



Crystal structure of 2-oxo-*N'*-phenyl-2*H*-chromene-3-carbohydrazide

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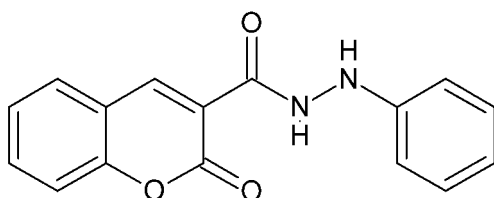
In the title compound, C₁₆H₁₂N₂O₃, the 2*H*-chromene moiety is essentially planar, with an r.m.s. deviation of the nine constituent atoms from the mean plane of 0.0093 Å, and makes a dihedral angle of 76.84 (3)° with the pendant phenyl ring. An intramolecular N—H···O hydrogen bond helps to determine the conformation of the side chain. In the crystal, N—H···O and N—H···N hydrogen bonds link the molecules, forming [100] chains.

Keywords: crystal structure; coumarins; bio-activity; coumarin scaffold compounds.

CCDC reference: 1438684

1. Related literature

For synthesis and bio-activity of coumarin scaffold compounds, see: Shivashankar *et al.* (2008*a,b*, 2009); Bansal *et al.* (2013); Jacquot *et al.* (2007); Bhavsar *et al.* (2011).



2. Experimental

2.1. Crystal data

C ₁₆ H ₁₂ N ₂ O ₃	$\gamma = 94.757 (2)^\circ$
$M_r = 280.28$	$V = 639.31 (4) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.6508 (2) \text{ \AA}$	Cu $K\alpha$ radiation
$b = 8.3906 (3) \text{ \AA}$	$\mu = 0.85 \text{ mm}^{-1}$
$c = 11.6388 (4) \text{ \AA}$	$T = 150 \text{ K}$
$\alpha = 96.504 (2)^\circ$	$0.19 \times 0.13 \times 0.05 \text{ mm}$
$\beta = 95.614 (2)^\circ$	

2.2. Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer	4865 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2015)	2371 independent reflections
$T_{\min} = 0.88$, $T_{\max} = 0.96$	2121 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.107$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
2371 reflections	
199 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots N2^i$	0.896 (18)	2.327 (18)	3.0498 (14)	137.7 (15)
$N1-H1N\cdots O2$	0.896 (18)	2.112 (18)	2.7544 (13)	127.8 (15)
$N2-H2N\cdots O2^{ii}$	0.911 (17)	2.243 (17)	3.1358 (14)	166.3 (14)

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

Data collection: *APEX2* (Bruker, 2015); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2144).

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supporting information

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S1. Comment

Coumarins are known to be biologically versatile compounds possessing several biological properties. Numerous research reports have indicated the coumarin nucleus as a potential candidate for development of anti-inflammatory (Shivashankar *et al.*, 2008*a,b*; Bansal *et al.*, 2013), antibacterial (Shivashankar *et al.*, 2008*b*), antifungal (Shivashankar *et al.*, 2009), anti-cancer (Jacquot *et al.*, 2007) and anti-HIV (Bhavsar *et al.*, 2011) agents. In light of such facts, we report in this study the synthesis and crystal structure of the title compound.

The 2*H*-chromene moiety is essentially planar with an r.m.s. deviation of the nine constituent atoms from the mean plane of 0.0093 Å. The dihedral angle between this plane and that of the pendant phenyl ring is 76.84 (3)°. The conformation of the hydrazide side-chain is partially determined by an intramolecular N1—H1N···O2 hydrogen bond (Fig. 1 and Table 1). The packing is assisted by intermolecular N2—H2N···O2ⁱ (i: $x - 1, y, z$) and N1—H1N···N2ⁱⁱ (ii: $-x + 1, -y + 1, -z$) hydrogen bonds (Fig. 2 and Table 1).

S2. Experimental

The title compound was obtained as an unexpected product from the reaction of 1-phenylpyrazolidine-3,5-dione (176 mg, 1 mmol), 2-hydroxybenzaldehyde (122 mg, 1 mmol) and *o*-toluidine (107 mg, 1 mmol). The reaction mixture was refluxed in 20 mL ethanol and monitored by TLC till completion. On cooling, the solid product was deposited, filtered off under vacuum and recrystallized from ethanol to afford colourless crystals in a sufficient quality for x-ray diffraction. *Mp* 471–473 K.

S3. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

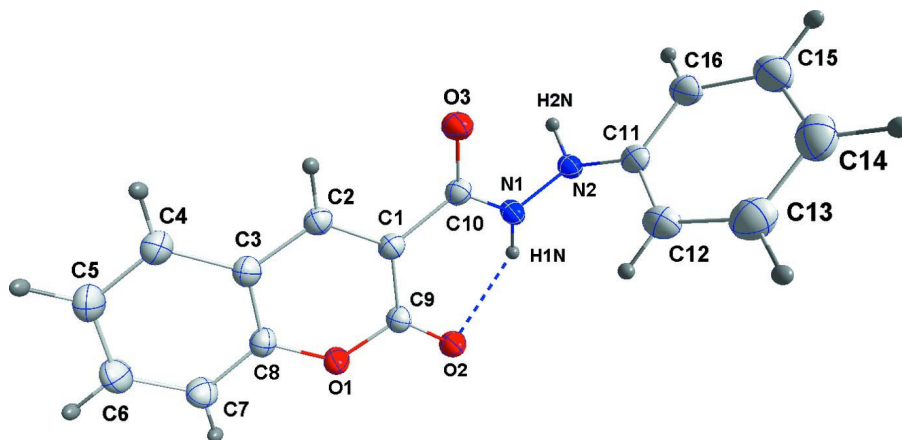


Figure 1

The title molecule with labeling scheme and 50% probability ellipsoids. The intramolecular N—H...O hydrogen bond is shown by a dotted line.

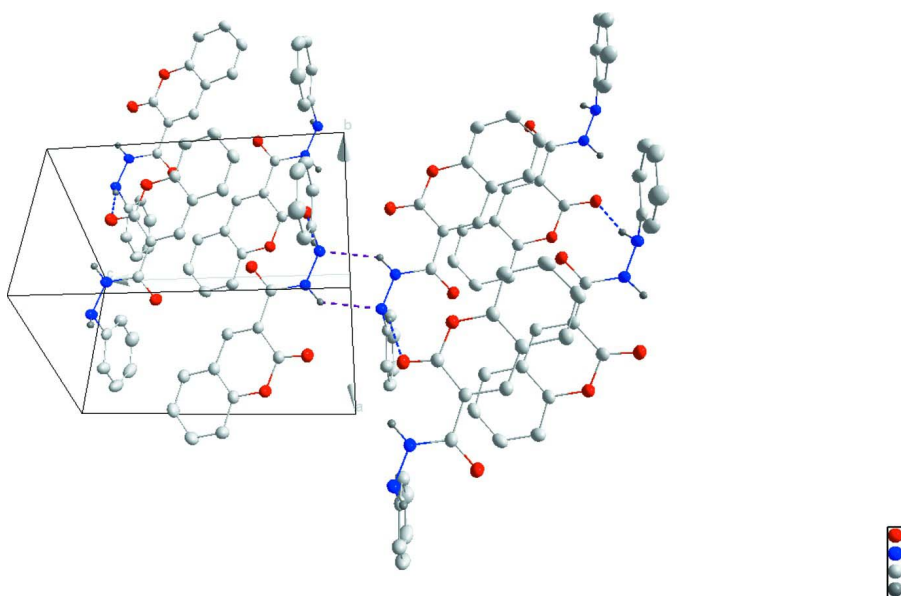


Figure 2

Packing viewed towards (110) with intermolecular N—H...O and N—H...N hydrogen bonds shown, respectively, as blue and purple dotted lines.

2-Oxo-*N'*-phenyl-2*H*-chromene-3-carbohydrazide

Crystal data

$C_{16}H_{12}N_2O_3$

$M_r = 280.28$

Triclinic, $P\bar{1}$

$a = 6.6508(2) \text{ \AA}$

$b = 8.3906(3) \text{ \AA}$

$c = 11.6388(4) \text{ \AA}$

$\alpha = 96.504(2)^\circ$

$\beta = 95.614(2)^\circ$

$\gamma = 94.757(2)^\circ$

$V = 639.31(4) \text{ \AA}^3$

$Z = 2$

$F(000) = 292$

$D_x = 1.456 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 3784 reflections

$\theta = 3.9\text{--}72.2^\circ$

$\mu = 0.85 \text{ mm}^{-1}$
 $T = 150 \text{ K}$

Tablet, colourless
 $0.19 \times 0.13 \times 0.05 \text{ mm}$

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS
 diffractometer
 Radiation source: INCOATEC $I\mu\text{S}$ micro-focus
 source
 Mirror monochromator
 Detector resolution: $10.4167 \text{ pixels mm}^{-1}$
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2015)

$T_{\text{min}} = 0.88, T_{\text{max}} = 0.96$
 4865 measured reflections
 2371 independent reflections
 2121 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 72.3^\circ, \theta_{\text{min}} = 3.9^\circ$
 $h = -7 \rightarrow 7$
 $k = -9 \rightarrow 10$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.107$
 $S = 1.06$
 2371 reflections
 199 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.1234P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{Å}^{-3}$
 Extinction correction: SHELXL2014 (Sheldrick
 2015b), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0129 (16)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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. H-atoms attached to carbon were placed in calculated positions ($C-H = 0.95 \text{ Å}$) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.05849 (13)	0.30993 (11)	0.29083 (7)	0.0271 (2)
O2	0.92419 (13)	0.42696 (12)	0.14651 (7)	0.0299 (2)
O3	0.43044 (13)	0.53972 (11)	0.33020 (7)	0.0292 (2)
N1	0.52900 (16)	0.50161 (13)	0.14932 (9)	0.0256 (3)
N2	0.35679 (16)	0.56386 (13)	0.09797 (9)	0.0245 (3)
C1	0.73626 (18)	0.41687 (14)	0.31115 (10)	0.0220 (3)
C2	0.74197 (18)	0.37498 (14)	0.42062 (10)	0.0233 (3)
H2	0.6338	0.3978	0.4656	0.028*
C3	0.90772 (18)	0.29704 (14)	0.46963 (10)	0.0240 (3)
C4	0.9195 (2)	0.24825 (16)	0.58159 (11)	0.0275 (3)

H4	0.8142	0.2670	0.6296	0.033*
C5	1.0850 (2)	0.17297 (16)	0.62133 (11)	0.0300 (3)
H5	1.0926	0.1391	0.6967	0.036*
C6	1.2410 (2)	0.14627 (16)	0.55202 (11)	0.0305 (3)
H6	1.3550	0.0960	0.5812	0.037*
C7	1.2321 (2)	0.19208 (16)	0.44109 (11)	0.0295 (3)
H7	1.3377	0.1730	0.3934	0.035*
C8	1.06438 (19)	0.26654 (15)	0.40167 (10)	0.0243 (3)
C9	0.90475 (18)	0.38814 (15)	0.24263 (10)	0.0236 (3)
C10	0.55367 (18)	0.49380 (14)	0.26524 (10)	0.0229 (3)
C11	0.35631 (19)	0.73381 (15)	0.11707 (9)	0.0246 (3)
C12	0.5353 (2)	0.83552 (17)	0.13527 (11)	0.0324 (3)
H12	0.6626	0.7916	0.1390	0.039*
C13	0.5280 (2)	1.00138 (18)	0.14802 (13)	0.0401 (4)
H13	0.6509	1.0703	0.1608	0.048*
C14	0.3449 (2)	1.06733 (18)	0.14238 (12)	0.0390 (4)
H14	0.3410	1.1810	0.1518	0.047*
C15	0.1668 (2)	0.96594 (18)	0.12283 (12)	0.0367 (3)
H15	0.0400	1.0106	0.1185	0.044*
C16	0.1711 (2)	0.80054 (16)	0.10956 (11)	0.0300 (3)
H16	0.0478	0.7323	0.0953	0.036*
H2N	0.242 (3)	0.5112 (19)	0.1179 (14)	0.033 (4)*
H1N	0.621 (3)	0.472 (2)	0.1014 (16)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0260 (5)	0.0348 (5)	0.0226 (4)	0.0082 (4)	0.0054 (3)	0.0060 (4)
O2	0.0274 (5)	0.0422 (6)	0.0222 (4)	0.0063 (4)	0.0070 (3)	0.0075 (4)
O3	0.0284 (5)	0.0393 (5)	0.0221 (4)	0.0101 (4)	0.0069 (4)	0.0040 (4)
N1	0.0259 (5)	0.0337 (6)	0.0194 (5)	0.0111 (4)	0.0041 (4)	0.0048 (4)
N2	0.0230 (5)	0.0297 (6)	0.0218 (5)	0.0067 (4)	0.0027 (4)	0.0048 (4)
C1	0.0234 (6)	0.0217 (6)	0.0207 (6)	0.0020 (4)	0.0035 (5)	0.0012 (4)
C2	0.0247 (6)	0.0240 (6)	0.0210 (6)	0.0021 (4)	0.0037 (5)	0.0012 (4)
C3	0.0264 (6)	0.0227 (6)	0.0222 (6)	0.0016 (5)	0.0014 (5)	0.0014 (5)
C4	0.0308 (7)	0.0289 (7)	0.0232 (6)	0.0030 (5)	0.0036 (5)	0.0043 (5)
C5	0.0358 (7)	0.0296 (7)	0.0243 (6)	0.0027 (5)	-0.0012 (5)	0.0065 (5)
C6	0.0301 (7)	0.0295 (7)	0.0317 (7)	0.0061 (5)	-0.0029 (5)	0.0056 (5)
C7	0.0274 (7)	0.0318 (7)	0.0299 (6)	0.0068 (5)	0.0031 (5)	0.0030 (5)
C8	0.0274 (6)	0.0240 (6)	0.0211 (6)	0.0015 (5)	0.0011 (5)	0.0029 (4)
C9	0.0244 (6)	0.0246 (6)	0.0215 (6)	0.0022 (4)	0.0020 (5)	0.0020 (4)
C10	0.0250 (6)	0.0239 (6)	0.0200 (6)	0.0026 (4)	0.0043 (5)	0.0022 (4)
C11	0.0297 (6)	0.0299 (7)	0.0156 (5)	0.0065 (5)	0.0048 (4)	0.0038 (4)
C12	0.0299 (7)	0.0364 (8)	0.0307 (7)	0.0051 (5)	0.0025 (5)	0.0025 (5)
C13	0.0423 (8)	0.0360 (8)	0.0409 (8)	-0.0017 (6)	0.0064 (6)	0.0015 (6)
C14	0.0562 (9)	0.0290 (7)	0.0345 (7)	0.0098 (6)	0.0128 (7)	0.0044 (5)
C15	0.0423 (8)	0.0398 (8)	0.0325 (7)	0.0181 (6)	0.0097 (6)	0.0079 (6)
C16	0.0292 (7)	0.0354 (7)	0.0272 (6)	0.0086 (5)	0.0051 (5)	0.0054 (5)

Geometric parameters (Å, °)

O1—C9	1.3687 (14)	C5—C6	1.3921 (19)
O1—C8	1.3774 (14)	C5—H5	0.9500
O2—C9	1.2158 (15)	C6—C7	1.3854 (18)
O3—C10	1.2245 (15)	C6—H6	0.9500
N1—C10	1.3528 (15)	C7—C8	1.3874 (18)
N1—N2	1.4064 (14)	C7—H7	0.9500
N1—H1N	0.896 (18)	C11—C12	1.3899 (19)
N2—C11	1.4184 (16)	C11—C16	1.3939 (17)
N2—H2N	0.911 (17)	C12—C13	1.388 (2)
C1—C2	1.3573 (16)	C12—H12	0.9500
C1—C9	1.4581 (17)	C13—C14	1.378 (2)
C1—C10	1.5027 (16)	C13—H13	0.9500
C2—C3	1.4315 (17)	C14—C15	1.384 (2)
C2—H2	0.9500	C14—H14	0.9500
C3—C8	1.3915 (18)	C15—C16	1.382 (2)
C3—C4	1.4064 (17)	C15—H15	0.9500
C4—C5	1.3809 (18)	C16—H16	0.9500
C4—H4	0.9500		
C9—O1—C8	122.88 (10)	O1—C8—C7	116.96 (11)
C10—N1—N2	120.49 (10)	O1—C8—C3	120.67 (11)
C10—N1—H1N	123.5 (11)	C7—C8—C3	122.37 (11)
N2—N1—H1N	116.0 (11)	O2—C9—O1	115.97 (10)
N1—N2—C11	115.52 (10)	O2—C9—C1	126.82 (11)
N1—N2—H2N	109.7 (10)	O1—C9—C1	117.20 (10)
C11—N2—H2N	112.6 (10)	O3—C10—N1	122.64 (11)
C2—C1—C9	119.98 (11)	O3—C10—C1	120.63 (11)
C2—C1—C10	117.83 (11)	N1—C10—C1	116.64 (10)
C9—C1—C10	122.18 (10)	C12—C11—C16	119.18 (12)
C1—C2—C3	121.33 (11)	C12—C11—N2	121.77 (11)
C1—C2—H2	119.3	C16—C11—N2	118.90 (12)
C3—C2—H2	119.3	C13—C12—C11	119.95 (13)
C8—C3—C4	118.47 (11)	C13—C12—H12	120.0
C8—C3—C2	117.82 (11)	C11—C12—H12	120.0
C4—C3—C2	123.71 (11)	C14—C13—C12	120.85 (14)
C5—C4—C3	119.62 (12)	C14—C13—H13	119.6
C5—C4—H4	120.2	C12—C13—H13	119.6
C3—C4—H4	120.2	C13—C14—C15	119.14 (13)
C4—C5—C6	120.62 (12)	C13—C14—H14	120.4
C4—C5—H5	119.7	C15—C14—H14	120.4
C6—C5—H5	119.7	C16—C15—C14	120.81 (13)
C7—C6—C5	120.82 (12)	C16—C15—H15	119.6
C7—C6—H6	119.6	C14—C15—H15	119.6
C5—C6—H6	119.6	C15—C16—C11	120.04 (13)
C6—C7—C8	118.10 (12)	C15—C16—H16	120.0
C6—C7—H7	121.0	C11—C16—H16	120.0

C8—C7—H7	121.0		
C10—N1—N2—C11	74.99 (14)	C2—C1—C9—O2	-175.93 (12)
C9—C1—C2—C3	-2.32 (18)	C10—C1—C9—O2	3.5 (2)
C10—C1—C2—C3	178.22 (10)	C2—C1—C9—O1	3.79 (17)
C1—C2—C3—C8	0.51 (18)	C10—C1—C9—O1	-176.78 (10)
C1—C2—C3—C4	-178.72 (11)	N2—N1—C10—O3	-0.14 (19)
C8—C3—C4—C5	0.41 (19)	N2—N1—C10—C1	176.63 (10)
C2—C3—C4—C5	179.64 (12)	C2—C1—C10—O3	11.13 (18)
C3—C4—C5—C6	0.6 (2)	C9—C1—C10—O3	-168.32 (11)
C4—C5—C6—C7	-1.1 (2)	C2—C1—C10—N1	-165.71 (11)
C5—C6—C7—C8	0.6 (2)	C9—C1—C10—N1	14.85 (17)
C9—O1—C8—C7	-178.13 (11)	N1—N2—C11—C12	28.34 (15)
C9—O1—C8—C3	1.88 (18)	N1—N2—C11—C16	-156.03 (11)
C6—C7—C8—O1	-179.58 (11)	C16—C11—C12—C13	1.27 (19)
C6—C7—C8—C3	0.4 (2)	N2—C11—C12—C13	176.89 (11)
C4—C3—C8—O1	179.08 (11)	C11—C12—C13—C14	-0.3 (2)
C2—C3—C8—O1	-0.19 (18)	C12—C13—C14—C15	-0.5 (2)
C4—C3—C8—C7	-0.91 (19)	C13—C14—C15—C16	0.3 (2)
C2—C3—C8—C7	179.82 (11)	C14—C15—C16—C11	0.7 (2)
C8—O1—C9—O2	176.16 (10)	C12—C11—C16—C15	-1.48 (18)
C8—O1—C9—C1	-3.59 (17)	N2—C11—C16—C15	-177.22 (11)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...N2 ⁱ	0.896 (18)	2.327 (18)	3.0498 (14)	137.7 (15)
N1—H1N...O2	0.896 (18)	2.112 (18)	2.7544 (13)	127.8 (15)
N2—H2N...O2 ⁱⁱ	0.911 (17)	2.243 (17)	3.1358 (14)	166.3 (14)

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