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Crystal structure of 2-(3-nitrophenyl)-1,3-dithiane

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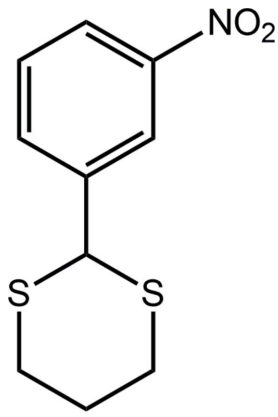
In the title compound, $C_{10}H_{11}NO_2S_2$, the 1,3-dithiane ring has a chair conformation with the 1,4-disposed C atoms being above and below the remaining four atoms. The nitrobenzene substituent occupies an equatorial position and forms a dihedral angle of 88.28 (5)° with the least-squares plane through the 1,3-dithiane ring. The nitro group is twisted out of the plane of the benzene ring to which it is connected, forming a dihedral angle of 10.12 (3)°. In the crystal, molecules aggregate into supramolecular zigzag chains (glide symmetry along the *c* axis) *via* nitro–benzene N—O···π [N—O···*Cg*(benzene) = 3.4279 (18) Å and angle at O = 93.95 (11)°] interactions. The chains pack with no specific intermolecular interactions between them.

Keywords: crystal structure; 1,3-dithiane; conformation; N—O···π interactions.

CCDC reference: 1048518

1. Related literature

For background to substituted 1,3-dithianes, see: Ballesteros *et al.* (2005). For nitro–aryl N—O···π interactions, see: Huang *et al.* (2008). For the structure of the closely related 3-bromo-substituted compound, see: Zukerman-Schpector *et al.* (2015).



2. Experimental

2.1. Crystal data

$C_{10}H_{11}NO_2S_2$
M_r = 241.32
Monoclinic, $P2_1/c$
a = 10.8547 (2) Å
b = 13.2655 (3) Å
c = 8.0891 (2) Å
β = 109.087 (1)°
V = 1100.74 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.46 mm^{-1}
T = 293 K
0.49 × 0.46 × 0.21 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.687, T_{max} = 0.745
7241 measured reflections
2035 independent reflections
1799 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.06
2035 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SIR2014* (Burla *et al.*, 2015); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *MarvinSketch* (ChemAxon, 2010) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5430).

References

Ballesteros, L., Noguez, O., Arroyo, G., Velasco, B., Delgado, F. & Miranda, R. (2005). *J. Mex. Chem. Soc.* **49**, 302–306.

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Burla, M. C., Caliandro, R., Carrozzini, B., Cascarano, G. L., Cuocci, C., Giacovazzo, C., Mallamo, M., Mazzone, A. & Polidori, G. (2015). *J. Appl. Cryst.* **48**, 306–309.

ChemAxon (2010). *Marvinsketch*. http://www.chemaxon.com.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Huang, L., Massa, L. & Karle, J. (2008). *Proc. Natl Acad. Sci.* **105**, 13720–13723.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Zukerman-Schpector, J., Caracelli, I., Stefani, H. A., Gozhina, O. & Tiekink, E. R. T. (2015). *Acta Cryst.* E**71**, o179–o180.

supporting information

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S1. Experimental

A solution of 3-nitrobenzaldehyde (0.037 mol, 1 equiv.) in chloroform (20 ml) was combined with an equimolar amount of propane-1,3-dithiol (3.7 ml, 0.037 mol) at room temperature. The solution was stirred for 1 h at this temperature, then cooled to -20 °C after which BF_3 etherate (0.46 ml, 0.0037 mol, 0.1 equiv.) was added drop-wise. The reaction solution was allowed to warm to room temperature and stirred overnight. After this time, the solution was washed three times each with water, 10% aqueous KOH, then water followed by drying over $MgSO_4$. Evaporation of the solvent furnishes a pure product as colourless crystals in 85% yield. To obtain crystals suitable for X-ray analysis, the product was crystallized from CH_3OH. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 (s, 1H), 8.18 (dt, J = 8.3, 2.6 Hz, 1H), 7.83 (ddd, J = 7.2, 3.6, 1.7 Hz, 1H), 7.63–7.48 (m, 1H), 5.26 (s, 1H), 3.10 (ddq, J = 14.4, 12.0, 2.3 Hz, 2H), 2.96 (ddp, J = 13.4, 5.1, 2.7 Hz, 2H), 2.23 (dtq, J = 14.0, 4.6, 2.3 Hz, 1H), 1.98 (dddd, J = 14.3, 12.1, 9.8, 6.0 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.39, 141.18, 133.99, 129.70, 123.36, 123.12, 50.19 (2 *x* C), 31.75, 24.78. *M*.pt: 368 K. IR (cm^{-1}): *v* 1525 (N—O); 1348 (N—O); 724 and 687 (C—S),

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2 U_{eq}(C).

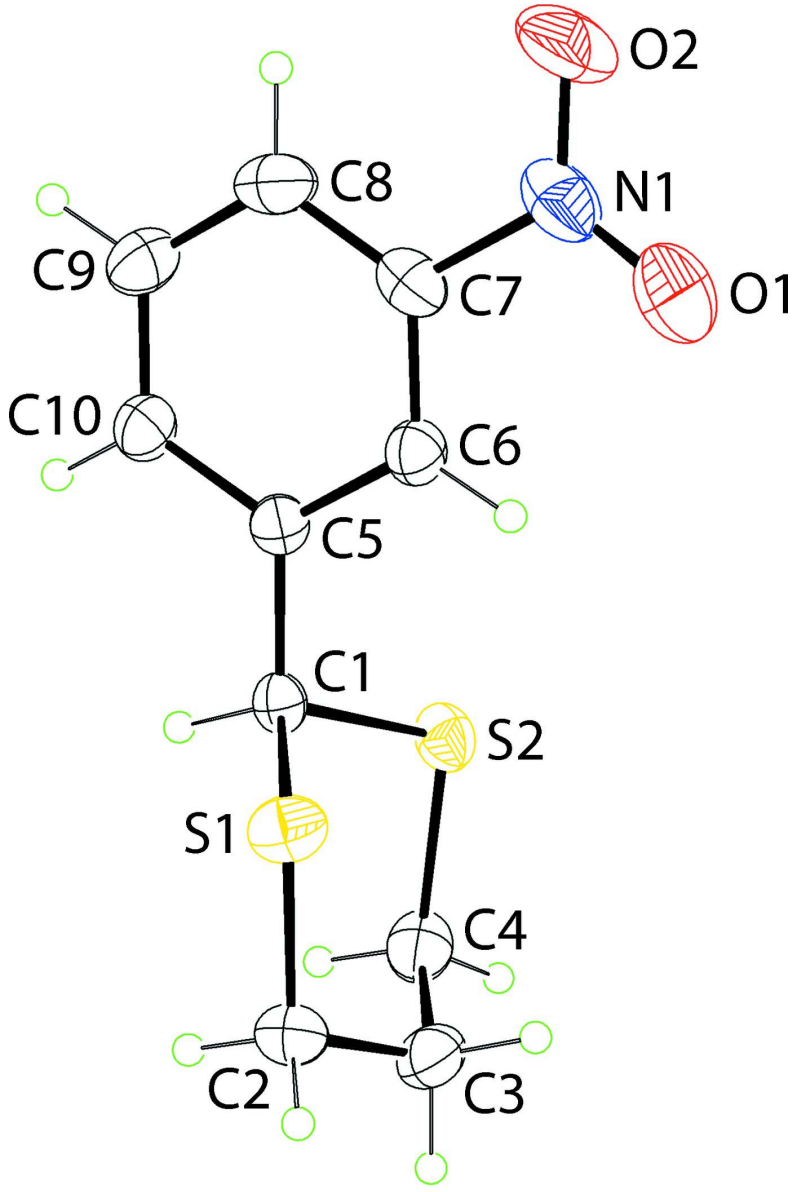

Figure 1

The molecular structure of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 35% probability level.

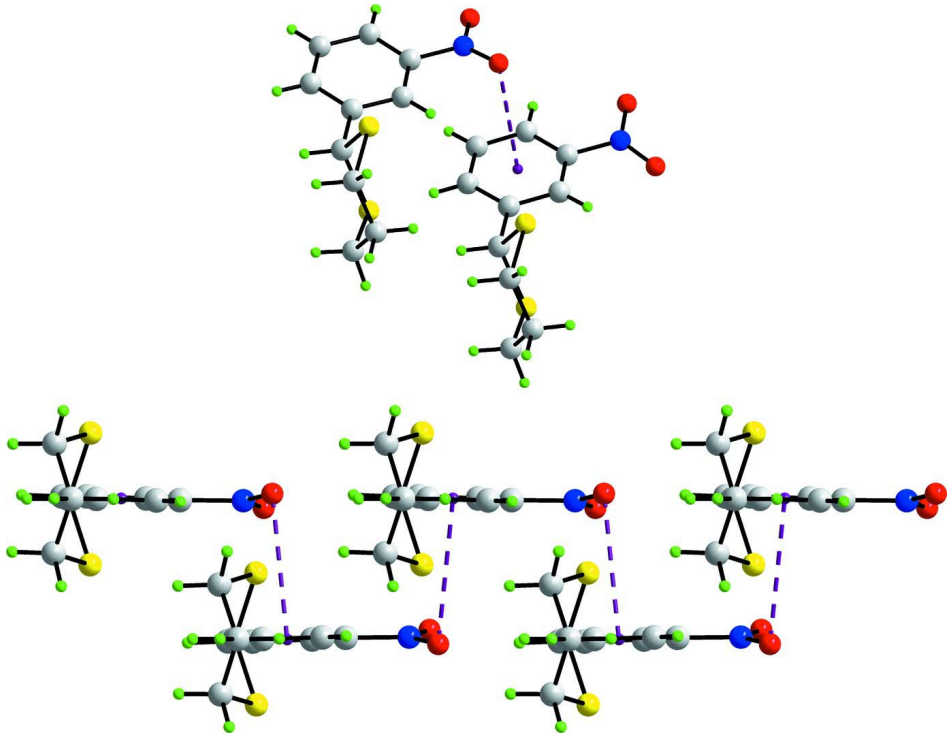

Figure 2

Upper view: detail of the nitro-N—O···π(benzene) interaction. Lower view: the zigzag supramolecular chain along the *c* axis (glide symmetry) mediated by nitro-N—O···π(benzene) interactions shown as purple dashed lines.

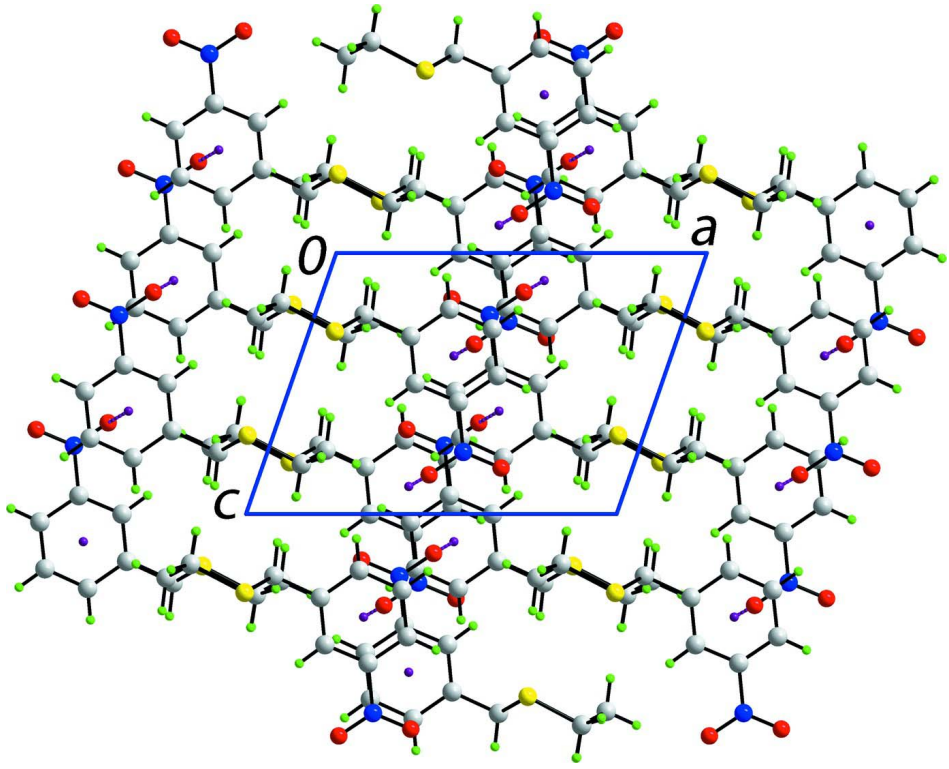

Figure 3

A view in projection down the *b* axis of the unit-cell contents. The nitro-N—O···π(benzene) interactions are shown as purple dashed lines.

2-(3-Nitrophenyl)-1,3-dithiane

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$\beta = 109.087$ (1)°
$V = 1100.74$ (4) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.456$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4425 reflections
$\theta = 2.5$–25.4°
$\mu = 0.46$ mm^{-1}
$T = 293$ K
Slab, colourless
0.49 × 0.46 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.687$, $T_{max} = 0.745$
7241 measured reflections
2035 independent reflections
1799 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.0°$
$h = -13\rightarrow12$
$k = -16\rightarrow16$
$l = -8\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.089$
$S = 1.06$
2035 reflections
137 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.3472P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick 2014, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0192 (17)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.93087 (4)	0.75719 (3)	0.69969 (5)	0.04460 (16)
S2	0.93019 (4)	0.98623 (3)	0.69835 (6)	0.04646 (16)
O1	0.54267 (18)	0.88063 (13)	0.15045 (19)	0.0830 (5)
O2	0.35484 (16)	0.85594 (14)	0.1765 (2)	0.0917 (6)
N1	0.47188 (18)	0.86855 (11)	0.2380 (2)	0.0608 (5)
C1	0.86732 (14)	0.87164 (10)	0.76325 (19)	0.0340 (3)
H1	0.8887	0.8719	0.8907	0.041*
C2	1.10282 (17)	0.96790 (14)	0.8068 (2)	0.0511 (4)
H2A	1.1494	1.0248	0.7804	0.061*
H2B	1.1198	0.9673	0.9322	0.061*
C3	1.15622 (17)	0.87200 (13)	0.7556 (2)	0.0519 (5)
H3A	1.1364	0.8713	0.6297	0.062*
H3B	1.2503	0.8720	0.8085	0.062*
C4	1.10278 (17)	0.77709 (14)	0.8096 (2)	0.0503 (4)
H4A	1.1185	0.7796	0.9347	0.060*
H4B	1.1503	0.7198	0.7868	0.060*
C5	0.72187 (15)	0.87186 (10)	0.6807 (2)	0.0353 (3)
C6	0.66533 (16)	0.86907 (11)	0.4999 (2)	0.0403 (4)
H6	0.7169	0.8669	0.4280	0.048*
C7	0.53161 (17)	0.86960 (11)	0.4295 (2)	0.0454 (4)
C8	0.45093 (17)	0.87258 (13)	0.5303 (3)	0.0538 (5)
H8	0.3607	0.8722	0.4790	0.065*
C9	0.50821 (18)	0.87617 (13)	0.7092 (3)	0.0539 (5)
H9	0.4561	0.8788	0.7803	0.065*
C10	0.64180 (17)	0.87591 (11)	0.7842 (2)	0.0441 (4)
H10	0.6789	0.8785	0.9053	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0499 (3)	0.0379 (2)	0.0436 (3)	0.00382 (16)	0.01201 (19)	−0.00589 (15)
S2	0.0441 (3)	0.0374 (3)	0.0589 (3)	−0.00061 (16)	0.0181 (2)	0.00780 (17)
O1	0.0889 (12)	0.1119 (14)	0.0382 (8)	0.0027 (9)	0.0071 (8)	0.0037 (7)
O2	0.0563 (9)	0.1212 (14)	0.0696 (10)	0.0056 (9)	−0.0178 (8)	−0.0135 (9)
N1	0.0629 (11)	0.0593 (10)	0.0454 (9)	0.0066 (7)	−0.0023 (8)	−0.0033 (7)
C1	0.0388 (8)	0.0344 (8)	0.0294 (7)	−0.0002 (6)	0.0119 (6)	−0.0002 (5)
C2	0.0416 (9)	0.0581 (11)	0.0532 (10)	−0.0114 (8)	0.0152 (8)	−0.0059 (8)
C3	0.0368 (8)	0.0729 (13)	0.0477 (10)	0.0044 (8)	0.0161 (7)	0.0004 (8)
C4	0.0467 (9)	0.0580 (11)	0.0433 (9)	0.0163 (8)	0.0107 (7)	0.0050 (7)
C5	0.0378 (8)	0.0338 (8)	0.0351 (8)	−0.0015 (6)	0.0128 (6)	0.0003 (5)
C6	0.0426 (9)	0.0438 (9)	0.0355 (8)	0.0009 (6)	0.0140 (7)	0.0009 (6)
C7	0.0456 (9)	0.0417 (9)	0.0408 (9)	−0.0002 (7)	0.0032 (7)	−0.0022 (6)
C8	0.0353 (9)	0.0542 (11)	0.0689 (12)	−0.0036 (7)	0.0131 (8)	−0.0020 (8)
C9	0.0462 (10)	0.0625 (12)	0.0608 (11)	−0.0065 (8)	0.0282 (9)	−0.0023 (8)
C10	0.0472 (9)	0.0490 (9)	0.0403 (9)	−0.0052 (7)	0.0201 (7)	−0.0006 (7)

Geometric parameters (Å, º)

S1—C4	1.8048 (18)	C3—H3B	0.9700
S1—C1	1.8102 (14)	C4—H4A	0.9700
S2—C2	1.8069 (18)	C4—H4B	0.9700
S2—C1	1.8128 (14)	C5—C6	1.389 (2)
O1—N1	1.214 (2)	C5—C10	1.391 (2)
O2—N1	1.215 (2)	C6—C7	1.375 (2)
N1—C7	1.471 (2)	C6—H6	0.9300
C1—C5	1.500 (2)	C7—C8	1.379 (3)
C1—H1	0.9800	C8—C9	1.377 (3)
C2—C3	1.511 (2)	C8—H8	0.9300
C2—H2A	0.9700	C9—C10	1.377 (3)
C2—H2B	0.9700	C9—H9	0.9300
C3—C4	1.509 (3)	C10—H10	0.9300
C3—H3A	0.9700		
C4—S1—C1	99.55 (8)	C3—C4—H4A	108.7
C2—S2—C1	100.11 (8)	S1—C4—H4A	108.7
O1—N1—O2	123.77 (18)	C3—C4—H4B	108.7
O1—N1—C7	117.90 (17)	S1—C4—H4B	108.7
O2—N1—C7	118.32 (19)	H4A—C4—H4B	107.6
C5—C1—S1	108.59 (10)	C6—C5—C10	119.13 (15)
C5—C1—S2	108.10 (10)	C6—C5—C1	120.46 (13)
S1—C1—S2	113.99 (8)	C10—C5—C1	120.41 (14)
C5—C1—H1	108.7	C7—C6—C5	118.65 (15)
S1—C1—H1	108.7	C7—C6—H6	120.7
S2—C1—H1	108.7	C5—C6—H6	120.7
C3—C2—S2	114.22 (12)	C6—C7—C8	122.93 (16)

C3—C2—H2A	108.7	C6—C7—N1	118.60 (16)
S2—C2—H2A	108.7	C8—C7—N1	118.46 (16)
C3—C2—H2B	108.7	C9—C8—C7	117.84 (16)
S2—C2—H2B	108.7	C9—C8—H8	121.1
H2A—C2—H2B	107.6	C7—C8—H8	121.1
C4—C3—C2	113.89 (15)	C10—C9—C8	120.77 (16)
C4—C3—H3A	108.8	C10—C9—H9	119.6
C2—C3—H3A	108.8	C8—C9—H9	119.6
C4—C3—H3B	108.8	C9—C10—C5	120.67 (16)
C2—C3—H3B	108.8	C9—C10—H10	119.7
H3A—C3—H3B	107.7	C5—C10—H10	119.7
C3—C4—S1	114.37 (12)		

C4—S1—C1—C5	178.82 (10)	C1—C5—C6—C7	−179.86 (13)
C4—S1—C1—S2	58.27 (10)	C5—C6—C7—C8	−0.1 (2)
C2—S2—C1—C5	−178.78 (10)	C5—C6—C7—N1	178.88 (13)
C2—S2—C1—S1	−57.95 (10)	O1—N1—C7—C6	−9.5 (2)
C1—S2—C2—C3	57.35 (14)	O2—N1—C7—C6	170.67 (16)
S2—C2—C3—C4	−65.25 (19)	O1—N1—C7—C8	169.54 (17)
C2—C3—C4—S1	66.18 (19)	O2—N1—C7—C8	−10.3 (2)
C1—S1—C4—C3	−58.63 (14)	C6—C7—C8—C9	0.7 (2)
S1—C1—C5—C6	−60.31 (15)	N1—C7—C8—C9	−178.35 (15)
S2—C1—C5—C6	63.82 (15)	C7—C8—C9—C10	−0.5 (3)
S1—C1—C5—C10	120.35 (13)	C8—C9—C10—C5	−0.1 (2)
S2—C1—C5—C10	−115.51 (13)	C6—C5—C10—C9	0.6 (2)
C10—C5—C6—C7	−0.5 (2)	C1—C5—C10—C9	179.98 (14)