

Benzyl 2-(4-bromoanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbothioate: first triclinic polymorph

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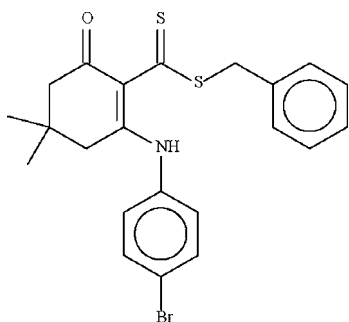
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.077; data-to-parameter ratio = 18.8.

The six-membered cyclohexene ring in the title compound, $\text{C}_{22}\text{H}_{22}\text{BrNOS}_2$, adopts an envelope conformation, with the C atom bearing the two methyl groups representing the flap. This atom deviates by 0.686 (4) Å from the plane passing through the other five atoms of the ring (r.m.s. deviation = 0.025 Å). The molecular conformation is stabilized by an intramolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bond.

Related literature

For previous work on this topic, see: El Ashry *et al.* (2005*a,b*, 2006, 2008*a,b*); El Ashry, Kassem *et al.* (2009). For the use of enamines in heterocyclic synthesis, see: Tominaga (1989); Tominaga *et al.* (1991). For another triclinic polymorph of the title compound, see El Ashry, Amer *et al.* (2009).



Experimental

Crystal data

$\text{C}_{22}\text{H}_{22}\text{BrNOS}_2$
 $M_r = 460.44$
Triclinic, $P\bar{1}$

$a = 9.3030$ (3) Å
 $b = 9.8099$ (3) Å
 $c = 12.3538$ (4) Å

$\alpha = 73.464$ (2)°
 $\beta = 72.391$ (2)°
 $\gamma = 89.916$ (2)°
 $V = 1025.73$ (6) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 2.22$ mm⁻¹
 $T = 100$ K
 $0.40 \times 0.04 \times 0.04$ mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.684$, $T_{\max} = 0.917$

9757 measured reflections
4709 independent reflections
3490 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.077$
 $S = 0.99$
4709 reflections
250 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{S2}$	0.88 (1)	2.10 (2)	2.905 (2)	151 (3)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2876).

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supporting information

Acta Cryst. (2009). E65, o597 [doi:10.1107/S160053680900614X]

Benzyl 2-(4-bromoanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate: first triclinic polymorph

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S1. Comment

The enaminothiocarboxylates are a class of compounds of crucial significance for designing functionalized molecules that are suitable for studying the biological activity of potentially useful chemicals. They are also important reactants in the synthesis of heterocyclic compounds (Tominaga, 1989; Tominaga *et al.*, 1991). Our interest in such class of compounds revolves around new glycosyl donors (El Ashry *et al.*, 2005a, 2005b, 2006, 2008a, 2008b, El Ashry, Kassem *et al.* 2009). The title compound is to be used in a model study to understand the scope of the reaction that uses cyclic enamines. A possibility exists that the representative title compound can lead to a fused cyclized product.

S2. Experimental

To a solution of 3-(4-bromoanilino)-5,5-dimethyl-cyclohex-2-en-1-one (0.1 mol) in DMSO (20 ml) and sodium hydroxide (0.1 mol) in water (1 ml) was added carbon disulphide (0.3 mol). The mixture was kept at 263 K for 20 min. Benzyl bromide (0.1 mol) was added. The mixture was left for 24 h, after which it was quenched with water (200 ml) and then acidified with 10% hydrochloric acid. The resulting precipitate was collected by filtration, dried and purified on a silica gel column (30% ethyl acetate in hexane) to give orange crystals (40% yield, mp 458 K). HRMS for $C_{22}H_{22}BrNOS_2$, calc.: 459.0326, found, m/z : 459.0316.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 to 1.5 $U(C)$. The methyl groups were allowed to rotate but not to tip.

The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N—H 0.88±0.01 Å; its isotropic displacement parameter was freely refined.

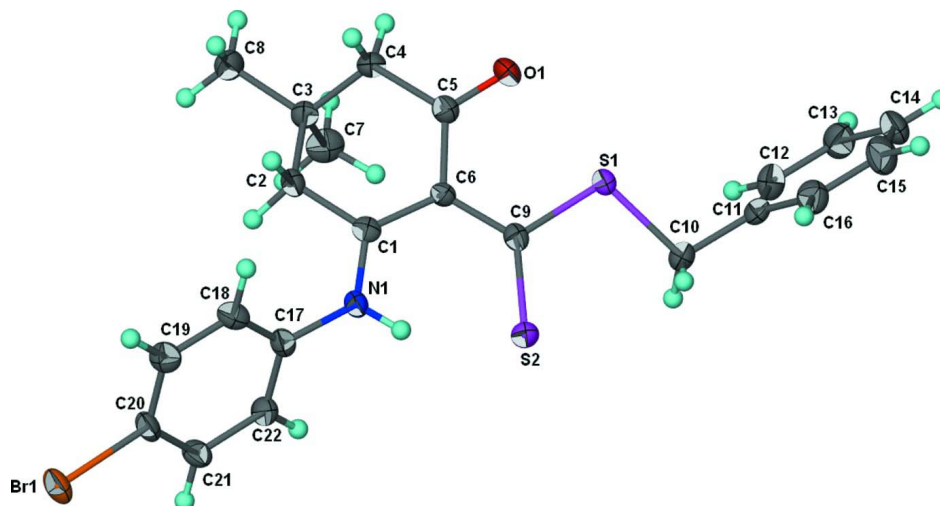


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) plot of $C_{22}H_{22}BrNOS_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Benzyl 2-(4-bromoanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbothioate

Crystal data

$C_{22}H_{22}BrNOS_2$

$M_r = 460.44$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.3030(3) \text{ \AA}$

$b = 9.8099(3) \text{ \AA}$

$c = 12.3538(4) \text{ \AA}$

$\alpha = 73.464(2)^\circ$

$\beta = 72.391(2)^\circ$

$\gamma = 89.916(2)^\circ$

$V = 1025.73(6) \text{ \AA}^3$

$Z = 2$

$F(000) = 472$

$D_x = 1.491 \text{ Mg m}^{-3}$

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

Cell parameters from 2124 reflections

$\theta = 2.4\text{--}25.2^\circ$

$\mu = 2.22 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Prism, yellow

$0.40 \times 0.04 \times 0.04 \text{ mm}$

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.684$, $T_{\max} = 0.917$

9757 measured reflections

4709 independent reflections

3490 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 1.8^\circ$

$h = -11 \rightarrow 12$

$k = -12 \rightarrow 12$

$l = -16 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.077$

$S = 0.99$

4709 reflections

250 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

$$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2 + 0.5453P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.47 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.38 \text{ e } \text{Å}^{-3}$$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.36770 (4)	0.20219 (3)	1.10143 (3)	0.02491 (9)
S1	0.64466 (8)	1.01099 (7)	0.25672 (6)	0.01823 (16)
S2	0.74397 (8)	0.76685 (8)	0.41055 (6)	0.02190 (17)
O1	0.3727 (2)	1.0432 (2)	0.34593 (18)	0.0319 (5)
N1	0.4826 (3)	0.6419 (2)	0.6181 (2)	0.0182 (5)
H1	0.5775 (16)	0.658 (3)	0.571 (2)	0.035 (10)*
C1	0.3918 (3)	0.7277 (3)	0.5732 (2)	0.0153 (6)
C2	0.2258 (3)	0.7000 (3)	0.6438 (2)	0.0181 (6)
H2A	0.2056	0.7509	0.7048	0.022*
H2B	0.2024	0.5966	0.6860	0.022*
C3	0.1189 (3)	0.7459 (3)	0.5701 (2)	0.0179 (6)
C4	0.1689 (3)	0.9016 (3)	0.5000 (2)	0.0196 (6)
H4A	0.1096	0.9325	0.4443	0.024*
H4B	0.1437	0.9594	0.5560	0.024*
C5	0.3345 (3)	0.9339 (3)	0.4300 (2)	0.0192 (6)
C6	0.4459 (3)	0.8392 (3)	0.4637 (2)	0.0149 (6)
C7	0.1277 (3)	0.6549 (3)	0.4872 (3)	0.0267 (7)
H7A	0.0547	0.6832	0.4439	0.040*
H7B	0.1040	0.5540	0.5340	0.040*
H7C	0.2302	0.6689	0.4304	0.040*
C8	-0.0436 (3)	0.7327 (3)	0.6537 (3)	0.0256 (7)
H8A	-0.1117	0.7662	0.6067	0.038*
H8B	-0.0483	0.7909	0.7072	0.038*
H8C	-0.0747	0.6326	0.7007	0.038*
C9	0.6016 (3)	0.8658 (3)	0.3878 (2)	0.0158 (6)
C10	0.8407 (3)	0.9962 (3)	0.1768 (2)	0.0211 (6)
H10A	0.9090	1.0131	0.2204	0.025*
H10B	0.8531	0.9004	0.1655	0.025*
C11	0.8740 (3)	1.1104 (3)	0.0584 (2)	0.0198 (6)
C12	0.8062 (3)	1.0952 (3)	-0.0244 (3)	0.0243 (7)
H12	0.7392	1.0127	-0.0055	0.029*
C13	0.8346 (3)	1.1978 (3)	-0.1334 (3)	0.0290 (7)
H13	0.7881	1.1855	-0.1891	0.035*
C14	0.9310 (4)	1.3184 (3)	-0.1609 (3)	0.0306 (8)
H14	0.9511	1.3891	-0.2358	0.037*
C15	0.9979 (4)	1.3362 (3)	-0.0802 (3)	0.0306 (8)

H15	1.0637	1.4194	-0.0992	0.037*
C16	0.9693 (3)	1.2323 (3)	0.0298 (3)	0.0250 (7)
H16	1.0156	1.2455	0.0853	0.030*
C17	0.4444 (3)	0.5381 (3)	0.7324 (2)	0.0168 (6)
C18	0.3865 (3)	0.5766 (3)	0.8354 (2)	0.0203 (6)
H18	0.3649	0.6724	0.8308	0.024*
C19	0.3599 (3)	0.4755 (3)	0.9452 (3)	0.0212 (6)
H19	0.3188	0.5013	1.0163	0.025*
C20	0.3937 (3)	0.3364 (3)	0.9507 (2)	0.0197 (6)
C21	0.4509 (3)	0.2970 (3)	0.8484 (2)	0.0202 (6)
H21	0.4732	0.2014	0.8531	0.024*
C22	0.4755 (3)	0.3981 (3)	0.7388 (2)	0.0202 (6)
H22	0.5137	0.3715	0.6678	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02959 (18)	0.02257 (16)	0.01791 (15)	-0.00197 (12)	-0.00961 (12)	0.00337 (12)
S1	0.0185 (4)	0.0162 (3)	0.0149 (3)	0.0032 (3)	-0.0018 (3)	-0.0005 (3)
S2	0.0162 (4)	0.0236 (4)	0.0205 (4)	0.0054 (3)	-0.0041 (3)	-0.0002 (3)
O1	0.0258 (12)	0.0285 (12)	0.0251 (12)	0.0089 (10)	-0.0013 (9)	0.0094 (9)
N1	0.0154 (13)	0.0186 (12)	0.0158 (12)	0.0009 (10)	-0.0043 (10)	0.0014 (10)
C1	0.0181 (15)	0.0129 (13)	0.0176 (14)	0.0016 (11)	-0.0075 (12)	-0.0066 (11)
C2	0.0177 (15)	0.0172 (14)	0.0153 (14)	0.0004 (11)	-0.0040 (11)	-0.0001 (11)
C3	0.0163 (15)	0.0191 (14)	0.0175 (14)	0.0018 (12)	-0.0060 (12)	-0.0035 (12)
C4	0.0173 (15)	0.0186 (14)	0.0209 (15)	0.0046 (12)	-0.0054 (12)	-0.0036 (12)
C5	0.0205 (15)	0.0197 (15)	0.0154 (14)	0.0034 (12)	-0.0049 (12)	-0.0027 (12)
C6	0.0153 (14)	0.0166 (13)	0.0126 (13)	0.0035 (11)	-0.0060 (11)	-0.0023 (11)
C7	0.0238 (17)	0.0280 (16)	0.0337 (18)	0.0017 (13)	-0.0137 (14)	-0.0124 (14)
C8	0.0162 (15)	0.0254 (16)	0.0288 (17)	0.0011 (13)	-0.0051 (13)	-0.0004 (13)
C9	0.0185 (14)	0.0148 (13)	0.0134 (13)	0.0004 (11)	-0.0048 (11)	-0.0034 (11)
C10	0.0168 (15)	0.0216 (15)	0.0199 (15)	0.0026 (12)	-0.0010 (12)	-0.0036 (12)
C11	0.0167 (15)	0.0192 (14)	0.0190 (15)	0.0046 (12)	0.0003 (12)	-0.0052 (12)
C12	0.0210 (16)	0.0254 (16)	0.0224 (16)	-0.0034 (13)	-0.0029 (13)	-0.0051 (13)
C13	0.0250 (17)	0.0389 (19)	0.0197 (16)	0.0040 (14)	-0.0079 (13)	-0.0026 (14)
C14	0.0350 (19)	0.0254 (17)	0.0206 (16)	0.0059 (15)	-0.0027 (14)	0.0032 (13)
C15	0.0343 (19)	0.0179 (15)	0.0315 (18)	-0.0040 (14)	-0.0017 (15)	-0.0042 (14)
C16	0.0258 (17)	0.0248 (16)	0.0229 (16)	0.0013 (13)	-0.0054 (13)	-0.0072 (13)
C17	0.0150 (14)	0.0180 (14)	0.0160 (14)	-0.0014 (11)	-0.0068 (11)	-0.0009 (11)
C18	0.0227 (16)	0.0164 (14)	0.0236 (15)	0.0029 (12)	-0.0106 (13)	-0.0054 (12)
C19	0.0236 (16)	0.0234 (15)	0.0189 (15)	0.0034 (13)	-0.0095 (12)	-0.0067 (12)
C20	0.0191 (15)	0.0189 (14)	0.0165 (14)	-0.0018 (12)	-0.0072 (12)	0.0033 (12)
C21	0.0220 (16)	0.0139 (14)	0.0232 (15)	0.0014 (12)	-0.0076 (12)	-0.0026 (12)
C22	0.0207 (15)	0.0221 (15)	0.0151 (14)	0.0040 (12)	-0.0037 (12)	-0.0035 (12)

Geometric parameters (Å, °)

Br1—C20	1.895 (3)	C8—H8C	0.9800
S1—C9	1.763 (3)	C10—C11	1.511 (4)
S1—C10	1.820 (3)	C10—H10A	0.9900
S2—C9	1.686 (3)	C10—H10B	0.9900
O1—C5	1.224 (3)	C11—C16	1.384 (4)
N1—C1	1.321 (3)	C11—C12	1.394 (4)
N1—C17	1.427 (3)	C12—C13	1.383 (4)
N1—H1	0.884 (10)	C12—H12	0.9500
C1—C6	1.424 (3)	C13—C14	1.382 (4)
C1—C2	1.506 (4)	C13—H13	0.9500
C2—C3	1.527 (4)	C14—C15	1.373 (4)
C2—H2A	0.9900	C14—H14	0.9500
C2—H2B	0.9900	C15—C16	1.396 (4)
C3—C4	1.518 (4)	C15—H15	0.9500
C3—C7	1.524 (4)	C16—H16	0.9500
C3—C8	1.532 (4)	C17—C18	1.382 (4)
C4—C5	1.504 (4)	C17—C22	1.388 (4)
C4—H4A	0.9900	C18—C19	1.385 (4)
C4—H4B	0.9900	C18—H18	0.9500
C5—C6	1.465 (4)	C19—C20	1.388 (4)
C6—C9	1.444 (4)	C19—H19	0.9500
C7—H7A	0.9800	C20—C21	1.379 (4)
C7—H7B	0.9800	C21—C22	1.385 (4)
C7—H7C	0.9800	C21—H21	0.9500
C8—H8A	0.9800	C22—H22	0.9500
C8—H8B	0.9800		
C9—S1—C10	104.06 (13)	C6—C9—S1	116.85 (19)
C1—N1—C17	127.3 (2)	S2—C9—S1	117.21 (16)
C1—N1—H1	113 (2)	C11—C10—S1	104.63 (18)
C17—N1—H1	120 (2)	C11—C10—H10A	110.8
N1—C1—C6	122.4 (2)	S1—C10—H10A	110.8
N1—C1—C2	116.2 (2)	C11—C10—H10B	110.8
C6—C1—C2	121.4 (2)	S1—C10—H10B	110.8
C1—C2—C3	114.7 (2)	H10A—C10—H10B	108.9
C1—C2—H2A	108.6	C16—C11—C12	118.4 (3)
C3—C2—H2A	108.6	C16—C11—C10	121.7 (3)
C1—C2—H2B	108.6	C12—C11—C10	119.9 (3)
C3—C2—H2B	108.6	C13—C12—C11	121.2 (3)
H2A—C2—H2B	107.6	C13—C12—H12	119.4
C4—C3—C7	111.0 (2)	C11—C12—H12	119.4
C4—C3—C2	106.0 (2)	C14—C13—C12	119.6 (3)
C7—C3—C2	111.0 (2)	C14—C13—H13	120.2
C4—C3—C8	109.5 (2)	C12—C13—H13	120.2
C7—C3—C8	109.9 (2)	C15—C14—C13	120.1 (3)
C2—C3—C8	109.4 (2)	C15—C14—H14	119.9

C5—C4—C3	115.2 (2)	C13—C14—H14	119.9
C5—C4—H4A	108.5	C14—C15—C16	120.2 (3)
C3—C4—H4A	108.5	C14—C15—H15	119.9
C5—C4—H4B	108.5	C16—C15—H15	119.9
C3—C4—H4B	108.5	C11—C16—C15	120.4 (3)
H4A—C4—H4B	107.5	C11—C16—H16	119.8
O1—C5—C6	121.5 (3)	C15—C16—H16	119.8
O1—C5—C4	117.6 (2)	C18—C17—C22	120.1 (2)
C6—C5—C4	120.9 (2)	C18—C17—N1	121.1 (2)
C1—C6—C9	124.3 (2)	C22—C17—N1	118.6 (2)
C1—C6—C5	116.5 (2)	C17—C18—C19	120.0 (3)
C9—C6—C5	119.2 (2)	C17—C18—H18	120.0
C3—C7—H7A	109.5	C19—C18—H18	120.0
C3—C7—H7B	109.5	C18—C19—C20	119.4 (3)
H7A—C7—H7B	109.5	C18—C19—H19	120.3
C3—C7—H7C	109.5	C20—C19—H19	120.3
H7A—C7—H7C	109.5	C21—C20—C19	120.9 (3)
H7B—C7—H7C	109.5	C21—C20—Br1	120.2 (2)
C3—C8—H8A	109.5	C19—C20—Br1	118.9 (2)
C3—C8—H8B	109.5	C20—C21—C22	119.4 (3)
H8A—C8—H8B	109.5	C20—C21—H21	120.3
C3—C8—H8C	109.5	C22—C21—H21	120.3
H8A—C8—H8C	109.5	C21—C22—C17	120.1 (3)
H8B—C8—H8C	109.5	C21—C22—H22	120.0
C6—C9—S2	125.9 (2)	C17—C22—H22	120.0
C17—N1—C1—C6	172.3 (3)	C10—S1—C9—S2	4.9 (2)
C17—N1—C1—C2	-8.4 (4)	C9—S1—C10—C11	174.14 (19)
N1—C1—C2—C3	-152.6 (2)	S1—C10—C11—C16	109.1 (3)
C6—C1—C2—C3	26.7 (4)	S1—C10—C11—C12	-69.9 (3)
C1—C2—C3—C4	-53.7 (3)	C16—C11—C12—C13	1.0 (4)
C1—C2—C3—C7	66.9 (3)	C10—C11—C12—C13	-179.9 (3)
C1—C2—C3—C8	-171.7 (2)	C11—C12—C13—C14	-0.5 (5)
C7—C3—C4—C5	-68.6 (3)	C12—C13—C14—C15	-0.2 (5)
C2—C3—C4—C5	52.0 (3)	C13—C14—C15—C16	0.3 (5)
C8—C3—C4—C5	169.9 (2)	C12—C11—C16—C15	-0.8 (4)
C3—C4—C5—O1	158.1 (3)	C10—C11—C16—C15	-179.9 (3)
C3—C4—C5—C6	-23.4 (4)	C14—C15—C16—C11	0.2 (5)
N1—C1—C6—C9	3.5 (4)	C1—N1—C17—C18	-58.6 (4)
C2—C1—C6—C9	-175.8 (2)	C1—N1—C17—C22	125.4 (3)
N1—C1—C6—C5	-174.7 (2)	C22—C17—C18—C19	0.2 (4)
C2—C1—C6—C5	6.0 (4)	N1—C17—C18—C19	-175.6 (2)
O1—C5—C6—C1	170.7 (3)	C17—C18—C19—C20	0.8 (4)
C4—C5—C6—C1	-7.8 (4)	C18—C19—C20—C21	-1.1 (4)
O1—C5—C6—C9	-7.6 (4)	C18—C19—C20—Br1	176.4 (2)
C4—C5—C6—C9	173.9 (2)	C19—C20—C21—C22	0.3 (4)
C1—C6—C9—S2	3.1 (4)	Br1—C20—C21—C22	-177.1 (2)
C5—C6—C9—S2	-178.7 (2)	C20—C21—C22—C17	0.7 (4)

C1—C6—C9—S1	-179.3 (2)	C18—C17—C22—C21	-1.0 (4)
C5—C6—C9—S1	-1.2 (3)	N1—C17—C22—C21	174.9 (2)
C10—S1—C9—C6	-172.8 (2)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...S2	0.88 (1)	2.10 (2)	2.905 (2)	151 (3)