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2-Hydroxy-*N*-(3-oxo-1-thia-4-aza-spiro[4.5]dec-4-yl)-2,2-diphenyl-acetamide

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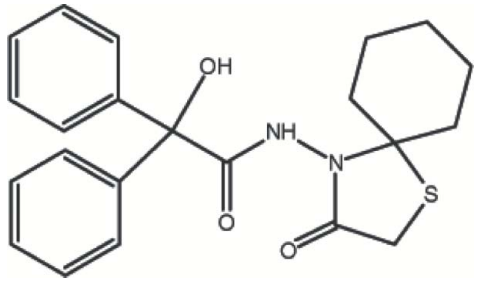
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.174; data-to-parameter ratio = 23.9.

In the molecule of the title compound, $C_{22}H_{24}N_2O_3S$, the dihedral angle between the two phenyl rings is 70.28 (11)°. The cyclohexane ring adopts a chair conformation while the thiazolidine ring assumes an envelope conformation. The crystal packing is stabilized by intramolecular N—H···O and intermolecular C—H···O and O—H···O hydrogen-bonding interactions. The structure also contains C—H···*Cg* interactions, where *Cg* is the centroid of the phenyl ring at $(1-x, -y, 1-z)$; for this contact, C···*Cg* = 3.721 (3) Å, H···*Cg* = 2.82 Å and C—H···*Cg* = 162°.

Related literature

For related literature, see: Allen *et al.* (1987); Andres *et al.* (2000); Ateş *et al.* (1997); Çapan *et al.* (1999); Cremer & Pople (1975); Güzel *et al.* (2006); Karalı, Terzioğlu & Gürsoy (1998); Srivastava *et al.* (2005); Ulusoy (2002); Ulusoy *et al.* (1997).



Experimental

Crystal data

$C_{22}H_{24}N_2O_3S$
M_r = 396.50
Monoclinic, $P2_1/c$
a = 12.1676 (2) Å
b = 9.3050 (2) Å
c = 17.9081 (3) Å
β = 95.4236 (11)°
V = 2018.47 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 294 (2) K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.963, T_{max} = 0.963
56688 measured reflections
6181 independent reflections
3693 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.174
S = 1.04
6181 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1	0.845 (19)	2.19 (2)	2.5886 (19)	109.1 (16)
O1—H1O···O2^{i}	0.82	1.95	2.7494 (18)	167
C1—H1···O1	0.93	2.33	2.697 (2)	103
C13—H13···O2	0.93	2.59	3.004 (2)	107
C16—H16*A*···O1^{ii}	0.97	2.54	3.466 (3)	159
C16—H16*B*···O3^{iii}	0.97	2.44	3.341 (3)	154

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y, -z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2465).

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supplementary materials

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2-Hydroxy-*N*-(3-oxo-1-thia-4-azaspiro[4.5]dec-4-yl)-2,2-diphenylacetamide

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Comment

4-Thiazolidinones and their spiroheterocyclic analogs have been shown to possess antibacterial (Ateş *et al.*, 1997; Andres *et al.*, 2000), antifungal (Ulusoy *et al.*, 1997; Çapan *et al.*, 1999) and antituberculosis (Ulusoy, 2002; Karalı *et al.*, 1998; Srivastava *et al.*, 2005) activities. In our previous report (Güzel *et al.*, 2006), we have synthesized and evaluated sixteen new 2-hydroxy-*N*-(3-oxo-1-thia-4-azaspiro[4.4]non-4-yl)/(3-oxo-1-thia-4-\ azaspiro[4.5]dec-4-yl)-2,2-diphenylacetamide derivatives, incorporating the thiazolidinone substructure, as potential antimycobacterials. We now report the crystal structure of the title compound, (I), (Fig. 1), which has a non-planar conformation.

All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the two phenyl rings is 70.28 (11)°. The five-membered ring (S1/N2/C15—C17) is not planar, with puckering parameters (Cremer & Pople, 1975) Q_2 = 0.2791 (18) Å and φ_2 = 171.1 (4) °. The C17—C22 cyclohexane ring has a normal chair conformation [puckering parameters: Q = 0.559 (2) Å, θ = 180.00 (2)° and φ = 341 (9) °].

The molecular conformation and crystal packing (Fig. 2) is stabilized by intramolecular N—H···O and intermolecular C—H···O and O—H···O hydrogen bonds (Table 1). The packing of (I) also features a C–H···π interaction, *viz*: C10—H10···*Cg*2(1 − *x*, −*y*, 1 − *z*), where *Cg* denotes the centre of the C1—C6 phenyl ring [C···*Cg* = 3.721 (3) Å, H···*Cg* = 2.82 Å and C–H···*Cg* = 162 °].

Experimental

A mixture of 2-hydroxy-2,2-diphenylacetohydrazide (0.005 mol), cyclohexanone (0.005 mol) and mercaptoacetic acid or α-mercaptopropionic acid (0.02 mol) was refluxed in 20 ml dry benzene for 5–6 h using a Dean-Stark water separator. Excess benzene was evaporated *in vacuo*. The resulting residue was titrated with saturated $NaHCO_3$ solution until CO_2 evolution ceased and was allowed to stand overnight or in some cases refrigerated until solidification. The solid thus obtained was recrystallized from ethanol.

Yield 47%; mp 493–495 K; IR(KBr) (ν, cm^{-1}): 3354 (O—H/N—H), 1685, 1726 (C═O). ^{1}H-NMR (DMSO-d_6, 500 MHz) δ (p.p.m.): 0.80–1.07 (m, 1H, spirodecane), 1.34–1.38 (m, 2H, spirodecane), 1.49–1.63 (m, 5H, spirodecane), 1.73, 1.76 (2 s, 2H, spirodecane), 3.54 (s, 2H, C_2—H_2), 6.81 (s, 1H, COH), 7.28–7.35 (m, 6H, Ar—H), 7.46 (s, 2H, Ar—H), 7.47–7.48 (m, 2H, Ar—H), 10.21 (s, 1H, CONH). ^{13}C-NMR (APT) (DMSO-d_6 / 125 MHz) δ (p.p.m.): 23.56 ($C_{7,9}$ spd.), 24.83 (C_8 spd.), 28.64 (C_2 spd.), 37.61 ($C_{6,10}$ spd.), 73.30 (C_5 spd.), 81.61 (C—OH), 128.05,128.25,128.30 (ar. CH), 144.40 (ar. C), 167.98 (amide C═O), 173.40 (lactam C═O). Analysis calculated for $C_{22}H_{24}N_2O_3S$ (396.494): C 66.64, H 6.10, N 7.07%. Found: C 66.21, H 6.22, N 6.69%.

Refinement

The NH H atom were found from a difference Fourier map and refined freely. The other H atoms were positioned geometrically, with C—H = 0.93–0.97Å and O—H = 0.82 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O).

Figures

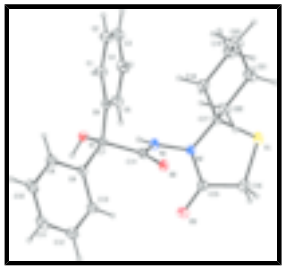

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 20% probability level (arbitrary spheres for the H atoms).

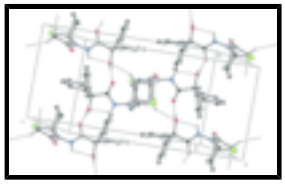

Fig. 2. View of the packing and hydrogen bonding interactions (dashed lines) for (I). H atoms not involved in these bonds have been omitted for clarity.

2-Hydroxy-*N*-(3-oxo-1-thia-4-azaspiro[4.5]dec-4-yl)-2,2-diphenylacetamide

Crystal data

$C_{22}H_{24}N_2O_3S$	F_{000} = 840
M_r = 396.50	D_x = 1.305 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.7107 Å
Hall symbol: -P 2ybc	Cell parameters from 9135 reflections
a = 12.1676 (2) Å	θ = 2.1–30.5º
b = 9.3050 (2) Å	μ = 0.19 mm^{-1}
c = 17.9081 (3) Å	T = 294 (2) K
β = 95.4236 (11)º	Prism, pale yellow
V = 2018.47 (6) Å^3	0.20 × 0.20 × 0.20 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	6181 independent reflections
Radiation source: Sealed Tube	3693 reflections with $I > 2\sigma(I)$
Monochromator: Graphite Monochromator	R_{int} = 0.067
Detector resolution: 10.0000 pixels mm^{-1}	θ_{max} = 30.7º
T = 294(2) K	θ_{min} = 2.3º
ω scans	h = −17→17

Absorption correction: multi-scan (SORTAV; Blessing, 1995)

T_{min} = 0.963, T_{max} = 0.963

56688 measured reflections

$k = -13 \rightarrow 11$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.174$

$S = 1.04$

6181 reflections

259 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 0.3246P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.28$ e Å^{-3}

$\Delta\rho_{min} = -0.32$ e Å^{-3}

Extinction correction: SHELXL97, $FC^*=KFC[1+0.001XFC^2\Lambda^3/\sin(2\Theta)]^{-1/4}$

Extinction coefficient: 0.0127 (15)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.80752 (5)	0.06874 (7)	0.00974 (3)	0.0757 (2)
O1	0.60444 (10)	0.26567 (12)	0.31383 (7)	0.0493 (4)
O2	0.57202 (11)	−0.04849 (13)	0.19848 (7)	0.0564 (4)
O3	0.52028 (12)	0.17812 (18)	0.06223 (9)	0.0772 (6)
N1	0.66222 (13)	0.16220 (17)	0.18918 (8)	0.0494 (5)
N2	0.69225 (12)	0.13232 (16)	0.11812 (8)	0.0484 (4)
C1	0.77259 (16)	0.1061 (2)	0.37823 (12)	0.0636 (7)
C2	0.85941 (19)	0.0326 (3)	0.41713 (15)	0.0809 (9)
C3	0.85038 (19)	−0.1121 (3)	0.43089 (13)	0.0778 (9)
C4	0.7553 (2)	−0.1830 (2)	0.40626 (13)	0.0738 (8)
C5	0.66836 (16)	−0.1106 (2)	0.36760 (11)	0.0592 (7)

C6	0.67673 (14)	0.03552 (18)	0.35312 (9)	0.0470 (5)
C7	0.58364 (13)	0.11450 (17)	0.30554 (9)	0.0440 (5)
C8	0.46878 (13)	0.07835 (18)	0.32601 (10)	0.0459 (5)
C9	0.44939 (17)	0.0643 (3)	0.40028 (12)	0.0698 (8)
C10	0.3446 (2)	0.0374 (3)	0.42036 (15)	0.0837 (10)
C11	0.25785 (18)	0.0262 (2)	0.36688 (17)	0.0777 (9)
C12	0.27456 (17)	0.0434 (2)	0.29364 (16)	0.0754 (9)
C13	0.38002 (16)	0.0703 (2)	0.27252 (12)	0.0582 (6)
C14	0.60133 (14)	0.06778 (18)	0.22493 (9)	0.0457 (5)
C15	0.61414 (16)	0.1364 (2)	0.05903 (11)	0.0568 (6)
C16	0.6604 (2)	0.0817 (3)	−0.01015 (12)	0.0760 (9)
C17	0.80025 (14)	0.06507 (18)	0.11158 (10)	0.0490 (5)
C18	0.89297 (16)	0.1561 (2)	0.15033 (12)	0.0604 (7)
C19	1.00600 (17)	0.0872 (2)	0.14582 (15)	0.0733 (8)
C20	1.00933 (19)	−0.0637 (3)	0.17748 (17)	0.0821 (9)
C21	0.91890 (18)	−0.1551 (2)	0.13800 (16)	0.0771 (9)
C22	0.80573 (16)	−0.0880 (2)	0.14201 (13)	0.0617 (7)
H1	0.77930	0.20390	0.36900	0.0760*
H1N	0.6718 (17)	0.247 (2)	0.2053 (11)	0.063 (6)*
H1O	0.54590	0.30960	0.30790	0.0740*
H2	0.92380	0.08140	0.43390	0.0970*
H3	0.90850	−0.16150	0.45680	0.0930*
H4	0.74910	−0.28090	0.41560	0.0890*
H5	0.60410	−0.15990	0.35130	0.0710*
H9	0.50780	0.07310	0.43740	0.0840*
H10	0.33320	0.02680	0.47070	0.1000*
H11	0.18750	0.00690	0.38050	0.0930*
H12	0.21510	0.03720	0.25710	0.0910*
H13	0.39040	0.08290	0.22210	0.0700*
H16A	0.64230	0.14710	−0.05170	0.0910*
H16B	0.62950	−0.01190	−0.02370	0.0910*
H18A	0.88030	0.16900	0.20260	0.0730*
H18B	0.89220	0.25030	0.12700	0.0730*
H19A	1.02250	0.08430	0.09390	0.0880*
H19B	1.06200	0.14530	0.17360	0.0880*
H20A	1.08050	−0.10680	0.17140	0.0990*
H20B	1.00030	−0.06010	0.23070	0.0990*
H21A	0.92010	−0.24960	0.16090	0.0930*
H21B	0.93260	−0.16690	0.08590	0.0930*
H22A	0.78830	−0.08730	0.19380	0.0740*
H22B	0.75070	−0.14630	0.11340	0.0740*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0720 (4)	0.1023 (5)	0.0557 (3)	0.0087 (3)	0.0218 (3)	−0.0021 (3)
O1	0.0509 (6)	0.0392 (6)	0.0576 (7)	0.0011 (5)	0.0048 (5)	−0.0026 (5)
O2	0.0601 (8)	0.0494 (7)	0.0618 (8)	−0.0122 (6)	0.0166 (6)	−0.0119 (6)

O3	0.0611 (9)	0.0903 (12)	0.0788 (10)	0.0182 (8)	−0.0009 (7)	−0.0057 (8)
N1	0.0578 (9)	0.0441 (8)	0.0484 (8)	−0.0049 (7)	0.0164 (6)	−0.0024 (6)
N2	0.0485 (8)	0.0522 (8)	0.0454 (7)	0.0019 (6)	0.0099 (6)	0.0003 (6)
C1	0.0505 (10)	0.0599 (12)	0.0784 (13)	−0.0005 (9)	−0.0039 (9)	0.0031 (10)
C2	0.0538 (12)	0.0871 (17)	0.0981 (18)	0.0035 (11)	−0.0126 (11)	0.0015 (13)
C3	0.0653 (13)	0.0869 (17)	0.0797 (15)	0.0255 (12)	−0.0016 (11)	0.0112 (12)
C4	0.0791 (15)	0.0571 (12)	0.0859 (16)	0.0150 (11)	0.0111 (12)	0.0159 (11)
C5	0.0567 (11)	0.0512 (11)	0.0701 (12)	0.0022 (8)	0.0076 (9)	0.0082 (9)
C6	0.0466 (9)	0.0471 (9)	0.0480 (9)	0.0035 (7)	0.0081 (7)	0.0020 (7)
C7	0.0457 (8)	0.0403 (8)	0.0465 (8)	−0.0020 (6)	0.0070 (6)	−0.0018 (6)
C8	0.0431 (8)	0.0429 (9)	0.0522 (9)	0.0027 (7)	0.0076 (7)	0.0016 (7)
C9	0.0540 (11)	0.1019 (18)	0.0553 (11)	0.0109 (11)	0.0147 (9)	0.0111 (11)
C10	0.0673 (14)	0.1045 (19)	0.0845 (16)	0.0194 (13)	0.0347 (12)	0.0258 (14)
C11	0.0544 (12)	0.0595 (13)	0.124 (2)	0.0012 (10)	0.0337 (13)	0.0064 (13)
C12	0.0468 (11)	0.0693 (14)	0.1084 (19)	0.0029 (10)	−0.0022 (11)	−0.0189 (12)
C13	0.0497 (10)	0.0600 (12)	0.0642 (11)	0.0046 (8)	0.0024 (8)	−0.0040 (9)
C14	0.0440 (8)	0.0449 (9)	0.0487 (9)	−0.0017 (7)	0.0076 (7)	0.0002 (7)
C15	0.0560 (11)	0.0582 (11)	0.0559 (10)	0.0037 (9)	0.0038 (8)	0.0014 (8)
C16	0.0789 (15)	0.0989 (18)	0.0498 (11)	0.0072 (13)	0.0047 (10)	−0.0041 (11)
C17	0.0477 (9)	0.0481 (9)	0.0524 (9)	0.0001 (7)	0.0111 (7)	0.0000 (7)
C18	0.0551 (10)	0.0511 (11)	0.0753 (13)	−0.0055 (8)	0.0079 (9)	−0.0041 (9)
C19	0.0494 (11)	0.0657 (13)	0.1045 (18)	−0.0076 (9)	0.0056 (11)	−0.0074 (12)
C20	0.0539 (12)	0.0706 (15)	0.120 (2)	0.0088 (10)	−0.0015 (12)	0.0030 (13)
C21	0.0608 (12)	0.0522 (12)	0.118 (2)	0.0049 (10)	0.0065 (12)	0.0013 (12)
C22	0.0532 (10)	0.0471 (10)	0.0852 (14)	−0.0025 (8)	0.0086 (9)	0.0042 (9)

Geometric parameters (Å, °)

S1—C16	1.796 (3)	C17—C18	1.524 (3)
S1—C17	1.8346 (19)	C18—C19	1.526 (3)
O1—C7	1.4344 (19)	C19—C20	1.513 (3)
O2—C14	1.221 (2)	C20—C21	1.512 (3)
O3—C15	1.213 (2)	C21—C22	1.520 (3)
O1—H1O	0.8200	C1—H1	0.9300
N1—N2	1.385 (2)	C2—H2	0.9300
N1—C14	1.349 (2)	C3—H3	0.9300
N2—C15	1.354 (2)	C4—H4	0.9300
N2—C17	1.470 (2)	C5—H5	0.9300
N1—H1N	0.845 (19)	C9—H9	0.9300
C1—C6	1.377 (3)	C10—H10	0.9300
C1—C2	1.389 (3)	C11—H11	0.9300
C2—C3	1.375 (4)	C12—H12	0.9300
C3—C4	1.368 (3)	C13—H13	0.9300
C4—C5	1.383 (3)	C16—H16A	0.9700
C5—C6	1.390 (3)	C16—H16B	0.9700
C6—C7	1.538 (2)	C18—H18A	0.9700
C7—C14	1.542 (2)	C18—H18B	0.9700
C7—C8	1.516 (2)	C19—H19A	0.9700
C8—C13	1.376 (3)	C19—H19B	0.9700

C8—C9	1.379 (3)	C20—H20A	0.9700
C9—C10	1.380 (3)	C20—H20B	0.9700
C10—C11	1.361 (4)	C21—H21A	0.9700
C11—C12	1.356 (4)	C21—H21B	0.9700
C12—C13	1.394 (3)	C22—H22A	0.9700
C15—C16	1.498 (3)	C22—H22B	0.9700
C17—C22	1.524 (3)		
C16—S1—C17	93.30 (9)	C6—C1—H1	120.00
C7—O1—H1O	109.00	C1—C2—H2	120.00
N2—N1—C14	120.64 (15)	C3—C2—H2	120.00
N1—N2—C15	119.06 (15)	C2—C3—H3	120.00
N1—N2—C17	118.30 (14)	C4—C3—H3	120.00
C15—N2—C17	121.08 (15)	C3—C4—H4	120.00
N2—N1—H1N	117.5 (13)	C5—C4—H4	120.00
C14—N1—H1N	120.7 (14)	C4—C5—H5	120.00
C2—C1—C6	120.64 (19)	C6—C5—H5	120.00
C1—C2—C3	120.1 (2)	C8—C9—H9	119.00
C2—C3—C4	119.6 (2)	C10—C9—H9	119.00
C3—C4—C5	120.65 (19)	C9—C10—H10	120.00
C4—C5—C6	120.25 (18)	C11—C10—H10	120.00
C1—C6—C5	118.73 (16)	C10—C11—H11	120.00
C1—C6—C7	120.65 (15)	C12—C11—H11	120.00
C5—C6—C7	120.51 (15)	C11—C12—H12	120.00
O1—C7—C6	107.32 (13)	C13—C12—H12	120.00
C6—C7—C14	102.90 (13)	C8—C13—H13	120.00
C8—C7—C14	112.17 (14)	C12—C13—H13	120.00
C6—C7—C8	114.05 (13)	S1—C16—H16A	110.00
O1—C7—C8	110.50 (13)	S1—C16—H16B	110.00
O1—C7—C14	109.54 (13)	C15—C16—H16A	110.00
C7—C8—C9	120.01 (16)	C15—C16—H16B	110.00
C7—C8—C13	121.64 (16)	H16A—C16—H16B	108.00
C9—C8—C13	118.14 (17)	C17—C18—H18A	109.00
C8—C9—C10	121.0 (2)	C17—C18—H18B	109.00
C9—C10—C11	120.3 (2)	C19—C18—H18A	109.00
C10—C11—C12	119.7 (2)	C19—C18—H18B	109.00
C11—C12—C13	120.6 (2)	H18A—C18—H18B	108.00
C8—C13—C12	120.2 (2)	C18—C19—H19A	109.00
O2—C14—N1	123.12 (15)	C18—C19—H19B	109.00
O2—C14—C7	123.48 (15)	C20—C19—H19A	109.00
N1—C14—C7	113.09 (14)	C20—C19—H19B	109.00
N2—C15—C16	110.24 (17)	H19A—C19—H19B	108.00
O3—C15—N2	124.51 (18)	C19—C20—H20A	110.00
O3—C15—C16	125.25 (19)	C19—C20—H20B	110.00
S1—C16—C15	107.72 (15)	C21—C20—H20A	109.00
S1—C17—N2	101.46 (11)	C21—C20—H20B	109.00
S1—C17—C18	109.82 (13)	H20A—C20—H20B	108.00
S1—C17—C22	111.61 (13)	C20—C21—H21A	109.00
N2—C17—C18	110.85 (14)	C20—C21—H21B	109.00
N2—C17—C22	112.24 (14)	C22—C21—H21A	109.00

C18—C17—C22	110.55 (15)	C22—C21—H21B	109.00
C17—C18—C19	111.98 (16)	H21A—C21—H21B	108.00
C18—C19—C20	111.24 (18)	C17—C22—H22A	109.00
C19—C20—C21	110.7 (2)	C17—C22—H22B	109.00
C20—C21—C22	111.67 (18)	C21—C22—H22A	109.00
C17—C22—C21	112.10 (16)	C21—C22—H22B	109.00
C2—C1—H1	120.00	H22A—C22—H22B	108.00
C17—S1—C16—C15	20.49 (17)	O1—C7—C8—C9	−80.3 (2)
C16—S1—C17—N2	−22.19 (14)	O1—C7—C8—C13	94.38 (19)
C16—S1—C17—C18	−139.52 (15)	C6—C7—C8—C9	40.7 (2)
C16—S1—C17—C22	97.53 (15)	C6—C7—C8—C13	−144.64 (16)
C14—N1—N2—C15	71.8 (2)	C14—C7—C8—C9	157.21 (18)
C14—N1—N2—C17	−94.09 (19)	C14—C7—C8—C13	−28.2 (2)
N2—N1—C14—O2	1.9 (3)	O1—C7—C14—O2	−167.35 (15)
N2—N1—C14—C7	175.65 (14)	O1—C7—C14—N1	18.93 (19)
N1—N2—C15—O3	7.5 (3)	C6—C7—C14—O2	78.74 (19)
N1—N2—C15—C16	−172.32 (17)	C6—C7—C14—N1	−94.98 (16)
C17—N2—C15—O3	172.97 (18)	C8—C7—C14—O2	−44.3 (2)
C17—N2—C15—C16	−6.8 (2)	C8—C7—C14—N1	142.02 (15)
N1—N2—C17—S1	−173.39 (12)	C7—C8—C9—C10	177.3 (2)
N1—N2—C17—C18	−56.8 (2)	C13—C8—C9—C10	2.5 (3)
N1—N2—C17—C22	67.3 (2)	C7—C8—C13—C12	−177.01 (16)
C15—N2—C17—S1	21.00 (18)	C9—C8—C13—C12	−2.3 (3)
C15—N2—C17—C18	137.58 (17)	C8—C9—C10—C11	−0.9 (4)
C15—N2—C17—C22	−98.3 (2)	C9—C10—C11—C12	−0.8 (4)
C6—C1—C2—C3	0.1 (4)	C10—C11—C12—C13	1.0 (3)
C2—C1—C6—C5	0.2 (3)	C11—C12—C13—C8	0.6 (3)
C2—C1—C6—C7	−175.90 (19)	O3—C15—C16—S1	168.39 (18)
C1—C2—C3—C4	−0.2 (4)	N2—C15—C16—S1	−11.8 (2)
C2—C3—C4—C5	0.1 (4)	S1—C17—C18—C19	−69.89 (19)
C3—C4—C5—C6	0.2 (3)	N2—C17—C18—C19	178.81 (16)
C4—C5—C6—C1	−0.3 (3)	C22—C17—C18—C19	53.7 (2)
C4—C5—C6—C7	175.79 (18)	S1—C17—C22—C21	69.1 (2)
C1—C6—C7—O1	−16.1 (2)	N2—C17—C22—C21	−177.74 (17)
C1—C6—C7—C8	−138.82 (17)	C18—C17—C22—C21	−53.4 (2)
C1—C6—C7—C14	99.44 (18)	C17—C18—C19—C20	−55.6 (3)
C5—C6—C7—O1	167.93 (15)	C18—C19—C20—C21	56.1 (3)
C5—C6—C7—C8	45.2 (2)	C19—C20—C21—C22	−56.0 (3)
C5—C6—C7—C14	−76.56 (19)	C20—C21—C22—C17	55.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1	0.845 (19)	2.19 (2)	2.5886 (19)	109.1 (16)
O1—H1O···O2^{i}	0.82	1.95	2.7494 (18)	167
C1—H1···O1	0.93	2.33	2.697 (2)	103
C13—H13···O2	0.93	2.59	3.004 (2)	107
C16—H16A···O1^{ii}	0.97	2.54	3.466 (3)	159

C16—H16B···O3^{iii}	0.97	2.44	3.341 (3)	154

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*+1, −*y*, −*z*.

Fig. 1

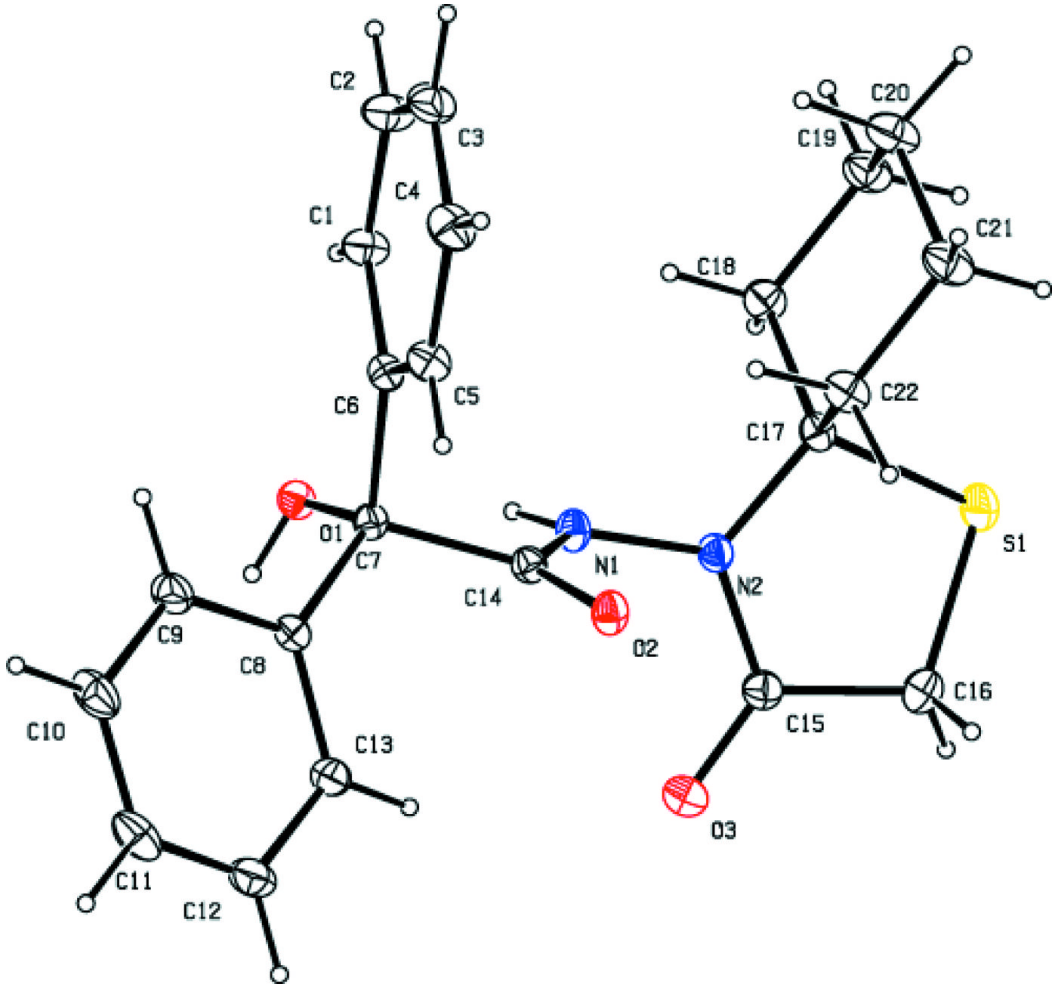

Fig. 2