Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(2-Furoyl)-3-(*o*-tolyl)thiourea

Rodrigo S. Corrêa,[a] O. Estévez-Hernández,[b]* J. Ellena[a] and J. Duque[b]

[a]Grupo de Cristalografía, Instituto de Física de São Carlos, Universidade de São Paulo, São Carlos, Brazil, and [b]Department of Structure Analysis, Institute of Materials, University of Havana, Cuba
Correspondence e-mail: osvaldo@imre.oc.uh.cu

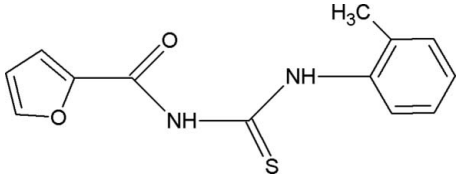
Received 27 June 2008; accepted 1 July 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.130; data-to-parameter ratio = 14.7.

The title compound, $C_{13}H_{12}N_2O_2S$, was synthesized from furoyl isothiocyanate and *o*-toluidine in dry acetone. The thiourea group is in the thioamide form. The central thiourea fragment makes dihedral angles of 2.6 (1) and 22.4 (1)° with the ketofuran group and the benzene ring, respectively. The molecular structure is stabilized by N—H···O hydrogen bonds. In the crystal structure, centrosymmetrically related molecules are linked by a pair of N—H···S hydrogen bonds to form a dimer with an $R_2^2(6)$ ring motif.

Related literature

For general background, see: Aly *et al.* (2007); Koch (2001); Estévez-Hernández *et al.* (2007). For related structures, see: Theodoro *et al.* (2008); Duque *et al.* (2008). For the synthesis, see: Otazo-Sánchez *et al.* (2001).



Experimental

Crystal data

$C_{13}H_{12}N_2O_2S$
M_r = 260.31
Monoclinic, $P2_1/c$
a = 6.0976 (1) Å
b = 16.6689 (6) Å
c = 13.1462 (4) Å
β = 108.765 (2)°
V = 1265.16 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 K
0.50 × 0.08 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: Gaussian (Coppens *et al.*, 1965)
T_{min} = 0.925, T_{max} = 0.983
8242 measured reflections
2408 independent reflections
1594 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.130
S = 1.02
2408 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.86	2.26	2.682 (3)	110
N1—H1···S1^{i}	0.86	2.80	3.639 (2)	165
N2—H2···O1	0.86	1.92	2.649 (2)	141

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; 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).

The authors thank the Crystallography Group, São Carlos Physics Institute, USP, and acknowledge financial support from Brazilian agency CNPq.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2623).

References

Aly, A. A., Ahmed, E. K., El-Mokadem, K. M. & Hegazy, M. E. F. (2007). *J. Sulfur Chem.* **28**, 73–93.
Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.
Duque, J., Estevez-Hernandez, O., Reguera, E., Corrêa, R. S. & Gutierrez Maria, P. (2008). *Acta Cryst.* E**64**, o1068.
Estévez-Hernández, O., Naranjo-Rodríguez, I., Hidalgo-Hidalgo de Cisneros, J. L. & Reguera, E. (2007). *Sens. Actuators B*, **123**, 488–494.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Koch, K. R. (2001). *Coord. Chem. Rev.* **216–217**, 473–488.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otazo-Sánchez, E., Pérez-Marín, L., Estévez-Hernández, O., Rojas-Lima, S. & Alonso-Chamorro, J. (2001). *J. Chem. Soc. Perkin Trans. 2*, pp. 2211–2218.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Theodoro, J. E., Mascarenhas, Y., Ellena, J., Estévez-Hernández, O. & Duque, J. (2008). *Acta Cryst.* E**64**, o1193.

supporting information

Acta Cryst. (2008). E**64**, o1414 [doi:10.1107/S1600536808020114]

1-(2-Furoyl)-3-(*o*-tolyl)thiourea

Rodrigo S. Corrêa, O. Estévez-Hernández, J. Ellena and J. Duque

S1. Comment

Thiourea and its derivatives form a versatile family of ligands suitable to form complexes with ions of transition metals through the S atom (Aly *et al.*, 2007). Of analytical interest is the potential application of these compounds as ionophores or chemical modifiers in potenciometric and amperometric sensors (Estévez-Hernández *et al.*, 2007). The derived crystal structures help to understand the behaviour of these ligands as ionophores and also the complex formation with salts of the heavy metals. The title compound (Fig.1) is another example of our newly synthesized furoylthiourea derivatives.

The title compound crystallizes in the thioamide form. The bond lengths and angles are within the ranges observed for similar compounds (Koch, 2001). The C2—S1 [1.652 (2) Å] and C1—O1 [1.221 (2) Å] bonds both show the expected double-bond character. The short values of the C2—N1 [1.400 (3) Å], C2—N2 [1.330 (3) Å] and C1—N1 [1.377 (2) Å] bonds indicate partial double bond character. These results can be explained by the existence of resonance in this part of the molecule. The furan carbonyl group [O1/O2/C1/C3-C6] is nearly coplanar with the plane of the thiourea fragment [N1/N2/C2/S1, dihedral angle 2.6 (1)°], whereas the C7-C12 benzene ring is inclined by 22.4 (1)°. The *trans-cis* geometry in the thiourea group is stabilized by the N2—H2···O1 and N1—H1···O2 intramolecular hydrogen bonds (Fig.1 and Table 1). The crystal structure is stabilized by two intermolecular N1—H1···S1 hydrogen bonds (Fig.2 and Table 1) between centrosymmetrically related molecules forming dimers stacked along the [100] direction.

S2. Experimental

The title compound was synthesized according to a previous report (Otazo-Sánchez *et al.*, 2001), by converting furoyl chloride into furoyl isothiocyanate and then condensing with *o*-toluidine. The resulting solid product was crystallized from ethanol yielding X-ray quality single crystals (m.p 387–388 K).

S3. Refinement

H atoms were placed in calculated positions with N-H = 0.86 Å and C-H = 0.93 Å (aromatic) or 0.96 Å (methyl), and refined in riding model, with U_{iso}(H) = 1.5U_{eq}(C_{methyl}) and 1.2U_{eq}(N,$C_{aromatic}$).

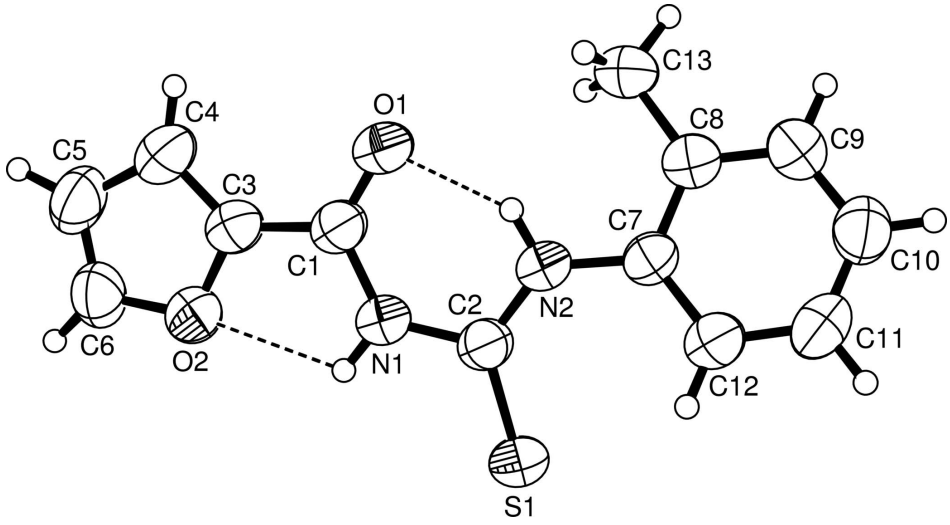


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids. Intramolecular hydrogen bonds are shown as dashed lines.

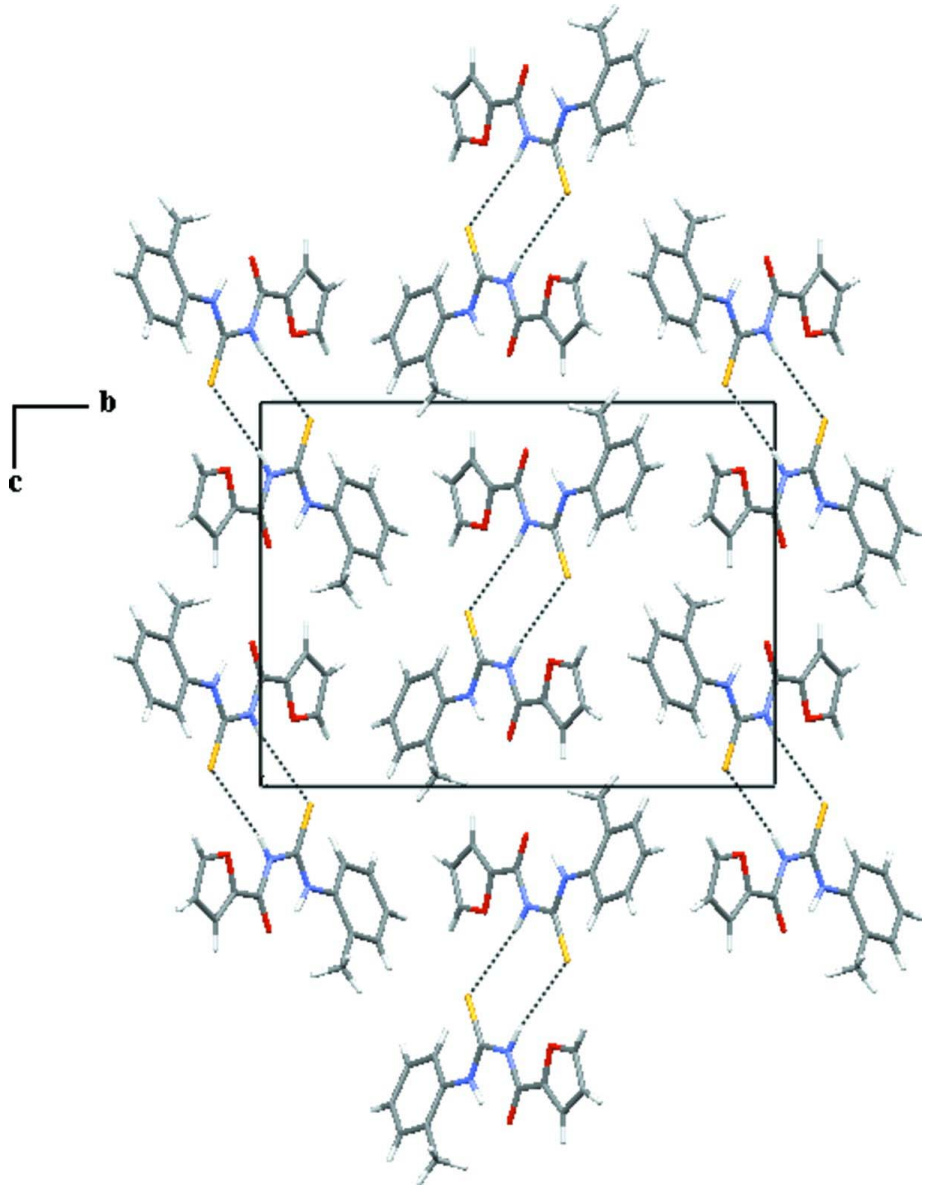


Figure 2

View of the crystal packing of the title compound. Intermolecular Hydrogen bonds are shown as dashed lines.

1-(2-Furoyl)-3-(*o*-tolyl)thiourea

Crystal data

$C_{13}H_{12}N_2O_2S$
$M_r = 260.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.0976\ (1)$ Å
$b = 16.6689\ (6)$ Å
$c = 13.1462\ (4)$ Å
$\beta = 108.765\ (2)°$
$V = 1265.16\ (6)$ Å^3
$Z = 4$

$F(000) = 544$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20621 reflections
$\theta = 2.9–25.7°$
$\mu = 0.25$ mm^{-1}
$T = 294$ K
Needle, colourless
0.50 × 0.08 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: gaussian (Coppens *et al.*, 1965)
T_{min} = 0.925, T_{max} = 0.983
8242 measured reflections

2408 independent reflections
1594 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.7°, θ_{min} = 2.9°
h = −6→7
k = −19→20
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.130
S = 1.03
2408 reflections
164 parameters

0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.1015P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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	0.22254 (12)	0.09680 (4)	0.04759 (5)	0.0800 (3)
N2	0.4198 (3)	0.09229 (10)	0.26210 (13)	0.0561 (4)
H2	0.408	0.0712	0.3198	0.067*
O2	−0.2979 (3)	−0.06394 (9)	0.17467 (12)	0.0679 (4)
O1	0.1899 (3)	0.01628 (10)	0.37313 (12)	0.0757 (5)
N1	0.0880 (3)	0.01904 (10)	0.18967 (13)	0.0578 (5)
H1	−0.0083	−0.0016	0.1328	0.069*
C7	0.6105 (3)	0.14441 (12)	0.27881 (15)	0.0528 (5)
C2	0.2552 (4)	0.07008 (12)	0.17262 (17)	0.0556 (5)
C1	0.0568 (4)	−0.00258 (13)	0.28515 (17)	0.0587 (5)
C12	0.7138 (4)	0.16151 (13)	0.20141 (17)	0.0631 (6)
H12	0.6557	0.1389	0.1333	0.076*
C3	−0.1472 (4)	−0.05056 (13)	0.27543 (17)	0.0594 (5)
C8	0.7009 (4)	0.17683 (13)	0.38190 (17)	0.0609 (6)
C9	0.8905 (4)	0.22716 (14)	0.40236 (19)	0.0728 (6)
H9	0.9524	0.2494	0.4705	0.087*
C4	−0.2232 (5)	−0.08806 (15)	0.3482 (2)	0.0774 (7)
H4	−0.1528	−0.0881	0.4224	0.093*
C10	0.9905 (4)	0.24531 (14)	0.3252 (2)	0.0737 (6)
H10	1.1166	0.2799	0.3409	0.088*
C11	0.9035 (4)	0.21225 (14)	0.2256 (2)	0.0703 (6)
H11	0.9719	0.2238	0.1734	0.084*
C13	0.5994 (4)	0.15856 (18)	0.46916 (17)	0.0835 (8)

H13A	0.671	0.1921	0.5303	0.125*
H13B	0.6264	0.1032	0.4896	0.125*
H13C	0.4357	0.1687	0.4435	0.125*
C6	−0.4688 (4)	−0.11084 (14)	0.1874 (2)	0.0769 (7)
H6	−0.5959	−0.1291	0.1316	0.092*
C5	−0.4295 (5)	−0.12709 (15)	0.2904 (2)	0.0831 (8)
H5	−0.5214	−0.1585	0.319	0.1*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0983 (5)	0.0930 (5)	0.0458 (4)	−0.0272 (4)	0.0189 (3)	0.0060 (3)
N2	0.0625 (10)	0.0623 (10)	0.0453 (10)	−0.0051 (9)	0.0199 (9)	0.0046 (8)
O2	0.0709 (10)	0.0737 (10)	0.0649 (10)	−0.0096 (8)	0.0302 (8)	−0.0045 (7)
O1	0.0788 (10)	0.0981 (13)	0.0498 (9)	−0.0183 (9)	0.0201 (8)	0.0072 (8)
N1	0.0627 (10)	0.0649 (11)	0.0469 (9)	−0.0066 (9)	0.0192 (8)	0.0012 (8)
C7	0.0572 (11)	0.0511 (12)	0.0512 (12)	0.0023 (10)	0.0190 (9)	0.0062 (9)
C2	0.0664 (13)	0.0525 (12)	0.0508 (13)	−0.0005 (10)	0.0231 (11)	0.0011 (9)
C1	0.0652 (13)	0.0602 (13)	0.0542 (13)	0.0000 (11)	0.0240 (11)	0.0040 (10)
C12	0.0688 (13)	0.0661 (14)	0.0583 (13)	0.0001 (11)	0.0262 (11)	0.0032 (10)
C3	0.0642 (13)	0.0624 (14)	0.0553 (13)	0.0021 (11)	0.0245 (11)	0.0016 (10)
C8	0.0639 (13)	0.0641 (14)	0.0541 (13)	−0.0005 (11)	0.0184 (10)	0.0032 (10)
C9	0.0739 (14)	0.0746 (16)	0.0652 (15)	−0.0132 (12)	0.0158 (12)	−0.0037 (12)
C4	0.0832 (17)	0.0879 (17)	0.0700 (16)	−0.0091 (14)	0.0370 (14)	0.0092 (13)
C10	0.0684 (14)	0.0705 (16)	0.0810 (17)	−0.0085 (12)	0.0223 (13)	0.0066 (13)
C11	0.0675 (14)	0.0750 (15)	0.0765 (17)	0.0015 (12)	0.0343 (13)	0.0164 (13)
C13	0.0874 (17)	0.113 (2)	0.0507 (13)	−0.0239 (15)	0.0226 (12)	−0.0079 (13)
C6	0.0716 (15)	0.0789 (17)	0.0887 (19)	−0.0192 (13)	0.0376 (14)	−0.0161 (14)
C5	0.0880 (18)	0.0825 (18)	0.094 (2)	−0.0170 (14)	0.0506 (16)	0.0027 (15)

Geometric parameters (Å, º)

S1—C2	1.652 (2)	C8—C9	1.383 (3)
N2—C2	1.330 (3)	C8—C13	1.500 (3)
N2—C7	1.411 (2)	C9—C10	1.375 (3)
N2—H2	0.86	C9—H9	0.93
O2—C6	1.355 (3)	C4—C5	1.403 (4)
O2—C3	1.366 (3)	C4—H4	0.93
O1—C1	1.221 (2)	C10—C11	1.361 (3)
N1—C1	1.377 (2)	C10—H10	0.93
N1—C2	1.400 (3)	C11—H11	0.93
N1—H1	0.86	C13—H13A	0.96
C7—C12	1.388 (3)	C13—H13B	0.96
C7—C8	1.397 (3)	C13—H13C	0.96
C1—C3	1.449 (3)	C6—C5	1.325 (4)
C12—C11	1.385 (3)	C6—H6	0.93
C12—H12	0.93	C5—H5	0.93
C3—C4	1.344 (3)		

C2—N2—C7	131.21 (17)	C10—C9—C8	122.1 (2)
C2—N2—H2	114.4	C10—C9—H9	118.9
C7—N2—H2	114.4	C8—C9—H9	118.9
C6—O2—C3	106.19 (18)	C3—C4—C5	106.5 (2)
C1—N1—C2	128.76 (18)	C3—C4—H4	126.7
C1—N1—H1	115.6	C5—C4—H4	126.7
C2—N1—H1	115.6	C11—C10—C9	119.5 (2)
C12—C7—C8	120.07 (19)	C11—C10—H10	120.2
C12—C7—N2	123.93 (19)	C9—C10—H10	120.2
C8—C7—N2	115.95 (17)	C10—C11—C12	120.4 (2)
N2—C2—N1	114.13 (17)	C10—C11—H11	119.8
N2—C2—S1	128.33 (16)	C12—C11—H11	119.8
N1—C2—S1	117.52 (16)	C8—C13—H13A	109.5
O1—C1—N1	123.5 (2)	C8—C13—H13B	109.5
O1—C1—C3	121.03 (19)	H13A—C13—H13B	109.5
N1—C1—C3	115.5 (2)	C8—C13—H13C	109.5
C11—C12—C7	120.0 (2)	H13A—C13—H13C	109.5
C11—C12—H12	120	H13B—C13—H13C	109.5
C7—C12—H12	120	C5—C6—O2	110.5 (2)
C4—C3—O2	109.6 (2)	C5—C6—H6	124.7
C4—C3—C1	132.6 (2)	O2—C6—H6	124.7
O2—C3—C1	117.80 (18)	C6—C5—C4	107.1 (2)
C9—C8—C7	117.88 (19)	C6—C5—H5	126.4
C9—C8—C13	120.0 (2)	C4—C5—H5	126.4
C7—C8—C13	122.15 (19)		

C2—N2—C7—C12	−24.6 (3)	N1—C1—C3—O2	−5.5 (3)
C2—N2—C7—C8	158.1 (2)	C12—C7—C8—C9	1.5 (3)
C7—N2—C2—N1	−177.53 (18)	N2—C7—C8—C9	178.90 (19)
C7—N2—C2—S1	0.8 (3)	C12—C7—C8—C13	−178.4 (2)
C1—N1—C2—N2	8.7 (3)	N2—C7—C8—C13	−1.0 (3)
C1—N1—C2—S1	−169.87 (17)	C7—C8—C9—C10	−0.3 (3)
C2—N1—C1—O1	−6.2 (4)	C13—C8—C9—C10	179.6 (2)
C2—N1—C1—C3	174.14 (18)	O2—C3—C4—C5	0.5 (3)
C8—C7—C12—C11	−1.5 (3)	C1—C3—C4—C5	−178.3 (2)
N2—C7—C12—C11	−178.73 (18)	C8—C9—C10—C11	−0.9 (4)
C6—O2—C3—C4	−0.1 (2)	C9—C10—C11—C12	0.9 (4)
C6—O2—C3—C1	178.91 (18)	C7—C12—C11—C10	0.3 (3)
O1—C1—C3—C4	−6.4 (4)	C3—O2—C6—C5	−0.3 (3)
N1—C1—C3—C4	173.3 (2)	O2—C6—C5—C4	0.6 (3)
O1—C1—C3—O2	174.84 (19)	C3—C4—C5—C6	−0.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	2.26	2.682 (3)	110

N1—H1···S1^{i}	0.86	2.80	3.639 (2)	165
N2—H2···O1	0.86	1.92	2.649 (2)	141

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