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8-Thia-1,6-diazabicyclo[4.3.0]nonane-7,9-dione

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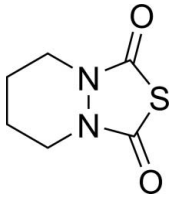
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.125; data-to-parameter ratio = 14.0.

There are two independent molecules, *A* and *B*, in the asymmetric unit of the title compound, $C_6H_8N_2O_2S$. In the crystal, pairs of intermolecular S···O contacts [3.286 (1) Å] link the *B* molecules into inversion dimers.

Related literature

For applications of the title compound, see: Yamaguchi *et al.* (1989). For the synthesis, see: Zhu *et al.* (2011). For bond-length data, see: Allen *et al.* (1987). For a review of carbonyl–carbonyl interactions, see: Allen *et al.* (1998).



Experimental

Crystal data

$C_6H_8N_2O_2S$
M_r = 172.20
Triclinic, $P\overline{1}$
a = 7.8400 (16) Å
b = 10.464 (2) Å
c = 10.514 (2) Å
α = 63.84 (3)°
β = 79.62 (3)°
γ = 89.42 (3)°
V = 759.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.896, T_{max} = 0.964
3018 measured reflections
2795 independent reflections
2092 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.125
S = 1.00
2795 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo,1995); 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: LX2196).

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supporting information

Acta Cryst. (2011). E**67**, o2771 [https://doi.org/10.1107/S1600536811038785]

8-Thia-1,6-diazabicyclo[4.3.0]nonane-7,9-dione

Bin-bin Xi, Lu Shi, Hai Huang, Qin Wang and Hong-Jun Zhu

S1. Comment

The title compound, 8-thia-1,6-diazabicyclo[4.3.0]nonane-7,9-dione, is an important intermediate for a kind of manufacturing agrochemical, especially for Fluthiacet-methyl as a super-effective, wide-spectral and safe herbicide (Yamaguchi *et al.*, 1989). We report herein the crystal structure of the title compound.

The asymmetric unit of the title compound is shown in Fig. 1 and there are two independent unique molecules [labeled A & B]. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The crystal packing (Fig. 2) is stabilized by an intermolecular S···O interaction between the sulfur and the O atom of the carbonyl group interpreted as similar to type-II carbonyl–carbonyl interaction (Allen *et al.*, 1998), with S1···O2^{i} distance of 3.286 (1) Å.

S2. Experimental

The title compound was prepared according to reported in literature (Zhu *et al.*, 2011). Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in ethanol at room temperature for ca. 6 d.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å and U_{iso}(H) =1.5U_{eq}(C).

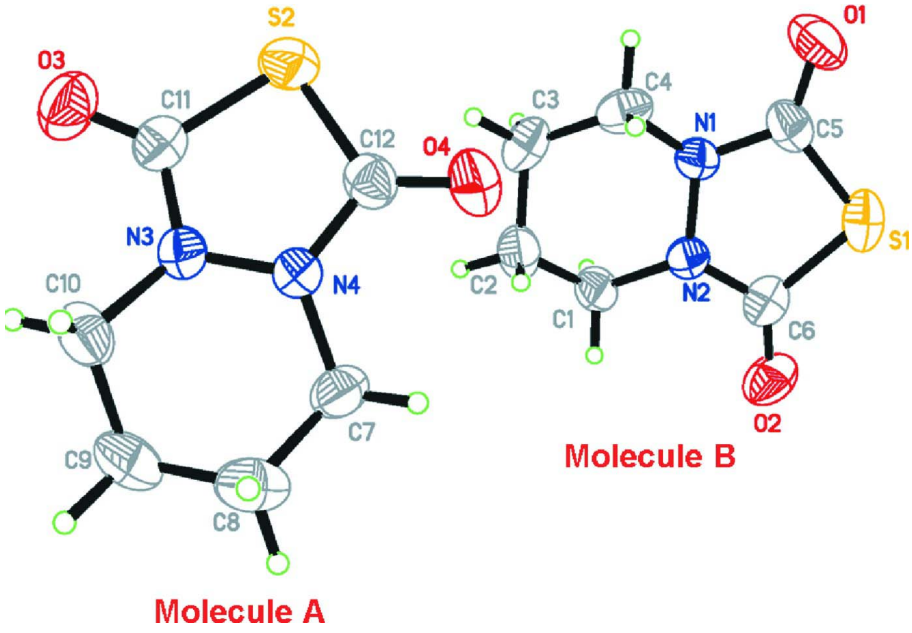

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

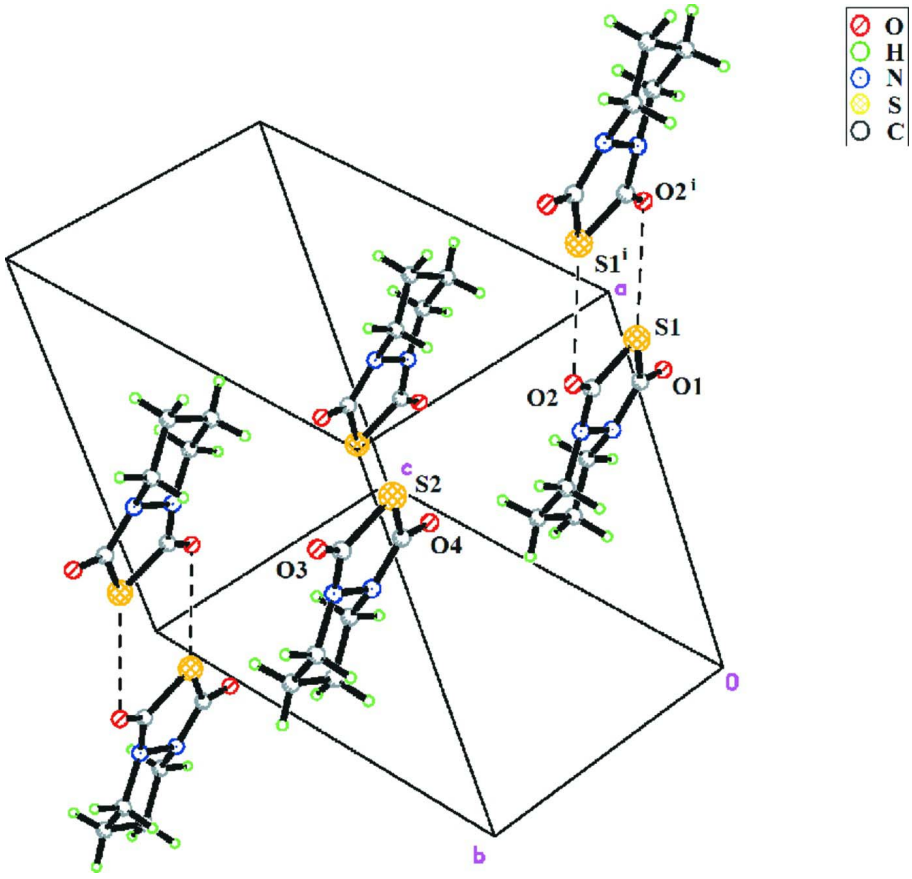

Figure 2

A view of the S···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) - *x*, - *y* + 2, - *z*.]

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$a = 7.8400$ (16) Å
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$c = 10.514$ (2) Å
$\alpha = 63.84$ (3)°
$\beta = 79.62$ (3)°
$\gamma = 89.42$ (3)°
$V = 759.2$ (3) Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.507$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.37$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.896$, $T_{max} = 0.964$
3018 measured reflections
2795 independent reflections
2092 reflections with $I > 2\sigma(I)$

R_{int} = 0.041
θ_{max} = 25.4°, θ_{min} = 2.2°
h = 0→9
k = −12→12
l = −12→12
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.125
S = 1.00
2795 reflections
200 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.327 (16)

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 $F^2 > \sigma(F^2)$ 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}
S1	−0.09731 (11)	0.82576 (8)	0.04086 (8)	0.0598 (3)
O1	−0.3185 (3)	0.5944 (3)	0.1791 (3)	0.0737 (7)
O2	0.2479 (3)	0.8734 (2)	−0.0219 (2)	0.0632 (6)
N1	−0.0292 (2)	0.5625 (2)	0.1507 (2)	0.0416 (5)
N2	0.1318 (3)	0.6432 (2)	0.1083 (2)	0.0411 (5)
C1	0.2837 (3)	0.5721 (3)	0.0768 (3)	0.0464 (6)
H1A	0.3892	0.6300	0.0591	0.056*
H1B	0.2803	0.5604	−0.0092	0.056*
C2	0.2842 (4)	0.4278 (3)	0.2034 (3)	0.0547 (7)
H2A	0.3027	0.4404	0.2860	0.066*
H2B	0.3789	0.3771	0.1791	0.066*
C3	0.1134 (4)	0.3408 (3)	0.2418 (3)	0.0529 (7)
H3A	0.1002	0.3205	0.1623	0.063*
H3B	0.1137	0.2505	0.3261	0.063*
C4	−0.0370 (4)	0.4199 (3)	0.2726 (3)	0.0491 (7)
H4A	−0.1456	0.3669	0.2878	0.059*
H4B	−0.0326	0.4290	0.3599	0.059*
C5	−0.1691 (3)	0.6421 (3)	0.1356 (3)	0.0497 (7)
C6	0.1243 (4)	0.7859 (3)	0.0365 (3)	0.0462 (6)

S2	0.16047 (9)	0.00770 (8)	0.62686 (8)	0.0517 (3)
O3	0.3930 (3)	−0.1856 (2)	0.6988 (2)	0.0625 (6)
O4	0.1494 (2)	0.2796 (2)	0.5788 (2)	0.0585 (5)
N3	0.4698 (2)	0.0396 (2)	0.6615 (2)	0.0396 (5)
N4	0.3934 (3)	0.1675 (2)	0.6431 (2)	0.0379 (5)
C7	0.5149 (3)	0.2948 (3)	0.5815 (3)	0.0462 (6)
H7A	0.4534	0.3749	0.5837	0.055*
H7B	0.5648	0.3187	0.4817	0.055*
C8	0.6568 (4)	0.2672 (3)	0.6669 (3)	0.0567 (8)
H8A	0.6083	0.2567	0.7629	0.068*
H8B	0.7430	0.3483	0.6203	0.068*
C9	0.7431 (3)	0.1341 (3)	0.6786 (3)	0.0541 (7)
H9A	0.8298	0.1158	0.7384	0.065*
H9B	0.8015	0.1481	0.5833	0.065*
C10	0.6102 (3)	0.0070 (3)	0.7438 (3)	0.0502 (7)
H10A	0.6655	−0.0756	0.7418	0.060*
H10B	0.5628	−0.0153	0.8437	0.060*
C11	0.3614 (3)	−0.0634 (3)	0.6686 (3)	0.0433 (6)
C12	0.2314 (3)	0.1752 (3)	0.6121 (3)	0.0412 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0698 (5)	0.0513 (5)	0.0626 (5)	0.0262 (4)	−0.0203 (4)	−0.0268 (4)
O1	0.0382 (12)	0.0922 (17)	0.0976 (17)	0.0078 (11)	−0.0077 (11)	−0.0508 (14)
O2	0.0749 (14)	0.0403 (11)	0.0593 (13)	−0.0102 (10)	0.0012 (11)	−0.0138 (10)
N1	0.0357 (11)	0.0393 (11)	0.0448 (12)	0.0011 (9)	−0.0052 (9)	−0.0153 (10)
N2	0.0369 (11)	0.0349 (11)	0.0453 (12)	0.0006 (9)	−0.0038 (9)	−0.0139 (9)
C1	0.0375 (14)	0.0472 (15)	0.0502 (15)	0.0045 (11)	0.0008 (11)	−0.0213 (13)
C2	0.0508 (17)	0.0486 (16)	0.0617 (18)	0.0155 (13)	−0.0147 (14)	−0.0209 (14)
C3	0.073 (2)	0.0344 (14)	0.0467 (15)	0.0053 (13)	−0.0151 (14)	−0.0130 (12)
C4	0.0545 (16)	0.0431 (15)	0.0406 (14)	−0.0101 (12)	−0.0044 (12)	−0.0120 (12)
C5	0.0418 (16)	0.0638 (18)	0.0531 (16)	0.0122 (13)	−0.0109 (13)	−0.0343 (15)
C6	0.0585 (17)	0.0418 (15)	0.0362 (13)	0.0039 (13)	−0.0048 (12)	−0.0172 (12)
S2	0.0443 (4)	0.0511 (4)	0.0589 (5)	−0.0031 (3)	−0.0175 (3)	−0.0208 (3)
O3	0.0769 (15)	0.0407 (11)	0.0712 (14)	0.0081 (10)	−0.0188 (11)	−0.0247 (10)
O4	0.0494 (11)	0.0536 (12)	0.0682 (13)	0.0176 (10)	−0.0212 (10)	−0.0200 (10)
N3	0.0358 (11)	0.0354 (11)	0.0451 (12)	0.0058 (9)	−0.0080 (9)	−0.0159 (9)
N4	0.0328 (10)	0.0335 (11)	0.0445 (12)	0.0028 (8)	−0.0076 (9)	−0.0150 (9)
C7	0.0446 (15)	0.0356 (13)	0.0543 (16)	−0.0040 (11)	−0.0011 (12)	−0.0195 (12)
C8	0.0418 (15)	0.0667 (19)	0.0708 (19)	−0.0067 (14)	−0.0050 (14)	−0.0409 (16)
C9	0.0312 (13)	0.076 (2)	0.0632 (18)	0.0066 (13)	−0.0108 (13)	−0.0377 (16)
C10	0.0381 (14)	0.0576 (16)	0.0520 (16)	0.0119 (12)	−0.0132 (12)	−0.0204 (14)
C11	0.0467 (15)	0.0393 (15)	0.0413 (14)	0.0016 (12)	−0.0056 (11)	−0.0167 (11)
C12	0.0386 (13)	0.0453 (14)	0.0359 (13)	0.0049 (12)	−0.0095 (11)	−0.0138 (11)

Geometric parameters (Å, º)

S1—C5	1.771 (3)	S2—C12	1.776 (3)
S1—C6	1.780 (3)	S2—C11	1.779 (3)
O1—C5	1.203 (3)	O3—C11	1.208 (3)
O2—C6	1.209 (3)	O4—C12	1.208 (3)
N1—C5	1.360 (3)	N3—C11	1.347 (3)
N1—N2	1.411 (3)	N3—N4	1.409 (3)
N1—C4	1.470 (3)	N3—C10	1.467 (3)
N2—C6	1.351 (3)	N4—C12	1.360 (3)
N2—C1	1.463 (3)	N4—C7	1.464 (3)
C1—C2	1.510 (4)	C7—C8	1.501 (4)
C1—H1A	0.9700	C7—H7A	0.9700
C1—H1B	0.9700	C7—H7B	0.9700
C2—C3	1.514 (4)	C8—C9	1.508 (4)
C2—H2A	0.9700	C8—H8A	0.9700
C2—H2B	0.9700	C8—H8B	0.9700
C3—C4	1.503 (4)	C9—C10	1.514 (4)
C3—H3A	0.9700	C9—H9A	0.9700
C3—H3B	0.9700	C9—H9B	0.9700
C4—H4A	0.9700	C10—H10A	0.9700
C4—H4B	0.9700	C10—H10B	0.9700
C5—S1—C6	91.50 (13)	C12—S2—C11	91.50 (12)
C5—N1—N2	114.0 (2)	C11—N3—N4	114.76 (19)
C5—N1—C4	121.3 (2)	C11—N3—C10	122.2 (2)
N2—N1—C4	114.5 (2)	N4—N3—C10	114.6 (2)
C6—N2—N1	114.8 (2)	C12—N4—N3	114.5 (2)
C6—N2—C1	121.8 (2)	C12—N4—C7	121.1 (2)
N1—N2—C1	115.11 (19)	N3—N4—C7	115.35 (19)
N2—C1—C2	109.5 (2)	N4—C7—C8	109.7 (2)
N2—C1—H1A	109.8	N4—C7—H7A	109.7
C2—C1—H1A	109.8	C8—C7—H7A	109.7
N2—C1—H1B	109.8	N4—C7—H7B	109.7
C2—C1—H1B	109.8	C8—C7—H7B	109.7
H1A—C1—H1B	108.2	H7A—C7—H7B	108.2
C1—C2—C3	110.6 (2)	C7—C8—C9	111.1 (2)
C1—C2—H2A	109.5	C7—C8—H8A	109.4
C3—C2—H2A	109.5	C9—C8—H8A	109.4
C1—C2—H2B	109.5	C7—C8—H8B	109.4
C3—C2—H2B	109.5	C9—C8—H8B	109.4
H2A—C2—H2B	108.1	H8A—C8—H8B	108.0
C4—C3—C2	110.8 (2)	C8—C9—C10	110.7 (2)
C4—C3—H3A	109.5	C8—C9—H9A	109.5
C2—C3—H3A	109.5	C10—C9—H9A	109.5
C4—C3—H3B	109.5	C8—C9—H9B	109.5
C2—C3—H3B	109.5	C10—C9—H9B	109.5
H3A—C3—H3B	108.1	H9A—C9—H9B	108.1

N1—C4—C3	109.9 (2)	N3—C10—C9	109.7 (2)
N1—C4—H4A	109.7	N3—C10—H10A	109.7
C3—C4—H4A	109.7	C9—C10—H10A	109.7
N1—C4—H4B	109.7	N3—C10—H10B	109.7
C3—C4—H4B	109.7	C9—C10—H10B	109.7
H4A—C4—H4B	108.2	H10A—C10—H10B	108.2
O1—C5—N1	125.0 (3)	O3—C11—N3	126.2 (3)
O1—C5—S1	125.5 (2)	O3—C11—S2	124.5 (2)
N1—C5—S1	109.5 (2)	N3—C11—S2	109.34 (18)
O2—C6—N2	125.7 (3)	O4—C12—N4	125.5 (2)
O2—C6—S1	125.1 (2)	O4—C12—S2	125.4 (2)
N2—C6—S1	109.2 (2)	N4—C12—S2	109.10 (18)
C5—N1—N2—C6	−11.3 (3)	C11—N3—N4—C12	−10.1 (3)
C4—N1—N2—C6	−157.2 (2)	C10—N3—N4—C12	−159.0 (2)
C5—N1—N2—C1	−160.4 (2)	C11—N3—N4—C7	−157.8 (2)
C4—N1—N2—C1	53.6 (3)	C10—N3—N4—C7	53.3 (3)
C6—N2—C1—C2	160.1 (2)	C12—N4—C7—C8	162.2 (2)
N1—N2—C1—C2	−53.2 (3)	N3—N4—C7—C8	−52.4 (3)
N2—C1—C2—C3	54.1 (3)	N4—C7—C8—C9	53.6 (3)
C1—C2—C3—C4	−56.3 (3)	C7—C8—C9—C10	−56.1 (3)
C5—N1—C4—C3	164.3 (2)	C11—N3—C10—C9	161.2 (2)
N2—N1—C4—C3	−52.4 (3)	N4—N3—C10—C9	−52.4 (3)
C2—C3—C4—N1	53.8 (3)	C8—C9—C10—N3	53.8 (3)
N2—N1—C5—O1	−171.7 (3)	N4—N3—C11—O3	−171.8 (2)
C4—N1—C5—O1	−28.3 (4)	C10—N3—C11—O3	−25.6 (4)
N2—N1—C5—S1	8.8 (3)	N4—N3—C11—S2	7.9 (3)
C4—N1—C5—S1	152.26 (19)	C10—N3—C11—S2	154.20 (19)
C6—S1—C5—O1	176.8 (3)	C12—S2—C11—O3	176.4 (2)
C6—S1—C5—N1	−3.76 (19)	C12—S2—C11—N3	−3.33 (19)
N1—N2—C6—O2	−172.2 (2)	N3—N4—C12—O4	−172.5 (2)
C1—N2—C6—O2	−25.3 (4)	C7—N4—C12—O4	−26.9 (4)
N1—N2—C6—S1	7.8 (3)	N3—N4—C12—S2	6.9 (3)
C1—N2—C6—S1	154.63 (19)	C7—N4—C12—S2	152.55 (18)
C5—S1—C6—O2	177.7 (3)	C11—S2—C12—O4	177.4 (2)
C5—S1—C6—N2	−2.26 (19)	C11—S2—C12—N4	−2.03 (19)