Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(11*R*)-13-(1-Naphthylmethylamino)-4,5-epoxy-11,13-dihydrocostunolide

Shama Nasim,[a] Sean Parkin[b] and Peter A. Crooks[a]*

[a]Department of Pharmaceutical Sciences, College of Pharmacy, University of Kentucky, Lexington, KY 40536, USA, and [b]Department of Chemistry, University of Kentucky, Lexington, KY 40506, USA
Correspondence e-mail: pcrooks@email.uky.edu

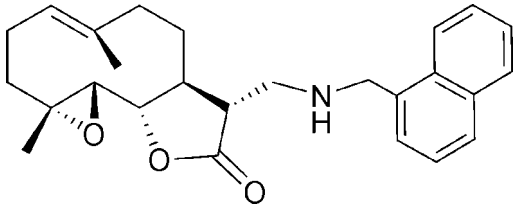
Received 21 September 2007; accepted 25 September 2007

Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.080; data-to-parameter ratio = 13.0.

Crystals of the title compound, $C_{26}H_{31}NO_3$, were obtained by the reaction of 1-naphthylmethylamine with parthenolide. X-ray crystal structure determination reveals a stacked conformation between the cyclodecane ring of the parthenolide moiety and the pendant naphthalene ring. The configuration of the new chiral center at C11 (the point of attachment of the naphthylmethylamino side group) in the title compound is *R*, establishing the stereospecificity of the amination reaction.

Related literature

For related literature, see: Allen *et al.* (1987); Crooks *et al.* (2005); Desiraju & Steiner (1999); Nasim *et al.* (2007); Parsons & Flack (2004); Flack (1983).



Experimental

Crystal data

$C_{26}H_{31}NO_3$
M_r = 405.52
Orthorhombic, $P2_12_12_1$
a = 9.3842 (6) Å
b = 11.9025 (7) Å
c = 18.6386 (13) Å
V = 2081.8 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 90.0 (2) K
0.30 × 0.28 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS* in *APEX2*; Bruker, 2006)
T_{min} = 0.826, T_{max} = 0.852
16527 measured reflections
3576 independent reflections
3503 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.081
S = 1.11
3576 reflections
276 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), with 1490 Friedel pairs
Flack parameter: 0.09 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1′—H1N′···O3	0.92 (2)	2.709 (17)	3.0778 (17)	104.9 (12)

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELX97* and local procedures.

Financial support from the Kentucky Lung Cancer Research Program is gratefully acknowleged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2301).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2006). *APEX2*. Bruker AXS Inc, Madison, Wisconsin, USA.
Crooks, P. A., Jordan, C. T. & Wei, X. (2005). US Patent Appl. Publ. Cont.-in-part of US Ser. No. 888 274.
Desiraju, G. R. & Steiner, T. D. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*, p. 13. Oxford University Press.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Nasim, S., Parkin, S. & Crooks, P. A. (2007). *Acta Cryst.* E**63**, o3922.
Parsons, S. & Flack, H. D. (2004). *Abstracts of the 22nd European Crystallography Meeting*, Budapest, 26–31 August 2004, Abstract No. MS22.
Sheldrick, G. M. (1995). *XP* in *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807046983

supplementary materials

Acta Cryst. (2007). E**63**, o4274 [**doi:10.1107/S1600536807046983**]

(11*R*)-13-(1-Naphthylmethylamino)-4,5-epoxy-11,13-dihydrocostunolide

S. Nasim, S. Parkin and P. A. Crooks

Comment

The title compound was synthesized as part of our ongoing drug discovery effort (Crooks *et al.*, 2005), by the reaction of 1-naphthylmethylamine with parthenolide, and was shown to be a single diastereomer by NMR spectroscopic analysis. The crystal structure of the title compound was determined to obtain the configuration of the newly formed chiral center at C-11. An *R* absolute configuration was found.

The naphthalene ring of the amino side-chain is stacked against the cyclodecene ring of the parthenolide backbone. We believe that such a stacked structure is also consistent with the conformation of the title compound in the solution state ($CDCl_3$), due to the unusual upfield resonance (4.66 p.p.m.) of the C-1 olefinic hydrogen as opposed to the usual value (*ca* 5.1–5.2 p.p.m.), in the NMR spectrum. This upfield shift is likely to be a result of anisotropic shielding of the C-1 hydrogen by the Pi cloud of the naphthalene ring. An intramolecular H-bonding is observed between N-1H and O3 (2.70 (17) A°, 3.07 (17) A°, 104.9 (12)°) of the carbonyl oxygen of the 5-membered lactone ring (Desiraju & Steiner, 1999). Other bond distances and angles within the molecule are quite regular (Allen *et al.*, 1987).

Experimental

The title compound was prepared acording to the previously reported procedure of Nasim *et al.* (2007). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a chloroform/hexane solution at room temperature. The title compound was obtained as white crystals. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 8.29 (d, 1H, J = 8.4 Hz), 7.83 (m, 2H), 7.57 (m, 4H), 4.66 (app. d, 1H, J = 10.2 Hz), 4.45 (d, 1H, J = 13.2 Hz) 4.10 (d, 1H, J = 13.2 Hz), 3.73 (t, 1H, J = 9.0 Hz), 3.24 (dd, 1H, J = 3.6, 12.6 Hz), 2.70 (dd, 1H, J = 12.6, 4.8 Hz), 2.51 (d, 1H, J = 9.0 Hz), 2.40–2.24 (m, 2H), 2.11–1.86 (m, 7H), 1.58 (s, 3H), 1.53–1.09 (m, 2H), 1.22 (s, 3H); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 176.7, 135.0, 134.3, 134.1, 132.1, 128.8, 128.2, 126.9, 126.0, 125.8, 125.3, 124.9, 124.6, 82.7, 66.4, 61.6, 51.9, 48.8, 45.8, 45.1, 40.7, 36.7, 30.2, 24.2, 17.4, 17.0.

Refinement

H atoms were found in difference Fourier maps and those attached to carbon atoms were subsequently placed in idealized positions with constrained C—H distances of 0.98 Å (RCH_3), 0.99 Å (R_2CH_2), 1.00 Å (R_3CH) and 0.95 Å ($C_{Ar}H$) with U_{iso}(H) values set to either $1.5U_{eq}$ (methyl) or $1.2U_{eq}$ of the attached C atom respectively. Since the NH hydrogen was clearly not planar, and there being no suitable riding model available, the coordinates of this H atom were refined but its U_{iso} was set to $1.5U_{eq}$ of the attached N atom. Since this crystal structure was known to be of an all light-atom chiral compound, Cu Kα *x*-rays were used so that the absolute configuration could be determined from the anomalous scattering of the oxygen atoms. The value of the Flack parameter (Flack, 1983) based on refinement with unmerged Friedel pairs, as determined by the Parsons' quotient method (Parsons & Flack, 2004) was *x*(*u*) = 0.13 (6).

Figures

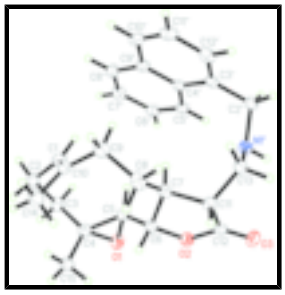


Fig. 1. A view of the asymmetric unit of the title compound, with displacement ellipsoids drawn at the 50% probability level; H atoms are shown as small spheres of arbitrary radii.

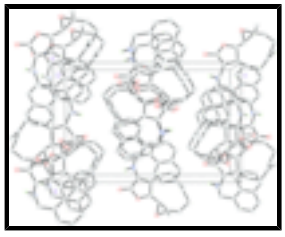


Fig. 2. A packing diagram, viewed down the *a* axis, hydrogen atoms have been omitted for clarity.

(11*R*)-13-(1-Naphthylmethylamino)-4,5-epoxy-11,13-dihydrocostunolide

Crystal data

$C_{26}H_{31}NO_3$	F_{000} = 872
M_r = 405.52	D_x = 1.294 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation λ = 1.54178 Å
Hall symbol: P 2ac 2ab	Cell parameters from 7441 reflections
a = 9.3842 (6) Å	θ = 4.4–67.0º
b = 11.9025 (7) Å	μ = 0.66 mm^{-1}
c = 18.6386 (13) Å	T = 90.0 (2) K
V = 2081.8 (2) Å^3	Cut block, colourless
Z = 4	0.30 × 0.28 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer	3576 independent reflections
Radiation source: fine-focus rotating anode	3503 reflections with $I > 2\sigma(I)$
Monochromator: graded multilayer optics	R_{int} = 0.034
T = 90.0(2) K	θ_{max} = 67.3º
φ and ω scans	θ_{min} = 4.4º
Absorption correction: multi-scan (SADABS in APEX2; Bruker, 2006)	h = −11→10
T_{min} = 0.826, T_{max} = 0.852	k = −14→14
16527 measured reflections	l = −20→22

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.31P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.034$	$(\Delta/\sigma)_{max} = 0.003$
$wR(F^2) = 0.081$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
3576 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
276 parameters	Extinction coefficient: 0.0179 (7)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 1490 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.09 (5)
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2\sigma(F^2)$ 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}
O1	0.73981 (11)	0.85246 (8)	0.94404 (6)	0.0238 (2)
C1	0.81331 (16)	0.62589 (13)	0.78893 (8)	0.0243 (3)
H1A	0.7323	0.5806	0.7989	0.029*
O2	0.82549 (12)	0.70644 (8)	1.05639 (5)	0.0245 (2)
C2	0.78375 (17)	0.73806 (13)	0.75591 (8)	0.0261 (3)
H2A	0.7151	0.7293	0.7160	0.031*
H2B	0.8730	0.7698	0.7362	0.031*
O3	0.82066 (12)	0.62291 (9)	1.16246 (6)	0.0294 (3)
C3	0.72219 (16)	0.81826 (12)	0.81239 (8)	0.0241 (3)
H3A	0.7175	0.8952	0.7923	0.029*
H3B	0.6241	0.7946	0.8247	0.029*
C4	0.81190 (15)	0.81904 (11)	0.87883 (8)	0.0203 (3)
C5	0.77511 (15)	0.73593 (11)	0.93375 (8)	0.0205 (3)
H5A	0.6921	0.6871	0.9212	0.025*

C6	0.87838 (15)	0.68344 (12)	0.98458 (8)	0.0205 (3)
H6A	0.9752	0.7169	0.9781	0.025*
C7	0.88646 (15)	0.55483 (11)	0.98032 (8)	0.0199 (3)
H7A	0.7902	0.5255	0.9670	0.024*
C8	0.99634 (16)	0.50330 (13)	0.92968 (8)	0.0269 (3)
H8A	1.0292	0.4316	0.9509	0.032*
H8B	1.0796	0.5542	0.9277	0.032*
C9	0.94802 (17)	0.48003 (12)	0.85268 (8)	0.0273 (3)
H9A	1.0153	0.4262	0.8305	0.033*
H9B	0.8532	0.4437	0.8542	0.033*
C10	0.93891 (16)	0.58185 (12)	0.80601 (8)	0.0228 (3)
C11	0.91569 (15)	0.52442 (12)	1.05852 (8)	0.0208 (3)
H11A	1.0210	0.5256	1.0666	0.025*
C12	0.85036 (15)	0.61909 (12)	1.09992 (8)	0.0219 (3)
C13	0.85856 (15)	0.41104 (12)	1.08246 (8)	0.0217 (3)
H13A	0.8896	0.3522	1.0483	0.026*
H13B	0.8975	0.3923	1.1303	0.026*
C14	1.08003 (16)	0.62865 (13)	0.78347 (9)	0.0280 (3)
H14A	1.0650	0.6952	0.7534	0.042*
H14B	1.1325	0.5718	0.7561	0.042*
H14C	1.1351	0.6496	0.8261	0.042*
C15	0.95707 (16)	0.86843 (12)	0.87159 (8)	0.0237 (3)
H15A	1.0067	0.8638	0.9177	0.036*
H15B	0.9491	0.9473	0.8570	0.036*
H15C	1.0109	0.8267	0.8353	0.036*
N1'	0.70323 (13)	0.41347 (10)	1.08575 (7)	0.0206 (3)
H1N'	0.6721 (18)	0.4342 (14)	1.1307 (11)	0.028 (4)*
C2'	0.63519 (15)	0.30803 (12)	1.06522 (8)	0.0216 (3)
H2'A	0.5347	0.3087	1.0814	0.026*
H2'B	0.6841	0.2449	1.0895	0.026*
C3'	0.63980 (15)	0.28988 (11)	0.98538 (8)	0.0193 (3)
C4'	0.56993 (14)	0.36495 (11)	0.93748 (8)	0.0183 (3)
C5'	0.48953 (15)	0.45765 (11)	0.96127 (8)	0.0195 (3)
H5'A	0.4802	0.4711	1.0113	0.023*
C6'	0.42482 (15)	0.52852 (12)	0.91389 (8)	0.0222 (3)
H6'A	0.3694	0.5896	0.9311	0.027*
C7'	0.43983 (15)	0.51149 (13)	0.83983 (8)	0.0243 (3)
H7'A	0.3969	0.5623	0.8070	0.029*
C8'	0.51576 (15)	0.42240 (12)	0.81488 (8)	0.0230 (3)
H8'A	0.5251	0.4112	0.7646	0.028*
C9'	0.58078 (14)	0.34651 (11)	0.86271 (8)	0.0194 (3)
C10'	0.65761 (15)	0.25253 (13)	0.83713 (8)	0.0228 (3)
H10A	0.6650	0.2397	0.7870	0.027*
C11'	0.72056 (16)	0.18102 (12)	0.88348 (9)	0.0252 (3)
H11B	0.7710	0.1176	0.8659	0.030*
C12'	0.71158 (15)	0.20023 (12)	0.95787 (8)	0.0235 (3)
H12A	0.7569	0.1492	0.9898	0.028*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0280 (5)	0.0214 (5)	0.0221 (5)	0.0066 (4)	0.0010 (4)	0.0003 (4)
C1	0.0242 (7)	0.0244 (7)	0.0243 (7)	−0.0060 (6)	0.0018 (6)	−0.0065 (6)
O2	0.0343 (6)	0.0183 (5)	0.0208 (5)	−0.0004 (4)	−0.0029 (4)	−0.0007 (4)
C2	0.0270 (8)	0.0314 (8)	0.0198 (7)	−0.0012 (7)	−0.0046 (6)	−0.0013 (6)
O3	0.0378 (6)	0.0303 (5)	0.0203 (5)	−0.0011 (5)	−0.0039 (5)	−0.0004 (4)
C3	0.0247 (8)	0.0238 (7)	0.0239 (7)	0.0020 (6)	−0.0028 (6)	0.0024 (6)
C4	0.0222 (7)	0.0170 (6)	0.0218 (7)	0.0035 (5)	−0.0001 (6)	−0.0022 (6)
C5	0.0199 (6)	0.0194 (7)	0.0222 (7)	0.0011 (6)	−0.0003 (6)	−0.0011 (5)
C6	0.0194 (7)	0.0204 (7)	0.0216 (7)	−0.0012 (5)	0.0002 (6)	0.0012 (6)
C7	0.0185 (7)	0.0187 (6)	0.0224 (8)	0.0007 (5)	0.0003 (6)	0.0014 (5)
C8	0.0260 (7)	0.0267 (7)	0.0280 (8)	0.0064 (6)	0.0044 (6)	0.0044 (6)
C9	0.0316 (8)	0.0201 (7)	0.0302 (8)	0.0024 (6)	0.0087 (7)	−0.0038 (6)
C10	0.0262 (8)	0.0227 (7)	0.0197 (7)	−0.0026 (6)	0.0039 (6)	−0.0060 (6)
C11	0.0187 (6)	0.0210 (7)	0.0227 (7)	−0.0006 (6)	−0.0033 (6)	0.0014 (6)
C12	0.0226 (7)	0.0206 (7)	0.0223 (8)	−0.0053 (6)	−0.0055 (6)	0.0007 (6)
C13	0.0189 (7)	0.0207 (6)	0.0257 (7)	−0.0002 (5)	−0.0033 (6)	0.0047 (6)
C14	0.0250 (7)	0.0279 (7)	0.0312 (8)	0.0001 (6)	0.0061 (7)	0.0002 (7)
C15	0.0281 (8)	0.0195 (6)	0.0236 (7)	−0.0019 (6)	−0.0018 (6)	0.0002 (6)
N1'	0.0191 (6)	0.0217 (6)	0.0212 (6)	−0.0008 (5)	0.0006 (5)	−0.0008 (5)
C2'	0.0199 (7)	0.0219 (7)	0.0229 (7)	−0.0046 (6)	−0.0010 (6)	0.0031 (6)
C3'	0.0153 (7)	0.0193 (6)	0.0231 (7)	−0.0044 (5)	0.0007 (6)	0.0009 (5)
C4'	0.0146 (6)	0.0178 (6)	0.0224 (7)	−0.0043 (5)	0.0008 (5)	−0.0009 (6)
C5'	0.0171 (6)	0.0198 (7)	0.0217 (7)	−0.0028 (5)	0.0007 (5)	−0.0029 (5)
C6'	0.0174 (7)	0.0182 (6)	0.0309 (8)	−0.0005 (5)	0.0000 (6)	−0.0031 (6)
C7'	0.0186 (7)	0.0265 (7)	0.0279 (7)	−0.0014 (6)	−0.0041 (6)	0.0053 (6)
C8'	0.0182 (7)	0.0303 (7)	0.0204 (7)	−0.0054 (6)	−0.0009 (6)	−0.0001 (6)
C9'	0.0144 (6)	0.0217 (7)	0.0220 (7)	−0.0038 (5)	0.0002 (5)	−0.0026 (5)
C10'	0.0198 (7)	0.0259 (7)	0.0228 (7)	−0.0034 (6)	0.0027 (6)	−0.0084 (6)
C11'	0.0218 (7)	0.0207 (7)	0.0332 (8)	0.0012 (6)	0.0028 (6)	−0.0061 (6)
C12'	0.0181 (7)	0.0213 (7)	0.0312 (8)	0.0000 (6)	−0.0002 (6)	0.0026 (6)

Geometric parameters (Å, °)

O1—C5	1.4388 (16)	C13—N1'	1.4592 (19)
O1—C4	1.4467 (17)	C13—H13A	0.9900
C1—C10	1.329 (2)	C13—H13B	0.9900
C1—C2	1.496 (2)	C14—H14A	0.9800
C1—H1A	0.9500	C14—H14B	0.9800
O2—C12	1.3393 (18)	C14—H14C	0.9800
O2—C6	1.4534 (17)	C15—H15A	0.9800
C2—C3	1.534 (2)	C15—H15B	0.9800
C2—H2A	0.9900	C15—H15C	0.9800
C2—H2B	0.9900	N1'—C2'	1.4591 (18)
O3—C12	1.1994 (18)	N1'—H1N'	0.92 (2)
C3—C4	1.497 (2)	C2'—C3'	1.504 (2)

C3—H3A	0.9900	C2'—H2'A	0.9900
C3—H3B	0.9900	C2'—H2'B	0.9900
C4—C5	1.465 (2)	C3'—C12'	1.362 (2)
C4—C15	1.490 (2)	C3'—C4'	1.423 (2)
C5—C6	1.492 (2)	C4'—C5'	1.408 (2)
C5—H5A	1.0000	C4'—C9'	1.414 (2)
C6—C7	1.5348 (19)	C5'—C6'	1.364 (2)
C6—H6A	1.0000	C5'—H5'A	0.9500
C7—C8	1.527 (2)	C6'—C7'	1.402 (2)
C7—C11	1.527 (2)	C6'—H6'A	0.9500
C7—H7A	1.0000	C7'—C8'	1.360 (2)
C8—C9	1.530 (2)	C7'—H7'A	0.9500
C8—H8A	0.9900	C8'—C9'	1.408 (2)
C8—H8B	0.9900	C8'—H8'A	0.9500
C9—C10	1.494 (2)	C9'—C10'	1.414 (2)
C9—H9A	0.9900	C10'—C11'	1.349 (2)
C9—H9B	0.9900	C10'—H10A	0.9500
C10—C14	1.497 (2)	C11'—C12'	1.408 (2)
C11—C12	1.497 (2)	C11'—H11B	0.9500
C11—C13	1.5190 (19)	C12'—H12A	0.9500
C11—H11A	1.0000		
C5—O1—C4	61.01 (9)	C7—C11—H11A	108.5
C10—C1—C2	127.96 (14)	O3—C12—O2	121.26 (14)
C10—C1—H1A	116.0	O3—C12—C11	128.65 (14)
C2—C1—H1A	116.0	O2—C12—C11	110.08 (12)
C12—O2—C6	110.62 (11)	N1'—C13—C11	110.32 (12)
C1—C2—C3	110.05 (12)	N1'—C13—H13A	109.6
C1—C2—H2A	109.7	C11—C13—H13A	109.6
C3—C2—H2A	109.7	N1'—C13—H13B	109.6
C1—C2—H2B	109.7	C11—C13—H13B	109.6
C3—C2—H2B	109.7	H13A—C13—H13B	108.1
H2A—C2—H2B	108.2	C10—C14—H14A	109.5
C4—C3—C2	111.08 (12)	C10—C14—H14B	109.5
C4—C3—H3A	109.4	H14A—C14—H14B	109.5
C2—C3—H3A	109.4	C10—C14—H14C	109.5
C4—C3—H3B	109.4	H14A—C14—H14C	109.5
C2—C3—H3B	109.4	H14B—C14—H14C	109.5
H3A—C3—H3B	108.0	C4—C15—H15A	109.5
O1—C4—C5	59.23 (9)	C4—C15—H15B	109.5
O1—C4—C15	113.28 (12)	H15A—C15—H15B	109.5
C5—C4—C15	123.04 (13)	C4—C15—H15C	109.5
O1—C4—C3	115.70 (12)	H15A—C15—H15C	109.5
C5—C4—C3	116.19 (13)	H15B—C15—H15C	109.5
C15—C4—C3	116.20 (13)	C2'—N1'—C13	114.15 (12)
O1—C5—C4	59.76 (9)	C2'—N1'—H1N'	109.3 (11)
O1—C5—C6	117.93 (12)	C13—N1'—H1N'	111.1 (11)
C4—C5—C6	124.99 (13)	N1'—C2'—C3'	111.73 (12)
O1—C5—H5A	114.3	N1'—C2'—H2'A	109.3
C4—C5—H5A	114.3	C3'—C2'—H2'A	109.3

C6—C5—H5A	114.3	N1'—C2'—H2'B	109.3
O2—C6—C5	106.50 (11)	C3'—C2'—H2'B	109.3
O2—C6—C7	104.62 (11)	H2'A—C2'—H2'B	107.9
C5—C6—C7	114.64 (12)	C12'—C3'—C4'	118.91 (13)
O2—C6—H6A	110.3	C12'—C3'—C2'	119.94 (13)
C5—C6—H6A	110.3	C4'—C3'—C2'	121.14 (12)
C7—C6—H6A	110.3	C5'—C4'—C9'	118.05 (13)
C8—C7—C11	111.95 (12)	C5'—C4'—C3'	122.77 (13)
C8—C7—C6	117.78 (12)	C9'—C4'—C3'	119.18 (12)
C11—C7—C6	101.30 (11)	C6'—C5'—C4'	121.28 (13)
C8—C7—H7A	108.5	C6'—C5'—H5'A	119.4
C11—C7—H7A	108.5	C4'—C5'—H5'A	119.4
C6—C7—H7A	108.5	C5'—C6'—C7'	120.22 (13)
C7—C8—C9	116.90 (13)	C5'—C6'—H6'A	119.9
C7—C8—H8A	108.1	C7'—C6'—H6'A	119.9
C9—C8—H8A	108.1	C8'—C7'—C6'	120.13 (14)
C7—C8—H8B	108.1	C8'—C7'—H7'A	119.9
C9—C8—H8B	108.1	C6'—C7'—H7'A	119.9
H8A—C8—H8B	107.3	C7'—C8'—C9'	120.73 (14)
C10—C9—C8	114.59 (13)	C7'—C8'—H8'A	119.6
C10—C9—H9A	108.6	C9'—C8'—H8'A	119.6
C8—C9—H9A	108.6	C8'—C9'—C10'	121.00 (13)
C10—C9—H9B	108.6	C8'—C9'—C4'	119.54 (13)
C8—C9—H9B	108.6	C10'—C9'—C4'	119.46 (13)
H9A—C9—H9B	107.6	C11'—C10'—C9'	120.44 (13)
C1—C10—C9	120.68 (14)	C11'—C10'—H10A	119.8
C1—C10—C14	124.80 (14)	C9'—C10'—H10A	119.8
C9—C10—C14	114.50 (13)	C10'—C11'—C12'	120.13 (13)
C12—C11—C13	111.88 (12)	C10'—C11'—H11B	119.9
C12—C11—C7	103.89 (11)	C12'—C11'—H11B	119.9
C13—C11—C7	115.32 (12)	C3'—C12'—C11'	121.85 (14)
C12—C11—H11A	108.5	C3'—C12'—H12A	119.1
C13—C11—H11A	108.5	C11'—C12'—H12A	119.1
C10—C1—C2—C3	−106.01 (18)	C6—O2—C12—O3	176.63 (13)
C1—C2—C3—C4	50.10 (16)	C6—O2—C12—C11	−3.70 (16)
C5—O1—C4—C15	−115.77 (13)	C13—C11—C12—O3	38.6 (2)
C5—O1—C4—C3	106.47 (14)	C7—C11—C12—O3	163.59 (15)
C2—C3—C4—O1	−155.94 (12)	C13—C11—C12—O2	−141.08 (12)
C2—C3—C4—C5	−89.27 (15)	C7—C11—C12—O2	−16.04 (15)
C2—C3—C4—C15	67.55 (16)	C12—C11—C13—N1'	48.63 (16)
C4—O1—C5—C6	116.24 (15)	C7—C11—C13—N1'	−69.81 (16)
C15—C4—C5—O1	99.28 (15)	C11—C13—N1'—C2'	144.82 (13)
C3—C4—C5—O1	−105.64 (13)	C13—N1'—C2'—C3'	−74.63 (16)
O1—C4—C5—C6	−104.71 (15)	N1'—C2'—C3'—C12'	117.47 (15)
C15—C4—C5—C6	−5.4 (2)	N1'—C2'—C3'—C4'	−62.64 (17)
C3—C4—C5—C6	149.66 (13)	C12'—C3'—C4'—C5'	177.91 (13)
C12—O2—C6—C5	143.65 (12)	C2'—C3'—C4'—C5'	−2.0 (2)
C12—O2—C6—C7	21.87 (15)	C12'—C3'—C4'—C9'	−2.02 (19)
O1—C5—C6—O2	52.48 (16)	C2'—C3'—C4'—C9'	178.08 (12)

C4—C5—C6—O2	123.53 (14)	C9'—C4'—C5'—C6'	−0.60 (19)
O1—C5—C6—C7	167.65 (12)	C3'—C4'—C5'—C6'	179.47 (13)
C4—C5—C6—C7	−121.30 (15)	C4'—C5'—C6'—C7'	−1.3 (2)
O2—C6—C7—C8	−152.32 (12)	C5'—C6'—C7'—C8'	1.8 (2)
C5—C6—C7—C8	91.42 (16)	C6'—C7'—C8'—C9'	−0.3 (2)
O2—C6—C7—C11	−29.90 (14)	C7'—C8'—C9'—C10'	178.80 (13)
C5—C6—C7—C11	−146.16 (12)	C7'—C8'—C9'—C4'	−1.6 (2)
C11—C7—C8—C9	152.93 (13)	C5'—C4'—C9'—C8'	2.07 (19)
C6—C7—C8—C9	−90.25 (17)	C3'—C4'—C9'—C8'	−177.99 (13)
C7—C8—C9—C10	76.25 (17)	C5'—C4'—C9'—C10'	−178.36 (12)
C2—C1—C10—C9	166.76 (14)	C3'—C4'—C9'—C10'	1.57 (19)
C2—C1—C10—C14	−11.4 (2)	C8'—C9'—C10'—C11'	179.37 (14)
C8—C9—C10—C1	−105.11 (17)	C4'—C9'—C10'—C11'	−0.2 (2)
C8—C9—C10—C14	73.23 (16)	C9'—C10'—C11'—C12'	−0.8 (2)
C8—C7—C11—C12	153.82 (12)	C4'—C3'—C12'—C11'	1.1 (2)
C6—C7—C11—C12	27.45 (14)	C2'—C3'—C12'—C11'	−178.98 (13)
C8—C7—C11—C13	−83.38 (15)	C10'—C11'—C12'—C3'	0.3 (2)
C6—C7—C11—C13	150.25 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1'—H1N'···O3	0.92 (2)	2.709 (17)	3.0778 (17)	104.9 (12)

Fig. 1

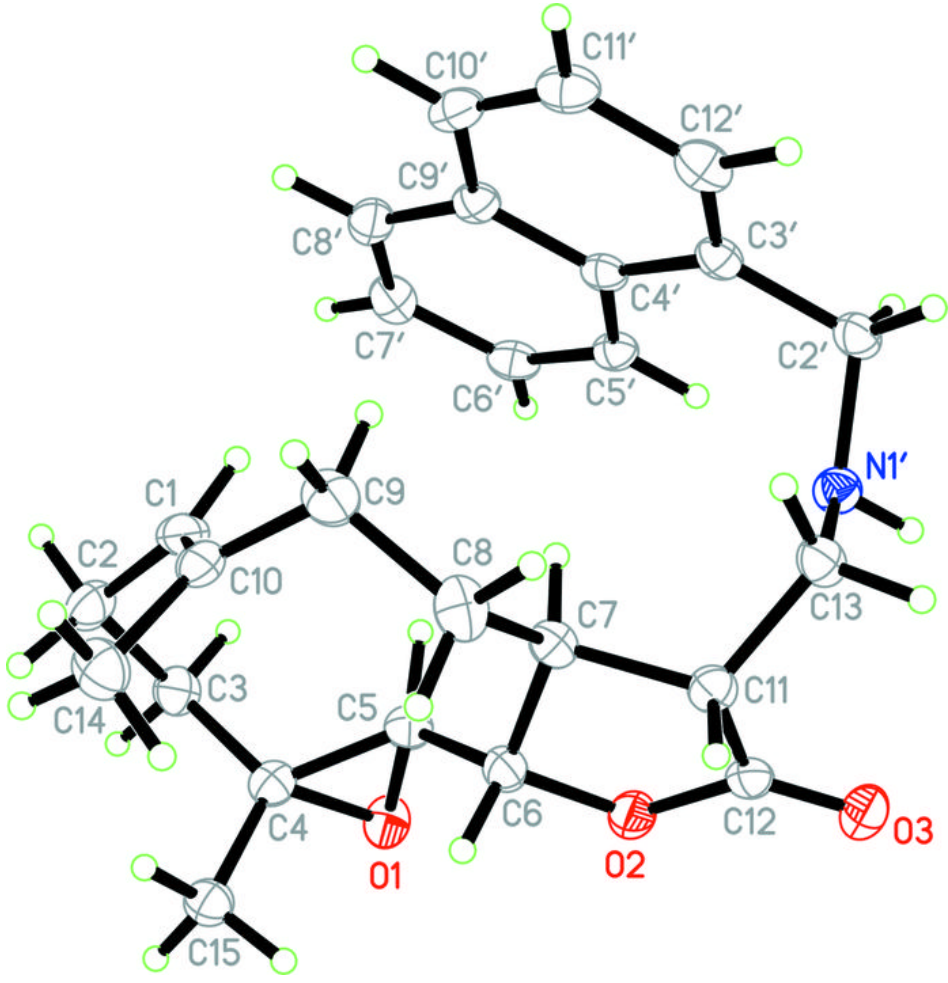

C10′
C11′
C12′
C9′
C8′
C3′
C4′
C7′
C2′
C6′
C5′
N1′
C1
C9
C2
C10
C8
C7
C13
C3
C11
C14
C5
C4
C6
C12
O3
O1
O2
C15

Fig. 2