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Methyl 2-bromo-3-(4-chlorobenzenesulfonamido)benzoate

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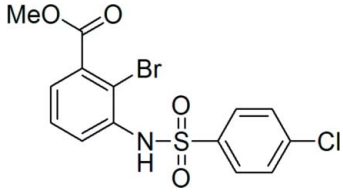
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 14.7.

In the crystal structure of the title compound, $C_{14}H_{11}BrClNO_4S$, the molecules form inversion dimers with $R_2^2(8)$ motifs through pairs of N—H···O hydrogen bonds. The benzene rings are not coplanar and subtend a dihedral angle of 66.27 (8)°. The carbomethoxy group makes a dihedral angle of 75.1 (1)° with the ring to which it is attached.

Related literature

Depending on their substitution patterns, sulfonamides display a wide array of biological activity. For their use as antimitotic, antibacterial and anti-obesity agents, see: Hu *et al.* (2008); Wydysh *et al.* (2009). For structures related to the development of novel antimicrobial agents, see: Kulkarni *et al.* (2012*a*,*b*).



Experimental

Crystal data

$C_{14}H_{11}BrClNO_4S$
M_r = 404.66
Monoclinic, $P2_1/c$
a = 7.9206 (2) Å
b = 9.4600 (3) Å
c = 20.0915 (6) Å
β = 94.505 (3)°
V = 1500.79 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 6.84 mm^{-1}
T = 123 K
1.06 × 0.88 × 0.52 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2010), based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.049, T_{max} = 0.198
5248 measured reflections
3012 independent reflections
2873 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.08
3012 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.80 (4)	2.22 (4)	2.978 (3)	158 (3)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2221).

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supporting information

Acta Cryst. (2013). E**69**, o311 [doi:10.1107/S1600536812048581]

Methyl 2-bromo-3-(4-chlorobenzenesulfonamido)benzoate

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S1. Comment

Depending on their substitution patterns, sulfonamides display a wide array of biological activity. These compounds have been used as antimitotic, antibacterial, anti-obesity agents. See: Hu *et al.* (2008); Wydysh *et al.* (2009). The crystal structure of the title compound has not previously been reported. The title compound was synthesized as an intermediate during our synthetic studies directed towards the development of novel antimicrobial agents (Kulkarni *et al.*, 2012*a*, 2012*b*).

In the title compound ($C_{14}H_{11}Br_1Cl_1N_1O_4S_1$), the molecules form dimers through N—H···O hydrogen bonding to form $R^2_2(8)$ motifs. The two phenyl rings are not coplanar and have a dihedral angle of 66.27 (8)°. The carbomethoxy group makes a dihedral angle of 75.1 (1)° with the ring to which it is attached.

S2. Experimental

A 25 ml one-neck flask equipped with a magnetic stir bar was charged with a solution of the methyl ester (230 mg, 1.0 mmole) in CH_2Cl_2 (4.0 ml). Pyridine (604 ml, 7.5 mmole) was added. The reaction mixture was cooled at 0 °C using ice-bath for 15 minutes. 4-chlorophenylsulfonyl chloride (253 mg, 1.2 mmole) was added to the reaction mixture. The reaction mixture was slowly warmed to RT and was stirred at RT for 4 h. The crude reaction mixture was poured into a separatory funnel containing 1 N HCl (20 ml) and CH_2Cl_2 (20 ml). The layers were separated, the organic layer was washed with water (2X 20 ml) and brine (1X 20 ml). It was dried over anhydr. Na_2SO_4. The solvent was evaporated *in vacuo*. The orange/brown oil thus obtained was purified using silica gel flash column chromatography. Elution with 40% EA in hexanes afforded the desired sulfonamide product as pale yellow crystals. mp 121–124 °C; ^{1}H-NMR ($CDCl_3$) d 7.80 (dd, *J* = 8.0, 2.0 Hz, 1H), 7. 68 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.51 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.41–7.31 (m, 4H), 3.86 (s, 3H). ^{13}C-NMR ($CDCl_3$) d 165.9, 139.9, 137.1, 135.5, 133.4, 129.4, 128.6, 128.0, 127.7, 125.4, 115.1, 52.6.

S3. Refinement

The amine H atom was seen in a difference Fourier map and refined isotropically with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H-atoms were positioned geometrically with C—H = 0.95 and 0.98 Å, for aromatic and CH_3 H-atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) [U_{iso}(H) = 1.5U_{eq}(C) for CH_3].

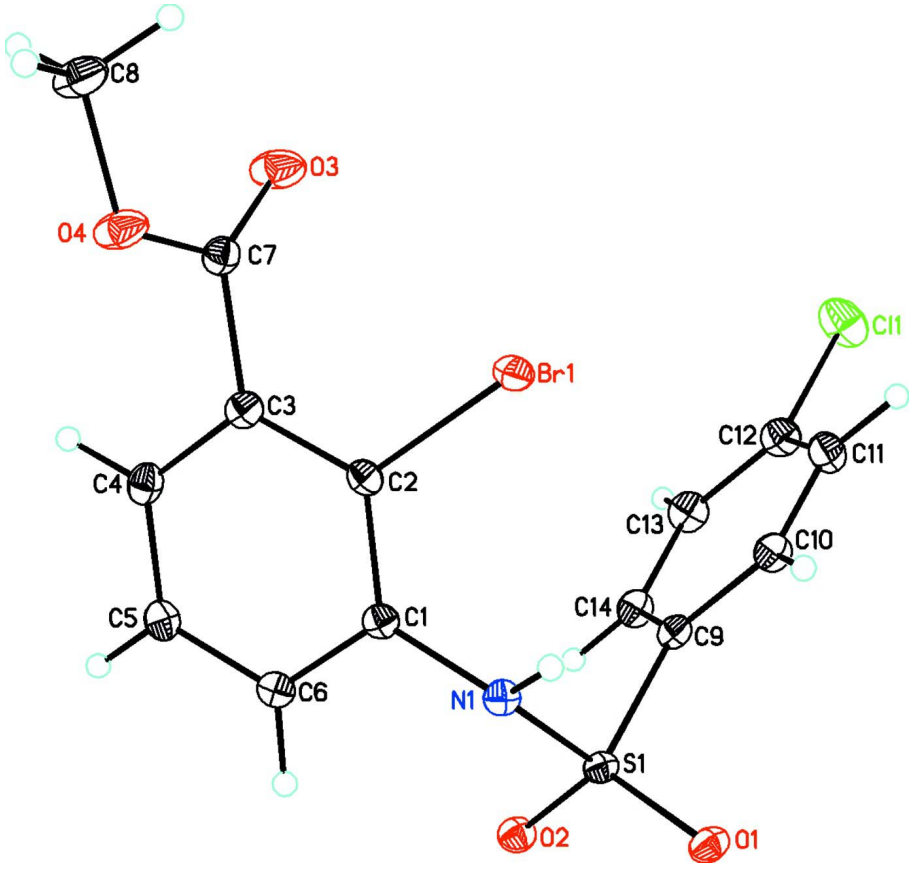

Figure 1

View of the molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-hydrogen atoms.

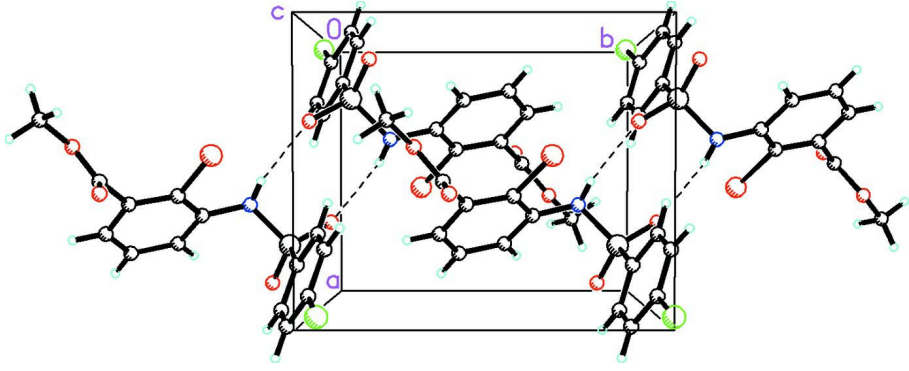

Figure 2

The packing view showing the hydrogen bonds network. Dashed lines indicate intermolecular N—H···O hydrogen bonds (see Table 1 for details).

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Crystal data

$C_{14}H_{11}BrClNO_4S$
$M_r = 404.66$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.9206$ (2) Å
$b = 9.4600$ (3) Å
$c = 20.0915$ (6) Å
$\beta = 94.505$ (3)°
$V = 1500.79$ (8) Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.791$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3046 reflections
$\theta = 4.4$–$75.7°$
$\mu = 6.84$ mm^{-1}
$T = 123$ K
Chunk, colorless
1.06 × 0.88 × 0.52 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2010), based on expressions derived by Clark & Reid (1995)]
$T_{min} = 0.049$, $T_{max} = 0.198$
5248 measured reflections
3012 independent reflections
2873 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 75.9°$, $\theta_{min} = 4.4°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 7$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.093$
$S = 1.08$
3012 reflections
205 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.5274P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0021 (3)

Special details

Experimental. CrysAlisPro (Agilent, 2010) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by R.C. Clark & J.S. Reid. (Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897)

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	0.55830 (3)	0.29987 (2)	0.362077 (11)	0.02554 (13)
Cl1	0.01337 (10)	−0.03081 (9)	0.18862 (3)	0.0433 (2)
S1	0.23124 (6)	0.09272 (5)	0.48834 (2)	0.01812 (15)
O1	0.3147 (2)	−0.03081 (17)	0.51690 (8)	0.0235 (3)
O2	0.0901 (2)	0.15202 (17)	0.51868 (8)	0.0230 (3)
O3	0.4253 (3)	0.6005 (2)	0.27820 (9)	0.0397 (5)
O4	0.5888 (3)	0.7061 (2)	0.35913 (9)	0.0352 (5)
N1	0.3799 (3)	0.2130 (2)	0.48786 (10)	0.0193 (4)
H1A	0.466 (5)	0.177 (3)	0.4775 (17)	0.027 (8)*
C1	0.3423 (3)	0.3532 (2)	0.46643 (10)	0.0185 (4)
C2	0.4146 (3)	0.4121 (2)	0.41128 (10)	0.0186 (4)
C3	0.3853 (3)	0.5527 (2)	0.39355 (11)	0.0206 (4)
C4	0.2772 (3)	0.6340 (2)	0.42946 (12)	0.0240 (4)
H4A	0.2549	0.7294	0.4170	0.029*
C5	0.2022 (3)	0.5763 (2)	0.48325 (12)	0.0229 (4)
H5A	0.1269	0.6315	0.5070	0.027*
C6	0.2372 (3)	0.4374 (3)	0.50239 (11)	0.0208 (4)
H6A	0.1890	0.3995	0.5404	0.025*
C7	0.4666 (3)	0.6198 (2)	0.33630 (11)	0.0219 (4)
C8	0.6814 (4)	0.7813 (3)	0.31100 (14)	0.0354 (6)
H8A	0.7891	0.8150	0.3326	0.053*
H8B	0.6143	0.8621	0.2936	0.053*
H8C	0.7035	0.7177	0.2742	0.053*
C9	0.1676 (3)	0.0580 (2)	0.40362 (11)	0.0203 (4)
C10	0.2808 (3)	−0.0105 (2)	0.36475 (12)	0.0233 (4)
H10A	0.3899	−0.0373	0.3835	0.028*
C11	0.2321 (3)	−0.0390 (3)	0.29849 (12)	0.0277 (5)
H11A	0.3065	−0.0871	0.2714	0.033*
C12	0.0727 (3)	0.0038 (3)	0.27216 (12)	0.0278 (5)
C13	−0.0388 (3)	0.0736 (3)	0.31041 (13)	0.0279 (5)
H13A	−0.1467	0.1028	0.2913	0.034*
C14	0.0093 (3)	0.1003 (2)	0.37711 (12)	0.0243 (5)
H14A	−0.0659	0.1473	0.4043	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03114 (18)	0.02542 (18)	0.02113 (17)	0.00412 (8)	0.00890 (10)	0.00167 (8)
Cl1	0.0431 (4)	0.0583 (4)	0.0263 (3)	0.0072 (3)	−0.0109 (3)	−0.0104 (3)
S1	0.0171 (3)	0.0196 (3)	0.0177 (3)	0.00109 (18)	0.00194 (18)	0.00302 (17)
O1	0.0231 (8)	0.0229 (8)	0.0248 (8)	0.0032 (6)	0.0033 (6)	0.0067 (6)
O2	0.0198 (7)	0.0256 (8)	0.0242 (8)	0.0008 (6)	0.0048 (6)	0.0020 (6)
O3	0.0456 (11)	0.0525 (12)	0.0200 (8)	−0.0195 (10)	−0.0034 (8)	0.0039 (8)
O4	0.0380 (10)	0.0442 (12)	0.0223 (9)	−0.0218 (9)	−0.0036 (7)	0.0062 (7)
N1	0.0168 (9)	0.0212 (9)	0.0197 (9)	0.0014 (7)	0.0005 (7)	0.0013 (7)

C1	0.0165 (9)	0.0217 (10)	0.0165 (9)	−0.0002 (8)	−0.0034 (7)	0.0007 (8)
C2	0.0168 (9)	0.0219 (10)	0.0167 (9)	0.0008 (8)	−0.0012 (7)	−0.0017 (8)
C3	0.0189 (10)	0.0230 (10)	0.0191 (10)	−0.0012 (8)	−0.0034 (8)	0.0013 (8)
C4	0.0238 (11)	0.0201 (10)	0.0271 (11)	0.0016 (8)	−0.0042 (9)	0.0010 (8)
C5	0.0192 (10)	0.0237 (11)	0.0257 (11)	0.0024 (8)	0.0008 (8)	−0.0030 (8)
C6	0.0175 (10)	0.0248 (10)	0.0199 (10)	0.0000 (8)	0.0003 (8)	0.0000 (8)
C7	0.0226 (10)	0.0212 (10)	0.0215 (10)	0.0010 (8)	−0.0015 (8)	0.0020 (8)
C8	0.0363 (14)	0.0401 (14)	0.0296 (13)	−0.0140 (11)	0.0019 (11)	0.0088 (11)
C9	0.0208 (10)	0.0186 (10)	0.0215 (10)	−0.0017 (8)	0.0005 (8)	0.0024 (8)
C10	0.0207 (10)	0.0244 (10)	0.0243 (11)	0.0018 (8)	−0.0008 (8)	0.0013 (8)
C11	0.0286 (12)	0.0285 (11)	0.0258 (12)	0.0039 (10)	0.0019 (9)	−0.0033 (9)
C12	0.0281 (12)	0.0307 (12)	0.0238 (11)	−0.0029 (9)	−0.0042 (9)	−0.0002 (9)
C13	0.0214 (11)	0.0315 (12)	0.0297 (12)	0.0002 (9)	−0.0054 (9)	0.0010 (9)
C14	0.0206 (10)	0.0244 (11)	0.0276 (11)	0.0017 (8)	−0.0001 (9)	0.0014 (9)

Geometric parameters (Å, º)

Br1—C2	1.892 (2)	C4—H4A	0.9500
Cl1—C12	1.738 (3)	C5—C6	1.391 (3)
S1—O2	1.4294 (16)	C5—H5A	0.9500
S1—O1	1.4397 (16)	C6—H6A	0.9500
S1—N1	1.638 (2)	C8—H8A	0.9800
S1—C9	1.767 (2)	C8—H8B	0.9800
O3—C7	1.201 (3)	C8—H8C	0.9800
O3—Br1^{i}	3.4022 (19)	C9—C14	1.383 (3)
O4—C7	1.320 (3)	C9—C10	1.394 (3)
O4—C8	1.446 (3)	C10—C11	1.383 (3)
N1—C1	1.418 (3)	C10—H10A	0.9500
N1—H1A	0.80 (4)	C11—C12	1.390 (4)
C1—C6	1.394 (3)	C11—H11A	0.9500
C1—C2	1.402 (3)	C12—C13	1.383 (4)
C2—C3	1.392 (3)	C13—C14	1.387 (4)
C3—C4	1.393 (3)	C13—H13A	0.9500
C3—C7	1.502 (3)	C14—H14A	0.9500
C4—C5	1.386 (3)		
O2—S1—O1	119.91 (10)	C1—C6—H6A	119.7
O2—S1—N1	108.45 (10)	O3—C7—O4	124.6 (2)
O1—S1—N1	104.97 (10)	O3—C7—C3	125.4 (2)
O2—S1—C9	108.08 (10)	O4—C7—C3	109.98 (19)
O1—S1—C9	108.68 (10)	O4—C8—H8A	109.5
N1—S1—C9	105.91 (10)	O4—C8—H8B	109.5
C7—O3—Br1^{i}	134.06 (17)	H8A—C8—H8B	109.5
C7—O4—C8	117.9 (2)	O4—C8—H8C	109.5
C1—N1—S1	121.14 (16)	H8A—C8—H8C	109.5
C1—N1—H1A	118 (2)	H8B—C8—H8C	109.5
S1—N1—H1A	110 (2)	C14—C9—C10	121.5 (2)
C6—C1—C2	118.7 (2)	C14—C9—S1	119.90 (18)

C6—C1—N1	119.76 (19)	C10—C9—S1	118.60 (17)
C2—C1—N1	121.51 (19)	C11—C10—C9	119.1 (2)
C3—C2—C1	120.81 (19)	C11—C10—H10A	120.4
C3—C2—Br1	119.86 (16)	C9—C10—H10A	120.4
C1—C2—Br1	119.31 (16)	C10—C11—C12	119.1 (2)
C2—C3—C4	119.4 (2)	C10—C11—H11A	120.5
C2—C3—C7	121.8 (2)	C12—C11—H11A	120.5
C4—C3—C7	118.8 (2)	C13—C12—C11	121.9 (2)
C5—C4—C3	120.3 (2)	C13—C12—Cl1	119.4 (2)
C5—C4—H4A	119.8	C11—C12—Cl1	118.8 (2)
C3—C4—H4A	119.8	C12—C13—C14	119.0 (2)
C4—C5—C6	120.0 (2)	C12—C13—H13A	120.5
C4—C5—H5A	120.0	C14—C13—H13A	120.5
C6—C5—H5A	120.0	C9—C14—C13	119.4 (2)
C5—C6—C1	120.7 (2)	C9—C14—H14A	120.3
C5—C6—H6A	119.7	C13—C14—H14A	120.3
O2—S1—N1—C1	−47.0 (2)	C8—O4—C7—C3	−179.6 (2)
O1—S1—N1—C1	−176.30 (17)	C2—C3—C7—O3	75.4 (3)
C9—S1—N1—C1	68.80 (19)	C4—C3—C7—O3	−104.3 (3)
S1—N1—C1—C6	64.4 (3)	C2—C3—C7—O4	−105.9 (3)
S1—N1—C1—C2	−118.6 (2)	C4—C3—C7—O4	74.3 (3)
C6—C1—C2—C3	1.4 (3)	O2—S1—C9—C14	5.6 (2)
N1—C1—C2—C3	−175.7 (2)	O1—S1—C9—C14	137.26 (19)
C6—C1—C2—Br1	179.72 (16)	N1—S1—C9—C14	−110.4 (2)
N1—C1—C2—Br1	2.6 (3)	O2—S1—C9—C10	−175.16 (17)
C1—C2—C3—C4	−2.6 (3)	O1—S1—C9—C10	−43.5 (2)
Br1—C2—C3—C4	179.04 (17)	N1—S1—C9—C10	68.8 (2)
C1—C2—C3—C7	177.6 (2)	C14—C9—C10—C11	−1.2 (4)
Br1—C2—C3—C7	−0.7 (3)	S1—C9—C10—C11	179.62 (18)
C2—C3—C4—C5	1.3 (3)	C9—C10—C11—C12	1.2 (4)
C7—C3—C4—C5	−178.9 (2)	C10—C11—C12—C13	−0.3 (4)
C3—C4—C5—C6	1.2 (3)	C10—C11—C12—Cl1	179.35 (19)
C4—C5—C6—C1	−2.5 (3)	C11—C12—C13—C14	−0.6 (4)
C2—C1—C6—C5	1.2 (3)	Cl1—C12—C13—C14	179.75 (19)
N1—C1—C6—C5	178.3 (2)	C10—C9—C14—C13	0.3 (4)
Br1^{i}—O3—C7—O4	−27.5 (4)	S1—C9—C14—C13	179.46 (18)
Br1^{i}—O3—C7—C3	150.97 (18)	C12—C13—C14—C9	0.6 (4)
C8—O4—C7—O3	−1.0 (4)		

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{ii}	0.80 (4)	2.22 (4)	2.978 (3)	158 (3)

Symmetry code: (ii) −*x*+1, −*y*, −*z*+1.