0.0102 (5)
C5	0.0217 (5)	0.0400 (7)	0.0266 (6)	−0.0090 (5)	0.0045 (4)	−0.0201 (5)
C6	0.0179 (5)	0.0282 (6)	0.0150 (5)	−0.0001 (4)	0.0000 (4)	−0.0036 (4)
C7	0.0189 (5)	0.0185 (5)	0.0151 (5)	−0.0058 (4)	0.0002 (4)	−0.0043 (4)
C8	0.0192 (5)	0.0217 (5)	0.0155 (5)	−0.0036 (4)	−0.0009 (4)	−0.0057 (4)
C9	0.0241 (5)	0.0199 (5)	0.0149 (5)	−0.0085 (4)	0.0000 (4)	−0.0050 (4)
N1	0.0196 (4)	0.0185 (4)	0.0142 (4)	−0.0041 (3)	−0.0031 (3)	−0.0027 (3)
N2	0.0196 (4)	0.0215 (5)	0.0118 (4)	−0.0018 (3)	−0.0011 (3)	−0.0034 (3)
N3	0.0253 (5)	0.0255 (5)	0.0240 (5)	−0.0037 (4)	0.0020 (4)	−0.0087 (4)
O1	0.0221 (4)	0.0263 (4)	0.0163 (4)	0.0009 (3)	−0.0027 (3)	−0.0043 (3)

Geometric parameters (Å, º)

C1—N1	1.2845 (13)	C5—H5A	0.99
C1—C2	1.5032 (14)	C5—H5B	0.99
C1—C6	1.5036 (14)	C6—H6A	0.99
C2—C3	1.5371 (15)	C6—H6B	0.99
C2—H2A	0.99	C7—O1	1.2306 (12)
C2—H2B	0.99	C7—N2	1.3442 (13)
C3—C4	1.5269 (16)	C7—C8	1.5209 (14)
C3—H3A	0.99	C8—C9	1.4622 (14)
C3—H3B	0.99	C8—H8A	0.99
C4—C5	1.5273 (17)	C8—H8B	0.99
C4—H4A	0.99	C9—N3	1.1457 (13)

C4—H4B	0.99	N1—N2	1.3938 (12)
C5—C6	1.5314 (16)	N2—H1	0.900 (14)

N1—C1—C2	116.83 (9)	C4—C5—H5B	109.4
N1—C1—C6	128.63 (9)	C6—C5—H5B	109.4
C2—C1—C6	114.13 (8)	H5A—C5—H5B	108.0
C1—C2—C3	108.83 (9)	C1—C6—C5	108.10 (9)
C1—C2—H2A	109.9	C1—C6—H6A	110.1
C3—C2—H2A	109.9	C5—C6—H6A	110.1
C1—C2—H2B	109.9	C1—C6—H6B	110.1
C3—C2—H2B	109.9	C5—C6—H6B	110.1
H2A—C2—H2B	108.3	H6A—C6—H6B	108.4
C4—C3—C2	111.01 (9)	O1—C7—N2	122.06 (9)
C4—C3—H3A	109.4	O1—C7—C8	121.97 (9)
C2—C3—H3A	109.4	N2—C7—C8	115.97 (9)
C4—C3—H3B	109.4	C9—C8—C7	111.64 (8)
C2—C3—H3B	109.4	C9—C8—H8A	109.3
H3A—C3—H3B	108.0	C7—C8—H8A	109.3
C3—C4—C5	111.44 (9)	C9—C8—H8B	109.3
C3—C4—H4A	109.3	C7—C8—H8B	109.3
C5—C4—H4A	109.3	H8A—C8—H8B	108.0
C3—C4—H4B	109.3	N3—C9—C8	177.32 (11)
C5—C4—H4B	109.3	C1—N1—N2	117.65 (9)
H4A—C4—H4B	108.0	C7—N2—N1	119.30 (9)
C4—C5—C6	111.25 (9)	C7—N2—H1	116.5 (8)
C4—C5—H5A	109.4	N1—N2—H1	123.6 (8)
C6—C5—H5A	109.4		

N1—C1—C2—C3	114.56 (10)	O1—C7—C8—C9	3.02 (14)
C6—C1—C2—C3	−58.72 (11)	N2—C7—C8—C9	−177.51 (9)
C1—C2—C3—C4	54.68 (12)	C2—C1—N1—N2	−173.88 (8)
C2—C3—C4—C5	−54.91 (12)	C6—C1—N1—N2	−1.72 (16)
C3—C4—C5—C6	55.98 (12)	O1—C7—N2—N1	−176.79 (9)
N1—C1—C6—C5	−113.10 (12)	C8—C7—N2—N1	3.75 (13)
C2—C1—C6—C5	59.23 (11)	C1—N1—N2—C7	176.44 (9)
C4—C5—C6—C1	−56.13 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O1^{i}	0.900 (14)	2.052 (15)	2.9399 (12)	168.4 (11)
C6—H6*B*···O1^{i}	0.99	2.32	3.2736 (13)	161
C8—H8*B*···N3^{ii}	0.99	2.41	3.3783 (14)	165

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