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(11*R*)-13-(Benzylamino)-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

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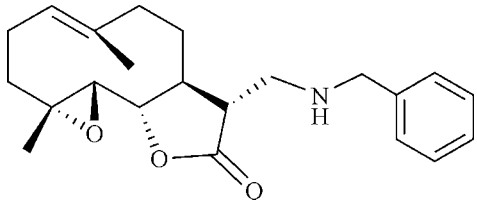
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.096; data-to-parameter ratio = 14.7.

The title compound, $C_{22}H_{29}NO_3$, was obtained by the reaction of benzylamine with parthenolide. X-ray crystal structure determination revealed that the configuration of the new chiral center at C11 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); Guzman *et al.* (2005); de Kraker *et al.* (2002); Parsons & Flack (2004); Flack (1983)



Experimental

Crystal data

$C_{22}H_{29}NO_3$
M_r = 355.46
Orthorhombic, $P2_12_12_1$
a = 8.3772 (6) Å
b = 13.7441 (10) Å
c = 16.9684 (12) Å
V = 1953.7 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 90.0 (2) K
0.35 × 0.08 × 0.08 mm

Data collection

Bruker–Nonius X8 Proteum diffractometer
Absorption correction: none
21942 measured reflections
3531 independent reflections
3446 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.08
3531 reflections
241 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), with 1493 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—H1*N*···O3	0.92 (2)	2.303 (17)	2.9784 (17)	130.2 (8)

Data collection: *APEX2* (Bruker–Nonius, 2004); 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/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2279).

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supplementary materials

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

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

Comment

Parthenolide, is a germacrene sesquiterpene lactone. It has been isolated from several different species of plant in the Asteraceae (Compositae) family, feverfew (Tanacetum parthenium) being one of them (de Kraker *et al.*, 2002). Its remarkable cytotoxic, antitumor, antiviral and antileishmanic properties have made it the focus of a large number of investigations. Its ability to induce tumor apoptosis through the inhibition of NF-kappaB make it a potential therapeutic for the treatment of leukemia (Guzman *et al.*, 2005). Its development in this regard has been impeded by its low polarity and low water solubility. We saw a potential solution to this problem in the formation of a conjugate adduct of an amine species at the C-13, α-methylene group of the γ-butyrolactone function of parthenolide (Crooks *et al.*, 2005). The title compound was synthesized as part of an ongoing drug discovery effort by reaction of benzylamine with parthenolide. The benzylamine adduct obtained was shown to be a single diastereomer by NMR analysis. The crystal structure of the title compound was determined to obtain the configuration of the newly formed stereocenter at C-11. An *R* absolute configuration was found. Bond distances and angles within the molecule are quite regular with average normal bonds (Allen *et al.*, 1987). A hydrogen bond is observed between N-1H and O3 (2.30 (17) A°, 2.97 (17) A°, 130.2 (8) °) (Table 1) of the carbonyl oxygen of the 5-membered lactone ring, which can be considered as a weak hydrogen bond (Desiraju & Steiner, 1999).

Experimental

The title compound was prepared by dissolving parthenolide (100 mg, 0.403 mmol) in MeOH (10 ml) and adding benzylamine (43.1 mg, 0.403 mmol) to the solution. The mixture was stirred at room temperature for 18 h. The solvent was evaporated and the title compound was purified by column chromatography over silica gel by eluting with 15% acetone in hexane. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in chloroform/hexane at room temperature. The title compound was obtained as white crystals. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 7.35–7.26 (m, 5H), 5.19 (app. d, 1H, J = 10.2), 3.92–3.75 (m, 3H), 3.02 (dd, 1H, J = 3.6 and 12.3 Hz), 2.74 (m, 2H), 2.44–1.78 (m, 11 H), 1.69 (s, 3H), 1.29 (s, 3H); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 176.7, 140.0, 134.6, 128.5, 128.1, 127.1, 125.2, 82.7, 66.5, 61.7, 54.1, 48.4, 46.7, 46.2, 41.2, 36.8, 30.2, 24.3, 17.5, 17.2.

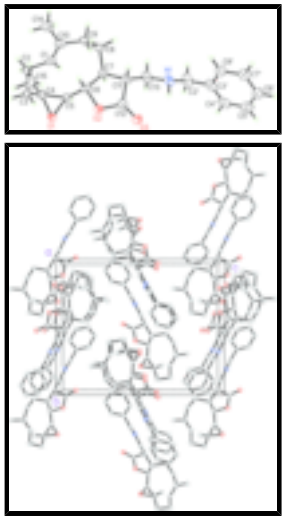
Refinement

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 0.98Å (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



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

Crystal data

$C_{22}H_{29}NO_3$	$F_{000} = 768$
$M_r = 355.46$	$D_x = 1.208$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation $\lambda = 1.54178$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 7188 reflections
$a = 8.3772$ (6) Å	$\theta = 4.1$–$68.0°$
$b = 13.7441$ (10) Å	$\mu = 0.63$ mm^{-1}
$c = 16.9684$ (12) Å	$T = 90.0$ (2) K
$V = 1953.7$ (2) Å^3	Rod, colourless
$Z = 4$	0.35 × 0.08 × 0.08 mm

Data collection

X8 Proteum diffractometer	3446 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus rotating anode	$R_{int} = 0.043$
Monochromator: Helios multilayer optics	$\theta_{max} = 68.0°$
$T = 90.0(2)$ K	$\theta_{min} = 4.1°$
ω and φ scans	$h = -10 \rightarrow 10$
Absorption correction: none	$k = -16 \rightarrow 16$
21942 measured reflections	$l = -20 \rightarrow 20$
3531 independent reflections	Standard reflections: ?

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.2363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
3531 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
241 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.0055 (5)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 0000 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.13 (6)

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.

Flack parameter from normal least-squares (unmerged data): is $x = 0.09232$; su = 0.07777 Flack parameter from Parson's quotients: CC = 15.45, GooF 1.2789, Flack $x = 0.1327$; su = 0.0656 Result from Parsons quotients given in this CIF.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.48723 (15)	0.32099 (8)	0.46644 (9)	0.0325 (3)
H1N	0.422 (3)	0.3350 (9)	0.4245 (12)	0.049*
O1	0.24522 (12)	0.80545 (7)	0.50257 (5)	0.0269 (2)
O2	0.26117 (12)	0.60530 (7)	0.47005 (6)	0.0282 (2)
O3	0.28650 (15)	0.47566 (7)	0.39368 (6)	0.0397 (3)
C1	0.54615 (17)	0.79131 (10)	0.68316 (8)	0.0260 (3)
H1	0.6343	0.7856	0.6482	0.031*
C2	0.46528 (19)	0.88816 (10)	0.68533 (8)	0.0309 (3)
H2A	0.5454	0.9401	0.6938	0.037*
H2B	0.3883	0.8901	0.7296	0.037*
C3	0.37714 (17)	0.90558 (10)	0.60700 (8)	0.0292 (3)
H3A	0.3096	0.9644	0.6116	0.035*

H3B	0.4560	0.9170	0.5645	0.035*
C4	0.27459 (16)	0.81977 (10)	0.58600 (8)	0.0266 (3)
C5	0.35340 (15)	0.74075 (9)	0.54297 (7)	0.0211 (3)
H5	0.4688	0.7525	0.5313	0.025*
C6	0.30846 (16)	0.63576 (10)	0.54922 (7)	0.0233 (3)
H6	0.2174	0.6275	0.5868	0.028*
C7	0.44529 (16)	0.56754 (10)	0.57132 (8)	0.0247 (3)
H7	0.5462	0.5941	0.5484	0.030*
C8	0.4736 (2)	0.54673 (11)	0.65905 (9)	0.0349 (4)
H8A	0.3689	0.5472	0.6861	0.042*
H8B	0.5178	0.4802	0.6640	0.042*
C9	0.58504 (19)	0.61685 (10)	0.70247 (9)	0.0331 (3)
H9A	0.6238	0.5848	0.7511	0.040*
H9B	0.6789	0.6300	0.6687	0.040*
C10	0.50933 (17)	0.71205 (11)	0.72440 (8)	0.0305 (3)
C11	0.40039 (16)	0.47539 (10)	0.52589 (8)	0.0263 (3)
H11	0.3246	0.4365	0.5587	0.032*
C12	0.31212 (17)	0.51417 (10)	0.45586 (9)	0.0277 (3)
C13	0.53912 (17)	0.41115 (11)	0.50311 (10)	0.0335 (3)
H13A	0.6091	0.4469	0.4661	0.040*
H13B	0.6025	0.3958	0.5508	0.040*
C14	0.3954 (3)	0.70842 (16)	0.79204 (11)	0.0502 (5)
H14A	0.3438	0.7720	0.7981	0.075*
H14B	0.4536	0.6922	0.8404	0.075*
H14C	0.3141	0.6587	0.7820	0.075*
C15	0.13073 (18)	0.80289 (13)	0.63629 (10)	0.0369 (4)
H15A	0.0642	0.8615	0.6366	0.055*
H15B	0.1645	0.7878	0.6902	0.055*
H15C	0.0692	0.7482	0.6150	0.055*
C2'	0.62254 (18)	0.26381 (12)	0.44003 (13)	0.0444 (4)
H2'1	0.6892	0.2464	0.4861	0.053*
H2'2	0.6885	0.3036	0.4039	0.053*
C3'	0.57217 (17)	0.17232 (10)	0.39836 (10)	0.0329 (3)
C4'	0.62586 (19)	0.15239 (12)	0.32291 (10)	0.0389 (4)
H4'	0.6913	0.1983	0.2964	0.047*
C5'	0.58531 (19)	0.06646 (13)	0.28581 (9)	0.0391 (4)
H5'	0.6237	0.0532	0.2342	0.047*
C6'	0.48934 (19)	0.00007 (12)	0.32352 (9)	0.0351 (3)
H6'	0.4614	−0.0590	0.2980	0.042*
C7'	0.43343 (18)	0.01924 (11)	0.39868 (9)	0.0329 (3)
H7'	0.3664	−0.0264	0.4247	0.039*
C8'	0.47545 (17)	0.10493 (10)	0.43568 (9)	0.0307 (3)
H8'	0.4375	0.1178	0.4874	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0196 (6)	0.0285 (6)	0.0494 (7)	−0.0004 (5)	−0.0044 (6)	0.0005 (5)

O1	0.0237 (5)	0.0322 (5)	0.0248 (5)	0.0057 (4)	−0.0060 (4)	0.0004 (4)
O2	0.0274 (5)	0.0280 (5)	0.0291 (5)	0.0004 (4)	−0.0113 (4)	−0.0012 (4)
O3	0.0516 (7)	0.0332 (5)	0.0345 (6)	0.0028 (5)	−0.0147 (5)	−0.0036 (4)
C1	0.0213 (7)	0.0332 (7)	0.0236 (6)	−0.0046 (5)	−0.0039 (5)	−0.0001 (5)
C2	0.0319 (8)	0.0324 (7)	0.0284 (7)	−0.0029 (6)	−0.0016 (6)	−0.0044 (5)
C3	0.0296 (7)	0.0284 (6)	0.0296 (7)	0.0051 (6)	−0.0017 (6)	−0.0031 (6)
C4	0.0207 (6)	0.0351 (7)	0.0238 (6)	0.0069 (5)	−0.0025 (5)	−0.0024 (5)
C5	0.0148 (6)	0.0274 (6)	0.0213 (6)	0.0011 (5)	−0.0027 (5)	0.0007 (5)
C6	0.0182 (6)	0.0300 (6)	0.0215 (6)	−0.0040 (5)	−0.0026 (5)	−0.0002 (5)
C7	0.0209 (6)	0.0256 (6)	0.0276 (7)	−0.0063 (5)	−0.0060 (5)	0.0071 (5)
C8	0.0408 (9)	0.0327 (7)	0.0311 (7)	−0.0118 (7)	−0.0147 (7)	0.0110 (6)
C9	0.0313 (8)	0.0329 (7)	0.0350 (7)	−0.0068 (6)	−0.0134 (6)	0.0088 (6)
C10	0.0266 (7)	0.0373 (7)	0.0277 (7)	−0.0061 (6)	−0.0065 (6)	0.0044 (6)
C11	0.0204 (6)	0.0251 (6)	0.0334 (7)	−0.0053 (5)	−0.0055 (5)	0.0045 (6)
C12	0.0238 (6)	0.0261 (6)	0.0331 (7)	−0.0016 (6)	−0.0048 (6)	0.0006 (5)
C13	0.0196 (7)	0.0279 (7)	0.0531 (9)	−0.0040 (6)	−0.0065 (6)	0.0043 (6)
C14	0.0538 (11)	0.0618 (11)	0.0350 (8)	−0.0059 (9)	0.0059 (8)	0.0136 (8)
C15	0.0239 (7)	0.0512 (9)	0.0357 (8)	0.0058 (7)	0.0040 (6)	−0.0041 (6)
C2'	0.0202 (7)	0.0344 (8)	0.0784 (12)	0.0009 (6)	0.0010 (8)	−0.0026 (8)
C3'	0.0150 (6)	0.0324 (7)	0.0514 (9)	0.0046 (5)	0.0011 (6)	0.0049 (7)
C4'	0.0224 (7)	0.0471 (9)	0.0473 (9)	0.0084 (6)	0.0067 (6)	0.0191 (7)
C5'	0.0255 (7)	0.0611 (10)	0.0308 (7)	0.0157 (7)	0.0014 (6)	0.0070 (7)
C6'	0.0260 (7)	0.0422 (8)	0.0371 (7)	0.0095 (6)	−0.0029 (6)	−0.0046 (6)
C7'	0.0233 (7)	0.0362 (7)	0.0391 (8)	−0.0006 (6)	0.0007 (6)	0.0022 (6)
C8'	0.0194 (7)	0.0364 (7)	0.0361 (7)	0.0028 (6)	0.0043 (6)	−0.0005 (6)

Geometric parameters (Å, °)

N1—C2'	1.450 (2)	C9—C10	1.501 (2)
N1—C13	1.4532 (19)	C9—H9A	0.9900
N1—H1N	0.92 (2)	C9—H9B	0.9900
O1—C5	1.4429 (15)	C10—C14	1.494 (2)
O1—C4	1.4502 (16)	C11—C12	1.4975 (19)
O2—C12	1.3450 (17)	C11—C13	1.510 (2)
O2—C6	1.4617 (15)	C11—H11	1.0000
O3—C12	1.1998 (18)	C13—H13A	0.9900
C1—C10	1.331 (2)	C13—H13B	0.9900
C1—C2	1.494 (2)	C14—H14A	0.9800
C1—H1	0.9500	C14—H14B	0.9800
C2—C3	1.539 (2)	C14—H14C	0.9800
C2—H2A	0.9900	C15—H15A	0.9800
C2—H2B	0.9900	C15—H15B	0.9800
C3—C4	1.502 (2)	C15—H15C	0.9800
C3—H3A	0.9900	C2'—C3'	1.503 (2)
C3—H3B	0.9900	C2'—H2'1	0.9900
C4—C5	1.4658 (18)	C2'—H2'2	0.9900
C4—C15	1.495 (2)	C3'—C8'	1.384 (2)
C5—C6	1.4950 (18)	C3'—C4'	1.384 (2)
C5—H5	1.0000	C4'—C5'	1.381 (3)

C6—C7	1.5277 (19)	C4'—H4'	0.9500
C6—H6	1.0000	C5'—C6'	1.374 (2)
C7—C11	1.5297 (18)	C5'—H5'	0.9500
C7—C8	1.5343 (18)	C6'—C7'	1.384 (2)
C7—H7	1.0000	C6'—H6'	0.9500
C8—C9	1.531 (2)	C7'—C8'	1.380 (2)
C8—H8A	0.9900	C7'—H7'	0.9500
C8—H8B	0.9900	C8'—H8'	0.9500
C2'—N1—C13	111.13 (12)	H9A—C9—H9B	107.6
C2'—N1—H1N	109.8 (8)	C1—C10—C14	125.41 (16)
C13—N1—H1N	109.4 (8)	C1—C10—C9	119.03 (13)
C5—O1—C4	60.88 (8)	C14—C10—C9	115.56 (14)
C12—O2—C6	110.20 (10)	C12—C11—C13	112.65 (12)
C10—C1—C2	127.67 (14)	C12—C11—C7	103.10 (10)
C10—C1—H1	116.2	C13—C11—C7	115.08 (11)
C2—C1—H1	116.2	C12—C11—H11	108.6
C1—C2—C3	109.56 (11)	C13—C11—H11	108.6
C1—C2—H2A	109.8	C7—C11—H11	108.6
C3—C2—H2A	109.8	O3—C12—O2	120.76 (13)
C1—C2—H2B	109.8	O3—C12—C11	128.99 (13)
C3—C2—H2B	109.8	O2—C12—C11	110.25 (11)
H2A—C2—H2B	108.2	N1—C13—C11	112.21 (11)
C4—C3—C2	110.92 (11)	N1—C13—H13A	109.2
C4—C3—H3A	109.5	C11—C13—H13A	109.2
C2—C3—H3A	109.5	N1—C13—H13B	109.2
C4—C3—H3B	109.5	C11—C13—H13B	109.2
C2—C3—H3B	109.5	H13A—C13—H13B	107.9
H3A—C3—H3B	108.0	C10—C14—H14A	109.5
O1—C4—C5	59.32 (8)	C10—C14—H14B	109.5
O1—C4—C15	113.54 (12)	H14A—C14—H14B	109.5
C5—C4—C15	122.17 (13)	C10—C14—H14C	109.5
O1—C4—C3	115.80 (12)	H14A—C14—H14C	109.5
C5—C4—C3	116.26 (12)	H14B—C14—H14C	109.5
C15—C4—C3	116.59 (13)	C4—C15—H15A	109.5
O1—C5—C4	59.80 (8)	C4—C15—H15B	109.5
O1—C5—C6	118.06 (10)	H15A—C15—H15B	109.5
C4—C5—C6	124.50 (11)	C4—C15—H15C	109.5
O1—C5—H5	114.4	H15A—C15—H15C	109.5
C4—C5—H5	114.4	H15B—C15—H15C	109.5
C6—C5—H5	114.4	N1—C2'—C3'	112.29 (12)
O2—C6—C5	106.23 (10)	N1—C2'—H2'1	109.1
O2—C6—C7	104.67 (10)	C3'—C2'—H2'1	109.1
C5—C6—C7	114.89 (11)	N1—C2'—H2'2	109.1
O2—C6—H6	110.3	C3'—C2'—H2'2	109.1
C5—C6—H6	110.3	H2'1—C2'—H2'2	107.9
C7—C6—H6	110.3	C8'—C3'—C4'	118.74 (15)
C6—C7—C11	101.53 (10)	C8'—C3'—C2'	120.59 (15)
C6—C7—C8	117.94 (12)	C4'—C3'—C2'	120.63 (15)
C11—C7—C8	111.88 (11)	C5'—C4'—C3'	120.73 (15)

C6—C7—H7	108.3	C5'—C4'—H4'	119.6
C11—C7—H7	108.3	C3'—C4'—H4'	119.6
C8—C7—H7	108.3	C6'—C5'—C4'	119.98 (15)
C9—C8—C7	116.34 (12)	C6'—C5'—H5'	120.0
C9—C8—H8A	108.2	C4'—C5'—H5'	120.0
C7—C8—H8A	108.2	C5'—C6'—C7'	120.04 (15)
C9—C8—H8B	108.2	C5'—C6'—H6'	120.0
C7—C8—H8B	108.2	C7'—C6'—H6'	120.0
H8A—C8—H8B	107.4	C8'—C7'—C6'	119.70 (14)
C10—C9—C8	114.22 (13)	C8'—C7'—H7'	120.2
C10—C9—H9A	108.7	C6'—C7'—H7'	120.2
C8—C9—H9A	108.7	C7'—C8'—C3'	120.81 (14)
C10—C9—H9B	108.7	C7'—C8'—H8'	119.6
C8—C9—H9B	108.7	C3'—C8'—H8'	119.6
C10—C1—C2—C3	−110.86 (17)	C2—C1—C10—C9	168.37 (13)
C1—C2—C3—C4	50.01 (16)	C8—C9—C10—C1	−104.86 (16)
C5—O1—C4—C15	−114.63 (14)	C8—C9—C10—C14	74.63 (17)
C5—O1—C4—C3	106.53 (13)	C6—C7—C11—C12	29.69 (13)
C2—C3—C4—O1	−152.77 (11)	C8—C7—C11—C12	156.36 (12)
C2—C3—C4—C5	−85.95 (15)	C6—C7—C11—C13	152.74 (12)
C2—C3—C4—C15	69.67 (16)	C8—C7—C11—C13	−80.59 (16)
C4—O1—C5—C6	115.63 (13)	C6—O2—C12—O3	−179.43 (13)
C15—C4—C5—O1	100.10 (14)	C6—O2—C12—C11	−0.25 (15)
C3—C4—C5—O1	−105.76 (13)	C13—C11—C12—O3	35.0 (2)
O1—C4—C5—C6	−105.09 (13)	C7—C11—C12—O3	159.65 (16)
C15—C4—C5—C6	−5.0 (2)	C13—C11—C12—O2	−144.11 (12)
C3—C4—C5—C6	149.15 (12)	C7—C11—C12—O2	−19.44 (15)
C12—O2—C6—C5	141.96 (11)	C2'—N1—C13—C11	175.89 (14)
C12—O2—C6—C7	20.01 (14)	C12—C11—C13—N1	−67.32 (15)
O1—C5—C6—O2	48.47 (14)	C7—C11—C13—N1	174.88 (12)
C4—C5—C6—O2	119.49 (13)	C13—N1—C2'—C3'	−177.25 (14)
O1—C5—C6—C7	163.67 (11)	N1—C2'—C3'—C8'	−57.4 (2)
C4—C5—C6—C7	−125.31 (13)	N1—C2'—C3'—C4'	124.74 (16)
O2—C6—C7—C11	−30.40 (13)	C8'—C3'—C4'—C5'	−0.7 (2)
C5—C6—C7—C11	−146.50 (11)	C2'—C3'—C4'—C5'	177.20 (14)
O2—C6—C7—C8	−152.99 (11)	C3'—C4'—C5'—C6'	0.7 (2)
C5—C6—C7—C8	90.90 (14)	C4'—C5'—C6'—C7'	−0.1 (2)
C6—C7—C8—C9	−88.67 (17)	C5'—C6'—C7'—C8'	−0.5 (2)
C11—C7—C8—C9	154.15 (14)	C6'—C7'—C8'—C3'	0.4 (2)
C7—C8—C9—C10	78.04 (18)	C4'—C3'—C8'—C7'	0.2 (2)
C2—C1—C10—C14	−11.1 (2)	C2'—C3'—C8'—C7'	−177.73 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O3	0.92 (2)	2.303 (17)	2.9784 (17)	130.2 (8)

Fig. 1

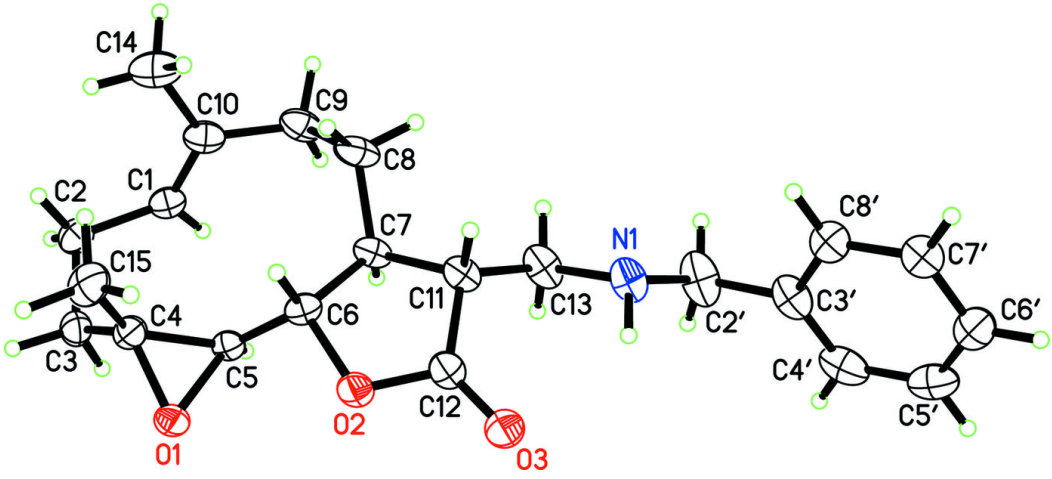

C14
C10
C9
C8
C1
C2
C7
C15
C4
C6
C11
C13
N1
C2′
C8′
C7′
C3′
C6′
C3
C5
C12
C4′
C5′
O1
O2
O3

Fig. 2