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An enantiopure azasteroidal derivative bearing an isoborneol sulfinyl residue

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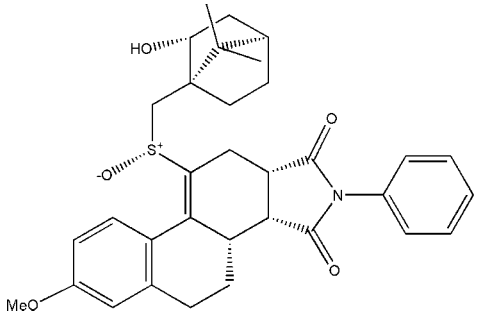
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.072; data-to-parameter ratio = 13.0.

The title compound (systematic name (3a*R*,3b*S*,11a*S*,R_S)-3a,3b,4,5,11,11a-hexahydro-10-{[[(1*S*,2*R*,4*R*)-2-hydroxy-7,7-dimethylbicyclo[2.2.1]hept-1-yl]methyl]sulfinyl}-7-methoxy-2-phenyl-1*H*-naphth[2,1-*e*]isoindole-1,3(2*H*)-dione), $C_{33}H_{37}NO_5S$, is an enantiopure sulfinyl adduct which represents a 16-azasteroid analogue and displays an estrone-like skeleton. The structure exhibits a strong intramolecular hydrogen bond and a C—H$\cdots\pi$ intermolecular interaction.

Related literature

For related literature, see: Aversa *et al.* (2001, 2005).



Experimental

Crystal data

$C_{33}H_{37}NO_5S$
M_r = 559.70
Monoclinic, $P2_1$
a = 11.271 (1) Å
b = 7.137 (1) Å
c = 17.513 (1) Å
β = 91.620 (1)°
V = 1408.2 (2) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 1.37 mm^{-1}
T = 100 (2) K
0.28 × 0.21 × 0.16 mm

Data collection

Oxford Diffraction Excalibur PX Ultra CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.710, T_{max} = 0.803
20509 measured reflections
4772 independent reflections
4326 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.072
S = 1.04
4772 reflections
368 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 1756 Friedel pairs
Flack parameter: −0.002 (11)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H*O*4$\cdots$O2	0.85 (3)	1.97 (3)	2.7847 (19)	161 (2)
C11*A*—H11*C*$\cdots$*Cg*1	1.00	2.81	3.546 (1)	142

Note: *Cg*1 is the centroid of the C13–C18 ring.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2066).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Aversa, M. C., Barattucci, A., Bonaccorsi, P., Bruno, G., Caruso, F. & Giannetto, P. (2001). *Tetrahedron Asymm.* **12**, 2901–2908.
Aversa, M. C., Barattucci, A., Bonaccorsi, P., Faggi, C., Gacs-Baitz, E., Marrocchi, A., Minuti, L. & Taticchi, A. (2005). *Tetrahedron*, **61**, 7719–7726.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Oxford Diffraction (2006). *CrysAlis CCD* (Version 1.171.31.2), *CrysAlis RED* (Version 1.171.31.2) and *ABSPACK* in *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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An enantiopure azasteroidal derivative bearing an isoborneol sulfinyl residue

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Comment

The Diels–Alder (DA) reaction is one of the best examined and well appreciated complexity-generating reactions. Some of us (Aversa *et al.*, 2001) have described the synthesis of enantiopure 4-[1-(alkyl sulfinyl)vinyl]-1,2-dihydronaphthalenes and their DA reactions with *N*-phenyl maleimide (NPM). The main product (I) of the NPM cycloaddition to (1*S*,2*R*,4*R*)-1-{[(*R*)-[1-(3,4-dihydro-6-methoxy-1- naphthalenyl)ethenyl]sulfinyl]methyl}-7,7-dimethylbicyclo[2.2.1]heptan-2-ol is easily separated from their diastereomers and obtained as crystals suitable for X-ray analysis, which allows the assignment of (*R*), (*S*) and (*S*) configurations respectively to the new stereogenic centres 3a, 3 b and 11*a*, generated during the DA cycloaddition. The results here reported confirm the structure previously assigned to adduct (I) just through comparison with an analogous compound which structure had been determined by X-ray studies (Aversa *et al.*, 2001). On the basis of our recent studies (Aversa *et al.*, 2005) an unquestionable confirmation of structure (I) appeared to be desiderable, and this has been possible with the low temperature data set presented herein. A strong intramolecular hydrogen bond is formed (O4–HO4···O2, Fig 1 and Table 1) and this may be an important factor in determining the preferred face of approach by the dienophile and thus the corresponding configurations of the new stereogenic centres. The most important intermolecular interaction is a C—H···π one where the hydrogen atom H11*c* points to the geometrical centroid of the [C13—C18]^{i} phenyl ring, i: −*x* + 2;*y* + 0,5;-*z* + 2 (Fig 2), with a H···Ct(Ph) distance of 2.81 Å and a C11*a*—H11*c*–Ct(Ph) angle of 142°.

Experimental

The preparation of adduct I was previously described in detail (Aversa *et al.*, 2001). Crystals suitable for X-ray analysis were obtained by spontaneous slow evaporation of the chromatographic column eluent (petrol/EtOAc 7:3).

Refinement

H atom on O4 was located in the difference Fourier map and refined freely. All other H atoms were clearly observable but were placed at ideal positions (C—H_3: 0.96 (1) Å, C—H_2: 0.97 (1) Å, C—H: 0.98 (1) Å, and allowed to ride with U(H) = 1.2 × U_{eq}(Host) for C—H_2 and C—H and U(H) = 1.5 × U_{eq}(Host) for C—H_3.

Figures

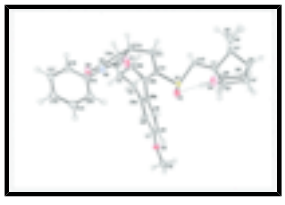


Fig. 1. Molecular structure of (I) showing the intramolecular hydrogen bond (dashed line) between O4—HO4 and O2. All non-H atoms have been labelled. Displacement ellipsoids are drawn at the 40% probability level.

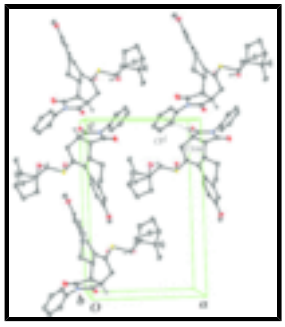


Fig. 2. Packing diagram of (I) drawn along the *b* axis. The CH-π intermolecular interaction between centroid Ct^{i} (i: −*x* + 2;*y* + 0,5;-*z* + 2) and C11*a*—H11*c* is shown in broken lines.

(3a*R*,3 b*S*,11*aS*,*R*$_S$)-3a,3 b,4,5,11,11*a*-hexahydro-10- {[[(1*S*,2*R*,4*R*)-2-hydroxy-7,7-dimethylbicyclo[2.2.1] hept-1-yl]methyl]sulfinyl}-7-methoxy-2-phenyl-1*H*-naphth[2,1-*e*] isoindole-1,3(2*H*)-dione

Crystal data

$C_{33}H_{37}NO_5S$	F_{000} = 596
M_r = 559.70	D_x = 1.320 Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation λ = 1.54178 Å
Hall symbol: P 2yb	Cell parameters from 13765 reflections
a = 11.271 (1) Å	θ = 3.9–72.2º
b = 7.137 (1) Å	μ = 1.37 mm^{-1}
c = 17.513 (1) Å	*T* = 100 (2) K
β = 91.620 (1)º	Prismatic, light yellow
V = 1408.2 (2) Å^3	0.28 × 0.21 × 0.16 mm
Z = 2	

Data collection

Oxford Diffraction Excalibur PX Ultra CCD diffractometer	4772 independent reflections
Radiation source: Enhance (Cu) X-ray Source	4326 reflections with $I > 2\sigma(I)$
Monochromator: Oxford Diffraction, Enhance ULTRA assembly	R_{int} = 0.034
Detector resolution: 8.1241 pixels mm^{-1}	θ_{max} = 72.3º
T = 100(2) K	θ_{min} = 3.9º
ω scans	*h* = −13→13
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	*k* = −8→6
T_{min} = 0.710, T_{max} = 0.803	*l* = −21→20
20509 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.072$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
4772 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}
368 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.002 (11)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.81555 (3)	0.41507 (5)	0.70796 (2)	0.01779 (9)
N2	1.13302 (12)	0.2560 (2)	0.92663 (8)	0.0216 (3)
O1	0.97546 (11)	0.4534 (2)	0.94801 (7)	0.0343 (3)
O2	0.83893 (11)	0.60772 (17)	0.74147 (7)	0.0261 (3)
O3	1.24918 (12)	0.0105 (2)	0.88840 (8)	0.0332 (3)
O4	0.61320 (13)	0.76651 (19)	0.74012 (8)	0.0318 (3)
O5	1.18741 (11)	0.27852 (19)	0.45217 (7)	0.0265 (3)
C1	1.01297 (15)	0.3044 (3)	0.92662 (9)	0.0240 (4)
C3	1.15251 (16)	0.0808 (3)	0.89372 (9)	0.0234 (4)
C3A	1.03364 (15)	−0.0007 (3)	0.86789 (10)	0.0225 (4)
H3A	1.0231	−0.1239	0.8941	0.027*
C3B	1.01803 (15)	−0.0319 (2)	0.78048 (9)	0.0203 (4)
H3B	0.9472	−0.1154	0.7742	0.024*
C4	1.11951 (15)	−0.1389 (2)	0.74457 (10)	0.0228 (4)
H4A	1.1247	−0.2667	0.7664	0.027*
H4B	1.1956	−0.0740	0.7559	0.027*
C5	1.09750 (15)	−0.1504 (2)	0.65824 (10)	0.0224 (4)
H5A	1.1608	−0.2255	0.6349	0.027*
H5B	1.0205	−0.2127	0.6470	0.027*
C5A	1.09625 (14)	0.0440 (2)	0.62493 (9)	0.0177 (3)
C6	1.14288 (14)	0.0803 (3)	0.55369 (9)	0.0206 (3)
H6	1.1762	−0.0195	0.5254	0.025*
C7	1.14154 (14)	0.2594 (3)	0.52332 (9)	0.0199 (3)

C8	1.09805 (13)	0.4080 (3)	0.56590 (8)	0.0181 (3)
H8	1.0993	0.5320	0.5462	0.022*
C9	1.05288 (13)	0.3728 (2)	0.63736 (9)	0.0181 (3)
H9	1.0260	0.4747	0.6671	0.022*
C9A	1.04601 (13)	0.1914 (2)	0.66660 (9)	0.0163 (3)
C9B	0.98456 (14)	0.1480 (2)	0.73786 (9)	0.0170 (3)
C10	0.89132 (14)	0.2434 (2)	0.76548 (9)	0.0177 (3)
C11	0.84265 (15)	0.1932 (3)	0.84179 (9)	0.0226 (4)
H11A	0.7862	0.0877	0.8355	0.027*
H11B	0.7988	0.3016	0.8621	0.027*
C11A	0.94198 (15)	0.1377 (3)	0.89859 (9)	0.0231 (4)
H11C	0.9046	0.0785	0.9437	0.028*
C12	1.20323 (16)	0.4640 (3)	0.42401 (10)	0.0276 (4)
H12A	1.1266	0.5291	0.4221	0.041*
H12B	1.2351	0.4585	0.3726	0.041*
H12C	1.2588	0.5319	0.4580	0.041*
C13	1.22603 (15)	0.3608 (3)	0.96544 (9)	0.0227 (4)
C14	1.25390 (16)	0.5396 (3)	0.94238 (10)	0.0260 (4)
H14	1.2117	0.5963	0.9007	0.031*
C15	1.34454 (16)	0.6360 (3)	0.98083 (11)	0.0306 (4)
H15	1.3644	0.7594	0.9655	0.037*
C16	1.40612 (16)	0.5528 (3)	1.04146 (11)	0.0316 (4)
H16	1.4691	0.6183	1.0669	0.038*
C17	1.37598 (16)	0.3747 (3)	1.06485 (10)	0.0302 (4)
H17	1.4176	0.3187	1.1069	0.036*
C18	1.28532 (16)	0.2770 (3)	1.02726 (10)	0.0265 (4)
H18	1.2641	0.1548	1.0435	0.032*
C1'	0.57486 (14)	0.4506 (2)	0.67893 (9)	0.0185 (3)
C2'	0.58386 (15)	0.6673 (3)	0.67131 (10)	0.0232 (4)
H2'	0.6426	0.6992	0.6316	0.028*
C3'	0.45756 (16)	0.7237 (3)	0.64210 (11)	0.0289 (4)
H3'1	0.4163	0.7994	0.6806	0.035*
H3'2	0.4607	0.7956	0.5939	0.035*
C4'	0.39582 (15)	0.5338 (3)	0.62942 (10)	0.0242 (4)
H4'	0.3074	0.5414	0.6244	0.029*
C5'	0.45540 (16)	0.4398 (3)	0.56100 (10)	0.0296 (4)
H5'1	0.4645	0.5292	0.5183	0.036*
H5'2	0.4093	0.3299	0.5425	0.036*
C6'	0.57822 (15)	0.3798 (3)	0.59575 (9)	0.0253 (4)
H6'1	0.5879	0.2420	0.5941	0.030*
H6'2	0.6438	0.4391	0.5681	0.030*
C7'	0.44189 (13)	0.4178 (3)	0.69833 (9)	0.0228 (3)
C8'	0.40543 (17)	0.4955 (4)	0.77559 (11)	0.0366 (5)
H8'1	0.4343	0.6244	0.7813	0.055*
H8'2	0.4399	0.4178	0.8167	0.055*
H8'3	0.3187	0.4940	0.7784	0.055*
C9'	0.40350 (17)	0.2118 (3)	0.69455 (13)	0.0370 (5)
H9'1	0.3173	0.2034	0.6997	0.056*
H9'2	0.4432	0.1419	0.7361	0.056*

H9'3	0.4255	0.1583	0.6454	0.056*
C10'	0.66464 (13)	0.3585 (2)	0.73332 (9)	0.0197 (4)
H10A	0.6536	0.2209	0.7318	0.024*
H10B	0.6509	0.4015	0.7861	0.024*
HO4	0.685 (2)	0.737 (4)	0.7490 (14)	0.045 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01489 (17)	0.0198 (2)	0.01866 (17)	−0.00013 (16)	0.00063 (13)	0.00070 (17)
N2	0.0214 (7)	0.0257 (8)	0.0176 (7)	0.0045 (6)	−0.0016 (5)	−0.0007 (6)
O1	0.0288 (7)	0.0459 (9)	0.0279 (7)	0.0129 (6)	−0.0060 (5)	−0.0163 (6)
O2	0.0236 (6)	0.0199 (7)	0.0345 (7)	−0.0041 (5)	−0.0025 (5)	−0.0017 (6)
O3	0.0254 (7)	0.0359 (8)	0.0377 (8)	0.0110 (6)	−0.0074 (5)	−0.0087 (6)
O4	0.0296 (7)	0.0253 (7)	0.0400 (8)	0.0047 (6)	−0.0070 (6)	−0.0128 (6)
O5	0.0313 (7)	0.0279 (7)	0.0207 (6)	0.0029 (5)	0.0089 (5)	0.0025 (5)
C1	0.0237 (9)	0.0365 (11)	0.0117 (8)	0.0061 (8)	−0.0005 (6)	0.0003 (8)
C3	0.0278 (9)	0.0241 (9)	0.0182 (8)	0.0038 (8)	−0.0019 (6)	0.0011 (7)
C3A	0.0238 (8)	0.0235 (9)	0.0200 (8)	0.0024 (7)	−0.0008 (7)	0.0049 (7)
C3B	0.0219 (8)	0.0195 (9)	0.0195 (8)	−0.0027 (6)	−0.0005 (6)	0.0026 (6)
C4	0.0260 (8)	0.0186 (9)	0.0239 (9)	0.0025 (7)	−0.0015 (7)	0.0032 (7)
C5	0.0247 (9)	0.0167 (8)	0.0258 (9)	0.0019 (7)	0.0013 (7)	−0.0007 (7)
C5A	0.0149 (7)	0.0170 (8)	0.0212 (8)	−0.0004 (6)	−0.0017 (6)	−0.0012 (7)
C6	0.0182 (8)	0.0217 (9)	0.0220 (8)	0.0025 (7)	0.0019 (6)	−0.0032 (7)
C7	0.0160 (7)	0.0271 (9)	0.0166 (7)	−0.0013 (7)	0.0016 (6)	−0.0006 (7)
C8	0.0160 (7)	0.0174 (8)	0.0209 (7)	−0.0010 (7)	−0.0004 (6)	0.0043 (8)
C9	0.0131 (7)	0.0205 (9)	0.0207 (8)	0.0005 (6)	0.0002 (6)	−0.0019 (6)
C9A	0.0143 (7)	0.0177 (8)	0.0168 (8)	−0.0024 (6)	−0.0003 (6)	−0.0005 (6)
C9B	0.0162 (7)	0.0168 (8)	0.0179 (8)	−0.0018 (6)	−0.0024 (6)	−0.0002 (7)
C10	0.0175 (7)	0.0197 (9)	0.0159 (7)	−0.0027 (7)	−0.0012 (6)	0.0002 (7)
C11	0.0184 (8)	0.0307 (10)	0.0187 (8)	−0.0010 (7)	0.0008 (6)	0.0031 (7)
C11A	0.0217 (8)	0.0309 (10)	0.0167 (8)	0.0024 (7)	0.0015 (6)	0.0046 (7)
C12	0.0264 (9)	0.0332 (11)	0.0234 (9)	0.0041 (7)	0.0060 (7)	0.0095 (8)
C13	0.0198 (8)	0.0318 (10)	0.0165 (8)	0.0044 (7)	0.0006 (6)	−0.0051 (7)
C14	0.0249 (9)	0.0340 (11)	0.0192 (8)	0.0052 (8)	0.0030 (7)	−0.0001 (8)
C15	0.0283 (9)	0.0327 (11)	0.0314 (10)	−0.0017 (8)	0.0080 (8)	−0.0025 (9)
C16	0.0220 (9)	0.0436 (12)	0.0294 (10)	−0.0001 (8)	0.0020 (7)	−0.0104 (9)
C17	0.0266 (9)	0.0403 (12)	0.0233 (9)	0.0085 (8)	−0.0043 (7)	−0.0033 (8)
C18	0.0265 (9)	0.0308 (10)	0.0222 (9)	0.0054 (8)	−0.0019 (7)	0.0010 (8)
C1'	0.0151 (7)	0.0200 (9)	0.0204 (8)	0.0013 (6)	0.0021 (6)	−0.0021 (7)
C2'	0.0230 (9)	0.0212 (9)	0.0253 (9)	0.0008 (7)	−0.0003 (7)	−0.0009 (7)
C3'	0.0263 (9)	0.0239 (9)	0.0363 (10)	0.0062 (8)	−0.0041 (8)	0.0003 (8)
C4'	0.0181 (8)	0.0265 (10)	0.0280 (9)	0.0053 (7)	−0.0019 (7)	−0.0021 (8)
C5'	0.0252 (9)	0.0367 (11)	0.0265 (9)	0.0060 (8)	−0.0068 (7)	−0.0059 (9)
C6'	0.0201 (8)	0.0327 (11)	0.0229 (8)	0.0055 (7)	−0.0016 (6)	−0.0074 (8)
C7'	0.0136 (7)	0.0268 (9)	0.0281 (8)	−0.0001 (8)	0.0023 (6)	0.0028 (9)
C8'	0.0228 (9)	0.0575 (14)	0.0300 (10)	0.0061 (9)	0.0098 (8)	0.0031 (9)
C9'	0.0211 (9)	0.0320 (11)	0.0576 (13)	−0.0065 (8)	−0.0051 (9)	0.0112 (10)

C10'	0.0155 (7)	0.0223 (9)	0.0214 (8)	0.0000 (6)	0.0010 (6)	−0.0010 (7)

Geometric parameters (Å, °)

S1—O2	1.5151 (13)	C12—H12A	0.9800
S1—C10	1.7879 (17)	C12—H12B	0.9800
S1—C10'	1.8156 (16)	C12—H12C	0.9800
N2—C1	1.396 (2)	C13—C14	1.377 (3)
N2—C3	1.397 (2)	C13—C18	1.391 (2)
N2—C13	1.442 (2)	C14—C15	1.390 (3)
O1—C1	1.208 (2)	C14—H14	0.9500
O3—C3	1.206 (2)	C15—C16	1.386 (3)
O4—C2'	1.428 (2)	C15—H15	0.9500
O4—HO4	0.85 (3)	C16—C17	1.381 (3)
O5—C7	1.3695 (19)	C16—H16	0.9500
O5—C12	1.426 (2)	C17—C18	1.388 (3)
C1—C11A	1.509 (3)	C17—H17	0.9500
C3—C3A	1.518 (2)	C18—H18	0.9500
C3A—C11A	1.537 (2)	C1'—C10'	1.519 (2)
C3A—C3B	1.552 (2)	C1'—C6'	1.543 (2)
C3A—H3A	1.0000	C1'—C2'	1.556 (3)
C3B—C4	1.526 (2)	C1'—C7'	1.564 (2)
C3B—C9B	1.527 (2)	C2'—C3'	1.552 (2)
C3B—H3B	1.0000	C2'—H2'	1.0000
C4—C5	1.527 (2)	C3'—C4'	1.537 (3)
C4—H4A	0.9900	C3'—H3'1	0.9900
C4—H4B	0.9900	C3'—H3'2	0.9900
C5—C5A	1.505 (2)	C4'—C7'	1.541 (2)
C5—H5A	0.9900	C4'—C5'	1.543 (2)
C5—H5B	0.9900	C4'—H4'	1.0000
C5A—C6	1.392 (2)	C5'—C6'	1.556 (2)
C5A—C9A	1.408 (2)	C5'—H5'1	0.9900
C6—C7	1.384 (3)	C5'—H5'2	0.9900
C6—H6	0.9500	C6'—H6'1	0.9900
C7—C8	1.394 (2)	C6'—H6'2	0.9900
C8—C9	1.387 (2)	C7'—C8'	1.529 (3)
C8—H8	0.9500	C7'—C9'	1.534 (3)
C9—C9A	1.395 (2)	C8'—H8'1	0.9800
C9—H9	0.9500	C8'—H8'2	0.9800
C9A—C9B	1.477 (2)	C8'—H8'3	0.9800
C9B—C10	1.353 (2)	C9'—H9'1	0.9800
C10—C11	1.502 (2)	C9'—H9'2	0.9800
C11—C11A	1.529 (2)	C9'—H9'3	0.9800
C11—H11A	0.9900	C10'—H10A	0.9900
C11—H11B	0.9900	C10'—H10B	0.9900
C11A—H11C	1.0000		
O2—S1—C10	109.18 (8)	H12B—C12—H12C	109.5
O2—S1—C10'	105.12 (7)	C14—C13—C18	121.20 (17)
C10—S1—C10'	98.37 (8)	C14—C13—N2	120.73 (16)

C1—N2—C3	112.66 (15)	C18—C13—N2	118.06 (16)
C1—N2—C13	124.26 (15)	C13—C14—C15	119.15 (17)
C3—N2—C13	122.55 (14)	C13—C14—H14	120.4
C2'—O4—HO4	103.1 (17)	C15—C14—H14	120.4
C7—O5—C12	117.49 (14)	C16—C15—C14	120.3 (2)
O1—C1—N2	124.44 (18)	C16—C15—H15	119.8
O1—C1—C11A	127.48 (16)	C14—C15—H15	119.8
N2—C1—C11A	108.04 (15)	C17—C16—C15	119.96 (18)
O3—C3—N2	123.88 (17)	C17—C16—H16	120.0
O3—C3—C3A	127.56 (17)	C15—C16—H16	120.0
N2—C3—C3A	108.55 (15)	C16—C17—C18	120.39 (17)
C3—C3A—C11A	104.23 (15)	C16—C17—H17	119.8
C3—C3A—C3B	115.05 (14)	C18—C17—H17	119.8
C11A—C3A—C3B	112.23 (14)	C17—C18—C13	118.96 (18)
C3—C3A—H3A	108.4	C17—C18—H18	120.5
C11A—C3A—H3A	108.4	C13—C18—H18	120.5
C3B—C3A—H3A	108.4	C10'—C1'—C6'	114.61 (13)
C4—C3B—C9B	113.40 (13)	C10'—C1'—C2'	116.10 (14)
C4—C3B—C3A	114.38 (14)	C6'—C1'—C2'	103.96 (14)
C9B—C3B—C3A	112.42 (14)	C10'—C1'—C7'	115.12 (13)
C4—C3B—H3B	105.2	C6'—C1'—C7'	101.84 (13)
C9B—C3B—H3B	105.2	C2'—C1'—C7'	103.44 (14)
C3A—C3B—H3B	105.2	O4—C2'—C3'	109.75 (14)
C3B—C4—C5	109.41 (14)	O4—C2'—C1'	115.75 (15)
C3B—C4—H4A	109.8	C3'—C2'—C1'	102.98 (14)
C5—C4—H4A	109.8	O4—C2'—H2'	109.4
C3B—C4—H4B	109.8	C3'—C2'—H2'	109.4
C5—C4—H4B	109.8	C1'—C2'—H2'	109.4
H4A—C4—H4B	108.2	C4'—C3'—C2'	103.08 (14)
C5A—C5—C4	109.48 (14)	C4'—C3'—H3'1	111.1
C5A—C5—H5A	109.8	C2'—C3'—H3'1	111.1
C4—C5—H5A	109.8	C4'—C3'—H3'2	111.1
C5A—C5—H5B	109.8	C2'—C3'—H3'2	111.1
C4—C5—H5B	109.8	H3'1—C3'—H3'2	109.1
H5A—C5—H5B	108.2	C3'—C4'—C7'	102.67 (14)
C6—C5A—C9A	119.57 (16)	C3'—C4'—C5'	106.87 (15)
C6—C5A—C5	121.28 (15)	C7'—C4'—C5'	103.31 (14)
C9A—C5A—C5	119.15 (14)	C3'—C4'—H4'	114.2
C7—C6—C5A	121.05 (15)	C7'—C4'—H4'	114.2
C7—C6—H6	119.5	C5'—C4'—H4'	114.2
C5A—C6—H6	119.5	C4'—C5'—C6'	102.58 (13)
O5—C7—C6	116.20 (15)	C4'—C5'—H5'1	111.3
O5—C7—C8	123.95 (16)	C6'—C5'—H5'1	111.3
C6—C7—C8	119.83 (15)	C4'—C5'—H5'2	111.3
C9—C8—C7	119.24 (16)	C6'—C5'—H5'2	111.3
C9—C8—H8	120.4	H5'1—C5'—H5'2	109.2
C7—C8—H8	120.4	C1'—C6'—C5'	103.51 (13)
C8—C9—C9A	121.65 (15)	C1'—C6'—H6'1	111.1
C8—C9—H9	119.2	C5'—C6'—H6'1	111.1

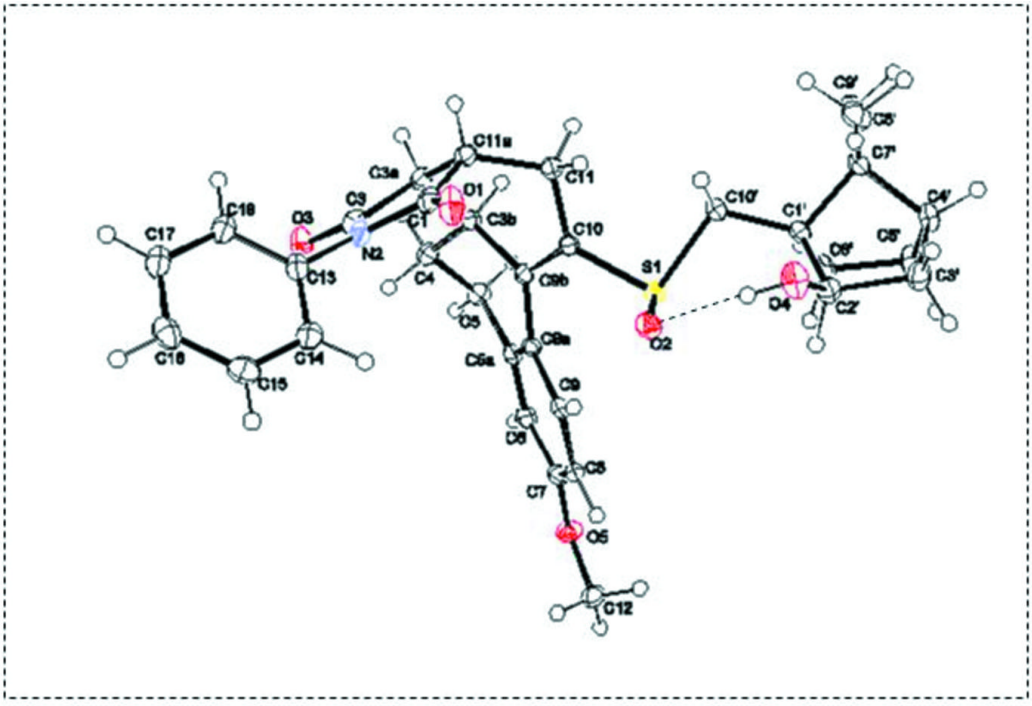
C9A—C9—H9	119.2	C1'—C6'—H6'2	111.1
C9—C9A—C5A	118.40 (14)	C5'—C6'—H6'2	111.1
C9—C9A—C9B	122.45 (14)	H6'1—C6'—H6'2	109.0
C5A—C9A—C9B	119.12 (15)	C8'—C7'—C9'	107.64 (17)
C10—C9B—C9A	125.76 (15)	C8'—C7'—C4'	113.91 (16)
C10—C9B—C3B	115.58 (14)	C9'—C7'—C4'	113.12 (15)
C9A—C9B—C3B	118.28 (14)	C8'—C7'—C1'	114.91 (15)
C9B—C10—C11	120.33 (15)	C9'—C7'—C1'	113.89 (15)
C9B—C10—S1	120.45 (12)	C4'—C7'—C1'	93.06 (13)
C11—C10—S1	118.95 (12)	C7'—C8'—H8'1	109.5
C10—C11—C11A	111.20 (13)	C7'—C8'—H8'2	109.5
C10—C11—H11A	109.4	H8'1—C8'—H8'2	109.5
C11A—C11—H11A	109.4	C7'—C8'—H8'3	109.5
C10—C11—H11B	109.4	H8'1—C8'—H8'3	109.5
C11A—C11—H11B	109.4	H8'2—C8'—H8'3	109.5
H11A—C11—H11B	108.0	C7'—C9'—H9'1	109.5
C1—C11A—C11	112.28 (15)	C7'—C9'—H9'2	109.5
C1—C11A—C3A	105.38 (14)	H9'1—C9'—H9'2	109.5
C11—C11A—C3A	115.21 (14)	C7'—C9'—H9'3	109.5
C1—C11A—H11C	107.9	H9'1—C9'—H9'3	109.5
C11—C11A—H11C	107.9	H9'2—C9'—H9'3	109.5
C3A—C11A—H11C	107.9	C1'—C10'—S1	111.24 (11)
O5—C12—H12A	109.5	C1'—C10'—H10A	109.4
O5—C12—H12B	109.5	S1—C10'—H10A	109.4
H12A—C12—H12B	109.5	C1'—C10'—H10B	109.4
O5—C12—H12C	109.5	S1—C10'—H10B	109.4
H12A—C12—H12C	109.5	H10A—C10'—H10B	108.0
C3—N2—C1—O1	−175.54 (17)	O1—C1—C11A—C3A	171.81 (17)
C13—N2—C1—O1	12.7 (3)	N2—C1—C11A—C3A	−10.36 (17)
C3—N2—C1—C11A	6.54 (18)	C10—C11—C11A—C1	73.88 (19)
C13—N2—C1—C11A	−165.26 (15)	C10—C11—C11A—C3A	−46.8 (2)
C1—N2—C3—O3	−179.26 (16)	C3—C3A—C11A—C1	10.09 (17)
C13—N2—C3—O3	−7.3 (3)	C3B—C3A—C11A—C1	−115.05 (15)
C1—N2—C3—C3A	0.23 (19)	C3—C3A—C11A—C11	134.44 (16)
C13—N2—C3—C3A	172.19 (14)	C3B—C3A—C11A—C11	9.3 (2)
O3—C3—C3A—C11A	172.82 (17)	C1—N2—C13—C14	−66.1 (2)
N2—C3—C3A—C11A	−6.65 (18)	C3—N2—C13—C14	122.91 (18)
O3—C3—C3A—C3B	−63.9 (2)	C1—N2—C13—C18	113.06 (19)
N2—C3—C3A—C3B	116.67 (16)	C3—N2—C13—C18	−58.0 (2)
C3—C3A—C3B—C4	50.4 (2)	C18—C13—C14—C15	1.3 (2)
C11A—C3A—C3B—C4	169.37 (14)	N2—C13—C14—C15	−179.61 (16)
C3—C3A—C3B—C9B	−80.78 (18)	C13—C14—C15—C16	0.1 (3)
C11A—C3A—C3B—C9B	38.17 (19)	C14—C15—C16—C17	−1.2 (3)
C9B—C3B—C4—C5	−45.98 (19)	C15—C16—C17—C18	0.9 (3)
C3A—C3B—C4—C5	−176.70 (14)	C16—C17—C18—C13	0.4 (3)
C3B—C4—C5—C5A	62.43 (18)	C14—C13—C18—C17	−1.6 (3)
C4—C5—C5A—C6	143.70 (15)	N2—C13—C18—C17	179.29 (15)
C4—C5—C5A—C9A	−36.8 (2)	C10'—C1'—C2'—O4	−37.7 (2)
C9A—C5A—C6—C7	0.5 (2)	C6'—C1'—C2'—O4	−164.56 (14)

C5—C5A—C6—C7	−179.98 (15)	C7'—C1'—C2'—O4	89.37 (16)
C12—O5—C7—C6	−171.10 (15)	C10'—C1'—C2'—C3'	−157.47 (14)
C12—O5—C7—C8	7.1 (2)	C6'—C1'—C2'—C3'	75.70 (15)
C5A—C6—C7—O5	−178.65 (14)	C7'—C1'—C2'—C3'	−30.38 (17)
C5A—C6—C7—C8	3.0 (2)	O4—C2'—C3'—C4'	−128.89 (15)
O5—C7—C8—C9	179.66 (14)	C1'—C2'—C3'—C4'	−5.09 (18)
C6—C7—C8—C9	−2.2 (2)	C2'—C3'—C4'—C7'	39.58 (17)
C7—C8—C9—C9A	−2.3 (2)	C2'—C3'—C4'—C5'	−68.77 (17)
C8—C9—C9A—C5A	5.8 (2)	C3'—C4'—C5'—C6'	73.14 (18)
C8—C9—C9A—C9B	−172.02 (14)	C7'—C4'—C5'—C6'	−34.75 (19)
C6—C5A—C9A—C9	−4.8 (2)	C10'—C1'—C6'—C5'	161.04 (15)
C5—C5A—C9A—C9	175.66 (15)	C2'—C1'—C6'—C5'	−71.19 (17)
C6—C5A—C9A—C9B	173.03 (14)	C7'—C1'—C6'—C5'	36.08 (18)
C5—C5A—C9A—C9B	−6.5 (2)	C4'—C5'—C6'—C1'	−1.26 (19)
C9—C9A—C9B—C10	29.3 (2)	C3'—C4'—C7'—C8'	63.04 (18)
C5A—C9A—C9B—C10	−148.51 (17)	C5'—C4'—C7'—C8'	174.07 (16)
C9—C9A—C9B—C3B	−158.17 (15)	C3'—C4'—C7'—C9'	−173.62 (15)
C5A—C9A—C9B—C3B	24.0 (2)	C5'—C4'—C7'—C9'	−62.58 (18)
C4—C3B—C9B—C10	177.05 (14)	C3'—C4'—C7'—C1'	−55.98 (15)
C3A—C3B—C9B—C10	−51.26 (19)	C5'—C4'—C7'—C1'	55.05 (16)
C4—C3B—C9B—C9A	3.7 (2)	C10'—C1'—C7'—C8'	62.0 (2)
C3A—C3B—C9B—C9A	135.43 (15)	C6'—C1'—C7'—C8'	−173.41 (17)
C9A—C9B—C10—C11	−175.47 (15)	C2'—C1'—C7'—C8'	−65.73 (19)
C3B—C9B—C10—C11	11.8 (2)	C10'—C1'—C7'—C9'	−62.9 (2)
C9A—C9B—C10—S1	10.6 (2)	C6'—C1'—C7'—C9'	61.76 (18)
C3B—C9B—C10—S1	−162.16 (12)	C2'—C1'—C7'—C9'	169.43 (16)
O2—S1—C10—C9B	−111.63 (14)	C10'—C1'—C7'—C4'	−179.85 (14)
C10'—S1—C10—C9B	139.08 (14)	C6'—C1'—C7'—C4'	−55.23 (15)
O2—S1—C10—C11	74.32 (14)	C2'—C1'—C7'—C4'	52.45 (15)
C10'—S1—C10—C11	−34.97 (15)	C6'—C1'—C10'—S1	65.08 (17)
C9B—C10—C11—C11A	37.0 (2)	C2'—C1'—C10'—S1	−56.24 (17)
S1—C10—C11—C11A	−148.90 (13)	C7'—C1'—C10'—S1	−177.27 (12)
O1—C1—C11A—C11	45.6 (2)	O2—S1—C10'—C1'	81.13 (12)
N2—C1—C11A—C11	−136.53 (14)	C10—S1—C10'—C1'	−166.30 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—HO4···O2	0.85 (3)	1.97 (3)	2.7847 (19)	161 (2)
C11A—H11C···Ct(Ph)	1.00	2.81	3.645 (1)	142

Fig. 1


C2'
C6'
C7'
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C10'
O4
C11a
C3a
C11
O1
C9
C1
C3b
C10
S1
O2
C18
O3
C17
N2
C13
C4
C9b
C5
C9a
C5a
C16
C14
C15
C9
C6
C8
C7
O5
C12

Fig. 2