



Crystal structure of ethyl 4-(2-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Shaaban K. Mohamed,^{a,b} Joel T. Mague,^c Mehmet Akkurt,^d Ahmed Khodairy^e and Eman A. Ahmed^{e*}

^aChemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, ^bChemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt, ^cDepartment of Chemistry, Tulane University, New Orleans, LA 70118, USA, ^dDepartment of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and ^eChemistry Department, Faculty of Science, Sohag University, 82524 Sohag, Egypt. *Correspondence e-mail: abdala_15@yahoo.com

Received 13 May 2015; accepted 23 May 2015

Edited by H. Ishida, Okayama University, Japan

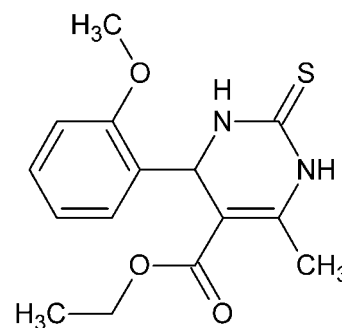
In the title compound, C₁₅H₁₈N₂O₃S, the hydroypyrimidine ring adopts a sofa conformation with the methine C atom as the flap. The benzene ring is almost perpendicular to the mean plane of the hydroypyrimidine ring, making a dihedral angle of 85.51 (8)°, and the methoxy O atom lies over the centre of the pyrimidine ring. In the crystal, weak N—H···S interactions form a zigzag chain running along the *b*-axis direction.

Keywords: crystal structure; Biginelli reactions; dihydropyrimidinones; three-component reactions; N—H···S interactions.

CCDC reference: 1402530

1. Related literature

For syntheses of dihydropyrimidinones and their analogous, see: Biginelli (1893); Varala *et al.* (2003); Gohain *et al.* (2004); Ahmed *et al.* (2009). For biological activities of hydroypyrimidinones, see: Salehi *et al.* (2006); Singh *et al.* (2010); Hed *et al.* (2009); Russowsky *et al.* (2007); Shah *et al.* (2009). For the synthesis of the title compound, see: Ahmed *et al.* (2012).



2. Experimental

2.1. Crystal data

C ₁₅ H ₁₈ N ₂ O ₃ S	$\gamma = 80.850 (1)^\circ$
$M_r = 306.37$	$V = 757.60 (3) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.9791 (2) \text{ \AA}$	Cu $K\alpha$ radiation
$b = 8.2031 (2) \text{ \AA}$	$\mu = 2.00 \text{ mm}^{-1}$
$c = 11.8405 (3) \text{ \AA}$	$T = 150 \text{ K}$
$\alpha = 81.987 (1)^\circ$	$0.25 \times 0.21 \times 0.12 \text{ mm}$
$\beta = 87.975 (1)^\circ$	

2.2. Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer Absorption correction: multi-scan (SADABS; Bruker, 2014) $T_{\min} = 0.73$, $T_{\max} = 0.79$	9145 measured reflections 2929 independent reflections 2773 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.021$
---	---

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	193 parameters
$wR(F^2) = 0.101$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\max} = 0.43 \text{ e \AA}^{-3}$
2929 reflections	$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···S1 ⁱ	0.91	2.46	3.3539 (13)	167
N2—H2A···S1 ⁱⁱ	0.91	2.58	3.4327 (14)	157

Symmetry codes: (i) $-x + 2, -y + 1, -z + 2$; (ii) $-x + 2, -y, -z + 2$.

Data collection: APEX2 (Bruker, 2014); cell refinement: SAINT (Bruker, 2014); data reduction: SAINT; program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: DIAMOND (Brandenburg & Putz, 2012); software used to prepare material for publication: SHELXL2014.

Acknowledgements

The support of NSF–MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5401).

References

- Ahmed, B., Khan, R. A., Habibullah & Keshari, M. (2009). *Tetrahedron Lett.* **50**, 2889–2892.
- Ahmed, E. A., Mohamed, M. A. A. & El-Saghier, A. M. M. (2012). *J. Am. Sci.* **8**, 815–818.
- Biginelli, P. (1893). *Gazz. Chim. Ital.* **23**, 360–416.
- Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2014). *APEX2, SAINT and SADABS*. Bruker AXS, Inc., Madison, Wisconsin, USA.
- Gohain, M., Prajapati, D. & Sandhu, J. S. (2004). *Synlett*, pp. 0235–0238.
- Hed, L. C., Sharma, R., Pareek, C. & Chaudhari, P. B. (2009). *Eur. J. Chem.* **6**, 770–774.
- Russowsky, D., Benvenuti, E. V., Roxo, G. S. & Grasel, F. (2007). *Lett. Org. Chem.* **4**, 39–42.
- Salehi, P., Dabiri, M., Khosropour, A. R. & Roozbehniya, P. (2006). *J. Iran. Chem. Soc.* **3**, 98–104.
- Shah, T. B., Gupte, A., Patel, M. R., Chaudhari, V. S., Patel, H. & Patel, V. C. (2009). *Indian J. Chem. Sect. B.* **48**, 88–96.
- Sheldrick, G. M. (2015a). *Acta Cryst. A* **71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst. C* **71**, 3–8.
- Singh, O. M., Devi, N. S., Devi, L. R. & Khumanthem, N. (2010). *Int. J. Drug Des. Discov.* **1**, 258–264.
- Varala, R., Alam, M. M. & Adapa, S. R. (2003). *Synlett*, pp. 67–70.

supporting information

Acta Cryst. (2015). E71, o444–o445 [doi:10.1107/S2056989015010026]

Crystal structure of ethyl 4-(2-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Shaaban K. Mohamed, Joel T. Mague, Mehmet Akkurt, Ahmed Khodairy and Eman A. Ahmed

S1. Comment

Dihydropyrimidin-2(1*H*)-one scaffold compounds are an important class of substances in organic and medicinal chemistry. Aryl-substituted 3, 4-dihydropyrimidin-2(1*H*)-ones and their sulfur analogue have been reported to possess diverse range of pharmacological activity (Salehi *et al.*, 2006) such as anticancer, anti HIV, antibacterial, antimalarial, antihypertensive, sedative, hypnotics, anticonvulsant, antithyroid, antihistaminic agents and antibiotics (Singh *et al.*, 2010; Hed *et al.*, 2009; Russowsky *et al.*, 2007; Shah *et al.*, 2009). This stimulated the invention of a wide range of synthetic methods for their preparation and chemical transformations. In recent years, several modified procedures have been reported to improve the efficiency of the Biginelli dihydropyrimidine synthesis (Biginelli, 1893) by using different catalysts *e.g.* Lewis acids (Varala *et al.*, 2003; Gohain *et al.*, 2004) or by using basic condition *via* phase transfer catalysis (Ahmed *et al.*, 2009). In this context, we report in this study the crystal structure of the title compound.

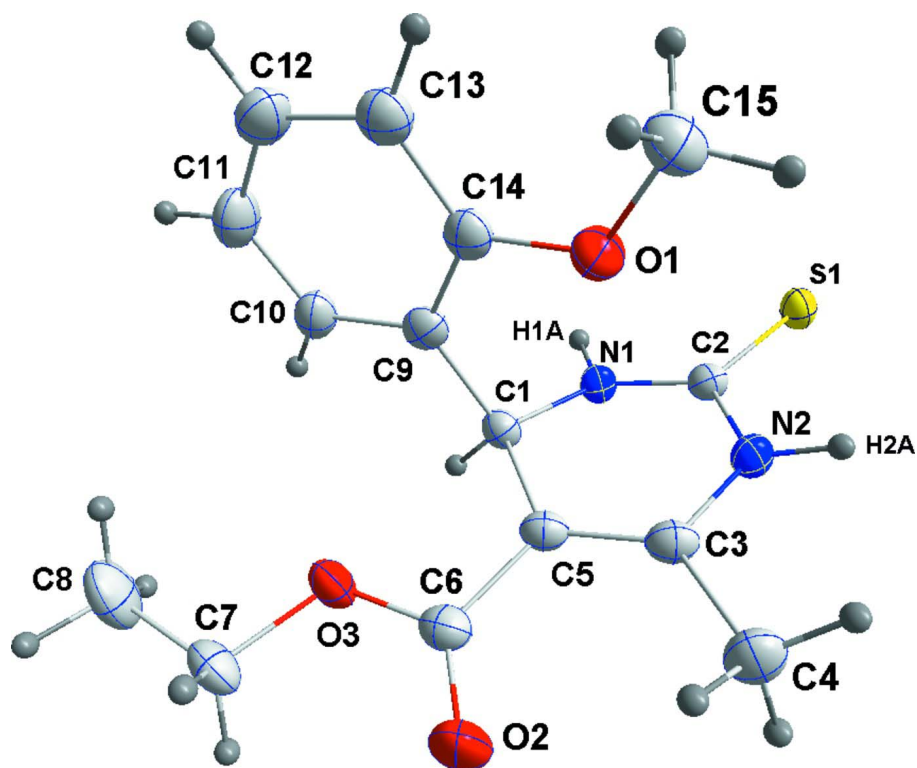
In the title compound (Fig. 1), the plane of the benzene ring is almost parallel to the C1...N2 vector with the methoxy oxygen atom (O1) lying over the centre of the pyrimidine ring. The pyrimidine ring has Cremer-Pople puckering parameters $Q = 0.201(2) \text{ \AA}$, $\theta = 62.2(5)^\circ$ and $\varphi = 42.9(2)^\circ$. In the crystal, weak N—H...S interactions (Table 1) form a chain running parallel to the *b* axis (Figs. 2 & 3).

S2. Experimental

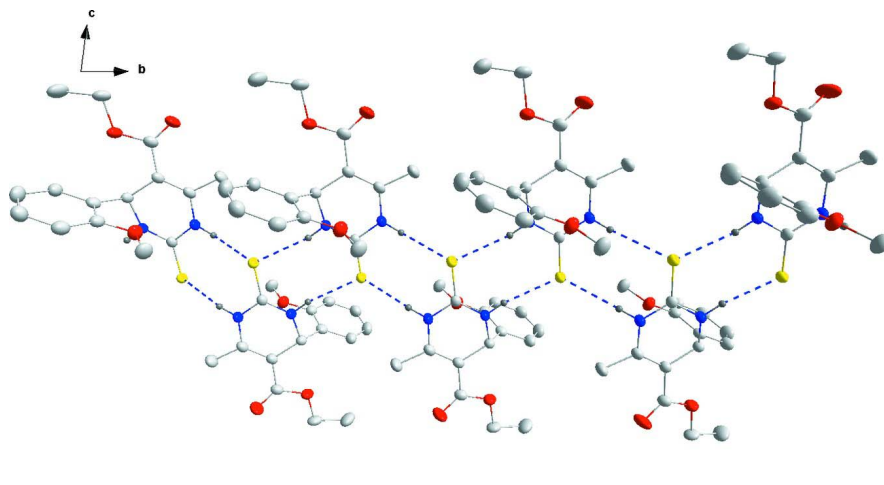
The title compound was prepared according to our reported method (Ahmed *et al.*, 2012). Colourless crystals suitable for X-ray analysis were grown from ethanol (*m.p.* 473–475 K, yield 98%).

S3. Refinement

H-atoms attached to C were placed in calculated positions (C—H = 0.95–1.00 Å), while those attached to N were placed in a difference map and their parameters adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 or 1.5 times those of the attached atoms.

**Figure 1**

The molecular structure of the title compound showing labeling scheme and 50% probability ellipsoids.

**Figure 2**

A section of the chain formed by N—H...S hydrogen bonds (dashed lines).

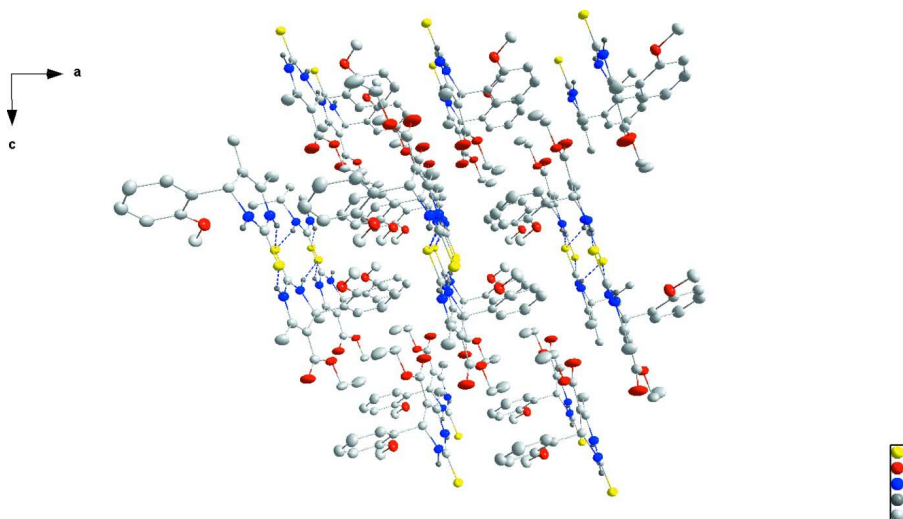


Figure 3

A packing diagram viewed along the *b* axis. N—H...S interactions are shown as dotted lines.

Ethyl 4-(2-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Crystal data

$C_{15}H_{18}N_2O_3S$

$M_r = 306.37$

Triclinic, $P\bar{1}$

$a = 7.9791(2) \text{ \AA}$

$b = 8.2031(2) \text{ \AA}$

$c = 11.8405(3) \text{ \AA}$

$\alpha = 81.987(1)^\circ$

$\beta = 87.975(1)^\circ$

$\gamma = 80.850(1)^\circ$

$V = 757.60(3) \text{ \AA}^3$

$Z = 2$

$F(000) = 324$

$D_x = 1.343 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 7936 reflections

$\theta = 3.8\text{--}72.2^\circ$

$\mu = 2.00 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Thick plate, colourless

$0.25 \times 0.21 \times 0.12 \text{ mm}$

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS
diffractometer

Radiation source: INCOATEC $I\mu S$ micro-focus
source

Mirror monochromator

Detector resolution: $10.4167 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.73$, $T_{\max} = 0.79$

9145 measured reflections

2929 independent reflections

2773 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 72.2^\circ$, $\theta_{\min} = 3.8^\circ$

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.101$

$S = 1.08$

2929 reflections

193 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.4254P]$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$$

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.

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) while those attached to nitrogen were placed in locations derived from a difference map and their parameters adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	1.08339 (5)	0.22865 (4)	1.02799 (3)	0.02400 (13)
O1	0.64475 (15)	0.28798 (15)	0.86937 (10)	0.0312 (3)
O2	0.8568 (2)	0.26940 (18)	0.47910 (11)	0.0494 (4)
O3	0.75790 (16)	0.52294 (15)	0.52331 (9)	0.0304 (3)
N1	0.96037 (16)	0.42742 (15)	0.84338 (11)	0.0213 (3)
H1A	0.9617	0.5109	0.8864	0.026*
N2	0.99441 (17)	0.14788 (16)	0.83198 (11)	0.0240 (3)
H2A	1.0080	0.0431	0.8710	0.029*
C1	0.85934 (19)	0.47576 (19)	0.73835 (13)	0.0216 (3)
H1	0.9084	0.5685	0.6916	0.026*
C2	1.00503 (19)	0.27318 (19)	0.89394 (13)	0.0209 (3)
C3	0.9414 (2)	0.1739 (2)	0.71936 (13)	0.0247 (3)
C4	0.9585 (3)	0.0158 (2)	0.66684 (15)	0.0351 (4)
H4A	0.8722	0.0271	0.6082	0.053*
H4B	0.9429	-0.0770	0.7259	0.053*
H4C	1.0717	-0.0061	0.6320	0.053*
C5	0.88282 (19)	0.3296 (2)	0.66990 (13)	0.0230 (3)
C6	0.8339 (2)	0.3648 (2)	0.54883 (14)	0.0277 (3)
C7	0.6888 (2)	0.5701 (2)	0.40933 (14)	0.0331 (4)
H7A	0.6051	0.4980	0.3959	0.040*
H7B	0.7806	0.5588	0.3512	0.040*
C8	0.6050 (3)	0.7480 (3)	0.40272 (18)	0.0486 (5)
H8A	0.5172	0.7580	0.4625	0.073*
H8B	0.5529	0.7841	0.3278	0.073*
H8C	0.6900	0.8183	0.4136	0.073*
C9	0.6761 (2)	0.5437 (2)	0.76670 (13)	0.0255 (3)
C10	0.6100 (2)	0.7098 (2)	0.72635 (15)	0.0304 (4)
H10	0.6797	0.7785	0.6817	0.036*
C11	0.4425 (3)	0.7744 (2)	0.75156 (17)	0.0382 (4)
H11	0.3972	0.8861	0.7228	0.046*

C12	0.3434 (2)	0.6753 (2)	0.81827 (16)	0.0370 (4)
H12	0.2298	0.7205	0.8357	0.044*
C13	0.4043 (2)	0.5117 (2)	0.86057 (15)	0.0320 (4)
H13	0.3342	0.4452	0.9068	0.038*
C14	0.5720 (2)	0.4459 (2)	0.83384 (13)	0.0269 (3)
C15	0.5435 (2)	0.1762 (2)	0.93170 (16)	0.0361 (4)
H15A	0.4988	0.2205	1.0013	0.054*
H15B	0.6133	0.0671	0.9521	0.054*
H15C	0.4489	0.1642	0.8845	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0304 (2)	0.0195 (2)	0.0222 (2)	-0.00308 (15)	-0.00427 (14)	-0.00340 (14)
O1	0.0296 (6)	0.0277 (6)	0.0355 (6)	-0.0073 (5)	0.0044 (5)	0.0011 (5)
O2	0.0744 (10)	0.0443 (8)	0.0274 (7)	0.0095 (7)	-0.0101 (6)	-0.0165 (6)
O3	0.0391 (7)	0.0314 (6)	0.0209 (6)	-0.0055 (5)	-0.0058 (5)	-0.0026 (5)
N1	0.0255 (6)	0.0180 (6)	0.0217 (6)	-0.0048 (5)	-0.0032 (5)	-0.0046 (5)
N2	0.0309 (7)	0.0181 (6)	0.0238 (6)	-0.0034 (5)	-0.0014 (5)	-0.0056 (5)
C1	0.0237 (7)	0.0218 (7)	0.0202 (7)	-0.0052 (6)	-0.0020 (6)	-0.0033 (6)
C2	0.0202 (7)	0.0206 (7)	0.0227 (7)	-0.0045 (6)	0.0013 (6)	-0.0044 (6)
C3	0.0261 (8)	0.0261 (8)	0.0241 (8)	-0.0069 (6)	0.0019 (6)	-0.0084 (6)
C4	0.0497 (11)	0.0273 (9)	0.0309 (9)	-0.0059 (8)	-0.0026 (8)	-0.0121 (7)
C5	0.0235 (7)	0.0256 (8)	0.0217 (7)	-0.0063 (6)	0.0014 (6)	-0.0067 (6)
C6	0.0278 (8)	0.0332 (9)	0.0233 (8)	-0.0059 (7)	0.0010 (6)	-0.0066 (6)
C7	0.0370 (9)	0.0430 (10)	0.0199 (8)	-0.0104 (8)	-0.0046 (7)	0.0000 (7)
C8	0.0683 (14)	0.0410 (11)	0.0346 (10)	-0.0088 (10)	-0.0160 (10)	0.0062 (8)
C9	0.0268 (8)	0.0292 (8)	0.0222 (7)	-0.0048 (6)	-0.0031 (6)	-0.0081 (6)
C10	0.0335 (9)	0.0276 (8)	0.0289 (8)	0.0007 (7)	-0.0053 (7)	-0.0055 (7)
C11	0.0398 (10)	0.0284 (9)	0.0436 (10)	0.0035 (8)	-0.0066 (8)	-0.0041 (8)
C12	0.0352 (9)	0.0377 (10)	0.0367 (9)	0.0019 (8)	0.0000 (7)	-0.0094 (8)
C13	0.0320 (9)	0.0368 (9)	0.0278 (8)	-0.0069 (7)	-0.0017 (7)	-0.0049 (7)
C14	0.0294 (8)	0.0286 (8)	0.0233 (8)	-0.0044 (7)	-0.0036 (6)	-0.0049 (6)
C15	0.0366 (10)	0.0323 (9)	0.0386 (10)	-0.0106 (8)	0.0047 (8)	0.0033 (8)

Geometric parameters (Å, °)

S1—C2	1.6969 (15)	C5—C6	1.476 (2)
O1—C14	1.349 (2)	C7—C8	1.498 (3)
O1—C15	1.430 (2)	C7—H7A	0.9900
O2—C6	1.205 (2)	C7—H7B	0.9900
O3—C6	1.340 (2)	C8—H8A	0.9800
O3—C7	1.4541 (19)	C8—H8B	0.9800
N1—C2	1.322 (2)	C8—H8C	0.9800
N1—C1	1.4783 (18)	C9—C14	1.397 (2)
N1—H1A	0.9098	C9—C10	1.403 (2)
N2—C2	1.3580 (19)	C10—C11	1.395 (3)
N2—C3	1.391 (2)	C10—H10	0.9500

N2—H2A	0.9098	C11—C12	1.376 (3)
C1—C5	1.522 (2)	C11—H11	0.9500
C1—C9	1.523 (2)	C12—C13	1.382 (3)
C1—H1	1.0000	C12—H12	0.9500
C3—C5	1.347 (2)	C13—C14	1.403 (2)
C3—C4	1.501 (2)	C13—H13	0.9500
C4—H4A	0.9800	C15—H15A	0.9800
C4—H4B	0.9800	C15—H15B	0.9800
C4—H4C	0.9800	C15—H15C	0.9800
C14—O1—C15	118.77 (14)	C8—C7—H7A	110.4
C6—O3—C7	116.45 (13)	O3—C7—H7B	110.4
C2—N1—C1	125.30 (12)	C8—C7—H7B	110.4
C2—N1—H1A	117.1	H7A—C7—H7B	108.6
C1—N1—H1A	114.7	C7—C8—H8A	109.5
C2—N2—C3	123.59 (14)	C7—C8—H8B	109.5
C2—N2—H2A	116.2	H8A—C8—H8B	109.5
C3—N2—H2A	119.5	C7—C8—H8C	109.5
N1—C1—C5	108.79 (12)	H8A—C8—H8C	109.5
N1—C1—C9	110.68 (12)	H8B—C8—H8C	109.5
C5—C1—C9	115.38 (12)	C14—C9—C10	118.77 (16)
N1—C1—H1	107.2	C14—C9—C1	121.76 (15)
C5—C1—H1	107.2	C10—C9—C1	119.46 (15)
C9—C1—H1	107.2	C11—C10—C9	120.32 (17)
N1—C2—N2	117.28 (14)	C11—C10—H10	119.8
N1—C2—S1	122.70 (11)	C9—C10—H10	119.8
N2—C2—S1	119.94 (12)	C12—C11—C10	119.55 (17)
C5—C3—N2	119.62 (14)	C12—C11—H11	120.2
C5—C3—C4	127.50 (15)	C10—C11—H11	120.2
N2—C3—C4	112.89 (14)	C11—C12—C13	121.84 (17)
C3—C4—H4A	109.5	C11—C12—H12	119.1
C3—C4—H4B	109.5	C13—C12—H12	119.1
H4A—C4—H4B	109.5	C12—C13—C14	118.62 (17)
C3—C4—H4C	109.5	C12—C13—H13	120.7
H4A—C4—H4C	109.5	C14—C13—H13	120.7
H4B—C4—H4C	109.5	O1—C14—C9	115.19 (15)
C3—C5—C6	121.63 (14)	O1—C14—C13	123.92 (15)
C3—C5—C1	120.93 (14)	C9—C14—C13	120.89 (16)
C6—C5—C1	117.42 (14)	O1—C15—H15A	109.5
O2—C6—O3	122.34 (16)	O1—C15—H15B	109.5
O2—C6—C5	126.96 (16)	H15A—C15—H15B	109.5
O3—C6—C5	110.70 (13)	O1—C15—H15C	109.5
O3—C7—C8	106.67 (14)	H15A—C15—H15C	109.5
O3—C7—H7A	110.4	H15B—C15—H15C	109.5
C2—N1—C1—C5	-25.4 (2)	C3—C5—C6—O3	171.37 (14)
C2—N1—C1—C9	102.32 (16)	C1—C5—C6—O3	-6.8 (2)
C1—N1—C2—N2	16.9 (2)	C6—O3—C7—C8	177.69 (16)

C1—N1—C2—S1	-166.47 (11)	N1—C1—C9—C14	-61.89 (18)
C3—N2—C2—N1	0.7 (2)	C5—C1—C9—C14	62.18 (19)
C3—N2—C2—S1	-176.02 (12)	N1—C1—C9—C10	116.92 (15)
C2—N2—C3—C5	-6.0 (2)	C5—C1—C9—C10	-119.01 (16)
C2—N2—C3—C4	173.96 (15)	C14—C9—C10—C11	-1.1 (2)
N2—C3—C5—C6	176.60 (14)	C1—C9—C10—C11	-179.90 (15)
C4—C3—C5—C6	-3.3 (3)	C9—C10—C11—C12	1.3 (3)
N2—C3—C5—C1	-5.3 (2)	C10—C11—C12—C13	-0.7 (3)
C4—C3—C5—C1	174.72 (16)	C11—C12—C13—C14	-0.3 (3)
N1—C1—C5—C3	18.7 (2)	C15—O1—C14—C9	-175.65 (14)
C9—C1—C5—C3	-106.35 (17)	C15—O1—C14—C13	4.3 (2)
N1—C1—C5—C6	-163.15 (13)	C10—C9—C14—O1	-179.93 (14)
C9—C1—C5—C6	71.79 (18)	C1—C9—C14—O1	-1.1 (2)
C7—O3—C6—O2	5.8 (2)	C10—C9—C14—C13	0.1 (2)
C7—O3—C6—C5	-174.41 (13)	C1—C9—C14—C13	178.93 (14)
C3—C5—C6—O2	-8.9 (3)	C12—C13—C14—O1	-179.41 (16)
C1—C5—C6—O2	172.98 (18)	C12—C13—C14—C9	0.5 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1 <i>A</i> ...S1 ⁱ	0.91	2.46	3.3539 (13)	167
N2—H2 <i>A</i> ...S1 ⁱⁱ	0.91	2.58	3.4327 (14)	157

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $-x+2, -y, -z+2$.