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Crystal structure of methyl (*Z*)-2-[(*Z*)-3-methyl-2-({(*E*)-1-[(*R**)-4-methylcyclohex-3-en-1-yl]ethylidene}hydrazinylidene)-4-oxothiazolidin-5-ylidene]acetate

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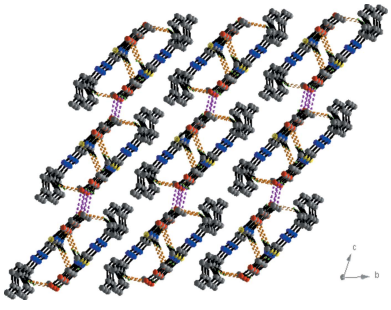
The new title 4-thiazolidinone derivative, $C_{16}H_{21}N_3O_3S$, was obtained from the cyclization reaction of 4-methyl-3-thiosemicarbazone and dimethyl acetylenedicarboxylate (DMAD). The cyclohexylidene ring has an envelope conformation with the stereogenic centre C atom as the flap. Its mean plane makes a dihedral angle of 56.23 (9)° with the thiazolidine ring mean plane. In the crystal, molecules are linked by C—H···O hydrogen bonds forming chains propagating in the [001] direction. Within the chains there are offset π–π interactions between the thiazolidine rings of inversion-related molecules [centroid–centroid distance = 3.703 (1) Å]. The chains are linked by further C—H···O hydrogen bonds, forming slabs parallel to the *ac* plane.

1. Chemical context

It has been reported that thiazolidinones exhibit antibacterial (Mayekar & Mulwad, 2008), antifungal (Omar *et al.* 2010), anticonvulsant (Bhat *et al.*, 2008), antitubercular (Babaoglu *et al.*, 2003), anti-inflammatory (Vigorita *et al.* 2003), anti-histaminic (Agrawal *et al.*, 2000), cardiovascular (Suzuki *et al.*, 1999) and anti-HIV (Rawal *et al.*, 2005) activities.

With the aim of preparing new thiazolidinone derivatives, we report herein on the synthesis (Fig. 1) and crystal structure of the title compound **3**, from 4-methyl-3-thiosemicarbazone **1**. Treatment of **1** with dimethyl acetylenedicarboxylate **2** in boiling ethanol for 1 h, afforded the thiazolidin-4-one **3** in 90% yield. Its structure has also been fully characterized by NMR spectroscopy while its relative stereochemistry was determined based mainly on the synthetic pathway and implied by the X-ray diffraction analysis.



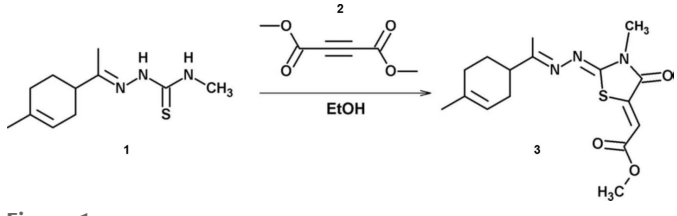
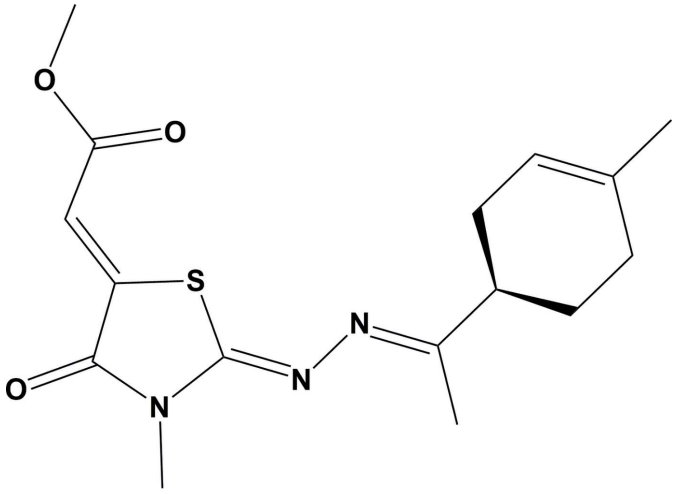


Figure 1
Reaction scheme for the synthesis of title compound **3**.



3

2. Structural commentary

The title compound **3**, is built up from an thiazolidinone ring linked to cyclohexylidene-hydrazone and methoxy-oxoethylidene units (Fig. 2). The compound crystallizes in the centrosymmetric space group $P\overline{1}$, and the stereogenic centre at C8 was assigned as having an *R* configuration. As expected, the thiazolidine ring and all the atoms attached to it (plane *A* = S1/C4/C5/N1/C6/N2/N3/O1/C3/C14) are roughly coplanar with an r.m.s. deviation of 0.036 Å. Its mean plane makes a dihedral angle of 56.0 (1)° with the mean plane of the cyclohexylidene ring (C8-C13). The methoxycarbonyl group (C1/O2/O3/C2) is also twisted slightly with respect to plane *A*, their mean planes being inclined to one another by 11.2 (2)°. The six-membered cyclohexylidene ring has an envelope conformation with atom C8 as the flap: puckering parameters are Q = 0.494 (2) Å, θ = 129.8 (2)° and φ = 180.8 (3)°. The C7═N3 and N2═C6 bond lengths are 1.282 (2) and 1.278 (2) Å, respectively, consistent with C═N double bonding. The C6—N2—N3—C7, C4—C3—C2—O3 and C3—C2—O3—C1 torsion angles are 175.5 (2), −172.4 (2) and 172.5 (2)°, respectively.

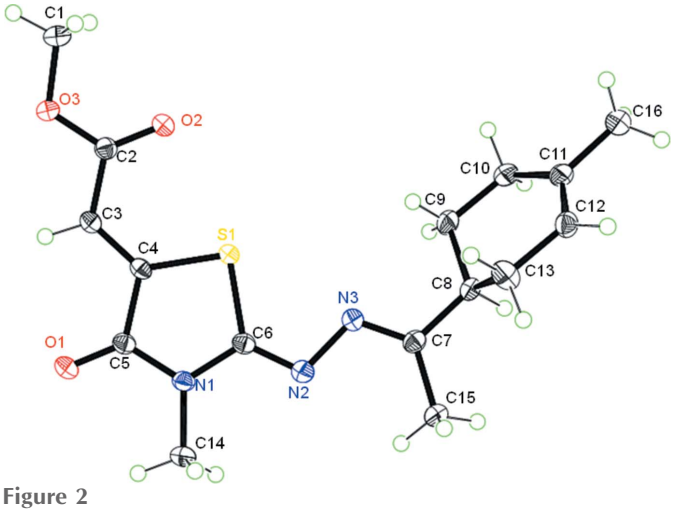


Figure 2
The molecular structure of the title compound **3**, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.95	2.59	3.4133 (19)	146
C1—H1*C*···O1^{ii}	0.98	2.51	3.208 (2)	128
C14—H14*A*···O2^{iii}	0.98	2.45	3.252 (2)	139
C14—H14*B*···O2^{iv}	0.98	2.48	3.409 (2)	159
C15—H15*A*···O3^{iv}	0.98	2.55	3.351 (2)	139

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

3. Supramolecular features

In the crystal, molecules are linked C3—H3···O3^{i} hydrogen bonds, forming chains propagating along [001]; see Table 1 and Fig. 3. Within the chains there are weak offset π–π stacking interactions between inversion-related thiazole rings [see Fig. 3; *Cg*1···*Cg*1($-x+1, -y+1, -z$) = 3.703 (1) Å,where *Cg*1 is the centroid of the S1/N1/C4–C6 ring, interplanar distance = 3.468 (1) Å, slippage = 1.298 Å]. The chains are linked by further C—H···O hydrogen bonds, forming slabs lying parallel to the *ac* plane (Table 1, Figs. 4 and 5).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.38, last update May 2017; Groom *et al.*, 2016) using a thiazolidone substituted by methoxy-oxoethyllidene and methylhydrazone as the main skeleton gave eight hits. The most relevant structures are methyl (2-{[1-(4-hydroxyphenyl)ethylidene]hydrazono}-4-oxo-3-phenyl-1,3-thiazolidin-5-ylidene)acetate (AGOMUG; Mohamed, Mague *et al.*, 2013), methyl (2-{[1-(4-methylphenyl)ethylidene]hydrazono}-4-oxo-3-phenyl-1,3-thiazolidin-5-ylidene)acetate (NIPPAF; Mague *et al.*, 2013) and dimethyl 2-[(4-{*N*-[5-(2-methoxy-2-oxoethylidene)-4-oxo-3-phenyl-1,3-thiazolidin-2-ylidene]ethanehydrazonoyl}phenyl)amino]but-2-enedioate (RIMDIC; Mohamed, Akkurt *et al.*, 2013).

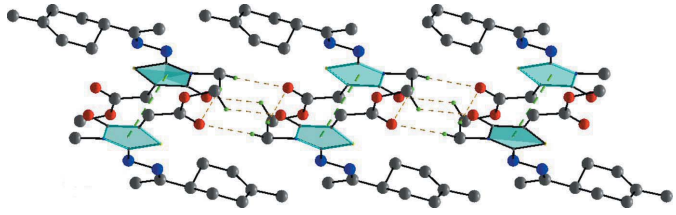


Figure 3
Partial crystal packing for title compound **3**, showing the C3—H3···O3^{i} hydrogen bonds and the offset π–π interactions between inversion-related molecules, forming chains in the [001] direction (dashed lines; only atom H3 has been included).

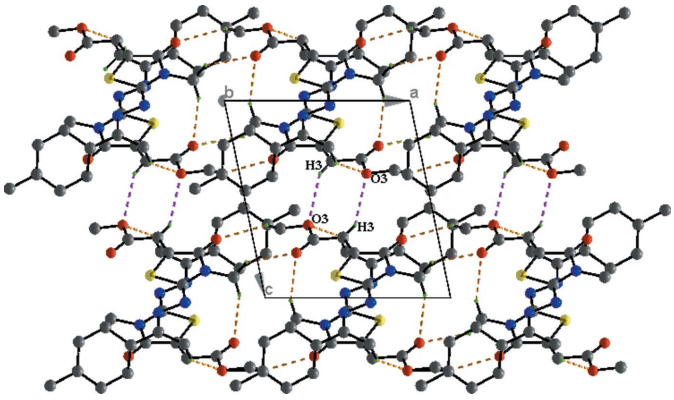


Figure 4
Packing and hydrogen-bonding interactions of the title compound viewed along the *b* axis. For clarity, only the H atoms involved in the hydrogen bonds (dashed lines) interactions have been included.

A comparison of the main C—N, N—N, C—S bond lengths in the title compound and the structures extracted from the CSD shows a good correlation. The C═N—N═C torsion angles indicate that in each case the four atoms are nearly planar, *viz*. 175.5 (2)° in the title compound, 172.1 (2)° in AGOMUG, −178.9 (2) and −165.5 (2)° in NIPPAF and −167.4 (5)° in RIMDIC.

5. Synthesis and crystallization

To a solution of 4-methyl-3-thiosemicarbazone (200 mg, 1.33 mmol) in ethanol (15 ml) was added dimethyl acetylenedicarboxylate (DMAD) (0.24 ml, 1.66 mmol). The mixture was stirred under reflux for 1 h, leading to the corresponding thiazolidinone. After cooling, the mixture was extracted with ethyl acetate (3 × 20 ml). The organic layer was washed with water, dried on anhydrous Na_2SO_4 and then evaporated under reduced pressure. The obtained residue was chromatographed on a silica gel column using hexane as eluent, to give compound **3** (yield 404 mg, 90%). Yellow prismatic crystals were obtained from a petroleum ether solution, by slow evaporation of the solvent at room temperature.

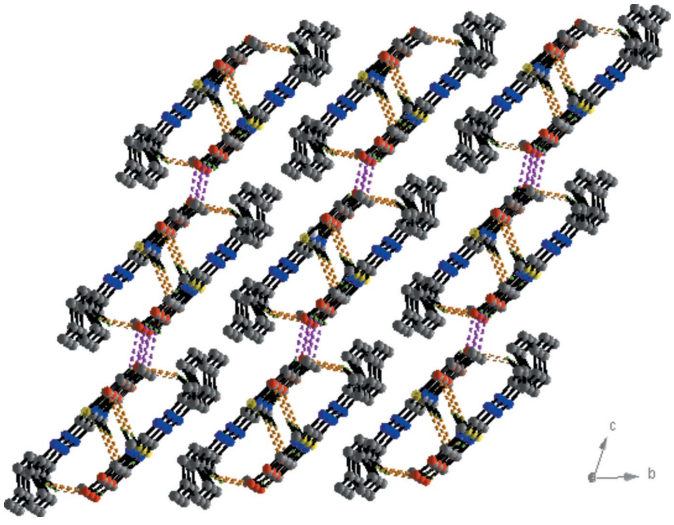


Figure 5
Packing and hydrogen-bonding interactions of the title compound, viewed along the *a* axis. For clarity, only the H atoms involved in hydrogen bonding (dashed lines) have been included.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{21}N_3O_3S$
M_r	335.42
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	9.0982 (2), 9.9556 (3), 10.5071 (3)
α, β, γ (°)	66.772 (1), 74.572 (1), 77.706 (1)
V (Å^3)	836.76 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.88
Crystal size (mm)	0.39 × 0.28 × 0.20
Data collection	
Diffractometer	D8 Venture CMOS area detector
Absorption correction	Numerical (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.733, 0.919
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30681, 3405, 3195
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.102, 1.06
No. of reflections	3405
No. of parameters	212
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.46

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg & Putz, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C-bound H atoms were placed in calculated positions with C—H = 0.95–1.00 Å, and refined in the riding-model approximation with U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{eq}(C) for other H atoms.

Funding information

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supporting information

Acta Cryst. (2017). E**73**, 1626-1629 [https://doi.org/10.1107/S2056989017014311]

Crystal structure of methyl (*Z*)-2-[(*Z*)-3-methyl-2-({(*E*)-1-[(*R**)-4-methylcyclohex-3-en-1-yl]ethylidene}hydrazinylidene)-4-oxothiazolidin-5-ylidene]acetate

Mourad Fawzi, Aziz Auhmani, Moulay Youssef Ait Itto, Abdelkhalek Riahi, Sylviane Chevreux and El Mostafa Ketatni

Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

(*Z*)-2-[(*Z*)-3-Methyl-2-({(*E*)-1-[(*R)-4-methylcyclohex-3-en-1-yl]ethylidene}hydrazinylidene)-4-oxothiazolidin-5-ylidene]acetate**

Crystal data

$C_{16}H_{21}N_3O_3S$
$M_r = 335.42$
Triclinic, $P\overline{1}$
$a = 9.0982$ (2) Å
$b = 9.9556$ (3) Å
$c = 10.5071$ (3) Å
$\alpha = 66.772$ (1)°
$\beta = 74.572$ (1)°
$\gamma = 77.706$ (1)°
$V = 836.76$ (4) Å^3
$Z = 2$
$F(000) = 356$
$D_x = 1.331$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9903 reflections
$\theta = 4.9$–$74.5°$
$\mu = 1.88$ mm^{-1}
$T = 100$ K
Prismatic, yellow
0.39 × 0.28 × 0.20 mm

Data collection

D8 Venture CMOS area detector diffractometer
Radiation source: microsource
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2012)
$T_{min} = 0.733$, $T_{max} = 0.919$
30681 measured reflections
3405 independent reflections
3195 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 74.5°$, $\theta_{min} = 4.7°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.06$
3405 reflections
212 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.6482P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.016$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.40715 (4)	0.68958 (4)	−0.11410 (4)	0.01854 (12)
O1	0.80122 (13)	0.54559 (13)	−0.29654 (13)	0.0255 (3)
O2	0.21903 (13)	0.59108 (14)	−0.22869 (13)	0.0262 (3)
O3	0.31331 (13)	0.45665 (12)	−0.36926 (12)	0.0219 (3)
N1	0.70665 (15)	0.66298 (14)	−0.13721 (14)	0.0192 (3)
N2	0.56362 (16)	0.77397 (15)	0.02334 (15)	0.0222 (3)
N3	0.40773 (15)	0.81672 (15)	0.07586 (15)	0.0215 (3)
C1	0.15960 (19)	0.4245 (2)	−0.3520 (2)	0.0267 (4)
H1A	0.1267	0.3563	−0.2554	0.040*
H1B	0.1594	0.3793	−0.4196	0.040*
H1C	0.0887	0.5160	−0.3692	0.040*
C2	0.32641 (18)	0.53614 (17)	−0.29626 (17)	0.0194 (3)
C3	0.48814 (18)	0.54559 (17)	−0.30630 (17)	0.0205 (3)
H3	0.5657	0.5084	−0.3690	0.025*
C4	0.52821 (17)	0.60574 (17)	−0.22856 (17)	0.0184 (3)
C5	0.69402 (18)	0.59946 (17)	−0.22780 (17)	0.0193 (3)
C6	0.56930 (18)	0.71493 (17)	−0.06627 (17)	0.0189 (3)
C7	0.38679 (18)	0.86854 (17)	0.17420 (17)	0.0201 (3)
C8	0.22330 (18)	0.90988 (17)	0.23980 (17)	0.0200 (3)
H8	0.2202	1.0019	0.2579	0.024*
C9	0.1056 (2)	0.9394 (2)	0.14898 (19)	0.0281 (4)
H9A	0.1337	1.0203	0.0579	0.034*
H9B	0.1069	0.8502	0.1285	0.034*
C10	−0.0542 (2)	0.9808 (2)	0.22322 (18)	0.0263 (4)
H10	−0.1269	1.0467	0.1702	0.032*
C11	−0.0941 (2)	0.92015 (19)	0.3727 (2)	0.0259 (4)
C12	0.01418 (19)	0.83015 (18)	0.45599 (18)	0.0235 (3)
H12A	−0.0295	0.7375	0.5188	0.028*
H12B	0.0236	0.8805	0.5175	0.028*
C13	0.1742 (2)	0.7892 (2)	0.38279 (19)	0.0289 (4)
H13A	0.1778	0.6962	0.3681	0.035*
H13B	0.2474	0.7725	0.4440	0.035*
C14	0.85481 (18)	0.66081 (19)	−0.10627 (19)	0.0243 (4)
H14A	0.9379	0.6357	−0.1775	0.036*

H14B	0.8625	0.5871	−0.0124	0.036*
H14C	0.8633	0.7581	−0.1082	0.036*
C15	0.51144 (19)	0.88538 (19)	0.23429 (18)	0.0239 (3)
H15A	0.5290	0.7966	0.3162	0.036*
H15B	0.4803	0.9709	0.2638	0.036*
H15C	0.6064	0.8996	0.1621	0.036*
C16	−0.2564 (2)	0.9523 (2)	0.4458 (2)	0.0317 (4)
H16A	−0.3170	0.8763	0.4573	0.048*
H16B	−0.3016	1.0488	0.3888	0.048*
H16C	−0.2564	0.9526	0.5390	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01467 (19)	0.0227 (2)	0.0196 (2)	−0.00267 (14)	−0.00178 (14)	−0.00991 (15)
O1	0.0160 (5)	0.0333 (6)	0.0293 (7)	−0.0041 (5)	0.0009 (5)	−0.0163 (5)
O2	0.0173 (6)	0.0360 (7)	0.0308 (7)	−0.0030 (5)	−0.0017 (5)	−0.0198 (5)
O3	0.0184 (5)	0.0264 (6)	0.0251 (6)	−0.0044 (4)	−0.0042 (4)	−0.0130 (5)
N1	0.0152 (6)	0.0218 (6)	0.0207 (7)	−0.0042 (5)	−0.0028 (5)	−0.0073 (5)
N2	0.0195 (7)	0.0258 (7)	0.0221 (7)	−0.0038 (5)	−0.0033 (5)	−0.0097 (6)
N3	0.0195 (7)	0.0250 (7)	0.0217 (7)	−0.0035 (5)	−0.0025 (5)	−0.0110 (6)
C1	0.0207 (8)	0.0324 (9)	0.0324 (10)	−0.0078 (7)	−0.0060 (7)	−0.0144 (8)
C2	0.0196 (8)	0.0206 (7)	0.0179 (8)	−0.0039 (6)	−0.0031 (6)	−0.0063 (6)
C3	0.0170 (7)	0.0240 (8)	0.0206 (8)	−0.0040 (6)	−0.0001 (6)	−0.0097 (6)
C4	0.0149 (7)	0.0197 (7)	0.0185 (8)	−0.0035 (6)	−0.0002 (6)	−0.0060 (6)
C5	0.0167 (7)	0.0206 (7)	0.0191 (8)	−0.0048 (6)	−0.0011 (6)	−0.0057 (6)
C6	0.0172 (7)	0.0190 (7)	0.0187 (8)	−0.0036 (6)	−0.0030 (6)	−0.0045 (6)
C7	0.0228 (8)	0.0196 (7)	0.0182 (8)	−0.0043 (6)	−0.0052 (6)	−0.0055 (6)
C8	0.0224 (8)	0.0216 (7)	0.0184 (8)	−0.0029 (6)	−0.0050 (6)	−0.0092 (6)
C9	0.0258 (9)	0.0407 (10)	0.0236 (9)	0.0015 (7)	−0.0088 (7)	−0.0180 (8)
C10	0.0229 (8)	0.0360 (9)	0.0225 (9)	−0.0026 (7)	−0.0083 (7)	−0.0111 (7)
C11	0.0227 (8)	0.0282 (8)	0.0349 (10)	−0.0060 (7)	−0.0035 (7)	−0.0197 (8)
C12	0.0267 (8)	0.0240 (8)	0.0210 (8)	−0.0058 (6)	−0.0024 (6)	−0.0096 (7)
C13	0.0260 (9)	0.0283 (9)	0.0239 (9)	0.0004 (7)	−0.0038 (7)	−0.0032 (7)
C14	0.0165 (8)	0.0305 (9)	0.0277 (9)	−0.0028 (6)	−0.0062 (6)	−0.0114 (7)
C15	0.0232 (8)	0.0284 (8)	0.0247 (8)	−0.0027 (6)	−0.0074 (7)	−0.0130 (7)
C16	0.0241 (9)	0.0427 (10)	0.0343 (10)	−0.0031 (7)	−0.0046 (7)	−0.0215 (9)

Geometric parameters (Å, º)

S1—C4	1.7469 (16)	C8—H8	1.0000
S1—C6	1.7736 (16)	C9—C10	1.512 (2)
O1—C5	1.212 (2)	C9—H9A	0.9900
O2—C2	1.211 (2)	C9—H9B	0.9900
O3—C2	1.3403 (19)	C10—C11	1.416 (3)
O3—C1	1.4495 (19)	C10—H10	0.9500
N1—C5	1.372 (2)	C11—C12	1.415 (2)
N1—C6	1.385 (2)	C11—C16	1.504 (2)

N1—C14	1.462 (2)	C12—C13	1.509 (2)
N2—C6	1.278 (2)	C12—H12A	0.9900
N2—N3	1.4174 (19)	C12—H12B	0.9900
N3—C7	1.282 (2)	C13—H13A	0.9900
C1—H1A	0.9800	C13—H13B	0.9900
C1—H1B	0.9800	C14—H14A	0.9800
C1—H1C	0.9800	C14—H14B	0.9800
C2—C3	1.467 (2)	C14—H14C	0.9800
C3—C4	1.340 (2)	C15—H15A	0.9800
C3—H3	0.9500	C15—H15B	0.9800
C4—C5	1.499 (2)	C15—H15C	0.9800
C7—C15	1.503 (2)	C16—H16A	0.9800
C7—C8	1.508 (2)	C16—H16B	0.9800
C8—C9	1.527 (2)	C16—H16C	0.9800
C8—C13	1.532 (2)		

C4—S1—C6	90.05 (7)	C10—C9—H9B	109.4
C2—O3—C1	115.77 (13)	C8—C9—H9B	109.4
C5—N1—C6	115.69 (13)	H9A—C9—H9B	108.0
C5—N1—C14	121.75 (13)	C11—C10—C9	119.25 (15)
C6—N1—C14	122.23 (13)	C11—C10—H10	120.4
C6—N2—N3	108.81 (13)	C9—C10—H10	120.4
C7—N3—N2	114.54 (13)	C12—C11—C10	122.07 (16)
O3—C1—H1A	109.5	C12—C11—C16	118.75 (16)
O3—C1—H1B	109.5	C10—C11—C16	119.18 (16)
H1A—C1—H1B	109.5	C11—C12—C13	118.89 (15)
O3—C1—H1C	109.5	C11—C12—H12A	107.6
H1A—C1—H1C	109.5	C13—C12—H12A	107.6
H1B—C1—H1C	109.5	C11—C12—H12B	107.6
O2—C2—O3	124.53 (14)	C13—C12—H12B	107.6
O2—C2—C3	124.24 (15)	H12A—C12—H12B	107.0
O3—C2—C3	111.22 (13)	C12—C13—C8	111.68 (14)
C4—C3—C2	121.12 (14)	C12—C13—H13A	109.3
C4—C3—H3	119.4	C8—C13—H13A	109.3
C2—C3—H3	119.4	C12—C13—H13B	109.3
C3—C4—C5	120.42 (14)	C8—C13—H13B	109.3
C3—C4—S1	127.81 (12)	H13A—C13—H13B	107.9
C5—C4—S1	111.70 (11)	N1—C14—H14A	109.5
O1—C5—N1	124.96 (14)	N1—C14—H14B	109.5
O1—C5—C4	125.02 (15)	H14A—C14—H14B	109.5
N1—C5—C4	110.01 (13)	N1—C14—H14C	109.5
N2—C6—N1	122.47 (14)	H14A—C14—H14C	109.5
N2—C6—S1	125.00 (12)	H14B—C14—H14C	109.5
N1—C6—S1	112.53 (11)	C7—C15—H15A	109.5
N3—C7—C15	125.43 (15)	C7—C15—H15B	109.5
N3—C7—C8	117.47 (14)	H15A—C15—H15B	109.5
C15—C7—C8	117.04 (14)	C7—C15—H15C	109.5
C7—C8—C9	115.06 (13)	H15A—C15—H15C	109.5

C7—C8—C13	109.94 (13)	H15B—C15—H15C	109.5
C9—C8—C13	108.72 (14)	C11—C16—H16A	109.5
C7—C8—H8	107.6	C11—C16—H16B	109.5
C9—C8—H8	107.6	H16A—C16—H16B	109.5
C13—C8—H8	107.6	C11—C16—H16C	109.5
C10—C9—C8	111.08 (14)	H16A—C16—H16C	109.5
C10—C9—H9A	109.4	H16B—C16—H16C	109.5
C8—C9—H9A	109.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.95	2.59	3.4133 (19)	146
C1—H1*C*···O1^{ii}	0.98	2.51	3.208 (2)	128
C14—H14*A*···O2^{iii}	0.98	2.45	3.252 (2)	139
C14—H14*B*···O2^{iv}	0.98	2.48	3.409 (2)	159
C15—H15*A*···O3^{iv}	0.98	2.55	3.351 (2)	139

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*−1; (ii) *x*−1, *y*, *z*; (iii) *x*+1, *y*, *z*; (iv) −*x*+1, −*y*+1, −*z*.