

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N,N'-Bis(3-methylphenyl)succinamide dihydrate

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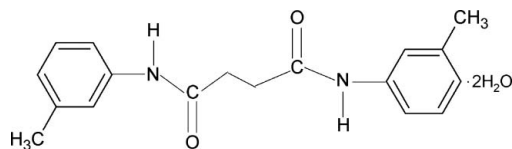
Received 23 May 2011; accepted 31 May 2011

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; disorder in main residue; R factor = 0.126; wR factor = 0.159; data-to-parameter ratio = 13.7.

The asymmetric unit of the title compound, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2 \cdot 2\text{H}_2\text{O}$, contains half a molecule with a center of symmetry at the mid-point of the central C—C bond. The N—H bonds in the amide fragments are *anti* to the *meta*-methyl groups in the adjacent benzene rings. The dihedral angle between the benzene ring and the NH—C(O)—CH₂ segment in the two halves of the molecule is 5.6 (4)°. In the crystal, the packing of molecules through O—H···O and N—H···O hydrogen-bonding interactions leads to the formation of layers parallel to the *bc* plane. The methyl group is disordered with respect to the 3- and 5-positions of the benzene ring, with site-occupation factors of 0.910 (8) and 0.090 (8).

Related literature

For the study of the effect of substituents on the structures of *N*-(aryl)-amides, see: Gowda *et al.* (2000); Saraswathi *et al.* (2011*a,b*). For the effect of substituents on the structures of *N*-(aryl)methanesulfonamides, see: Gowda *et al.* (2007). For similar structures, see: Pierrot *et al.* (1984).



Experimental

Crystal data

 $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2 \cdot 2\text{H}_2\text{O}$
 $M_r = 332.39$
 Monoclinic, $P2_1/c$
 $a = 13.401$ (4) Å
 $b = 4.937$ (2) Å
 $c = 14.446$ (4) Å
 $\beta = 108.67$ (3)°

 $V = 905.5$ (5) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.48 \times 0.12 \times 0.04$ mm

Data collection

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

 Diffraction, 2009
 $T_{\min} = 0.960$, $T_{\max} = 0.997$
 2857 measured reflections
 1679 independent reflections
 797 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.126$
 $wR(F^2) = 0.159$
 $S = 1.23$
 1679 reflections
 123 parameters

 10 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

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···O2 ⁱ	0.86	2.10	2.946 (6)	169
O2—H21···O2 ⁱⁱ	0.82	2.08	2.836 (4)	153
O2—H22···O1	0.84	1.87	2.713 (5)	178

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; 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*.

BSS thanks the University Grants Commission, Government of India, New Delhi, for the award of a research fellowship under its faculty improvement program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2494).

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supporting information

Acta Cryst. (2011). E67, o1591 [doi:10.1107/S1600536811020940]

N,N'-Bis(3-methylphenyl)succinamide dihydrate

B. S. Saraswathi, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are important constituents of many biologically significant compounds. As a part of studying the substituent effects on the structures of this class of compounds (Gowda *et al.*, 2000, 2007; Saraswathi *et al.*, 2011*a, b*), in the present work, the structure of *N,N*-bis(3-methylphenyl)-succinamide dihydrate, (I), has been determined (Fig.1). The asymmetric unit of (I) contains half a molecule with a center of symmetry at the mid-point of the central C—C bond, similar to that observed in bis(2-chlorophenylaminocarbonylmethyl)disulfide, (II), (Pierrot *et al.*, 1984), *N,N*-bis(2-methylphenyl)-succinamide, (III), (Saraswathi *et al.*, 2011*a*) *N,N*-bis(3-chlorophenyl)-succinamide (IV) (Saraswathi *et al.*, 2011*b*).

In the C—NH—C(O)—C segment, the amide O atom is *anti* to the H atoms attached to the adjacent C atom. The N—H bonds in the amide fragments are also *anti* to the *meta*-methyl groups in the adjacent benzene rings, similar to that observed with respect to the *ortho*-methyl groups in (III) and the *meta*-chloro groups in (IV).

The dihedral angle between the benzene ring and the NH—C(O)—CH₂ segment in the two halves of the molecule is 5.6 (4)°, compared to the values of 62.1 (2)° in (III) and 32.8 (1)° in (IV). The striking difference may be due to the fact that the title compound is the dihydrate, *i.e.* is composed of the amide and lattice water molecules, which, unlike in other compounds, influence the molecular conformation through hydrogen bonding interactions.

The torsion angles of N1—C7—C8—C8a and O1—C7—C8—C8a in (I) are 175.9 (6)° and -5.3 (9)°, compared to the values of 150.9 (3)° and -30.5 (4)° in (III) and -175.4 (2)° and 5.9 (4)° in (IV). The differences in the torsion angles may be due to the steric hindrances caused by the different substituents.

Similarly, the torsion angles of C2—C1—N1—C7 and C6—C1—N1—C7 are 5.4 (9)° and -173.6 (6)°, compared to the values of -64.0 (4)° and 117.6 (3)° in (III) and -35.0 (3)° and 147.5 (2)° in (IV).

The crystal packing of (I), through N1—H1N···O2, O2—H21···O2 and O2—H22···O1 hydrogen bonding (Table 1), leads to the formation of layers parallel to the *bc* plane and is shown in Fig. 2.

S2. Experimental

Succinic anhydride (0.01 mol) in toluene (25 ml) was treated dropwise with *m*-toluidine (0.01 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for one hour and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove unreacted *m*-toluidine. The resultant *N*-(3-methylphenyl)succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. The compound was recrystallized to a constant melting point from ethanol. The purity of the compound was checked by elemental analysis and characterized by its infrared and NMR spectra.

The *N*-(3-methylphenyl)succinamic acid obtained was then treated with phosphorous oxychloride and excess of *m*-toluidine at room temperature with constant stirring. The resultant mixture was stirred for 4 h, kept aside for additional 6

h for completion of the reaction and poured slowly into crushed ice with constant stirring. It was kept aside for a day. The resultant solid, *N,N*-bis(3-methylphenyl)-succinamide dihydrate, was filtered under suction, washed thoroughly with water, dilute sodium hydroxide solution and finally with water. It was recrystallized to a constant melting point from a mixture of acetone and chloroform. The purity of the compound was checked by elemental analysis, and characterized by its infrared and NMR spectra.

Needle-like colorless single crystals used in the X-ray diffraction studies were grown in a mixture of acetone and chloroform at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (2) Å. The structure was modelled with stoichiometric chemical composition applying an approximation of full occupancy of H5 and no corresponding partly occupied hydrogen atom at C3. The C9/H3 group in an alternative orientation was idealized and refined using a AFIX 3 (positional optimization of the entire group only by translation, no rotations) in SHELXL. The U^{ij} components of C9' were assumed to be identical with that of C5 (EADP C5 C9') and were restrained to approximate isotropic behaviour. Atom C9 was refined using a split model. The corresponding site-occupation factors were refined so that their sum was unity [0.910 (8) and 0.090 (8)]. A DELU restraint was used for all U^{ij} . The water molecule was refined as a rigid group with respect to x,y,z and its orientation (AFIX 6). The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å, the methyl C—H = 0.96 Å and the methylene C—H = 0.97 Å. $U_{iso}(H)$ values of the methyl group and the water molecule were set at 1.5 U_{eq} of the parent atom. The other H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

The crystals available for X-ray studies were of rather poor quality and weak scatterers at high theta value resulting in relatively high R values.

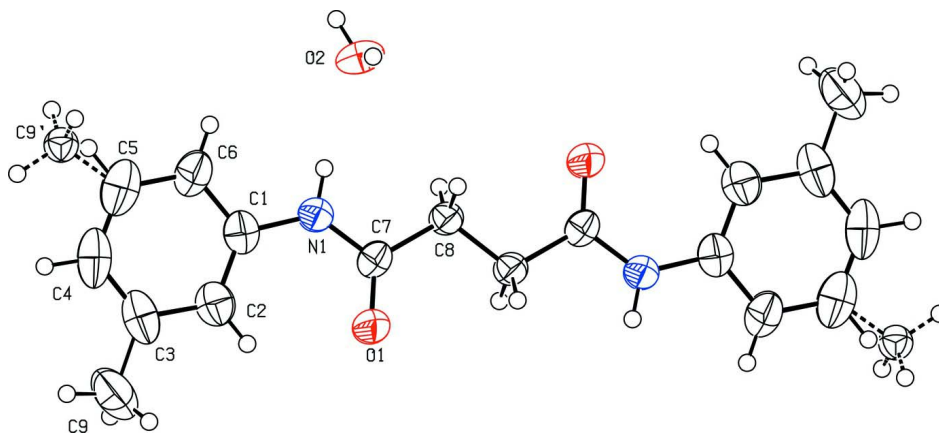
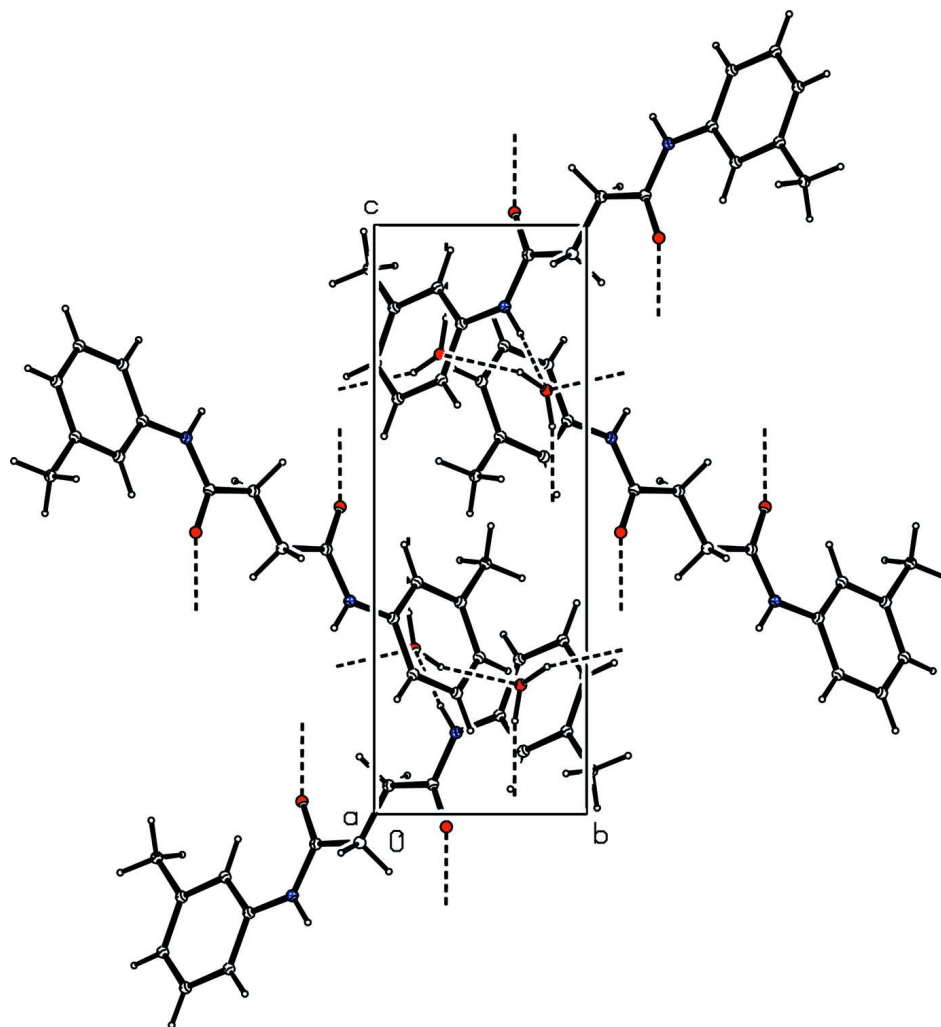


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level. The disordered methyl group at the 3 and 5 positions of the phenyl ring is shown with both orientations.

**Figure 2**

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

N,N'-Bis(3-methylphenyl)butane-1,4-diamide dihydrate

Crystal data

$C_{18}H_{20}N_2O_2 \cdot 2H_2O$

$M_r = 332.39$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 13.401 (4) \text{ \AA}$

$b = 4.937 (2) \text{ \AA}$

$c = 14.446 (4) \text{ \AA}$

$\beta = 108.67 (3)^\circ$

$V = 905.5 (5) \text{ \AA}^3$

$Z = 2$

$F(000) = 356$

$D_x = 1.219 \text{ Mg m}^{-3}$

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

Cell parameters from 433 reflections

$\theta = 2.9\text{--}28.2^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Needle, colourless

$0.48 \times 0.12 \times 0.04 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω scans
Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.960$, $T_{\max} = 0.997$

2857 measured reflections
1679 independent reflections
797 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$
 $\theta_{\max} = 25.7^\circ$, $\theta_{\min} = 2.9^\circ$
 $h = -10 \rightarrow 16$
 $k = -6 \rightarrow 4$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.126$
 $wR(F^2) = 0.159$
 $S = 1.23$
1679 reflections
123 parameters
10 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 1.5559P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.008$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.1286 (3)	0.1598 (8)	0.4781 (2)	0.0590 (12)	
N1	0.1521 (3)	0.1149 (9)	0.6385 (3)	0.0471 (13)	
H1N	0.1313	0.1791	0.6846	0.056*	
C1	0.2292 (4)	-0.0896 (12)	0.6675 (4)	0.0467 (15)	
C2	0.2784 (4)	-0.2019 (13)	0.6062 (4)	0.0582 (17)	
H2	0.2614	-0.1412	0.5421	0.070*	
C3	0.3535 (5)	-0.4063 (13)	0.6396 (6)	0.0668 (19)	
C4	0.3776 (5)	-0.4944 (14)	0.7340 (6)	0.076 (2)	
H4	0.4281	-0.6288	0.7572	0.091*	
C5	0.3280 (5)	-0.3861 (15)	0.7938 (6)	0.081 (2)	
H5	0.3439	-0.4510	0.8574	0.098*	
C6	0.2547 (5)	-0.1828 (13)	0.7627 (4)	0.0625 (18)	
H6	0.2226	-0.1086	0.8051	0.075*	
C7	0.1061 (4)	0.2251 (11)	0.5504 (4)	0.0408 (14)	
C8	0.0227 (4)	0.4323 (11)	0.5484 (3)	0.0407 (14)	
H8A	-0.0337	0.3441	0.5655	0.049*	

H8B	0.0530	0.5691	0.5975	0.049*	
C9	0.4042 (6)	-0.5247 (16)	0.5729 (5)	0.098 (3)	0.910 (8)
H9A	0.4538	-0.3979	0.5624	0.147*	0.910 (8)
H9B	0.3516	-0.5661	0.5116	0.147*	0.910 (8)
H9C	0.4403	-0.6878	0.6010	0.147*	0.910 (8)
C9'	0.367 (4)	-0.529 (11)	0.878 (4)	0.041 (18)	0.090 (8)
H9'A	0.4200	-0.6566	0.8750	0.061*	0.090 (8)
H9'B	0.3956	-0.4078	0.9317	0.061*	0.090 (8)
H9'C	0.3080	-0.6241	0.8861	0.061*	0.090 (8)
O2	0.0524 (3)	0.1884 (9)	0.2803 (2)	0.0583 (12)	
H21	0.0046	0.3006	0.2619	0.087*	
H22	0.0756	0.1747	0.3415	0.087*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.071 (3)	0.067 (3)	0.038 (2)	0.022 (2)	0.016 (2)	-0.002 (2)
N1	0.054 (3)	0.047 (3)	0.040 (3)	0.010 (3)	0.015 (2)	-0.002 (2)
C1	0.038 (3)	0.038 (4)	0.057 (4)	-0.006 (3)	0.006 (3)	-0.005 (3)
C2	0.052 (4)	0.055 (4)	0.067 (4)	0.000 (4)	0.018 (3)	0.002 (4)
C3	0.043 (4)	0.053 (5)	0.101 (6)	0.005 (4)	0.018 (4)	0.003 (4)
C4	0.057 (5)	0.056 (5)	0.098 (6)	0.010 (4)	0.001 (4)	0.020 (5)
C5	0.080 (6)	0.070 (6)	0.080 (5)	0.009 (5)	0.006 (4)	0.023 (5)
C6	0.067 (4)	0.058 (5)	0.054 (4)	0.007 (4)	0.007 (3)	0.009 (4)
C7	0.046 (4)	0.035 (4)	0.037 (3)	-0.004 (3)	0.007 (3)	0.000 (3)
C8	0.051 (3)	0.035 (4)	0.036 (3)	0.006 (3)	0.014 (3)	-0.001 (3)
C9	0.086 (6)	0.099 (7)	0.121 (7)	0.037 (5)	0.051 (5)	0.005 (6)
C9'	0.041 (18)	0.040 (19)	0.040 (18)	0.000 (5)	0.013 (7)	0.001 (5)
O2	0.079 (3)	0.062 (3)	0.037 (2)	0.012 (2)	0.023 (2)	0.008 (2)

Geometric parameters (Å, °)

O1—C7	1.219 (5)	C6—H6	0.9300
N1—C7	1.340 (6)	C7—C8	1.509 (6)
N1—C1	1.409 (6)	C8—C8 ⁱ	1.492 (8)
N1—H1N	0.8600	C8—H8A	0.9700
C1—C2	1.378 (7)	C8—H8B	0.9700
C1—C6	1.385 (7)	C9—H9A	0.9600
C2—C3	1.398 (8)	C9—H9B	0.9600
C2—H2	0.9300	C9—H9C	0.9600
C3—C4	1.368 (8)	C9'—H9'A	0.9600
C3—C9	1.466 (8)	C9'—H9'B	0.9600
C4—C5	1.358 (8)	C9'—H9'C	0.9600
C4—H4	0.9300	O2—H21	0.8235
C5—C6	1.375 (8)	O2—H22	0.8398
C5—H5	0.9300		
C7—N1—C1	130.1 (5)	C1—C6—H6	120.3

C7—N1—H1N	115.0	O1—C7—N1	122.8 (5)
C1—N1—H1N	115.0	O1—C7—C8	123.2 (5)
C2—C1—C6	119.1 (6)	N1—C7—C8	114.0 (5)
C2—C1—N1	123.6 (5)	C8 ⁱ —C8—C7	113.5 (5)
C6—C1—N1	117.2 (5)	C8 ⁱ —C8—H8A	108.9
C1—C2—C3	120.6 (6)	C7—C8—H8A	108.9
C1—C2—H2	119.7	C8 ⁱ —C8—H8B	108.9
C3—C2—H2	119.7	C7—C8—H8B	108.9
C4—C3—C2	119.2 (6)	H8A—C8—H8B	107.7
C4—C3—C9	121.1 (7)	C3—C9—H9A	109.5
C2—C3—C9	119.7 (7)	C3—C9—H9B	109.5
C5—C4—C3	120.1 (7)	H9A—C9—H9B	109.5
C5—C4—H4	119.9	C3—C9—H9C	109.5
C3—C4—H4	119.9	H9A—C9—H9C	109.5
C4—C5—C6	121.5 (7)	H9B—C9—H9C	109.5
C4—C5—H5	119.2	H9'A—C9'—H9'B	109.5
C6—C5—H5	119.2	H9'A—C9'—H9'C	109.5
C5—C6—C1	119.4 (6)	H9'B—C9'—H9'C	109.5
C5—C6—H6	120.3	H21—O2—H22	112.4
C7—N1—C1—C2	5.4 (9)	C3—C4—C5—C6	-1.5 (11)
C7—N1—C1—C6	-173.6 (5)	C4—C5—C6—C1	1.3 (10)
C6—C1—C2—C3	-0.3 (8)	C2—C1—C6—C5	-0.4 (9)
N1—C1—C2—C3	-179.4 (5)	N1—C1—C6—C5	178.7 (5)
C1—C2—C3—C4	0.1 (9)	C1—N1—C7—O1	-1.5 (9)
C1—C2—C3—C9	179.4 (6)	C1—N1—C7—C8	177.3 (5)
C2—C3—C4—C5	0.8 (10)	O1—C7—C8—C8 ⁱ	-5.3 (9)
C9—C3—C4—C5	-178.5 (7)	N1—C7—C8—C8 ⁱ	175.9 (6)

Symmetry code: (i) $-x, -y+1, -z+1$.

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

<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>
N1—H1N \cdots O2 ⁱⁱ	0.86	2.10	2.946 (6)	169
O2—H21 \cdots O2 ⁱⁱⁱ	0.82	2.08	2.836 (4)	153
O2—H22 \cdots O1	0.84	1.87	2.713 (5)	178

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