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2-Amino-4-(4-chlorophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

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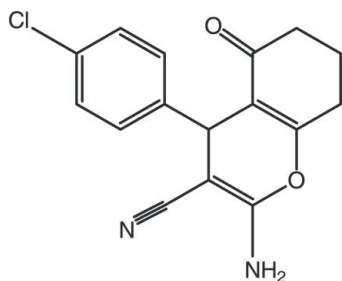
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.049; wR factor = 0.122; data-to-parameter ratio = 14.5.

In the title molecule, $\text{C}_{16}\text{H}_{13}\text{ClN}_2\text{O}_2$, the cyclohexene ring is in a sofa conformation. The pyran ring is essentially planar [maximum deviation = 0.038 (2) Å] and forms a dihedral angle of 89.68 (10)° with the benzene ring. In the crystal, molecules are linked by pairs of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, forming inversion dimers with $R_2^2(12)$ ring motifs. These dimers are further linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into chains along [110]. Weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds are also present.

Related literature

For pharmaceutical background to 2-amino-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile derivatives, see: Gao *et al.* (2001); Xu *et al.* (2011); Luan *et al.* (2011); Wang & Zhu, (2007); O'Callaghan *et al.* (1995). For similar structures, see: Tu *et al.* (2001); Qiao *et al.* (2011); Kong *et al.* (2011); Hu *et al.* (2012). For standard bond lengths, see: Allen *et al.* (1987). For geometric analysis, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{13}\text{ClN}_2\text{O}_2$
 $M_r = 300.73$
 Monoclinic, $C2/c$
 $a = 13.753$ (4) Å
 $b = 11.077$ (3) Å
 $c = 19.370$ (6) Å
 $\beta = 107.856$ (5)°
 $V = 2808.7$ (14) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 150$ K
 $0.43 \times 0.27 \times 0.07$ mm

Data collection

Bruker APEX 2000 CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.914$, $T_{\max} = 0.981$
 10643 measured reflections
 2755 independent reflections
 2124 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.122$
 $S = 1.02$
 2755 reflections
 190 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{N2}^{\text{i}}$	0.88	2.25	3.132 (3)	177
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{ii}}$	0.88	2.15	2.955 (2)	151
$\text{C3}-\text{H3}\cdots\text{N2}^{\text{iii}}$	0.95	2.48	3.226 (3)	135

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (iii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; 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, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5452).

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supplementary materials

Acta Cryst. (2012). E68, o1414–o1415 [doi:10.1107/S1600536812015838]

2-Amino-4-(4-chlorophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

Shaaban K. Mohamed, Mehmet Akkurt, Antar A. Abdelhamid, Kuldip Singh and M. A. Allahverdiyev

Comment

The existence of an amino group and cyano group in tetrahydrochromenone compounds make them great substrates for building up multi organic transformations (Luan *et al.*, 2011), preparing poly-functionalized substituted pyran derivatives (Wang & Zhu, 2007) and designing of poly-heterocyclic compounds (O'Callaghan *et al.*, 1995). Moreover, such derivatives of tetrahydrochromenones have attracted strong interests of pharmacists and biologists because of their potential application in the treatment of psoriatic arthritis and rheumatoid arthritis (Xu *et al.*, 2011). They also have wide biological applications such as anti-anaphylaxis, anti-achondroplasty and anti-cancer activity (Gao *et al.*, 2001). To continue to our interest in the synthesis of biologically active compounds we report herein the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The (C8–C13) cyclohexene ring is in a sofa conformation with puckering parameters (Cremer & Pople, 1975) of $Q_T = 0.466$ (3) Å, $\theta = 58.3$ (2) ° and $\varphi = 173.5$ (3) °. The pyran ring (O2/C7/C8/C13–C15) is essentially planar with a maximum deviation of 0.038 (2) Å for C7, and forms a dihedral angle of 89.68 (10)° with the benzene ring (C1–C6). The bond lengths (Allen *et al.*, 1987) and angles are similar to those for reported structures (Tu *et al.*, 2001; Qiao *et al.*, 2011; Kong *et al.*, 2011; Hu *et al.*, 2012).

In the crystal, molecules are linked by pairs of intermolecular N—H···N hydrogen bonds, forming inversion dimers with $R_2^2(12)$ ring motifs (Bernstein *et al.*, 1995; Etter *et al.*, 1990), and these dimers are connected by weak C—H···N and N—H···O hydrogen bonds, generating one-dimensional chains along [110] (Table 1, Fig. 2).

Experimental

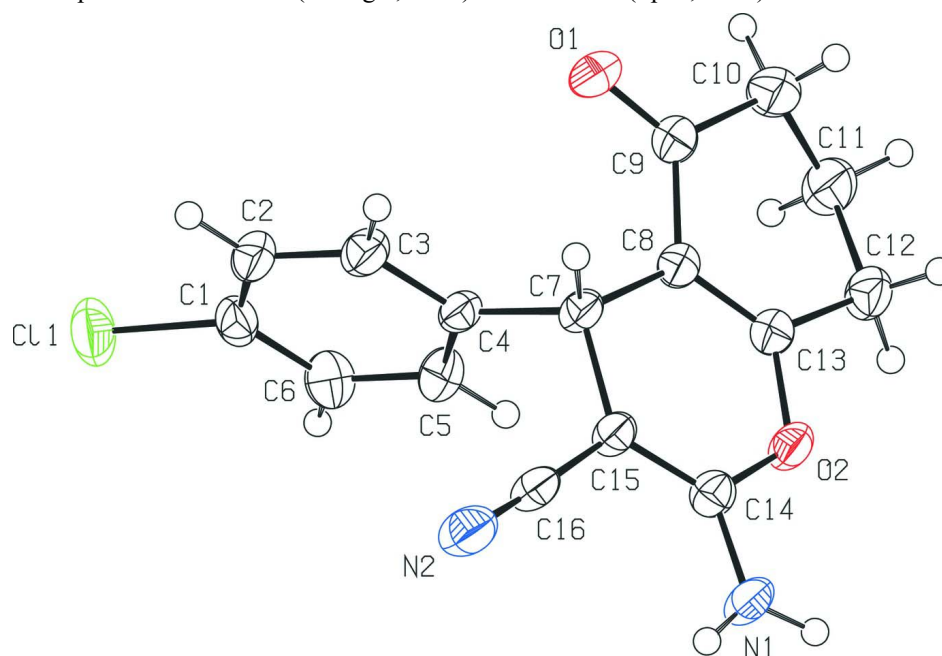
The title compound (I) was formed during a three component reaction of an equimolar ratios of (4-chlorobenzylidene)propanedinitrile (1 mmol), (4-aminophenyl)methanol (1 mmol) and cyclohexane-1,3-dione (1 mmol). The reaction mixture was heated in ethanol at 351 K. The reaction was monitored with TLC until completed after 5 h, then left in fume cupboard at room temperature until solvent evaporated. The resulting solid mass was recrystallized from ethanol to afford good quality crystals suitable for X-ray diffraction. [Yield: 83%, m.p.: 513 K].

Refinement

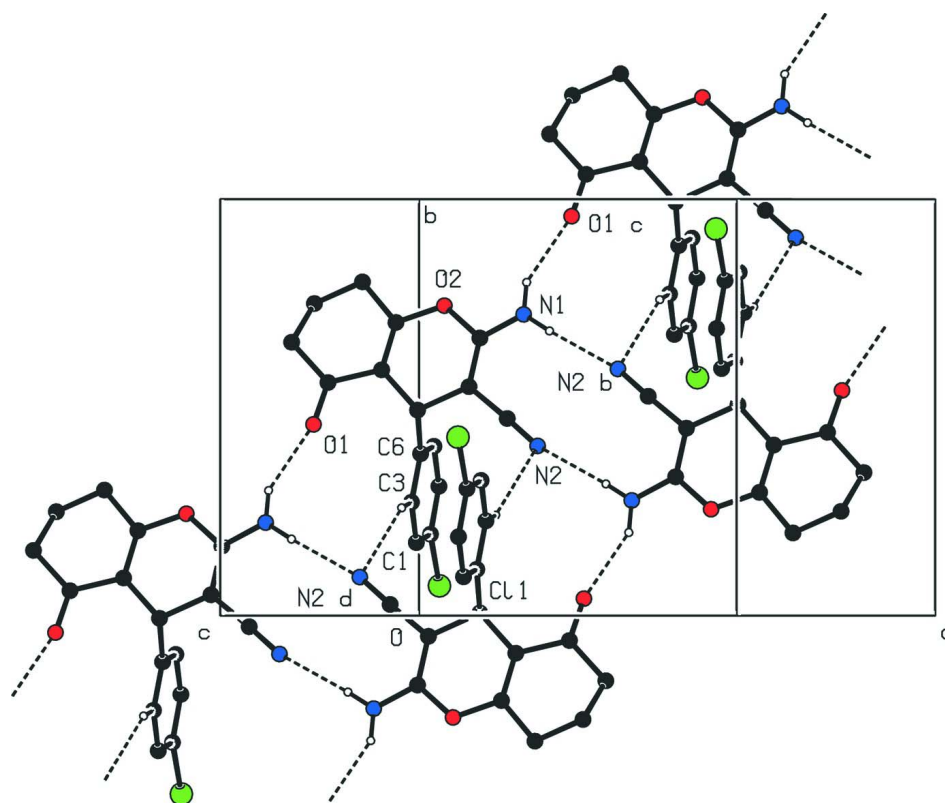
All H atoms were positioned geometrically and refined using as riding model with N—H = 0.88 Å for NH₂, C—H = 0.95 Å for aromatic, C—H = 0.99 Å for methylene and C—H = 1.00 Å for methine, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE* (Bruker, 2005); 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, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

**Figure 1**

A view of the molecule of (I), with displacement ellipsoids for non-H atoms drawn at the 50% probability level.


Figure 2

Part of the crystal structure of (I) with hydrogen bonds shown as dashed lines. H atoms not involved in H-bonding are omitted for clarity. Symmetry codes: (b) $-x+1, -y+1, -z+1$; (c) $x+1/2, y+1/2, z$; (d) $-x+1/2, -y+1/2, -z+1$.

2-Amino-4-(4-chlorophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3- carbonitrile

Crystal data

$C_{16}H_{13}ClN_2O_2$

$M_r = 300.73$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 13.753 (4) \text{ \AA}$

$b = 11.077 (3) \text{ \AA}$

$c = 19.370 (6) \text{ \AA}$

$\beta = 107.856 (5)^\circ$

$V = 2808.7 (14) \text{ \AA}^3$

$Z = 8$

$F(000) = 1248$

$D_x = 1.422 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 833 reflections

$\theta = 2.2\text{--}28.2^\circ$

$\mu = 0.28 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Plate, colourless

$0.43 \times 0.27 \times 0.07 \text{ mm}$

Data collection

Bruker APEX 2000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.914, T_{\max} = 0.981$

10643 measured reflections

2755 independent reflections

2124 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

$\theta_{\text{max}} = 26.0^\circ, \theta_{\text{min}} = 2.2^\circ$

$h = -16 \rightarrow 16$

$k = -13 \rightarrow 13$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H-atom parameters constrained
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2755 reflections	$(\Delta/\sigma)_{\max} < 0.001$
190 parameters	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.12556 (5)	0.07141 (5)	0.22343 (4)	0.0510 (2)
O1	-0.04515 (11)	0.45923 (13)	0.41373 (8)	0.0395 (5)
O2	0.20240 (10)	0.74455 (12)	0.39806 (8)	0.0320 (4)
N1	0.37218 (13)	0.72339 (15)	0.44001 (9)	0.0347 (6)
N2	0.41870 (14)	0.40760 (17)	0.49094 (11)	0.0440 (7)
C1	0.13012 (15)	0.19541 (19)	0.28039 (12)	0.0337 (7)
C2	0.12955 (16)	0.17534 (19)	0.35014 (13)	0.0374 (7)
C3	0.13788 (15)	0.27229 (18)	0.39614 (12)	0.0332 (7)
C4	0.14684 (14)	0.38895 (17)	0.37333 (11)	0.0274 (6)
C5	0.14494 (16)	0.40667 (18)	0.30168 (11)	0.0348 (7)
C6	0.13673 (16)	0.3107 (2)	0.25501 (12)	0.0378 (7)
C7	0.16120 (14)	0.49363 (17)	0.42613 (11)	0.0270 (6)
C8	0.08175 (14)	0.59034 (17)	0.40049 (10)	0.0274 (6)
C9	-0.02395 (15)	0.55980 (18)	0.39594 (11)	0.0311 (7)
C10	-0.10332 (17)	0.6565 (2)	0.37309 (14)	0.0470 (8)
C11	-0.07795 (17)	0.7496 (2)	0.32396 (14)	0.0465 (8)
C12	0.02871 (15)	0.80147 (18)	0.35920 (12)	0.0360 (7)
C13	0.10411 (15)	0.70407 (18)	0.38660 (11)	0.0286 (6)
C14	0.28249 (15)	0.66677 (18)	0.42763 (10)	0.0285 (6)
C15	0.26669 (14)	0.54985 (17)	0.44117 (11)	0.0271 (6)
C16	0.35154 (15)	0.47326 (19)	0.46903 (11)	0.0304 (7)
H1A	0.42960	0.68370	0.45910	0.0420*
H1B	0.37370	0.80030	0.42910	0.0420*
H2	0.12350	0.09570	0.36650	0.0450*
H3	0.13750	0.25880	0.44450	0.0400*
H5	0.14940	0.48630	0.28470	0.0420*

H6	0.13560	0.32360	0.20630	0.0450*
H7	0.15570	0.46120	0.47300	0.0320*
H10A	-0.11020	0.69730	0.41680	0.0560*
H10B	-0.16990	0.61890	0.34740	0.0560*
H11A	-0.12890	0.81560	0.31430	0.0560*
H11B	-0.08110	0.71180	0.27700	0.0560*
H12A	0.04920	0.85040	0.32320	0.0430*
H12B	0.02750	0.85500	0.39980	0.0430*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0542 (4)	0.0358 (4)	0.0625 (4)	0.0042 (3)	0.0171 (3)	-0.0181 (3)
O1	0.0357 (8)	0.0312 (9)	0.0545 (10)	-0.0086 (7)	0.0182 (7)	0.0011 (7)
O2	0.0301 (8)	0.0212 (7)	0.0432 (8)	-0.0032 (6)	0.0090 (6)	0.0040 (6)
N1	0.0288 (9)	0.0249 (9)	0.0471 (11)	-0.0069 (7)	0.0070 (8)	0.0050 (8)
N2	0.0314 (10)	0.0353 (11)	0.0633 (13)	-0.0011 (8)	0.0116 (9)	0.0139 (10)
C1	0.0238 (10)	0.0288 (12)	0.0460 (13)	0.0027 (9)	0.0071 (9)	-0.0091 (10)
C2	0.0363 (12)	0.0224 (11)	0.0545 (14)	-0.0011 (9)	0.0155 (10)	0.0015 (10)
C3	0.0345 (12)	0.0275 (11)	0.0383 (12)	-0.0036 (9)	0.0120 (9)	0.0030 (9)
C4	0.0224 (10)	0.0224 (10)	0.0366 (11)	-0.0036 (8)	0.0078 (8)	0.0000 (8)
C5	0.0405 (12)	0.0240 (11)	0.0390 (12)	-0.0003 (9)	0.0111 (10)	0.0028 (9)
C6	0.0410 (12)	0.0350 (13)	0.0359 (12)	0.0044 (10)	0.0095 (10)	-0.0001 (10)
C7	0.0279 (10)	0.0222 (10)	0.0309 (11)	-0.0038 (8)	0.0091 (8)	0.0027 (8)
C8	0.0268 (10)	0.0234 (11)	0.0319 (11)	-0.0009 (8)	0.0089 (8)	-0.0005 (8)
C9	0.0303 (11)	0.0285 (12)	0.0344 (11)	-0.0032 (9)	0.0099 (9)	-0.0045 (9)
C10	0.0290 (12)	0.0374 (13)	0.0759 (18)	0.0014 (10)	0.0182 (11)	0.0005 (12)
C11	0.0323 (13)	0.0334 (13)	0.0691 (17)	0.0046 (10)	0.0085 (12)	0.0082 (12)
C12	0.0365 (12)	0.0261 (11)	0.0441 (13)	0.0016 (9)	0.0104 (10)	0.0042 (10)
C13	0.0282 (11)	0.0261 (11)	0.0316 (11)	-0.0014 (9)	0.0095 (8)	-0.0025 (9)
C14	0.0278 (11)	0.0277 (11)	0.0287 (11)	-0.0018 (8)	0.0066 (8)	-0.0004 (8)
C15	0.0256 (10)	0.0230 (10)	0.0311 (11)	-0.0035 (8)	0.0064 (8)	0.0008 (8)
C16	0.0273 (11)	0.0262 (11)	0.0356 (12)	-0.0077 (9)	0.0067 (9)	0.0032 (9)

Geometric parameters (\AA , $^\circ$)

C11—C1	1.751 (2)	C8—C13	1.343 (3)
O1—C9	1.227 (3)	C9—C10	1.496 (3)
O2—C13	1.376 (3)	C10—C11	1.515 (3)
O2—C14	1.377 (3)	C11—C12	1.528 (3)
N1—C14	1.338 (3)	C12—C13	1.478 (3)
N2—C16	1.150 (3)	C14—C15	1.352 (3)
N1—H1B	0.8800	C15—C16	1.410 (3)
N1—H1A	0.8800	C2—H2	0.9500
C1—C6	1.381 (3)	C3—H3	0.9500
C1—C2	1.372 (3)	C5—H5	0.9500
C2—C3	1.378 (3)	C6—H6	0.9500
C3—C4	1.383 (3)	C7—H7	1.0000
C4—C7	1.518 (3)	C10—H10A	0.9900
C4—C5	1.394 (3)	C10—H10B	0.9900

C5—C6	1.378 (3)	C11—H11A	0.9900
C7—C8	1.502 (3)	C11—H11B	0.9900
C7—C15	1.523 (3)	C12—H12A	0.9900
C8—C9	1.469 (3)	C12—H12B	0.9900
C13—O2—C14	118.95 (15)	O2—C14—C15	121.62 (19)
H1A—N1—H1B	120.00	C7—C15—C14	123.71 (18)
C14—N1—H1A	120.00	C7—C15—C16	117.10 (17)
C14—N1—H1B	120.00	C14—C15—C16	119.17 (19)
C11—C1—C2	118.92 (16)	N2—C16—C15	177.8 (2)
C2—C1—C6	121.4 (2)	C1—C2—H2	120.00
C11—C1—C6	119.64 (17)	C3—C2—H2	120.00
C1—C2—C3	119.1 (2)	C2—C3—H3	119.00
C2—C3—C4	121.3 (2)	C4—C3—H3	119.00
C3—C4—C7	120.28 (18)	C4—C5—H5	119.00
C3—C4—C5	118.24 (18)	C6—C5—H5	119.00
C5—C4—C7	121.46 (17)	C1—C6—H6	121.00
C4—C5—C6	121.16 (19)	C5—C6—H6	121.00
C1—C6—C5	118.8 (2)	C4—C7—H7	108.00
C8—C7—C15	109.00 (16)	C8—C7—H7	108.00
C4—C7—C8	113.00 (17)	C15—C7—H7	108.00
C4—C7—C15	111.14 (16)	C9—C10—H10A	109.00
C7—C8—C9	117.51 (17)	C9—C10—H10B	109.00
C7—C8—C13	123.17 (19)	C11—C10—H10A	109.00
C9—C8—C13	119.15 (18)	C11—C10—H10B	109.00
O1—C9—C8	120.54 (19)	H10A—C10—H10B	108.00
C8—C9—C10	118.15 (18)	C10—C11—H11A	110.00
O1—C9—C10	121.2 (2)	C10—C11—H11B	110.00
C9—C10—C11	112.7 (2)	C12—C11—H11A	109.00
C10—C11—C12	110.5 (2)	C12—C11—H11B	110.00
C11—C12—C13	110.97 (17)	H11A—C11—H11B	108.00
O2—C13—C8	123.17 (18)	C11—C12—H12A	109.00
O2—C13—C12	111.50 (17)	C11—C12—H12B	109.00
C8—C13—C12	125.3 (2)	C13—C12—H12A	109.00
N1—C14—C15	127.46 (19)	C13—C12—H12B	109.00
O2—C14—N1	110.92 (17)	H12A—C12—H12B	108.00
C13—O2—C14—N1	-175.33 (16)	C4—C7—C8—C13	-117.7 (2)
C13—O2—C14—C15	4.4 (3)	C15—C7—C8—C9	-168.89 (17)
C14—O2—C13—C8	-2.4 (3)	C15—C7—C8—C13	6.4 (3)
C14—O2—C13—C12	176.27 (17)	C8—C7—C15—C16	177.19 (17)
C11—C1—C2—C3	177.03 (18)	C13—C8—C9—C10	2.3 (3)
C11—C1—C6—C5	-177.07 (18)	C7—C8—C13—C12	178.03 (19)
C2—C1—C6—C5	1.3 (3)	C9—C8—C13—O2	171.79 (18)
C6—C1—C2—C3	-1.4 (3)	C7—C8—C13—O2	-3.5 (3)
C1—C2—C3—C4	-0.1 (3)	C7—C8—C9—O1	1.2 (3)
C2—C3—C4—C7	-177.0 (2)	C7—C8—C9—C10	177.81 (18)
C2—C3—C4—C5	1.4 (3)	C13—C8—C9—O1	-174.36 (19)
C3—C4—C7—C15	112.8 (2)	C9—C8—C13—C12	-6.7 (3)

C3—C4—C7—C8	-124.3 (2)	O1—C9—C10—C11	-154.9 (2)
C3—C4—C5—C6	-1.5 (3)	C8—C9—C10—C11	28.4 (3)
C5—C4—C7—C8	57.4 (3)	C9—C10—C11—C12	-53.7 (3)
C5—C4—C7—C15	-65.5 (2)	C10—C11—C12—C13	48.8 (2)
C7—C4—C5—C6	176.9 (2)	C11—C12—C13—O2	161.65 (18)
C4—C5—C6—C1	0.1 (3)	C11—C12—C13—C8	-19.7 (3)
C4—C7—C15—C14	120.7 (2)	O2—C14—C15—C7	-0.6 (3)
C4—C7—C15—C16	-57.6 (2)	O2—C14—C15—C16	177.71 (18)
C8—C7—C15—C14	-4.5 (3)	N1—C14—C15—C7	179.11 (19)
C4—C7—C8—C9	67.0 (2)	N1—C14—C15—C16	-2.6 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots N2 ⁱ	0.88	2.25	3.132 (3)	177
N1—H1B \cdots O1 ⁱⁱ	0.88	2.15	2.955 (2)	151
C3—H3 \cdots N2 ⁱⁱⁱ	0.95	2.48	3.226 (3)	135

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1/2, y+1/2, z$; (iii) $-x+1/2, -y+1/2, -z+1$.