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1,4-Bis[2-(1,3-benzothiazol-2-yl)-phenoxy]butane

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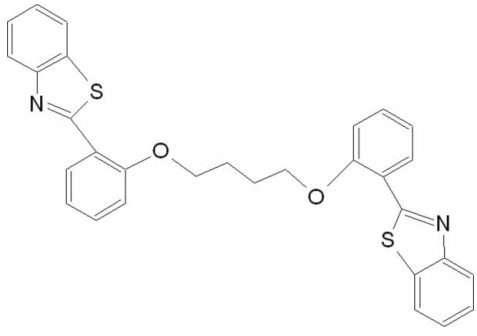
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.069; data-to-parameter ratio = 14.3.

The molecule of the title compound, $C_{30}H_{24}N_2O_2S_2$, adopts a *transoid* conformation consistent with the inversion centre located at the mid-point of the central C—C single bond, resulting in one half molecule in the asymmetric unit. The dihedral angle between the coplanar benzothiazole ring system and the benzene ring is 11.06 (7)°. In the crystal structure, molecules are linked by weak intermolecular π–π interactions between thiazole and benzene rings to form a three-dimensional network.

Related literature

For general background, see: Delmas *et al.* (2002); Karalı *et al.* (2004); Weinstock *et al.* (1987); Chopade *et al.* (2002); Di Nunno *et al.* (2000); Gökhan *et al.* (2004). For related structures, see: Sieroń *et al.* (1999); Usman *et al.* (2003). For related literature, see: Temel *et al.* (2008).



Experimental

Crystal data

$C_{30}H_{24}N_2O_2S_2$
M_r = 508.63
Monoclinic, $P2_1/c$
a = 14.3251 (13) Å
b = 4.8992 (3) Å
c = 17.4954 (17) Å
β = 102.522 (7)°
V = 1198.65 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 296 K
0.80 × 0.36 × 0.08 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.442, T_{max} = 0.936
14397 measured reflections
2339 independent reflections
1456 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.069
S = 0.84
2339 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
The observed π–π interaction distances (Å) for the title compound..

$Cg-Cg^{i}$	$d_{centroids}$	$d_{perpendicular}$
$Cg1-Cg2^{i}$	3.775 (11)	3.515
$Cg1-Cg3^{i}$	3.7934 (12)	3.59

$Cg1$, $Cg2$ and $Cg3$ are the centroids of atoms S1/N1/C1/C6/C7, (C1–C6) and (C8–C13) rings, respectively. Symmetry code: (i) $x, 1+y, z$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2423).

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supporting information

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1,4-Bis[2-(1,3-benzothiazol-2-yl)phenoxy]butane

Orhan Büyükgüngör, Arzu Özek, Senem Karahan and Elif Subaşı

S1. Comment

Benzothiazole derivatives possess a broad spectrum of pharmacological activity, including antibacterial, antifungal (Delmas *et al.*, 2002; Karalı *et al.*, 2004), dopaminergic (Weinstock *et al.*, 1987), anticonvulsant (Chopade *et al.*, 2002), antiadrenergic (Di Nunno *et al.*, 2000) and analgesic anti-inflammatory activities (Gökhan *et al.*, 2004). We report herein the synthesis and structure of the title compound, (I), which is a new benzothiazole derivative.

The molecule of the title compound, (I), (Fig. 1) displays an inversion centre with a half molecule in the asymmetric unit. The benzene ring and its fused thiazole ring are nearly coplanar, with the maximum deviation from the least-squares plane through S1/N1/C1—C7 occurring at S1 [0.033 (9) Å]. However, the molecule itself is nonplanar; the dihedral angle between the coplanar benzothiazole ring system and benzene ring is 11.06 (7)°. The N1—C7 [1.299 (2) Å] bond indicates double-bond character, whereas the S—C bond lengths are indicative of significant single-bond character. The S1—C1 [1.7231 (19) Å] bond is shorter than S1—C7 [1.7552 (18) Å], due to the fact that C7 is sp^2 hybridized, whereas C1 is part of the aromatic ring. A similar effect was observed for *cis*-bis(2-amino-1,3-benzothiazole-N^3)bis- (formato-O,*O′*)copper(II) [(II); Sieroń *et al.*, 1999] and diacetatobis- (2-aminobenzothiazole)zinc(II) [(III); Usman *et al.*, 2003]. The corresponding N—C and S—C values are [N1—C2 = 1.321 (3) Å, S1—C2 = 1.742 (3) Å and S1—C8 = 1.741 (3) Å, in (II)] and [N1—C1 = 1.311 (3) Å, N3—C8 = 1.317 (3) Å, S1—C1 = 1.747 (2) Å, S1—C2 = 1.733 (3) Å, S2—C8 = 1.751 (2) Å and S2—C9 = 1.749 (3) Å, in (III)].

In the crystal structure, the molecules are linked by weak intermolecular $\pi\cdots\pi$ interactions (Table 1) between thiazole and benzene rings to form a three-dimensional network (Fig. 2).

S2. Experimental

The title compound, (I), was prepared by the literature method (Temel *et al.*, 2008). It was obtained from the photochemical reaction of $M(CO)_5$ THF (*M*= Cr) (132 mg, 0.5 mmol) with *N*,*N′*-bis(2-aminothiophenol)-1,4-bis(2-carboxaldehyde- phenoxy) butane (153 mg, 0.3 mmol) in THF for 2 h at room temperature. UV irradiation was performed with a medium-pressure (125 W) mercury lamp through a quartz-walled immersion well reactor, which was cooled by circulating water. After the photochemical reaction, the solvent was removed under vacuum afford a solid residue which was dissolved in CH_2Cl_2 and then petroleum ether was added for the purification process. The solution was allowed to cool in a deep-freezer. Small colorless crystals grown in the CH_2Cl_2 /petroleum ether solution were filtered off and finally dried.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

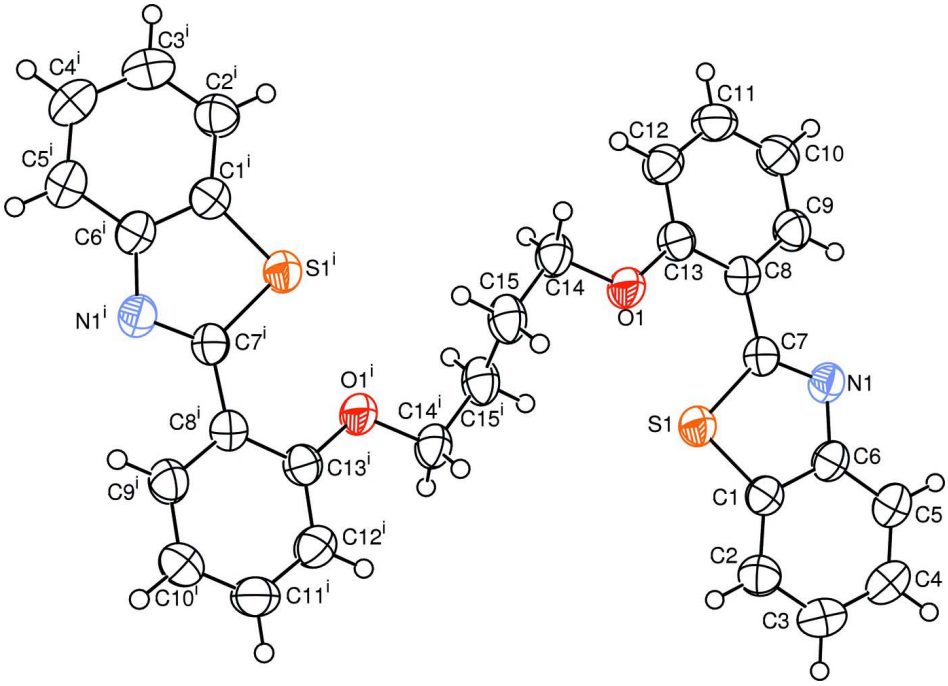

Figure 1

A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry code: (i) -*x*, -*y*, -*z*].

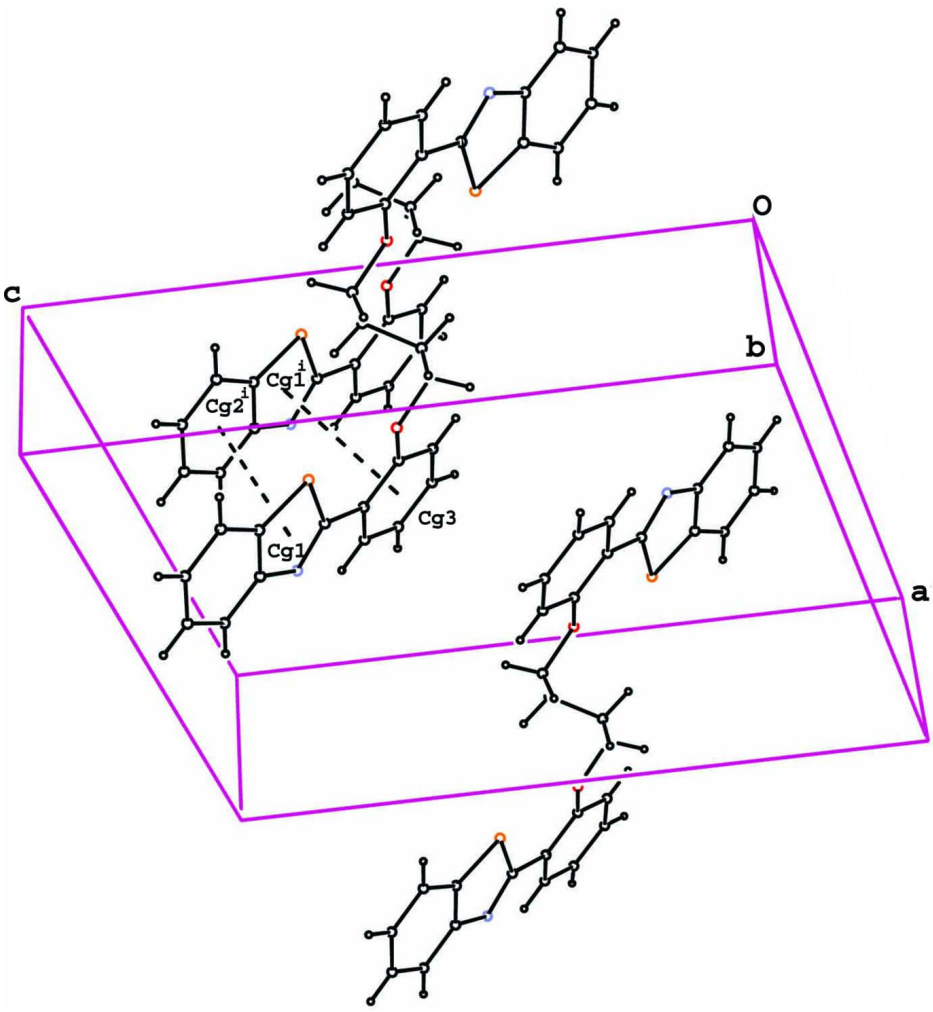

Figure 2

A partial packing diagram of (I), showing the $\pi\cdots\pi$ bonds (drawn as dashed lines) [symmetry code: (i) x, $1 + y$, z].

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Crystal data

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a = 14.3251 (13) Å
b = 4.8992 (3) Å
c = 17.4954 (17) Å
β = 102.522 (7)°
V = 1198.65 (18) Å^3
Z = 2
F(000) = 532
D_x = 1.409 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 14397 reflections
θ = 1.7–28.0°
μ = 0.26 mm^{-1}
T = 296 K
Thin long plate, colorless
0.80 × 0.36 × 0.08 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scans

Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.442, T_{max} = 0.936
14397 measured reflections
2339 independent reflections
1456 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 26.0°, θ_{min} = 2.4°
$h = -16 \rightarrow 17$
$k = -6 \rightarrow 6$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.069
S = 0.84
2339 reflections
163 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Experimental. 322 frames, detector distance = 100 mm

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.15719 (4)	0.95193 (10)	0.66141 (3)	0.05013 (15)
O1	0.18060 (9)	0.6078 (3)	0.54404 (7)	0.0548 (4)
N1	0.33188 (11)	1.0657 (3)	0.72790 (8)	0.0474 (4)
C1	0.17643 (14)	1.1896 (4)	0.73578 (10)	0.0449 (5)
C2	0.11011 (15)	1.3333 (4)	0.76826 (11)	0.0558 (5)
H2	0.0448	1.3057	0.7506	0.067*
C3	0.14437 (17)	1.5159 (4)	0.82685 (12)	0.0610 (6)
H3	0.1015	1.6124	0.8495	0.073*
C4	0.24200 (17)	1.5602 (4)	0.85322 (11)	0.0593 (5)
H4	0.2634	1.6883	0.8923	0.071*
C5	0.30670 (15)	1.4168 (4)	0.82207 (11)	0.0546 (5)
H5	0.3719	1.4453	0.8402	0.066*
C6	0.27417 (14)	1.2284 (4)	0.76311 (10)	0.0454 (5)
C7	0.28134 (13)	0.9108 (4)	0.67424 (10)	0.0420 (4)
C8	0.32640 (13)	0.7109 (4)	0.63100 (10)	0.0421 (4)
C9	0.42400 (14)	0.6654 (4)	0.65603 (11)	0.0524 (5)
H9	0.4582	0.7634	0.6985	0.063*
C10	0.47127 (14)	0.4785 (4)	0.61944 (12)	0.0596 (6)

H10	0.5365	0.4496	0.6375	0.071*
C11	0.42174 (16)	0.3352 (4)	0.55621 (12)	0.0599 (6)
H11	0.4537	0.2111	0.5308	0.072*
C12	0.32498 (16)	0.3741 (4)	0.53009 (11)	0.0545 (5)
H12	0.2917	0.2754	0.4874	0.065*
C13	0.27700 (13)	0.5594 (4)	0.56711 (10)	0.0453 (4)
C14	0.12712 (14)	0.4466 (4)	0.48140 (10)	0.0518 (5)
H14A	0.1321	0.2546	0.4953	0.062*
H14B	0.1517	0.4722	0.4344	0.062*
C15	0.02453 (14)	0.5376 (4)	0.46736 (11)	0.0556 (5)
H15A	−0.0104	0.4565	0.4190	0.067*
H15B	0.0221	0.7342	0.4607	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0431 (3)	0.0516 (3)	0.0532 (3)	−0.0016 (3)	0.0050 (2)	−0.0062 (2)
O1	0.0427 (8)	0.0624 (9)	0.0539 (8)	0.0000 (6)	−0.0010 (6)	−0.0175 (7)
N1	0.0471 (10)	0.0445 (9)	0.0480 (9)	−0.0022 (8)	0.0044 (7)	−0.0040 (8)
C1	0.0494 (13)	0.0390 (11)	0.0462 (11)	−0.0003 (9)	0.0103 (9)	0.0044 (8)
C2	0.0520 (13)	0.0539 (13)	0.0614 (13)	0.0044 (10)	0.0122 (10)	−0.0004 (10)
C3	0.0730 (16)	0.0560 (14)	0.0575 (12)	0.0126 (11)	0.0215 (10)	−0.0008 (10)
C4	0.0809 (17)	0.0459 (11)	0.0496 (11)	0.0013 (12)	0.0104 (11)	−0.0067 (10)
C5	0.0599 (14)	0.0488 (12)	0.0516 (11)	−0.0042 (10)	0.0041 (9)	−0.0043 (10)
C6	0.0525 (13)	0.0387 (11)	0.0426 (10)	−0.0012 (9)	0.0053 (9)	0.0018 (8)
C7	0.0425 (11)	0.0426 (11)	0.0392 (9)	−0.0024 (9)	0.0053 (8)	0.0031 (8)
C8	0.0421 (12)	0.0410 (11)	0.0421 (10)	−0.0013 (9)	0.0065 (8)	0.0019 (8)
C9	0.0444 (12)	0.0537 (12)	0.0557 (12)	−0.0011 (10)	0.0034 (9)	−0.0036 (9)
C10	0.0443 (12)	0.0659 (15)	0.0670 (13)	0.0061 (11)	0.0087 (10)	−0.0047 (11)
C11	0.0583 (15)	0.0610 (14)	0.0635 (13)	0.0095 (11)	0.0197 (11)	−0.0063 (11)
C12	0.0565 (14)	0.0574 (13)	0.0483 (11)	−0.0001 (10)	0.0087 (9)	−0.0087 (9)
C13	0.0425 (12)	0.0474 (11)	0.0446 (10)	0.0007 (9)	0.0060 (8)	0.0021 (9)
C14	0.0518 (12)	0.0559 (12)	0.0440 (10)	−0.0085 (10)	0.0024 (8)	−0.0065 (9)
C15	0.0510 (13)	0.0652 (13)	0.0452 (11)	−0.0105 (10)	−0.0012 (8)	0.0023 (10)

Geometric parameters (Å, º)

C1—S1	1.7231 (19)	C9—C10	1.376 (3)
C1—C2	1.398 (3)	C9—H9	0.9300
C1—C6	1.391 (3)	C10—C11	1.371 (3)
C2—C3	1.369 (3)	C10—H10	0.9300
C2—H2	0.9300	C11—C12	1.375 (3)
C3—C4	1.391 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.382 (3)
C4—C5	1.367 (3)	C12—H12	0.9300
C4—H4	0.9300	C13—O1	1.373 (2)
C5—C6	1.387 (3)	C14—O1	1.431 (2)
C5—H5	0.9300	C14—C15	1.504 (3)

C6—N1	1.385 (2)	C14—H14A	0.9700
C7—S1	1.7552 (18)	C14—H14B	0.9700
C7—N1	1.299 (2)	C15—C15^{i}	1.511 (4)
C7—C8	1.469 (2)	C15—H15A	0.9700
C8—C9	1.390 (3)	C15—H15B	0.9700
C8—C13	1.399 (2)		

C6—C1—C2	120.89 (18)	C11—C10—C9	119.68 (19)
C6—C1—S1	109.65 (14)	C11—C10—H10	120.2
C2—C1—S1	129.46 (16)	C9—C10—H10	120.2
C3—C2—C1	117.9 (2)	C10—C11—C12	120.33 (19)
C3—C2—H2	121.0	C10—C11—H11	119.8
C1—C2—H2	121.0	C12—C11—H11	119.8
C2—C3—C4	121.45 (19)	C11—C12—C13	120.22 (19)
C2—C3—H3	119.3	C11—C12—H12	119.9
C4—C3—H3	119.3	C13—C12—H12	119.9
C5—C4—C3	120.5 (2)	O1—C13—C12	123.13 (17)
C5—C4—H4	119.8	O1—C13—C8	116.46 (16)
C3—C4—H4	119.8	C12—C13—C8	120.41 (18)
C4—C5—C6	119.4 (2)	O1—C14—C15	107.66 (15)
C4—C5—H5	120.3	O1—C14—H14A	110.2
C6—C5—H5	120.3	C15—C14—H14A	110.2
N1—C6—C5	125.23 (18)	O1—C14—H14B	110.2
N1—C6—C1	114.94 (16)	C15—C14—H14B	110.2
C5—C6—C1	119.82 (18)	H14A—C14—H14B	108.5
N1—C7—C8	121.55 (17)	C14—C15—C15^{i}	113.7 (2)
N1—C7—S1	114.57 (13)	C14—C15—H15A	108.8
C8—C7—S1	123.83 (13)	C15^{i}—C15—H15A	108.8
C9—C8—C13	117.81 (17)	C14—C15—H15B	108.8
C9—C8—C7	117.97 (16)	C15^{i}—C15—H15B	108.8
C13—C8—C7	124.20 (17)	H15A—C15—H15B	107.7
C10—C9—C8	121.54 (18)	C7—N1—C6	111.40 (16)
C10—C9—H9	119.2	C13—O1—C14	117.86 (14)
C8—C9—H9	119.2	C1—S1—C7	89.39 (9)

C6—C1—C2—C3	0.9 (3)	C10—C11—C12—C13	0.4 (3)
S1—C1—C2—C3	−179.08 (15)	C11—C12—C13—O1	−179.97 (18)
C1—C2—C3—C4	0.5 (3)	C11—C12—C13—C8	0.6 (3)
C2—C3—C4—C5	−1.4 (3)	C9—C8—C13—O1	179.64 (16)
C3—C4—C5—C6	0.8 (3)	C7—C8—C13—O1	0.9 (3)
C4—C5—C6—N1	−178.41 (18)	C9—C8—C13—C12	−0.9 (3)
C4—C5—C6—C1	0.7 (3)	C7—C8—C13—C12	−179.58 (17)
C2—C1—C6—N1	177.62 (16)	O1—C14—C15—C15^{i}	−69.4 (3)
S1—C1—C6—N1	−2.4 (2)	C8—C7—N1—C6	−177.23 (15)
C2—C1—C6—C5	−1.6 (3)	S1—C7—N1—C6	0.4 (2)
S1—C1—C6—C5	178.43 (14)	C5—C6—N1—C7	−179.52 (17)
N1—C7—C8—C9	8.4 (3)	C1—C6—N1—C7	1.3 (2)
S1—C7—C8—C9	−168.92 (14)	C12—C13—O1—C14	3.6 (3)

N1—C7—C8—C13	−172.87 (18)	C8—C13—O1—C14	−176.95 (16)
S1—C7—C8—C13	9.8 (2)	C15—C14—O1—C13	179.99 (15)
C13—C8—C9—C10	0.2 (3)	C6—C1—S1—C7	2.04 (14)
C7—C8—C9—C10	179.00 (18)	C2—C1—S1—C7	−177.94 (18)
C8—C9—C10—C11	0.8 (3)	N1—C7—S1—C1	−1.44 (14)
C9—C10—C11—C12	−1.1 (3)	C8—C7—S1—C1	176.08 (15)

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