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N′-(3,3,7,7-Tetramethyl-1,5-dioxaspiro[5,5]undecane-8-ylidene)-*p*-toluenesulfonohydrazide

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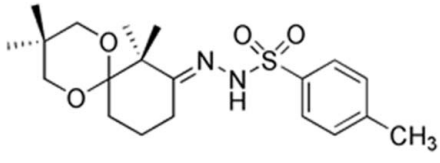
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.127; data-to-parameter ratio = 17.3.

The title compound, $C_{20}H_{30}N_2O_4S$, crystallizes as an *E* isomer with respect to the C=N bond. Both the cyclohexane and the dioxane rings adopt distorted chair conformations. In the crystal structure, molecules form dimers *via* inversion-related S=O···H—N hydrogen bonds. Additional C—H···O hydrogen bonds link these units into stacks down the *b* axis.

Related literature

For background to the use of tosylhydrazones in synthesis, see: Chamberlin *et al.* (1978); Fabris *et al.* (1999); Törmäkangas *et al.* (2002). For the stability of their isomers, see: Laus *et al.* (2006); Roy & Nangia (2007).



Experimental

Crystal data

$C_{20}H_{30}N_2O_4S$
M_r = 394.52
Triclinic, $P\overline{1}$
a = 7.437 (2) Å
b = 10.045 (3) Å
c = 14.299 (4) Å
α = 98.397 (4)°
β = 92.789 (4)°
γ = 98.676 (3)°
V = 1041.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 293 (2) K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.978, T_{max} = 0.986
5883 measured reflections
4212 independent reflections
3556 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.127
S = 1.04
4212 reflections
244 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*B*···O4	0.86	2.30	2.9425 (19)	132
C9—H9*A*···O3^{i}	0.97	2.68	3.3305 (19)	125
C15—H15*A*···O1^{ii}	0.97	2.60	3.4543 (19)	147
C19—H19*C*···O3^{ii}	0.96	2.59	3.290 (2)	130

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: SJ2404).

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supplementary materials

Acta Cryst. (2007). E**63**, o4931 [**doi:10.1107/S160053680705893X**]

N'-(3,3,7,7-Tetramethyl-1,5-dioxaspiro[5,5]undecane-8-ylidene)-*p*-toluenesulfonohydrazide

X. Zhu, L. Zou, Y. Xu, J. Han and C. Zhang

Comment

Tosylhydrazone is recognized as a key intermediate in the synthesis of many natural products (Chamberlin *et al.*, 1978), such as terpenes and taxol (Fabris *et al.*, 1999; Törmäkangas *et al.*, 2002). The title compound (I) obtained by condensation of 3,3,7,7-tetramethyl-1,5-dioxaspiro[5,5]undecan-8-one and tosylhydrazide in absolute ethanol was found to be a mixture of the *E*- and *Z*-isomers in $CHCl_3$ solution in a 9:1 ratio by ^1H NMR. Structural information on (I) is important as the configuration will influence attack by electrophilic reagents on the carbon atom in (C═N).

In this instance, the structure of the title compound (I), Fig. 1, shows only the *E*-isomer with respect to the C═N bond, suggesting that this is the thermodynamically stable form (Laus *et al.*, 2006; Roy & Nangia, 2007). Both the cyclohexane and the dioxane rings adopt distorted chair conformations. In the crystal structure, molecules form centrosymmetric dimers *via* S═O···H—N hydrogen bonds, Fig. 2. Additional C—H···O hydrogen bonds, Table 1, link these units into stacks down the *b* axis.

Experimental

The synthesis of 3 3,7,7-teramethyl-8-(tosylhydrazono)-1,5-dioxaspiro[5,5]undecane was done according to the procedure reported by Törmäkangas *et al.* (2002) with 1,3-cyclohexanedione as substrate which reacted with MeI to afford 2,2-dimethyl-1,3-cyclohexanedione in 58% yield. The target molecule was characterized by IR, ^1H NMR and elemental analysis. Single crystals were grown from MeOH:H_2O 3:1 *v*/*v*. Calcd. for $C_{20}H_{30}N_2O_4S$: C 60.89, H 7.66, N 7.10%; Analysis found: C 60.99, H 7.96, N 7.07%.

Refinement

The crystals were weakly diffracting and few high angle reflections were obtained which explains the low fraction of data obtained in this determination. All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, $U_{iso} = 1.2U_{eq}$(C) for aromatic 0.97 Å, $U_{iso} = 1.2U_{eq}$(C) for CH_2, 0.96 Å, $U_{iso} = 1.5U_{eq}$(C) for CH_3 atoms and 0.86 Å, $U_{iso} = 1.2U_{eq}$(N) for the NH atom.

Figures

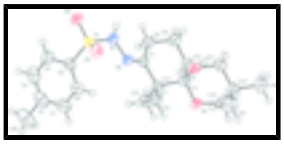

Fig. 1. The molecule structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

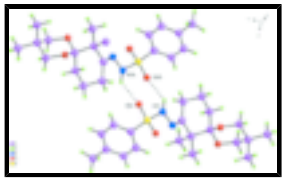

Fig. 2. Formation of dimers *via* N—H···O hydrogen bonds (dashed lines).

N'-(3,3,7,7-Tetramethyl-1,5-dioxaspiro[5,5]undecane-8-ylidene)- *p*-toluenesulfonohydrazide

Crystal data

$C_{20}H_{30}N_2O_4S$	$Z = 2$
$M_r = 394.52$	$F_{000} = 424$
Triclinic, $P\bar{1}$	$D_x = 1.258$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 415.65-416.55 K
$a = 7.437$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.045$ (3) Å	Cell parameters from 25 reflections
$c = 14.299$ (4) Å	$\theta = 2.1$–26.5º
$\alpha = 98.397$ (4)º	$\mu = 0.18$ mm^{-1}
$\beta = 92.789$ (4)º	$T = 293$ (2) K
$\gamma = 98.676$ (3)º	Block, colourless
$V = 1041.9$ (5) Å^3	0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer	4212 independent reflections
Radiation source: fine-focus sealed tube	3556 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.013$
$T = 298$(2) K	$\theta_{max} = 26.5$º
ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -9 \rightarrow 9$
$T_{min} = 0.978$, $T_{max} = 0.986$	$k = -12 \rightarrow 12$
5883 measured reflections	$l = -17 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.127$	$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.3202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
4212 reflections	$\Delta\rho_{max} = 0.48$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}

2 restraints
Primary atom site location: structure-invariant direct methods

Extinction correction: none

Special details

Experimental. For (I): IR (KBr disk, v, cm^{-1}): 3203, 2987, 2975, 2950, 2920, 2870, 1635. ^{1}H NMR (500 MHz, δ_H): [*E*-isomer in $CDCl_3$] 7.8380 (d, 2H, *J* = 8.15), 7.2861 (d, 2H, *J* = 8.00), 3.5510 (d, 2H, *J* = 11.25), 3.2546 (d, 2H, *J* = 11.55), 2.4224 (s, 3H), 2.2573 (t, 2H), 2.0290 (t, 2H), 1.5170 (m, 2H), 1.1378 (s, 3H), 1.1314 (s, 3H), 0.6917 (s, 3H); [*Z*-isomer in $CDCl_3$] 7.8020 (d, 2H *J* = 8.60), 7.3619 (d, 2H, *J* = 7.85), 3.6292 (d, 2H, *J*=11.40), 3.3368 (d, 2H, *J* = 11.40), 2.4538 (s, 3H), 2.4123 (t, 2H), 2.2126 (t, 2H), 1.6720 (m, 2H), 1.2021 (s, 6H), 1.1628 (s, 3H), 0.7221 (s, 3H).

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.74930 (4)	0.94640 (3)	0.92474 (2)	0.03409 (7)
O1	0.35368 (11)	0.85518 (8)	0.53518 (6)	0.0381 (2)
O2	0.26324 (12)	0.62269 (8)	0.48085 (6)	0.0387 (2)
O4	0.72649 (13)	1.04073 (9)	1.00690 (6)	0.0438 (2)
N1	0.55776 (14)	0.93342 (10)	0.85828 (7)	0.0379 (3)
H1B	0.4704	0.9770	0.8746	0.045*
N2	0.55154 (13)	0.84407 (10)	0.77244 (7)	0.0347 (2)
O3	0.89725 (12)	0.97570 (9)	0.86819 (6)	0.0444 (2)
C8	0.21772 (18)	0.84174 (16)	0.75584 (10)	0.0505 (4)
H8A	0.2414	0.9270	0.7994	0.061*
H8B	0.1582	0.7725	0.7897	0.061*
C6	0.7747 (2)	0.67650 (14)	0.89064 (11)	0.0522 (4)
H6A	0.7918	0.6892	0.8284	0.063*
C12	0.39561 (16)	0.69706 (12)	0.63791 (8)	0.0343 (3)
C11	0.26753 (16)	0.72936 (11)	0.55853 (8)	0.0331 (3)
C5	0.75496 (17)	0.78523 (12)	0.95861 (9)	0.0387 (3)
C10	0.07722 (17)	0.74173 (14)	0.59111 (9)	0.0420 (3)
H10A	0.0001	0.7590	0.5390	0.050*
H10B	0.0232	0.6568	0.6104	0.050*
C7	0.39707 (16)	0.80343 (12)	0.72640 (8)	0.0348 (3)
C2	0.7486 (2)	0.52802 (15)	1.00909 (13)	0.0621 (4)
C13	0.58742 (17)	0.69355 (15)	0.60413 (10)	0.0458 (3)
H13A	0.6332	0.7793	0.5850	0.069*
H13B	0.5826	0.6217	0.5514	0.069*

H13C	0.6667	0.6775	0.6549	0.069*
C15	0.2712 (2)	0.89224 (13)	0.45192 (10)	0.0478 (4)
H15A	0.3388	0.9773	0.4392	0.057*
H15B	0.1475	0.9065	0.4631	0.057*
C16	0.1801 (2)	0.64865 (13)	0.39464 (9)	0.0474 (4)
H16A	0.0516	0.6510	0.4020	0.057*
H16B	0.1896	0.5745	0.3443	0.057*
C9	0.08827 (17)	0.85731 (16)	0.67373 (10)	0.0485 (4)
H9A	−0.0325	0.8601	0.6959	0.058*
H9B	0.1291	0.9431	0.6520	0.058*
C14	0.3242 (2)	0.55659 (14)	0.66514 (11)	0.0529 (4)
H14A	0.2039	0.5565	0.6866	0.079*
H14B	0.4042	0.5385	0.7149	0.079*
H14C	0.3202	0.4873	0.6108	0.079*
C17	0.2679 (2)	0.78305 (14)	0.36608 (10)	0.0520 (4)
C18	0.4619 (3)	0.77578 (19)	0.33833 (13)	0.0755 (5)
H18A	0.4594	0.7063	0.2843	0.113*
H18B	0.5328	0.7543	0.3904	0.113*
H18C	0.5156	0.8622	0.3229	0.113*
C4	0.7365 (2)	0.76697 (16)	1.05132 (11)	0.0576 (4)
H4A	0.7257	0.8404	1.0974	0.069*
C3	0.7342 (3)	0.63871 (18)	1.07526 (13)	0.0702 (5)
H3A	0.7226	0.6269	1.1381	0.084*
C1	0.7688 (2)	0.54881 (16)	0.91645 (14)	0.0650 (5)
H1A	0.7786	0.4751	0.8705	0.078*
C19	0.1499 (3)	0.8148 (2)	0.28468 (12)	0.0850 (6)
H19A	0.0279	0.8166	0.3034	0.128*
H19B	0.1481	0.7458	0.2304	0.128*
H19C	0.1993	0.9020	0.2689	0.128*
C20	0.7411 (3)	0.38686 (18)	1.03574 (18)	0.0925 (7)
H20A	0.7254	0.3910	1.1023	0.139*
H20B	0.8527	0.3536	1.0216	0.139*
H20C	0.6405	0.3265	1.0003	0.139*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03184 (14)	0.03793 (14)	0.03056 (14)	0.00472 (11)	−0.00087 (11)	0.00080 (11)
O1	0.0394 (4)	0.0290 (4)	0.0427 (5)	−0.0001 (3)	0.0005 (4)	0.0019 (3)
O2	0.0502 (5)	0.0299 (4)	0.0326 (4)	0.0048 (3)	−0.0033 (4)	−0.0031 (3)
O4	0.0455 (5)	0.0453 (5)	0.0360 (5)	0.0064 (4)	−0.0027 (4)	−0.0065 (4)
N1	0.0346 (5)	0.0441 (5)	0.0332 (5)	0.0135 (4)	−0.0032 (4)	−0.0055 (4)
N2	0.0345 (5)	0.0387 (5)	0.0289 (5)	0.0081 (4)	−0.0003 (4)	−0.0024 (4)
O3	0.0366 (4)	0.0539 (5)	0.0419 (5)	0.0031 (4)	0.0038 (4)	0.0087 (4)
C8	0.0358 (6)	0.0733 (9)	0.0395 (7)	0.0189 (6)	0.0000 (6)	−0.0102 (6)
C6	0.0593 (8)	0.0482 (7)	0.0512 (8)	0.0183 (6)	0.0030 (7)	0.0049 (6)
C12	0.0330 (5)	0.0347 (5)	0.0335 (6)	0.0076 (4)	0.0003 (5)	−0.0018 (5)
C11	0.0333 (5)	0.0286 (5)	0.0338 (6)	0.0005 (4)	0.0013 (5)	−0.0015 (5)

C5	0.0339 (6)	0.0404 (6)	0.0412 (6)	0.0048 (5)	−0.0014 (5)	0.0069 (5)
C10	0.0293 (5)	0.0532 (7)	0.0403 (7)	0.0032 (5)	−0.0018 (5)	0.0017 (6)
C7	0.0339 (5)	0.0381 (6)	0.0324 (6)	0.0099 (5)	0.0029 (5)	0.0006 (5)
C2	0.0439 (7)	0.0532 (7)	0.0920 (11)	0.0028 (6)	−0.0056 (8)	0.0294 (8)
C13	0.0379 (6)	0.0545 (7)	0.0432 (7)	0.0173 (5)	0.0019 (6)	−0.0086 (6)
C15	0.0596 (8)	0.0349 (6)	0.0491 (8)	0.0079 (6)	0.0009 (7)	0.0079 (6)
C16	0.0607 (8)	0.0413 (7)	0.0354 (7)	0.0067 (6)	−0.0089 (6)	−0.0047 (5)
C9	0.0318 (6)	0.0658 (8)	0.0470 (8)	0.0173 (6)	0.0021 (6)	−0.0039 (6)
C14	0.0696 (9)	0.0387 (6)	0.0499 (8)	0.0087 (6)	−0.0020 (7)	0.0069 (6)
C17	0.0732 (9)	0.0460 (7)	0.0382 (7)	0.0138 (7)	0.0022 (7)	0.0071 (6)
C18	0.0947 (12)	0.0684 (10)	0.0696 (10)	0.0189 (9)	0.0350 (9)	0.0155 (8)
C4	0.0737 (10)	0.0537 (8)	0.0446 (8)	0.0063 (7)	0.0025 (7)	0.0100 (6)
C3	0.0802 (11)	0.0719 (9)	0.0627 (9)	0.0060 (9)	0.0015 (9)	0.0322 (8)
C1	0.0676 (9)	0.0448 (7)	0.0823 (12)	0.0177 (7)	−0.0032 (9)	0.0019 (8)
C19	0.1363 (17)	0.0746 (11)	0.0488 (9)	0.0362 (11)	−0.0118 (10)	0.0109 (8)
C20	0.0689 (11)	0.0668 (9)	0.1502 (18)	0.0068 (9)	−0.0059 (12)	0.0529 (11)

Geometric parameters (Å, °)

S1—O3	1.4175 (10)	C13—H13A	0.9600
S1—O4	1.4317 (9)	C13—H13B	0.9600
S1—N1	1.6508 (11)	C13—H13C	0.9600
S1—C5	1.7621 (14)	C15—C17	1.5185 (19)
O1—C11	1.4209 (14)	C15—H15A	0.9700
O1—C15	1.4349 (17)	C15—H15B	0.9700
O2—C11	1.4217 (14)	C16—C17	1.529 (2)
O2—C16	1.4276 (16)	C16—H16A	0.9700
N1—N2	1.4051 (14)	C16—H16B	0.9700
N1—H1B	0.8600	C9—H9A	0.9700
N2—C7	1.2752 (15)	C9—H9B	0.9700
C8—C7	1.5055 (18)	C14—H14A	0.9600
C8—C9	1.523 (2)	C14—H14B	0.9600
C8—H8A	0.9700	C14—H14C	0.9600
C8—H8B	0.9700	C17—C19	1.523 (2)
C6—C1	1.381 (2)	C17—C18	1.524 (3)
C6—C5	1.3825 (19)	C18—H18A	0.9600
C6—H6A	0.9300	C18—H18B	0.9600
C12—C7	1.5306 (16)	C18—H18C	0.9600
C12—C13	1.5319 (18)	C4—C3	1.378 (2)
C12—C14	1.5424 (18)	C4—H4A	0.9300
C12—C11	1.5481 (17)	C3—H3A	0.9300
C11—C10	1.5284 (18)	C1—H1A	0.9300
C5—C4	1.375 (2)	C19—H19A	0.9600
C10—C9	1.5204 (19)	C19—H19B	0.9600
C10—H10A	0.9700	C19—H19C	0.9600
C10—H10B	0.9700	C20—H20A	0.9600
C2—C3	1.372 (2)	C20—H20B	0.9600
C2—C1	1.383 (3)	C20—H20C	0.9600
C2—C20	1.515 (2)		

O3—S1—O4	120.03 (6)	C17—C15—H15A	109.3
O3—S1—N1	108.24 (6)	O1—C15—H15B	109.3
O4—S1—N1	103.97 (6)	C17—C15—H15B	109.3
O3—S1—C5	108.47 (6)	H15A—C15—H15B	107.9
O4—S1—C5	108.66 (6)	O2—C16—C17	112.57 (11)
N1—S1—C5	106.70 (6)	O2—C16—H16A	109.1
C11—O1—C15	113.75 (9)	C17—C16—H16A	109.1
C11—O2—C16	113.98 (9)	O2—C16—H16B	109.1
N2—N1—S1	113.38 (8)	C17—C16—H16B	109.1
N2—N1—H1B	123.3	H16A—C16—H16B	107.8
S1—N1—H1B	123.3	C10—C9—C8	112.49 (12)
C7—N2—N1	117.94 (10)	C10—C9—H9A	109.1
C7—C8—C9	114.13 (11)	C8—C9—H9A	109.1
C7—C8—H8A	108.7	C10—C9—H9B	109.1
C9—C8—H8A	108.7	C8—C9—H9B	109.1
C7—C8—H8B	108.7	H9A—C9—H9B	107.8
C9—C8—H8B	108.7	C12—C14—H14A	109.5
H8A—C8—H8B	107.6	C12—C14—H14B	109.5
C1—C6—C5	119.12 (15)	H14A—C14—H14B	109.5
C1—C6—H6A	120.4	C12—C14—H14C	109.5
C5—C6—H6A	120.4	H14A—C14—H14C	109.5
C7—C12—C13	111.75 (9)	H14B—C14—H14C	109.5
C7—C12—C14	107.26 (10)	C15—C17—C19	109.54 (13)
C13—C12—C14	108.04 (11)	C15—C17—C18	109.67 (13)
C7—C12—C11	108.97 (10)	C19—C17—C18	111.10 (15)
C13—C12—C11	110.32 (10)	C15—C17—C16	106.71 (11)
C14—C12—C11	110.45 (10)	C19—C17—C16	108.74 (13)
O1—C11—O2	110.50 (9)	C18—C17—C16	110.97 (13)
O1—C11—C10	110.62 (10)	C17—C18—H18A	109.5
O2—C11—C10	112.07 (9)	C17—C18—H18B	109.5
O1—C11—C12	105.13 (9)	H18A—C18—H18B	109.5
O2—C11—C12	106.17 (9)	C17—C18—H18C	109.5
C10—C11—C12	112.06 (10)	H18A—C18—H18C	109.5
C4—C5—C6	120.23 (13)	H18B—C18—H18C	109.5
C4—C5—S1	120.45 (11)	C5—C4—C3	119.29 (15)
C6—C5—S1	119.30 (11)	C5—C4—H4A	120.4
C9—C10—C11	110.23 (10)	C3—C4—H4A	120.4
C9—C10—H10A	109.6	C2—C3—C4	121.93 (16)
C11—C10—H10A	109.6	C2—C3—H3A	119.0
C9—C10—H10B	109.6	C4—C3—H3A	119.0
C11—C10—H10B	109.6	C6—C1—C2	121.52 (15)
H10A—C10—H10B	108.1	C6—C1—H1A	119.2
N2—C7—C8	125.89 (11)	C2—C1—H1A	119.2
N2—C7—C12	115.70 (10)	C17—C19—H19A	109.5
C8—C7—C12	118.20 (10)	C17—C19—H19B	109.5
C3—C2—C1	117.87 (15)	H19A—C19—H19B	109.5
C3—C2—C20	121.48 (18)	C17—C19—H19C	109.5
C1—C2—C20	120.65 (16)	H19A—C19—H19C	109.5
C12—C13—H13A	109.5	H19B—C19—H19C	109.5

C12—C13—H13B	109.5	C2—C20—H20A	109.5
H13A—C13—H13B	109.5	C2—C20—H20B	109.5
C12—C13—H13C	109.5	H20A—C20—H20B	109.5
H13A—C13—H13C	109.5	C2—C20—H20C	109.5
H13B—C13—H13C	109.5	H20A—C20—H20C	109.5
O1—C15—C17	111.71 (11)	H20B—C20—H20C	109.5
O1—C15—H15A	109.3		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O4^{i}	0.86	2.30	2.9425 (19)	132
C9—H9A···O3^{ii}	0.97	2.68	3.3305 (19)	125
C15—H15A···O1^{iii}	0.97	2.60	3.4543 (19)	147
C19—H19C···O3^{iii}	0.96	2.59	3.290 (2)	130

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

Fig. 1

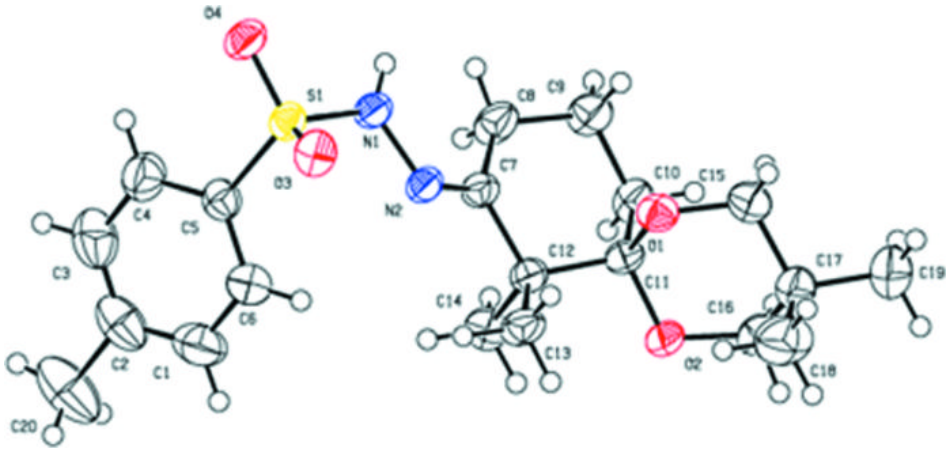
O4
S1
N1
O3
N2
C8
C9
C7
C10
C15
C4
C5
C3
C12
O1
C17
C19
C11
C14
C16
C6
C2
C1
C13
O2
C18
C20

Fig. 2