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N,*N*-Bis(5-bromo-2-thienylsulfonyl)-3-nitroaniline

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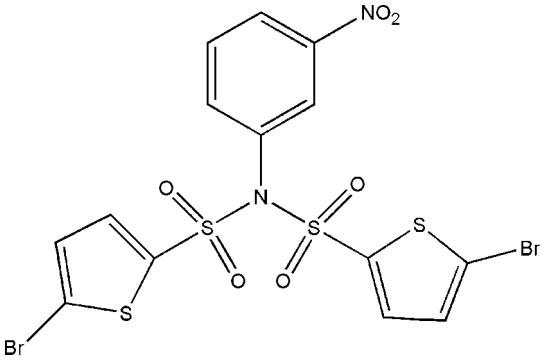
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.036; wR factor = 0.095; data-to-parameter ratio = 13.6.

In the title compound, $C_{14}H_8Br_2N_2O_6S_4$, all bond lengths and angles are within normal ranges. The benzene ring and the two thiophene rings make dihedral angles of 35.97 (15) and 37.92 (19)°, and the dihedral angle between the two thiophene rings is 62.47 (14)°.

Related literature

For related literature, see Allen *et al.* (1987); Gayathri *et al.* (2006); Krishnaiah *et al.* (1995); Yan *et al.* (2007); Yu (2006).



Experimental

Crystal data

$C_{14}H_8Br_2N_2O_6S_4$
M_r = 588.30
Triclinic, $P\overline{1}$
a = 8.5147 (7) Å
b = 9.3674 (7) Å
c = 13.7585 (11) Å
α = 103.109 (1)°
β = 100.810 (1)°
γ = 105.155 (1)°
V = 995.42 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.53 mm^{-1}
T = 298 (2) K
0.29 × 0.26 × 0.21 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.294, T_{max} = 0.392
5264 measured reflections
3451 independent reflections
2895 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.095
S = 1.02
3451 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2106).

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). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Gayathri, D., Velmurugan, D., Ravikumar, K., Poornachandran, M. & Raghunathan, R. (2006). *Acta Cryst.* E**62**, o4454–o4455.
Krishnaiah, M., Narayana Raju, K. V., Lu, I.-L., Chen, Y.-S. & Narasinga Rao, S. (1995). *Acta Cryst.* C**51**, 2429–2430.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yan, X.-W., Hu, M.-L. & Xiong, J. (2007). *Acta Cryst.* E**63**, o678–o679.
Yu, Y.-Y. (2006). *Acta Cryst.* E**62**, o2308–o2309.

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supplementary materials

Acta Cryst. (2007). E**63**, o3177 [**doi:10.1107/S1600536807024683**]

N,*N*-Bis(5-bromo-2-thienylsulfonyl)-3-nitroaniline

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Comment

Some sulfonamide compounds exhibit germicidal activities (Gayathri *et al.*, 2006; Krishnaiah *et al.*, 1995; Yu, 2006). Some crystal structures involving sulfonamide groups have been published, including a recent report from our laboratory (Yan *et al.*, 2007). As an extension of this research, we report here the synthesis and crystal structure of the title compound (I).

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987) and are unremarkable when compared with those found in our previous report (Yan *et al.*, 2007). The benzene ring (C5—C10) and the two thiophene groups (C1—C4/S1, C11—C14/S4) are essentially planar with r. m. s. deviations of 0.0025 Å, 0.0032 Å and 0.0036 Å each. The benzene ring and the two thiophene rings (C1—C4/S1, C11—C14/S4) make dihedral angles of 35.97 (15)° and 37.92 (19)°, respectively. The dihedral angle between the two thiophene rings is 62.47 (14)°.

Experimental

5-Bromothiophene-2-sulfonyl chloride (5 mmol, 1.304) in ethyl acetate (20 ml) was added dropwise to 3-nitroaniline (5 mmol, 1.38 g) in acetone (20 ml) at room temperature. The pure solid product was obtained after 24 h reaction and column chromatographic separation. Single crystals were obtained from 95% ethanol after 10 days.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å with $U_{iso} = 1.2U_{eq}$(parent atom).

Figures

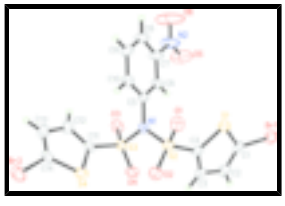


Fig. 1. Molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

N,*N*-Bis(5-bromo-2-thienylsulfonyl)-3-nitroaniline

Crystal data

$C_{14}H_8Br_2N_2O_6S_4$	$Z = 2$
$M_r = 588.30$	$F_{000} = 576$
Triclinic, $P\overline{1}$	$D_x = 1.963$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.5147 (7) Å	Cell parameters from 2347 reflections
b = 9.3674 (7) Å	θ = 2.4–25.0º
c = 13.7585 (11) Å	μ = 4.53 mm^{-1}
α = 103.109 (1)º	T = 298 (2) K
β = 100.810 (1)º	Block, colorless
γ = 105.155 (1)º	0.29 × 0.26 × 0.21 mm
V = 995.42 (14) Å^3	

Data collection

Bruker APEX area-detector diffractometer	3451 independent reflections
Radiation source: fine-focus sealed tube	2895 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.013
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	h = −8→10
T_{min} = 0.294, T_{max} = 0.392	k = −11→10
5264 measured reflections	l = −16→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.036	H-atom parameters constrained
$wR(F^2)$ = 0.095	$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 1.0353P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
3451 reflections	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
253 parameters	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.80658 (6)	0.57752 (6)	−0.01657 (3)	0.06367 (16)
Br2	0.16643 (7)	0.79432 (6)	0.73552 (3)	0.06776 (17)
S1	0.70495 (14)	0.75108 (11)	0.16962 (8)	0.0508 (3)
S2	0.56667 (11)	0.75343 (11)	0.35446 (7)	0.0415 (2)
S3	0.19996 (11)	0.59462 (10)	0.30359 (7)	0.0402 (2)
S4	0.24634 (15)	0.65019 (13)	0.53339 (8)	0.0549 (3)
O1	0.6610 (3)	0.9124 (3)	0.3806 (2)	0.0577 (7)
O2	0.5703 (4)	0.6730 (3)	0.4300 (2)	0.0583 (7)
O3	0.0704 (3)	0.5875 (3)	0.2195 (2)	0.0554 (7)
O4	0.2608 (4)	0.4679 (3)	0.3058 (2)	0.0553 (7)
O5	0.2349 (8)	0.8484 (5)	−0.0312 (3)	0.141 (2)
O6	0.2078 (12)	1.0655 (7)	−0.0019 (4)	0.220 (4)
N1	0.3660 (4)	0.7446 (3)	0.3097 (2)	0.0377 (6)
N2	0.2335 (8)	0.9620 (5)	0.0268 (3)	0.1021 (18)
C1	0.7121 (5)	0.5809 (5)	0.0948 (3)	0.0462 (9)
C2	0.6533 (5)	0.4597 (5)	0.1300 (3)	0.0544 (10)
H2	0.6492	0.3593	0.0987	0.065*
C3	0.5988 (5)	0.5025 (5)	0.2192 (3)	0.0521 (10)
H3	0.5536	0.4337	0.2536	0.062*
C4	0.6197 (4)	0.6552 (4)	0.2491 (3)	0.0412 (8)
C5	0.3332 (4)	0.8701 (4)	0.2738 (3)	0.0389 (8)
C6	0.3002 (5)	0.8562 (4)	0.1699 (3)	0.0489 (9)
H6	0.3000	0.7679	0.1224	0.059*
C7	0.2675 (7)	0.9768 (5)	0.1384 (3)	0.0620 (12)
C8	0.2669 (6)	1.1098 (5)	0.2055 (3)	0.0618 (12)
H8	0.2449	1.1897	0.1815	0.074*
C9	0.2998 (6)	1.1209 (5)	0.3093 (3)	0.0565 (11)
H9	0.2993	1.2093	0.3564	0.068*
C10	0.3337 (5)	1.0025 (4)	0.3444 (3)	0.0472 (9)
H10	0.3566	1.0112	0.4147	0.057*
C11	0.1428 (4)	0.6570 (4)	0.4158 (3)	0.0408 (8)
C12	0.0271 (5)	0.7303 (5)	0.4243 (3)	0.0580 (11)
H12	−0.0394	0.7456	0.3683	0.070*
C13	0.0170 (5)	0.7811 (6)	0.5264 (3)	0.0619 (11)
H13	−0.0566	0.8329	0.5460	0.074*
C14	0.1281 (5)	0.7448 (4)	0.5924 (3)	0.0482 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0770 (3)	0.0778 (3)	0.0497 (3)	0.0413 (3)	0.0284 (2)	0.0152 (2)
Br2	0.0943 (4)	0.0683 (3)	0.0411 (2)	0.0204 (3)	0.0258 (2)	0.0162 (2)
S1	0.0673 (7)	0.0480 (5)	0.0455 (6)	0.0249 (5)	0.0244 (5)	0.0139 (4)
S2	0.0417 (5)	0.0467 (5)	0.0323 (5)	0.0134 (4)	0.0067 (4)	0.0076 (4)

S3	0.0446 (5)	0.0374 (5)	0.0342 (5)	0.0087 (4)	0.0082 (4)	0.0090 (4)
S4	0.0752 (7)	0.0623 (6)	0.0394 (5)	0.0343 (6)	0.0164 (5)	0.0224 (5)
O1	0.0503 (16)	0.0471 (15)	0.0572 (18)	0.0034 (13)	0.0109 (13)	−0.0036 (13)
O2	0.0656 (18)	0.0781 (19)	0.0389 (15)	0.0320 (15)	0.0112 (13)	0.0234 (14)
O3	0.0511 (16)	0.0652 (18)	0.0363 (14)	0.0059 (13)	0.0010 (12)	0.0120 (13)
O4	0.0740 (19)	0.0354 (13)	0.0612 (18)	0.0190 (13)	0.0255 (15)	0.0147 (12)
O5	0.312 (7)	0.120 (3)	0.044 (2)	0.141 (4)	0.060 (3)	0.034 (2)
O6	0.531 (14)	0.148 (5)	0.076 (3)	0.213 (7)	0.104 (6)	0.073 (3)
N1	0.0404 (16)	0.0373 (15)	0.0401 (16)	0.0140 (13)	0.0133 (13)	0.0159 (13)
N2	0.213 (6)	0.078 (3)	0.056 (3)	0.085 (4)	0.053 (3)	0.041 (2)
C1	0.045 (2)	0.056 (2)	0.040 (2)	0.0260 (18)	0.0100 (16)	0.0075 (17)
C2	0.062 (3)	0.049 (2)	0.057 (3)	0.028 (2)	0.019 (2)	0.0085 (19)
C3	0.053 (2)	0.049 (2)	0.063 (3)	0.0225 (18)	0.020 (2)	0.0208 (19)
C4	0.0401 (19)	0.047 (2)	0.0367 (19)	0.0181 (16)	0.0074 (15)	0.0088 (16)
C5	0.046 (2)	0.0353 (18)	0.0369 (19)	0.0133 (15)	0.0129 (15)	0.0119 (15)
C6	0.079 (3)	0.0369 (19)	0.038 (2)	0.0264 (19)	0.0231 (19)	0.0102 (16)
C7	0.108 (4)	0.050 (2)	0.041 (2)	0.037 (2)	0.024 (2)	0.0197 (19)
C8	0.102 (4)	0.043 (2)	0.052 (3)	0.037 (2)	0.024 (2)	0.0185 (19)
C9	0.084 (3)	0.044 (2)	0.049 (2)	0.034 (2)	0.025 (2)	0.0085 (18)
C10	0.061 (2)	0.047 (2)	0.0336 (19)	0.0197 (18)	0.0144 (17)	0.0056 (16)
C11	0.0413 (19)	0.0407 (19)	0.0367 (19)	0.0057 (15)	0.0104 (15)	0.0126 (15)
C12	0.041 (2)	0.083 (3)	0.051 (2)	0.023 (2)	0.0114 (18)	0.021 (2)
C13	0.052 (2)	0.086 (3)	0.052 (3)	0.028 (2)	0.022 (2)	0.015 (2)
C14	0.057 (2)	0.041 (2)	0.042 (2)	0.0040 (17)	0.0207 (18)	0.0107 (17)

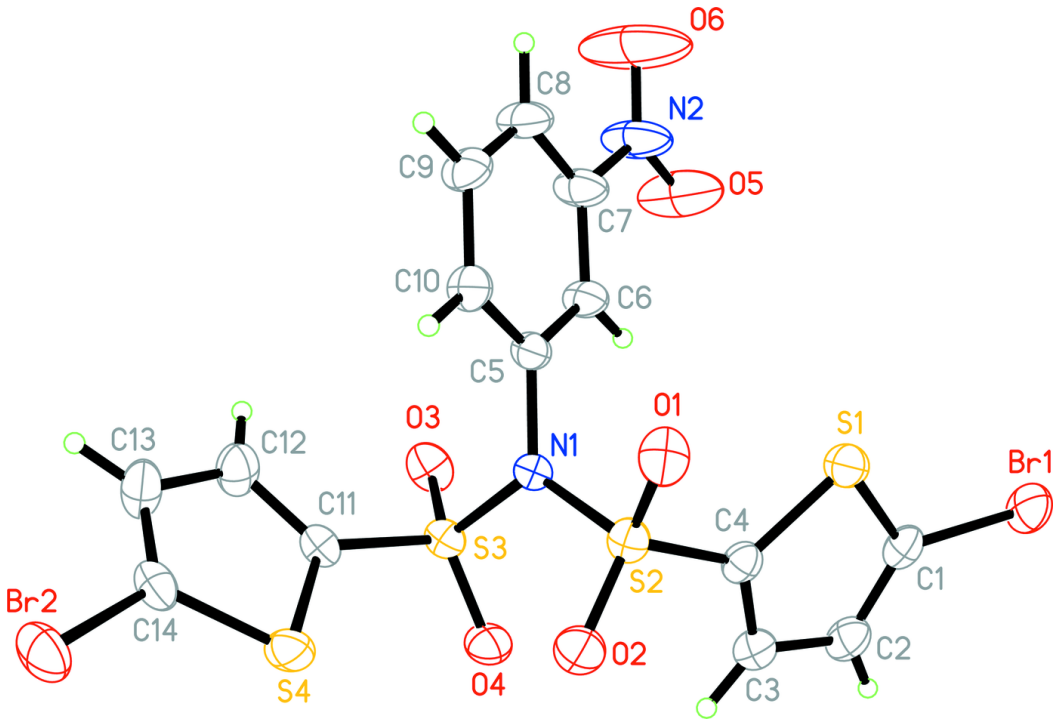
Geometric parameters (Å, °)

Br1—C1	1.857 (4)	C2—C3	1.404 (6)
Br2—C14	1.861 (4)	C2—H2	0.930
S1—C4	1.713 (4)	C3—C4	1.349 (5)
S1—C1	1.713 (4)	C3—H3	0.930
S2—O2	1.415 (3)	C5—C6	1.374 (5)
S2—O1	1.420 (3)	C5—C10	1.389 (5)
S2—N1	1.677 (3)	C6—C7	1.372 (5)
S2—C4	1.735 (4)	C6—H6	0.930
S3—O3	1.417 (3)	C7—C8	1.374 (6)
S3—O4	1.417 (3)	C8—C9	1.378 (6)
S3—N1	1.686 (3)	C8—H8	0.930
S3—C11	1.727 (4)	C9—C10	1.380 (5)
S4—C14	1.704 (4)	C9—H9	0.930
S4—C11	1.715 (4)	C10—H10	0.930
O5—N2	1.181 (5)	C11—C12	1.348 (5)
O6—N2	1.182 (5)	C12—C13	1.404 (6)
N1—C5	1.444 (4)	C12—H12	0.930
N2—C7	1.475 (6)	C13—C14	1.349 (6)
C1—C2	1.348 (6)	C13—H13	0.930
C4—S1—C1	89.88 (19)	S1—C4—S2	120.6 (2)
O2—S2—O1	120.68 (18)	C6—C5—C10	120.7 (3)
O2—S2—N1	107.56 (16)	C6—C5—N1	119.4 (3)

O1—S2—N1	104.87 (16)	C10—C5—N1	119.9 (3)
O2—S2—C4	109.33 (18)	C7—C6—C5	117.9 (3)
O1—S2—C4	107.98 (18)	C7—C6—H6	121.1
N1—S2—C4	105.36 (16)	C5—C6—H6	121.1
O3—S3—O4	121.60 (18)	C6—C7—C8	123.3 (4)
O3—S3—N1	105.18 (16)	C6—C7—N2	117.7 (4)
O4—S3—N1	106.70 (16)	C8—C7—N2	119.0 (4)
O3—S3—C11	107.58 (18)	C7—C8—C9	117.8 (4)
O4—S3—C11	110.68 (17)	C7—C8—H8	121.1
N1—S3—C11	103.50 (16)	C9—C8—H8	121.1
C14—S4—C11	90.14 (19)	C8—C9—C10	120.8 (3)
C5—N1—S2	118.8 (2)	C8—C9—H9	119.6
C5—N1—S3	118.3 (2)	C10—C9—H9	119.6
S2—N1—S3	122.91 (17)	C9—C10—C5	119.5 (4)
O5—N2—O6	121.7 (5)	C9—C10—H10	120.3
O5—N2—C7	119.4 (4)	C5—C10—H10	120.3
O6—N2—C7	118.9 (4)	C12—C11—S4	112.0 (3)
C2—C1—S1	112.9 (3)	C12—C11—S3	125.2 (3)
C2—C1—Br1	126.3 (3)	S4—C11—S3	122.5 (2)
S1—C1—Br1	120.7 (2)	C11—C12—C13	113.2 (4)
C1—C2—C3	112.2 (4)	C11—C12—H12	123.4
C1—C2—H2	123.9	C13—C12—H12	123.4
C3—C2—H2	123.9	C14—C13—C12	111.1 (4)
C4—C3—C2	112.0 (4)	C14—C13—H13	124.4
C4—C3—H3	124.0	C12—C13—H13	124.4
C2—C3—H3	124.0	C13—C14—S4	113.5 (3)
C3—C4—S1	113.0 (3)	C13—C14—Br2	126.8 (3)
C3—C4—S2	126.4 (3)	S4—C14—Br2	119.7 (2)
O2—S2—N1—C5	149.9 (3)	S3—N1—C5—C10	97.3 (4)
O1—S2—N1—C5	20.3 (3)	C10—C5—C6—C7	−0.1 (6)
C4—S2—N1—C5	−93.5 (3)	N1—C5—C6—C7	179.2 (4)
O2—S2—N1—S3	−30.6 (3)	C5—C6—C7—C8	0.2 (7)
O1—S2—N1—S3	−160.2 (2)	C5—C6—C7—N2	179.5 (5)
C4—S2—N1—S3	86.0 (2)	O5—N2—C7—C6	0.2 (9)
O3—S3—N1—C5	28.7 (3)	O6—N2—C7—C6	−178.8 (7)
O4—S3—N1—C5	159.1 (3)	O5—N2—C7—C8	179.5 (6)
C11—S3—N1—C5	−84.1 (3)	O6—N2—C7—C8	0.5 (10)
O3—S3—N1—S2	−150.8 (2)	C6—C7—C8—C9	−0.4 (8)
O4—S3—N1—S2	−20.4 (3)	N2—C7—C8—C9	−179.7 (5)
C11—S3—N1—S2	96.4 (2)	C7—C8—C9—C10	0.5 (7)
C4—S1—C1—C2	−0.3 (3)	C8—C9—C10—C5	−0.4 (7)
C4—S1—C1—Br1	−176.5 (2)	C6—C5—C10—C9	0.2 (6)
S1—C1—C2—C3	0.6 (5)	N1—C5—C10—C9	−179.1 (4)
Br1—C1—C2—C3	176.5 (3)	C14—S4—C11—C12	0.9 (3)
C1—C2—C3—C4	−0.6 (5)	C14—S4—C11—S3	174.4 (2)
C2—C3—C4—S1	0.3 (4)	O3—S3—C11—C12	−18.7 (4)
C2—C3—C4—S2	179.4 (3)	O4—S3—C11—C12	−153.7 (3)
C1—S1—C4—C3	0.0 (3)	N1—S3—C11—C12	92.3 (4)
C1—S1—C4—S2	−179.1 (2)	O3—S3—C11—S4	168.6 (2)

O2—S2—C4—C3	26.3 (4)	O4—S3—C11—S4	33.6 (3)
O1—S2—C4—C3	159.3 (3)	N1—S3—C11—S4	−80.4 (2)
N1—S2—C4—C3	−89.1 (4)	S4—C11—C12—C13	−0.9 (5)
O2—S2—C4—S1	−154.7 (2)	S3—C11—C12—C13	−174.2 (3)
O1—S2—C4—S1	−21.7 (3)	C11—C12—C13—C14	0.5 (6)
N1—S2—C4—S1	90.0 (2)	C12—C13—C14—S4	0.2 (5)
S2—N1—C5—C6	97.4 (4)	C12—C13—C14—Br2	177.6 (3)
S3—N1—C5—C6	−82.1 (4)	C11—S4—C14—C13	−0.6 (3)
S2—N1—C5—C10	−83.2 (4)	C11—S4—C14—Br2	−178.2 (2)

Fig. 1


O6
C8
N2
C9
O5
C7
C10
C6
C5
O3
O1
S1
C13
C12
N1
Br1
C11
S3
C4
S2
C1
Br2
C14
O2
C2
S4
O4
C3