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N-(2-Chloro-5-methylphenyl)succinamic acid

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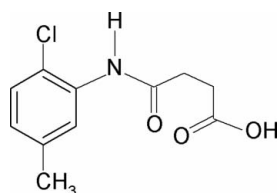
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.062; wR factor = 0.128; data-to-parameter ratio = 15.3.

In the title compound, $\text{C}_{11}\text{H}_{12}\text{ClNO}_3$, the conformation of the N—H bond in the amide segment is *syn* with respect to the *ortho*-Cl atom. The amide and carboxyl C=O groups are *syn* to each other. Furthermore, the C=O and O—H bonds of the carboxyl group are in *syn* positions with respect to each other. The dihedral angle between the benzene ring and the amide group is $47.8(2)^\circ$. In the crystal, molecules are connected by pairs of O—H \cdots O hydrogen bonds, forming inversion dimers. The dimers are further linked by N—H \cdots O hydrogen bonds into double chains along the *b*-axis direction.

Related literature

For our previous studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2001); Saraswathi *et al.* (2011), on *N*-(aryl)-methanesulfonamides, see: Jayalakshmi & Gowda (2004), on *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2005) and on *N*-chloroarylamides, see: Gowda *et al.* (1996). For modes of hydrogen bonding in the structures of carboxylic acids, see: Leiserowitz (1976). For the centrosymmetrical dimeric hydrogen-bonding association of carboxylic groups, see: Jagannathan *et al.* (1994).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{12}\text{ClNO}_3$
 $M_r = 241.67$

 Monoclinic, $C2/c$
 $a = 23.780(5)$ Å
 $b = 4.7784(7)$ Å
 $c = 23.892(5)$ Å
 $\beta = 121.20(1)^\circ$
 $V = 2322.2(8)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.32$ mm⁻¹
 $T = 293$ K
 $0.42 \times 0.10 \times 0.08$ mm

Data collection

 Oxford Diffraction Xcalibur with Sapphire CCD detector diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

 Diffraction, 2009)
 $T_{\min} = 0.877$, $T_{\max} = 0.975$
 4399 measured reflections
 2322 independent reflections
 1680 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.128$
 $S = 1.19$
 2322 reflections
 152 parameters
 2 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3O \cdots O2 ⁱ	0.83 (2)	1.83 (2)	2.652 (4)	172 (5)
N1—H1N \cdots O1 ⁱⁱ	0.86 (2)	2.08 (2)	2.910 (4)	163 (3)

 Symmetry codes: (i) $-x - \frac{1}{2}, -y + \frac{3}{2}, -z$; (ii) $x, y + 1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2035).

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Acta Cryst. (2012). E68, o221 [doi:10.1107/S1600536811054638]

N*-(2-Chloro-5-methylphenyl)succinamic acid*B. Thimme Gowda, Sabine Foro and U. Chaithanya****S1. Comment**

The amide and sulfonamide moieties are important constituents of many biologically important compounds. As a part of our studies of the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2001; Saraswathi *et al.*, 2011), *N*-(aryl)-methanesulfonamides (Jayalakshmi & Gowda, 2004), *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2005) and *N*-chloro-arylsulfonamides (Gowda *et al.*, 1996), in the present work, the crystal structure of *N*-(2-chloro-5-methylphenyl)succinamic acid (I) has been determined (Fig. 1). The conformation of the N—H bond in the amide segment is *syn* to the *ortho*-chloro group and *anti* to the *meta*-methyl group in the benzene ring, similar to the *syn* conformation observed between the amide hydrogen and the *ortho*-Cl and *anti* conformation between the amide hydrogen and the *meta*-Cl in the benzene ring of *N*-(2,5-dichlorophenyl)-succinamic acid (II) (Saraswathi *et al.*, 2011).

Further, the conformations of the amide oxygen and the carboxyl oxygen of the acid segment are *syn* to each other.

The C=O and O—H bonds of the acid group are in *syn* position to each other, similar to that observed in (II).

The dihedral angle between the phenyl ring and the amide group is 47.8 (2)°.

The intermolecular O—H⋯O and N—H⋯O hydrogen bonds pack the molecules into infinite chains along the *b*-axis direction (Table 1, Fig.2).

The modes of interlinking carboxylic acids by hydrogen bonds is described elsewhere (Leiserowitz, 1976). The packing of molecules involving dimeric hydrogen-bonded association of each carboxyl group with a centrosymmetrically related neighbor has also been observed (Jagannathan *et al.*, 1994).

S2. Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated with the solution of 2-chloro,5-methyl-aniline (0.01 mole) also in toluene (20 ml), added dropwise with permanent stirring. The resulting mixture was stirred for about one hour and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with diluted hydrochloric acid to remove the unreacted 2-chloro-5-methyl-aniline. The resultant title compound was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. The product was recrystallized to constant melting point from ethanol. The purity of the compound was checked and characterized by its infrared and NMR spectra.

Rod like colorless single crystals used in X-ray diffraction studies were grown from ethanolic solution by slow evaporation at room temperature.

S3. Refinement

The H atoms of the NH and OH groups were located in a difference map and later restrained to the distances N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, respectively. The other H atoms were positioned with idealized geometry and refined using a riding model with the aromatic C—H = 0.93 Å and methylene C—H = 0.97 Å. All H atoms were refined with

isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

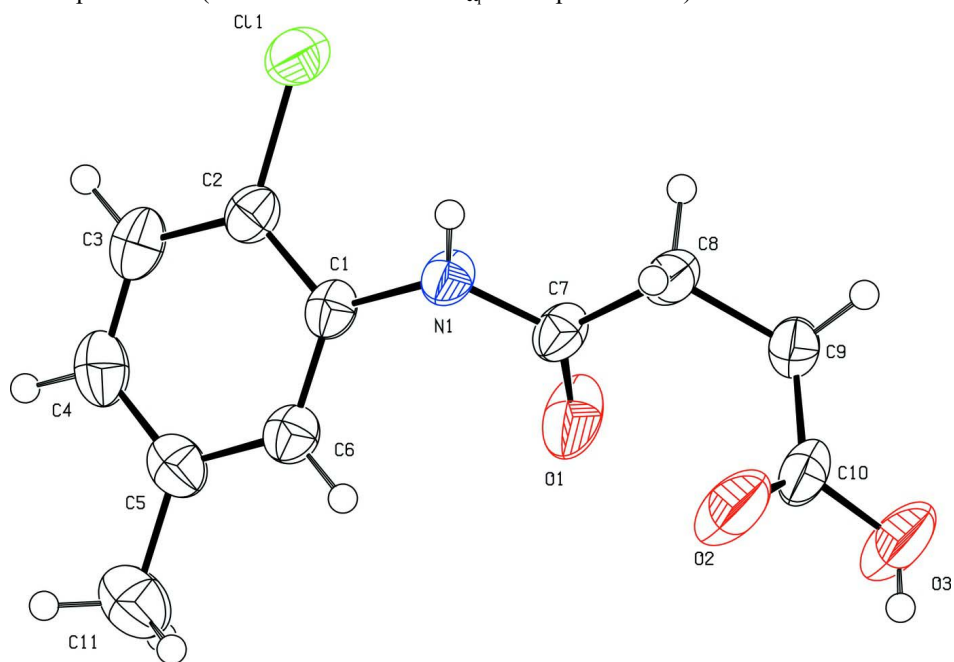


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

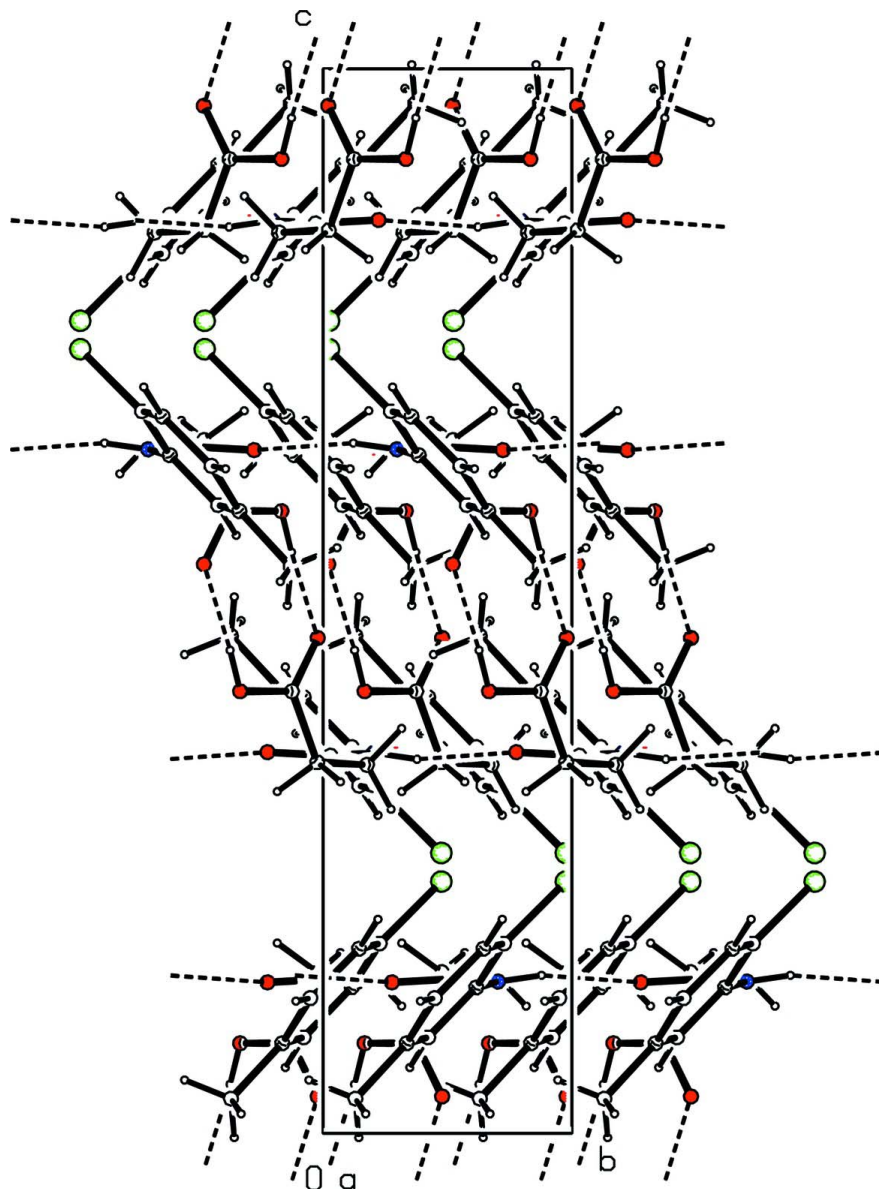


Figure 2

Molecular packing of the title compound with hydrogen bonds shown as dashed lines.

***N*-(2-Chloro-5-methylphenyl)succinamic acid**

Crystal data

$C_{11}H_{12}ClNO_3$

$M_r = 241.67$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 23.780\ (5)\ \text{\AA}$

$b = 4.7784\ (7)\ \text{\AA}$

$c = 23.892\ (5)\ \text{\AA}$

$\beta = 121.20\ (1)^\circ$

$V = 2322.2\ (8)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1008$

$D_x = 1.382\ \text{Mg m}^{-3}$

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

Cell parameters from 1604 reflections

$\theta = 2.6\text{--}27.9^\circ$

$\mu = 0.32\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Rod, colourless

$0.42 \times 0.10 \times 0.08\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur with Sapphire CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 ω and φ scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)
 $T_{\min} = 0.877$, $T_{\max} = 0.975$

4399 measured reflections
2322 independent reflections
1680 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -29 \rightarrow 28$
 $k = -4 \rightarrow 5$
 $l = -20 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.128$
 $S = 1.19$
2322 reflections
152 parameters
2 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
 $w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 6.3165P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.044$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.14042 (4)	1.47549 (19)	0.23661 (4)	0.0481 (3)
O1	-0.02815 (14)	0.7770 (5)	0.14193 (17)	0.0703 (9)
O2	-0.18862 (14)	0.9786 (7)	0.03462 (14)	0.0749 (9)
O3	-0.21661 (15)	0.6674 (7)	0.08465 (14)	0.0783 (10)
H3O	-0.2438 (19)	0.623 (10)	0.0462 (12)	0.094*
N1	0.01311 (13)	1.2030 (5)	0.14261 (14)	0.0363 (6)
H1N	0.0095 (16)	1.378 (4)	0.1481 (16)	0.044*
C1	0.06702 (14)	1.1156 (6)	0.13712 (15)	0.0330 (7)
C2	0.12917 (15)	1.2274 (7)	0.17838 (15)	0.0362 (7)
C3	0.18210 (16)	1.1446 (8)	0.17339 (18)	0.0473 (9)
H3	0.2235	1.2205	0.2011	0.057*
C4	0.17314 (17)	0.9488 (8)	0.12704 (18)	0.0487 (9)
H4	0.2090	0.8916	0.1243	0.058*
C5	0.11180 (17)	0.8360 (7)	0.08458 (17)	0.0423 (8)
C6	0.05926 (15)	0.9222 (7)	0.09027 (16)	0.0377 (8)

H6	0.0177	0.8484	0.0620	0.045*
C7	-0.03027 (15)	1.0299 (7)	0.14512 (16)	0.0377 (7)
C8	-0.08153 (15)	1.1768 (7)	0.15378 (18)	0.0418 (8)
H8A	-0.1036	1.3159	0.1196	0.050*
H8B	-0.0599	1.2737	0.1955	0.050*
C9	-0.13225 (16)	0.9767 (8)	0.15140 (17)	0.0452 (8)
H9A	-0.1096	0.8164	0.1788	0.054*
H9B	-0.1553	1.0697	0.1698	0.054*
C10	-0.18136 (16)	0.8754 (8)	0.08466 (19)	0.0450 (9)
C11	0.1020 (2)	0.6300 (8)	0.03259 (19)	0.0605 (11)
H11A	0.1074	0.4431	0.0494	0.073*
H11B	0.1339	0.6646	0.0201	0.073*
H11C	0.0586	0.6509	-0.0049	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0457 (5)	0.0473 (5)	0.0465 (5)	-0.0107 (4)	0.0206 (4)	-0.0069 (4)
O1	0.0751 (19)	0.0230 (14)	0.147 (3)	-0.0019 (13)	0.081 (2)	-0.0023 (16)
O2	0.0717 (18)	0.081 (2)	0.0607 (17)	-0.0415 (17)	0.0259 (15)	-0.0011 (17)
O3	0.072 (2)	0.084 (2)	0.0667 (19)	-0.0466 (18)	0.0269 (16)	-0.0004 (18)
N1	0.0339 (13)	0.0231 (13)	0.0559 (17)	-0.0001 (12)	0.0261 (13)	-0.0033 (13)
C1	0.0331 (16)	0.0245 (16)	0.0432 (18)	0.0027 (13)	0.0210 (14)	0.0072 (14)
C2	0.0366 (16)	0.0327 (18)	0.0381 (17)	-0.0027 (14)	0.0185 (14)	0.0039 (15)
C3	0.0334 (17)	0.053 (2)	0.055 (2)	-0.0001 (16)	0.0220 (16)	0.0081 (19)
C4	0.0430 (19)	0.052 (2)	0.061 (2)	0.0101 (18)	0.0338 (18)	0.012 (2)
C5	0.056 (2)	0.0349 (19)	0.0450 (19)	0.0061 (16)	0.0326 (17)	0.0065 (16)
C6	0.0382 (17)	0.0322 (18)	0.0430 (18)	0.0004 (14)	0.0212 (14)	0.0030 (15)
C7	0.0319 (16)	0.0283 (18)	0.0519 (19)	-0.0012 (14)	0.0210 (14)	0.0002 (16)
C8	0.0342 (16)	0.0336 (19)	0.059 (2)	-0.0058 (15)	0.0254 (16)	-0.0103 (17)
C9	0.0421 (18)	0.045 (2)	0.060 (2)	-0.0081 (17)	0.0346 (17)	-0.0092 (18)
C10	0.0332 (17)	0.040 (2)	0.065 (2)	-0.0062 (15)	0.0281 (17)	0.0000 (18)
C11	0.080 (3)	0.052 (2)	0.064 (3)	0.011 (2)	0.047 (2)	0.004 (2)

Geometric parameters (Å, °)

C11—C2	1.740 (3)	C4—H4	0.9300
O1—C7	1.214 (4)	C5—C6	1.387 (4)
O2—C10	1.220 (4)	C5—C11	1.506 (5)
O3—C10	1.300 (4)	C6—H6	0.9300
O3—H3O	0.832 (19)	C7—C8	1.511 (4)
N1—C7	1.347 (4)	C8—C9	1.517 (4)
N1—C1	1.418 (4)	C8—H8A	0.9700
N1—H1N	0.858 (18)	C8—H8B	0.9700
C1—C6	1.388 (4)	C9—C10	1.487 (5)
C1—C2	1.390 (4)	C9—H9A	0.9700
C2—C3	1.382 (4)	C9—H9B	0.9700
C3—C4	1.379 (5)	C11—H11A	0.9600

C3—H3	0.9300	C11—H11B	0.9600
C4—C5	1.384 (5)	C11—H11C	0.9600
C10—O3—H3O	109 (4)	O1—C7—C8	122.3 (3)
C7—N1—C1	125.0 (3)	N1—C7—C8	114.3 (3)
C7—N1—H1N	117 (2)	C7—C8—C9	112.6 (3)
C1—N1—H1N	118 (2)	C7—C8—H8A	109.1
C6—C1—C2	118.4 (3)	C9—C8—H8A	109.1
C6—C1—N1	121.5 (3)	C7—C8—H8B	109.1
C2—C1—N1	120.1 (3)	C9—C8—H8B	109.1
C3—C2—C1	120.7 (3)	H8A—C8—H8B	107.8
C3—C2—C11	119.6 (3)	C10—C9—C8	114.4 (3)
C1—C2—C11	119.7 (2)	C10—C9—H9A	108.7
C4—C3—C2	119.6 (3)	C8—C9—H9A	108.7
C4—C3—H3	120.2	C10—C9—H9B	108.7
C2—C3—H3	120.2	C8—C9—H9B	108.7
C3—C4—C5	121.2 (3)	H9A—C9—H9B	107.6
C3—C4—H4	119.4	O2—C10—O3	123.0 (4)
C5—C4—H4	119.4	O2—C10—C9	123.6 (3)
C4—C5—C6	118.3 (3)	O3—C10—C9	113.4 (3)
C4—C5—C11	120.9 (3)	C5—C11—H11A	109.5
C6—C5—C11	120.9 (3)	C5—C11—H11B	109.5
C5—C6—C1	121.8 (3)	H11A—C11—H11B	109.5
C5—C6—H6	119.1	C5—C11—H11C	109.5
C1—C6—H6	119.1	H11A—C11—H11C	109.5
O1—C7—N1	123.4 (3)	H11B—C11—H11C	109.5
C7—N1—C1—C6	49.1 (5)	C4—C5—C6—C1	-0.2 (5)
C7—N1—C1—C2	-132.0 (3)	C11—C5—C6—C1	-178.8 (3)
C6—C1—C2—C3	-0.9 (5)	C2—C1—C6—C5	1.0 (5)
N1—C1—C2—C3	-179.8 (3)	N1—C1—C6—C5	179.9 (3)
C6—C1—C2—C11	178.8 (2)	C1—N1—C7—O1	-1.1 (6)
N1—C1—C2—C11	-0.2 (4)	C1—N1—C7—C8	177.6 (3)
C1—C2—C3—C4	-0.1 (5)	O1—C7—C8—C9	-5.4 (5)
C11—C2—C3—C4	-179.7 (3)	N1—C7—C8—C9	175.8 (3)
C2—C3—C4—C5	1.0 (5)	C7—C8—C9—C10	-75.1 (4)
C3—C4—C5—C6	-0.8 (5)	C8—C9—C10—O2	-12.7 (5)
C3—C4—C5—C11	177.8 (3)	C8—C9—C10—O3	168.1 (3)

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>
O3—H3O...O2 ⁱ	0.83 (2)	1.83 (2)	2.652 (4)	172 (5)
N1—H1N...O1 ⁱⁱ	0.86 (2)	2.08 (2)	2.910 (4)	163 (3)

Symmetry codes: (i) $-x-1/2, -y+3/2, -z$; (ii) $x, y+1, z$.