Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(5-Bromo-2-methoxybenzylidene)-2-methoxybenzohydrazide

Xue-Song Lin* and Ya-Li Sang

Department of Chemistry, Chifeng University, Chifeng 024001, People's Republic of China
Correspondence e-mail: xuesong_lin@126.com

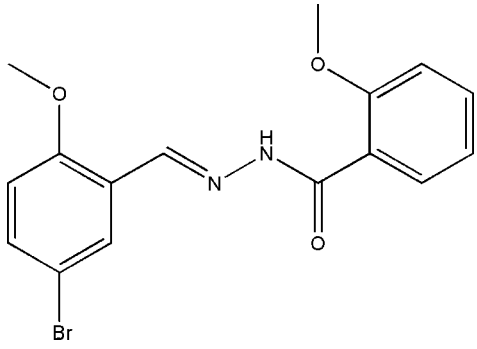
Received 13 June 2009; accepted 15 June 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.096; data-to-parameter ratio = 16.3.

The title hydrazone compound, $C_{16}H_{15}BrN_2O_3$, adopts an E configuration about the C=N double bond. The molecule is twisted, the dihedral angle between the two substituted benzene rings being 22.0 (2)°. In the crystal structure, molecules are linked through intermolecular N—H···O hydrogen bonds, forming chains along the c axis.

Related literature

For the biological properties of the hydrazone compounds, see: Khattab *et al.* (2005); Küçükgüzel *et al.* (2003); Çukurovalı *et al.* (2006). For the structures of hydrazone derivatives, see: Fun *et al.* (2008); Wei *et al.* (2009); Khaledi *et al.* (2008); Yang *et al.* (2008). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{15}BrN_2O_3$
M_r = 363.21
Monoclinic, $P2_1/c$
a = 13.3286 (3) Å
b = 11.4816 (3) Å
c = 10.1233 (2) Å
β = 99.128 (1)°
V = 1529.59 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.70 mm^{-1}
T = 298 K
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.614, T_{max} = 0.642
9188 measured reflections
3323 independent reflections
2281 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.01
3323 reflections
204 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.894 (10)	2.179 (19)	2.997 (3)	152 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

We are gratefully acknowledge Chifeng University for a research funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2816).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2002). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Çukurovalı, A., Yılmaz, I., Gür, S. & Kazaz, C. (2006). *Eur. J. Med. Chem.* **41**, 201–207.
Fun, H.-K., Patil, P. S., Rao, J. N., Kalluraya, B. & Chantrapromma, S. (2008). *Acta Cryst.* E**64**, o1707.
Khaledi, H., Mohd Ali, H. & Ng, S. W. (2008). *Acta Cryst.* E**64**, o2481.
Khattab, S. N. (2005). *Molecules*, **10**, 1218–1228.
Küçükgüzel, S. G., Mazi, A., Şahin, F., Öztürk, S. & Stables, J. (2003). *Eur. J. Med. Chem.* **38**, 1005–1013.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wei, Y.-J., Wang, F.-W. & Zhu, Q.-Y. (2009). *Acta Cryst.* E**65**, o688.
Yang, T., Cao, G.-B., Xiang, J.-M. & Zhang, L.-H. (2008). *Acta Cryst.* E**64**, o1186.

supporting information

Acta Cryst. (2009). E**65**, o1649 [doi:10.1107/S1600536809022971]

N′-(5-Bromo-2-methoxybenzylidene)-2-methoxybenzohydrazide

Xue-Song Lin and Ya-Li Sang

S1. Comment

Hydrazone and Schiff base compounds derived from the reaction of aldehydes with hydrazides have been widely investigated both for their crystal structures and biological properties (Khattab *et al.*, 2005; Küçükgüzel *et al.*, 2003; Çukurovalı *et al.*, 2006). In the last few years, a large number of hydrazone derivatives have been reported (Fun *et al.*, 2008; Wei *et al.*, 2009; Khaledi *et al.*, 2008; Yang *et al.*, 2008). However, the hydrazone compounds derived the 5-bromo-2-methoxybenzaldehyde have never been reported. In this paper, the crystal structure of the title new hydrazone compound, (I), derived from the reaction of 5-bromo-2-methoxybenzaldehyde and 2-methoxybenzohydrazide, is reported.

The molecular structure of (I) is shown as Fig. 1. The molecule adopts an *E* configuration about the C═N double bond. The molecule is twisted about the C8—N1—N2—C9 moiety, with the dihedral angle between the C1—C6 and C10—C15 benzene rings of 22.0 (2)°. All the bond lengths are within normal values (Allen *et al.*, 1987).

In the crystal structure of the compound, molecules are linked through intermolecular N–H···O hydrogen bonds (Table 1), forming chains along the *c* axis, as shown in Fig. 2.

S2. Experimental

5-Bromo-2-methoxybenzaldehyde (1.0 mmol, 215.0 mg) and 2-methoxybenzohydrazide (1.0 mmol, 166.2 mg) were mixed and refluxed in methanol (50 ml). The mixture was stirred for 1 h to give a clear colourless solution. Colourless crystals of (I) were formed by slow evaporation of the solution in air for a few days.

S3. Refinement

H2 attached to N2 was located in a difference map and refined with N–H distance restraint of 0.90 (1) Å. The other H atoms were positioned geometrically [d(C–H) = 0.93–0.96 Å], and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C_{methyl}).

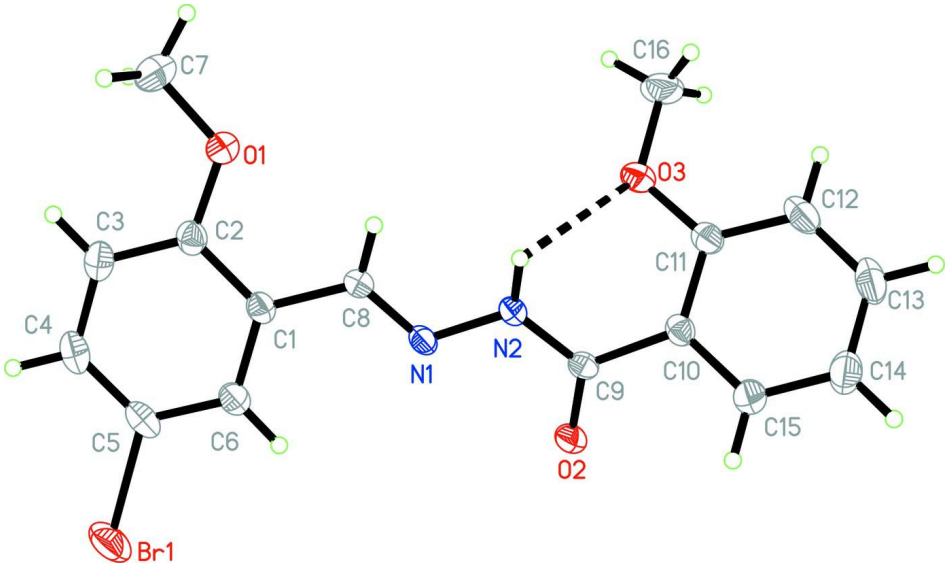


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

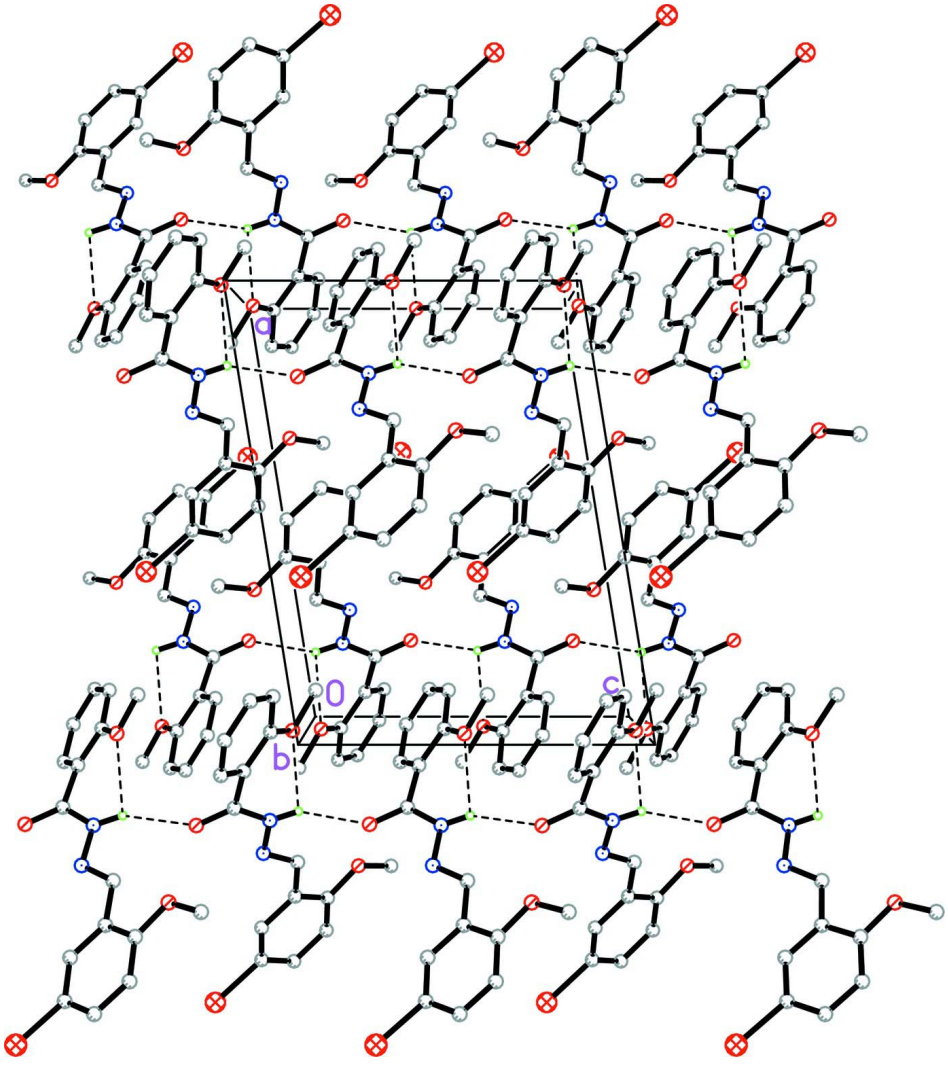


Figure 2

The crystal packing of (I). Hydrogen atoms not involved in hydrogen bonding have been omitted. Intermolecular hydrogen bonds are shown as dashed lines.

N′-(5-Bromo-2-methoxybenzylidene)-2-methoxybenzohydrazide

Crystal data

$C_{16}H_{15}BrN_2O_3$
$M_r = 363.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.3286\ (3)$ Å
$b = 11.4816\ (3)$ Å
$c = 10.1233\ (2)$ Å
$\beta = 99.128\ (1)°$
$V = 1529.59\ (6)$ Å^3
$Z = 4$

$F(000) = 736$
$D_x = 1.577$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2540 reflections
$\theta = 2.4–27.4°$
$\mu = 2.70$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.614, T_{max} = 0.642
9188 measured reflections
3323 independent reflections
2281 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.0°, θ_{min} = 1.6°
h = −16→15
k = −14→12
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.01
3323 reflections
204 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 1.0409P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.36301 (2)	1.06194 (4)	0.08660 (4)	0.07080 (16)
O1	0.67425 (14)	1.12056 (19)	0.5818 (2)	0.0511 (5)
O2	0.80411 (13)	0.73514 (17)	0.15200 (18)	0.0419 (5)
O3	1.01119 (14)	0.80174 (18)	0.4696 (2)	0.0497 (5)
N1	0.72756 (15)	0.88017 (18)	0.3201 (2)	0.0327 (5)
N2	0.80859 (15)	0.80683 (19)	0.3608 (2)	0.0322 (5)
C1	0.61419 (18)	1.0218 (2)	0.3825 (3)	0.0335 (6)
C2	0.60017 (19)	1.1107 (2)	0.4729 (3)	0.0377 (6)
C3	0.5157 (2)	1.1822 (2)	0.4485 (3)	0.0447 (7)
H3	0.5068	1.2409	0.5089	0.054*
C4	0.4447 (2)	1.1667 (3)	0.3350 (3)	0.0484 (7)
H4	0.3873	1.2138	0.3194	0.058*
C5	0.4594 (2)	1.0810 (3)	0.2451 (3)	0.0438 (7)
C6	0.54289 (19)	1.0089 (2)	0.2674 (3)	0.0383 (6)
H6	0.5516	0.9515	0.2054	0.046*

C7	0.6646 (3)	1.2090 (3)	0.6772 (4)	0.0734 (11)
H7A	0.6630	1.2839	0.6347	0.110*
H7B	0.7214	1.2054	0.7483	0.110*
H7C	0.6027	1.1974	0.7129	0.110*
C8	0.70088 (18)	0.9428 (2)	0.4122 (3)	0.0340 (6)
H8	0.7366	0.9383	0.4988	0.041*
C9	0.84257 (17)	0.7374 (2)	0.2700 (2)	0.0300 (5)
C10	0.93090 (18)	0.6607 (2)	0.3210 (2)	0.0331 (6)
C11	1.01527 (19)	0.6938 (3)	0.4138 (3)	0.0376 (6)
C12	1.0973 (2)	0.6182 (3)	0.4424 (3)	0.0517 (8)
H12	1.1548	0.6414	0.5010	0.062*
C13	1.0941 (3)	0.5100 (3)	0.3852 (3)	0.0588 (9)
H13	1.1493	0.4600	0.4056	0.071*
C14	1.0107 (3)	0.4748 (3)	0.2987 (3)	0.0576 (9)
H14	1.0080	0.4002	0.2626	0.069*
C15	0.9304 (2)	0.5502 (2)	0.2649 (3)	0.0447 (7)
H15	0.8747	0.5267	0.2034	0.054*
C16	1.0993 (2)	0.8449 (4)	0.5536 (4)	0.0690 (10)
H16A	1.1117	0.7995	0.6342	0.103*
H16B	1.0887	0.9248	0.5757	0.103*
H16C	1.1568	0.8395	0.5076	0.103*
H2	0.830 (3)	0.802 (3)	0.4490 (12)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0439 (2)	0.0908 (3)	0.0690 (3)	0.00890 (18)	−0.01763 (15)	0.0059 (2)
O1	0.0405 (11)	0.0619 (14)	0.0490 (12)	0.0068 (10)	0.0010 (9)	−0.0198 (10)
O2	0.0364 (10)	0.0531 (12)	0.0333 (10)	0.0071 (9)	−0.0033 (8)	−0.0053 (9)
O3	0.0324 (10)	0.0606 (14)	0.0520 (12)	0.0012 (9)	−0.0057 (9)	−0.0146 (10)
N1	0.0240 (10)	0.0347 (12)	0.0372 (12)	0.0022 (9)	−0.0016 (8)	0.0008 (10)
N2	0.0280 (10)	0.0361 (12)	0.0309 (11)	0.0053 (9)	−0.0006 (9)	−0.0003 (10)
C1	0.0268 (12)	0.0352 (14)	0.0387 (14)	0.0005 (10)	0.0050 (10)	0.0012 (11)
C2	0.0302 (13)	0.0409 (15)	0.0433 (15)	−0.0017 (12)	0.0103 (11)	0.0002 (12)
C3	0.0411 (15)	0.0387 (17)	0.0570 (19)	0.0061 (13)	0.0161 (14)	−0.0006 (14)
C4	0.0343 (14)	0.0463 (18)	0.066 (2)	0.0095 (13)	0.0111 (14)	0.0104 (15)
C5	0.0279 (13)	0.0518 (18)	0.0496 (17)	−0.0003 (12)	−0.0007 (12)	0.0129 (14)
C6	0.0322 (13)	0.0392 (15)	0.0424 (15)	0.0007 (12)	0.0022 (11)	0.0018 (13)
C7	0.066 (2)	0.086 (3)	0.067 (2)	0.002 (2)	0.0068 (18)	−0.036 (2)
C8	0.0280 (12)	0.0366 (15)	0.0354 (14)	0.0003 (11)	−0.0003 (10)	−0.0040 (12)
C9	0.0254 (11)	0.0327 (14)	0.0316 (13)	−0.0044 (10)	0.0030 (10)	0.0011 (11)
C10	0.0298 (12)	0.0402 (15)	0.0299 (13)	0.0040 (11)	0.0063 (10)	0.0027 (11)
C11	0.0309 (13)	0.0519 (18)	0.0302 (13)	0.0037 (12)	0.0055 (10)	0.0004 (12)
C12	0.0344 (15)	0.076 (2)	0.0432 (17)	0.0123 (15)	0.0004 (12)	0.0061 (16)
C13	0.0533 (19)	0.069 (2)	0.0544 (19)	0.0303 (18)	0.0104 (15)	0.0104 (18)
C14	0.067 (2)	0.0481 (19)	0.057 (2)	0.0191 (17)	0.0089 (17)	−0.0003 (16)
C15	0.0456 (16)	0.0454 (18)	0.0417 (16)	0.0073 (14)	0.0026 (12)	−0.0025 (13)
C16	0.0376 (17)	0.099 (3)	0.066 (2)	−0.0064 (18)	−0.0055 (15)	−0.033 (2)

Geometric parameters (Å, º)

Br1—C5	1.902 (3)	C6—H6	0.9300
O1—C2	1.363 (3)	C7—H7A	0.9600
O1—C7	1.421 (4)	C7—H7B	0.9600
O2—C9	1.223 (3)	C7—H7C	0.9600
O3—C11	1.367 (3)	C8—H8	0.9300
O3—C16	1.425 (3)	C9—C10	1.495 (3)
N1—C8	1.273 (3)	C10—C15	1.390 (4)
N1—N2	1.380 (3)	C10—C11	1.398 (3)
N2—C9	1.348 (3)	C11—C12	1.389 (4)
N2—H2	0.894 (10)	C12—C13	1.369 (5)
C1—C6	1.390 (4)	C12—H12	0.9300
C1—C2	1.402 (4)	C13—C14	1.363 (5)
C1—C8	1.462 (3)	C13—H13	0.9300
C2—C3	1.383 (4)	C14—C15	1.377 (4)
C3—C4	1.380 (4)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.376 (4)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C5—C6	1.376 (4)	C16—H16C	0.9600
C2—O1—C7	118.5 (2)	N1—C8—C1	120.2 (2)
C11—O3—C16	118.6 (2)	N1—C8—H8	119.9
C8—N1—N2	115.0 (2)	C1—C8—H8	119.9
C9—N2—N1	119.2 (2)	O2—C9—N2	123.0 (2)
C9—N2—H2	124 (2)	O2—C9—C10	120.7 (2)
N1—N2—H2	116 (2)	N2—C9—C10	116.3 (2)
C6—C1—C2	118.7 (2)	C15—C10—C11	118.3 (2)
C6—C1—C8	121.3 (2)	C15—C10—C9	116.3 (2)
C2—C1—C8	120.0 (2)	C11—C10—C9	125.3 (2)
O1—C2—C3	124.0 (3)	O3—C11—C12	124.2 (3)
O1—C2—C1	115.7 (2)	O3—C11—C10	116.3 (2)
C3—C2—C1	120.3 (3)	C12—C11—C10	119.5 (3)
C4—C3—C2	120.3 (3)	C13—C12—C11	120.5 (3)
C4—C3—H3	119.9	C13—C12—H12	119.7
C2—C3—H3	119.9	C11—C12—H12	119.7
C5—C4—C3	119.5 (3)	C14—C13—C12	120.6 (3)
C5—C4—H4	120.3	C14—C13—H13	119.7
C3—C4—H4	120.3	C12—C13—H13	119.7
C6—C5—C4	121.2 (3)	C13—C14—C15	119.7 (3)
C6—C5—Br1	119.3 (2)	C13—C14—H14	120.1
C4—C5—Br1	119.5 (2)	C15—C14—H14	120.1
C5—C6—C1	120.1 (3)	C14—C15—C10	121.3 (3)
C5—C6—H6	120.0	C14—C15—H15	119.4
C1—C6—H6	120.0	C10—C15—H15	119.4
O1—C7—H7A	109.5	O3—C16—H16A	109.5
O1—C7—H7B	109.5	O3—C16—H16B	109.5

H7A—C7—H7B	109.5	H16A—C16—H16B	109.5
O1—C7—H7C	109.5	O3—C16—H16C	109.5
H7A—C7—H7C	109.5	H16A—C16—H16C	109.5
H7B—C7—H7C	109.5	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.89 (1)	2.18 (2)	2.997 (3)	152 (3)

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