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## Structure Reports

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# (11*R*,13*R*)-13-(Tetralin-1-ylamino)-4,5-epoxy-11,13-dihydrocostunolide

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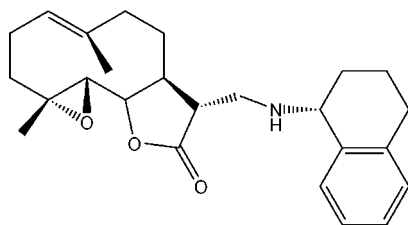
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 Key indicators: single-crystal X-ray study;  $T = 90$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.111; data-to-parameter ratio = 14.3.

The title compound [systematic name: (12*R*)-4,8-dimethyl-12-[(1'*R*)-1',2',3',4'-tetrahydro-1'-naphthyl]aminomethyl]-3,14-dioxatricyclo[9.3.0.0<sup>2,4</sup>]tetradec-7-en-13-one},  $\text{C}_{25}\text{H}_{33}\text{NO}_3$ , was formed from the reaction of (1*R*)-1-aminotetralin with parthenolide in methanolic solution. X-ray crystal structure analysis determined that the configuration of the new chiral center in the title compound was *R*.

## Related literature

 For related literature, see: Allen *et al.* (1987); Crooks *et al.* (2005); Desiraju & Steiner (1999); Nasim *et al.* (2007*a,b*).


## Experimental

### Crystal data

 $\text{C}_{25}\text{H}_{33}\text{NO}_3$ 
 $M_r = 395.52$ 

 Orthorhombic,  $P2_12_12_1$ 
 $a = 8.4952$  (13) Å

 $b = 13.1852$  (19) Å

 $c = 18.771$  (3) Å

 $V = 2102.6$  (6) Å<sup>3</sup>
 $Z = 4$ 

 Cu  $K\alpha$  radiation  
 $\mu = 0.64$  mm<sup>-1</sup>
 $T = 90.0$  (2) K  
 $0.30 \times 0.28 \times 0.18$  mm

### Data collection

 Bruker X8 Proteum diffractometer  
 Absorption correction: multi-scan  
 (SADABS in APEX2; Bruker  
 Nonius, 2004)  
 $T_{\min} = 0.782$ ,  $T_{\max} = 0.894$ 

 26461 measured reflections  
 3849 independent reflections  
 3813 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.110$   
 $S = 1.06$   
 3849 reflections  
 269 parameters  
 H atoms treated by a mixture of  
 independent and constrained  
 refinement

 $\Delta\rho_{\text{max}} = 0.34$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.22$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 1623 Friedel pairs  
 Flack parameter: 0.06 (5)

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                         | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1N}\cdots\text{O3}$ | 1.01 (3) | 2.32 (3)    | 2.992 (2)   | 123.5 (18)    |

Data collection: APEX2 (Bruker Nonius, 2004); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP in SHELXTL (Sheldrick, 2008); 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: HG2376).

## References

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**supplementary materials**

*Acta Cryst.* (2008). E64, o639 [ doi:10.1107/S1600536808003322 ]

## (11*R*,13*R*)-13-(Tetralin-1-ylamino)-4,5-epoxy-11,13-dihydrocostunolide

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

### Comment

Due to the interesting biological activity of parthenolide, we have synthesized a series of amino analogs of parthenolide (Crooks *et al.*, 2005). In order to confirm the configuration of the newly formed methine carbon at C-11 in these molecules, and to obtain more detailed information on the structural conformation of the molecule that may be of value in structure activity relationship studies, the X-ray structure determination of the title compound has been carried out and the results are presented below. The absolute stereochemistry of the newly formed methine at C-11 was found to be *R*, which is typical in structurally related C-11 aminoparthenolide analogs that result from the reaction of an secondary amino compound with parthenolide (Nasim *et al.*, 2007*a*, 2007*b*). Bond distances and angles within the molecule were quite regular with normal bond lengths (Allen *et al.*, 1987). A hydrogen bond is observed between N-1H and O3 of the carbonyl oxygen of the 5-membered lactone ring (Desiraju *et al.*, 1999) (2.32 (3) Å, 2.99 (2) Å, 123.5 (18)°) (Table 1).

### Experimental

The title compound was prepared utilizing the general procedure reported earlier (Nasim *et al.*, 2007*a*, 2007*b*).

### Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.95 Å (C<sub>sp2</sub>—H), 1.00 Å (*R*<sub>3</sub>CH), 0.99 Å (*R*<sub>2</sub>CH<sub>2</sub>), 0.98 Å (RCH<sub>3</sub>) except for the NH hydrogen coordinates, which were refined. *U*<sub>iso</sub>(H) values were set to 1.2*U*<sub>eq</sub> or 1.5*U*<sub>eq</sub> (RCH<sub>3</sub> only) of the attached atom.

Figures

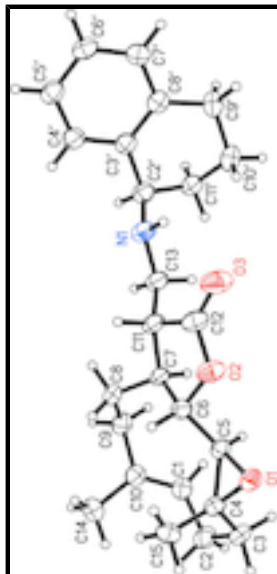


Fig. 1. A view of the asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level; H atoms are shown as small spheres of arbitrary radii.

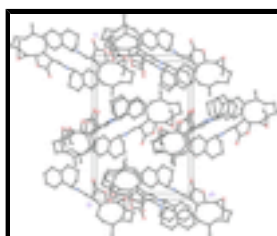


Fig. 2. A packing diagram, viewed down the a axis, hydrogen atoms have been omitted for clarity.

**(12*R*)-4,8-dimethyl-12-[(1'*R*)-1',2',3',4'-tetrahydro-1'-naphthyl]aminomethyl]-3,14-dioxatricyclo[9.3.0.0<sup>2,4</sup>]tetradec-7-en-13-one**

*Crystal data*

$C_{25}H_{33}NO_3$

$M_r = 395.52$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 8.4952$  (13) Å

$b = 13.1852$  (19) Å

$c = 18.771$  (3) Å

$V = 2102.6$  (6) Å<sup>3</sup>

$Z = 4$

$F(000) = 856$

$D_x = 1.249$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 9023 reflections

$\theta = 4.1$ – $68.6^\circ$

$\mu = 0.64$  mm<sup>-1</sup>

$T = 90$  K

Cut block, colourless

$0.30 \times 0.28 \times 0.18$  mm

*Data collection*

Bruker X8 Proteum  
diffractometer

Radiation source: fine-focus rotating anode

Helios multilayer optics

3849 independent reflections

3813 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.033$

Detector resolution: 18 pixels mm<sup>-1</sup>       $\theta_{\max} = 68.6^\circ$ ,  $\theta_{\min} = 4.1^\circ$   
 $\omega$  and  $\varphi$  scans       $h = -10 \rightarrow 10$   
Absorption correction: multi-scan       $k = -15 \rightarrow 15$   
(SADABS in APEX2; Bruker Nonius, 2004)       $l = -22 \rightarrow 22$   
 $T_{\min} = 0.782$ ,  $T_{\max} = 0.894$   
26461 measured 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  
 $R[F^2 > 2\sigma(F^2)] = 0.043$        $w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 0.7419P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.110$        $(\Delta/\sigma)_{\max} < 0.001$   
 $S = 1.06$        $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$   
3849 reflections       $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$   
269 parameters      Extinction correction: SHELXL97 (Sheldrick, 2008),  
 $F_c^* = kFc[1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
0 restraints      Extinction coefficient: 0.0147 (7)  
Primary atom site location: structure-invariant direct methods      Absolute structure: Flack (1983)  
Secondary atom site location: difference Fourier map      Flack parameter: 0.06 (5)

### 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| N1  | 0.5361 (2)   | 0.68044 (12) | 0.44880 (10) | 0.0365 (4)                       |
| H1N | 0.597 (3)    | 0.6648 (19)  | 0.4042 (13)  | 0.044*                           |
| O1  | 0.79418 (15) | 0.18237 (9)  | 0.50676 (7)  | 0.0315 (3)                       |
| O2  | 0.76245 (16) | 0.38600 (10) | 0.45744 (7)  | 0.0363 (3)                       |
| O3  | 0.7062 (2)   | 0.50746 (12) | 0.38018 (8)  | 0.0508 (4)                       |
| C1  | 0.5083 (2)   | 0.23023 (14) | 0.67140 (10) | 0.0317 (4)                       |
| H1  | 0.4217       | 0.2389       | 0.6400       | 0.038*                           |
| C2  | 0.5720 (3)   | 0.12589 (15) | 0.67700 (11) | 0.0389 (5)                       |

## supplementary materials

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|      |            |              |              |            |
|------|------------|--------------|--------------|------------|
| H2A  | 0.4851     | 0.0776       | 0.6862       | 0.047*     |
| H2B  | 0.6468     | 0.1220       | 0.7173       | 0.047*     |
| C3   | 0.6562 (3) | 0.09719 (14) | 0.60747 (11) | 0.0355 (4) |
| H3A  | 0.7194     | 0.0351       | 0.6152       | 0.043*     |
| H3B  | 0.5770     | 0.0824       | 0.5702       | 0.043*     |
| C4   | 0.7615 (2) | 0.18070 (13) | 0.58256 (9)  | 0.0286 (4) |
| C5   | 0.6906 (2) | 0.25699 (13) | 0.53651 (9)  | 0.0259 (4) |
| H5   | 0.5768     | 0.2449       | 0.5260       | 0.031*     |
| C6   | 0.7381 (2) | 0.36493 (13) | 0.53249 (10) | 0.0275 (4) |
| H6   | 0.8375     | 0.3764       | 0.5598       | 0.033*     |
| C7   | 0.6115 (2) | 0.43955 (12) | 0.55609 (10) | 0.0264 (4) |
| H7   | 0.5066     | 0.4092       | 0.5449       | 0.032*     |
| C8   | 0.6106 (2) | 0.47212 (14) | 0.63395 (10) | 0.0339 (4) |
| H8A  | 0.5834     | 0.5450       | 0.6361       | 0.041*     |
| H8B  | 0.7186     | 0.4646       | 0.6531       | 0.041*     |
| C9   | 0.4981 (3) | 0.41421 (15) | 0.68229 (10) | 0.0358 (4) |
| H9A  | 0.4775     | 0.4555       | 0.7254       | 0.043*     |
| H9B  | 0.3967     | 0.4052       | 0.6571       | 0.043*     |
| C10  | 0.5572 (2) | 0.31281 (15) | 0.70481 (10) | 0.0319 (4) |
| C11  | 0.6388 (2) | 0.52750 (13) | 0.50509 (10) | 0.0316 (4) |
| H11  | 0.7222     | 0.5723       | 0.5257       | 0.038*     |
| C12  | 0.7038 (3) | 0.47736 (15) | 0.44038 (11) | 0.0368 (4) |
| C13  | 0.4960 (2) | 0.59112 (14) | 0.48985 (11) | 0.0341 (4) |
| H13A | 0.4181     | 0.5502       | 0.4631       | 0.041*     |
| H13B | 0.4470     | 0.6121       | 0.5354       | 0.041*     |
| C14  | 0.6724 (3) | 0.31561 (18) | 0.76475 (11) | 0.0470 (5) |
| H14A | 0.7063     | 0.2464       | 0.7760       | 0.070*     |
| H14B | 0.6226     | 0.3460       | 0.8067       | 0.070*     |
| H14C | 0.7639     | 0.3563       | 0.7509       | 0.070*     |
| C15  | 0.9019 (2) | 0.20263 (16) | 0.62710 (11) | 0.0378 (5) |
| H15A | 0.9661     | 0.1412       | 0.6315       | 0.057*     |
| H15B | 0.8679     | 0.2247       | 0.6745       | 0.057*     |
| H15C | 0.9642     | 0.2564       | 0.6047       | 0.057*     |
| C2'  | 0.4037 (2) | 0.74860 (14) | 0.43624 (11) | 0.0334 (4) |
| H2'  | 0.3648     | 0.7720       | 0.4838       | 0.040*     |
| C3'  | 0.4645 (2) | 0.84025 (14) | 0.39714 (10) | 0.0299 (4) |
| C4'  | 0.5552 (2) | 0.90996 (15) | 0.43477 (11) | 0.0352 (4) |
| H4'  | 0.5774     | 0.8975       | 0.4836       | 0.042*     |
| C5'  | 0.6133 (2) | 0.99567 (15) | 0.40354 (11) | 0.0359 (4) |
| H5'  | 0.6759     | 1.0419       | 0.4301       | 0.043*     |
| C6'  | 0.5793 (2) | 1.01419 (15) | 0.33243 (11) | 0.0336 (4) |
| H6'  | 0.6179     | 1.0738       | 0.3100       | 0.040*     |
| C7'  | 0.4899 (2) | 0.94620 (15) | 0.29450 (10) | 0.0329 (4) |
| H7'  | 0.4676     | 0.9593       | 0.2458       | 0.040*     |
| C8'  | 0.4317 (2) | 0.85882 (14) | 0.32608 (10) | 0.0306 (4) |
| C9'  | 0.3252 (2) | 0.79086 (16) | 0.28324 (10) | 0.0365 (4) |
| H9'1 | 0.3710     | 0.7807       | 0.2353       | 0.044*     |
| H9'2 | 0.2216     | 0.8242       | 0.2773       | 0.044*     |
| C10' | 0.3021 (3) | 0.68777 (16) | 0.31904 (11) | 0.0398 (5) |

|      |            |              |              |            |
|------|------------|--------------|--------------|------------|
| H10A | 0.2149     | 0.6508       | 0.2956       | 0.048*     |
| H10B | 0.3991     | 0.6468       | 0.3142       | 0.048*     |
| C11' | 0.2645 (2) | 0.70309 (14) | 0.39664 (10) | 0.0346 (4) |
| H11A | 0.2364     | 0.6371       | 0.4184       | 0.042*     |
| H11B | 0.1725     | 0.7488       | 0.4012       | 0.042*     |

Atomic displacement parameters ( $\text{\AA}^2$ )

|      | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$    |
|------|-------------|-------------|-------------|--------------|--------------|-------------|
| N1   | 0.0365 (9)  | 0.0270 (8)  | 0.0461 (9)  | 0.0004 (7)   | -0.0010 (7)  | 0.0058 (7)  |
| O1   | 0.0324 (6)  | 0.0278 (6)  | 0.0343 (6)  | 0.0068 (6)   | 0.0029 (5)   | -0.0040 (5) |
| O2   | 0.0362 (7)  | 0.0304 (7)  | 0.0421 (7)  | 0.0045 (6)   | 0.0123 (6)   | 0.0074 (6)  |
| O3   | 0.0659 (11) | 0.0398 (8)  | 0.0465 (9)  | 0.0060 (8)   | 0.0143 (8)   | 0.0147 (7)  |
| C1   | 0.0326 (9)  | 0.0326 (9)  | 0.0299 (8)  | -0.0051 (8)  | 0.0050 (7)   | 0.0005 (7)  |
| C2   | 0.0499 (12) | 0.0284 (9)  | 0.0383 (10) | -0.0087 (9)  | 0.0034 (9)   | 0.0038 (8)  |
| C3   | 0.0433 (11) | 0.0207 (8)  | 0.0424 (11) | 0.0012 (8)   | -0.0013 (9)  | 0.0006 (8)  |
| C4   | 0.0293 (9)  | 0.0231 (8)  | 0.0333 (9)  | 0.0058 (7)   | 0.0009 (7)   | -0.0009 (7) |
| C5   | 0.0229 (8)  | 0.0237 (8)  | 0.0311 (8)  | 0.0018 (7)   | 0.0010 (7)   | -0.0026 (7) |
| C6   | 0.0226 (8)  | 0.0251 (8)  | 0.0347 (9)  | -0.0021 (7)  | -0.0016 (7)  | 0.0040 (7)  |
| C7   | 0.0246 (8)  | 0.0195 (8)  | 0.0350 (9)  | -0.0003 (7)  | -0.0022 (7)  | 0.0006 (7)  |
| C8   | 0.0423 (11) | 0.0220 (8)  | 0.0376 (10) | -0.0012 (8)  | -0.0042 (8)  | -0.0033 (8) |
| C9   | 0.0395 (11) | 0.0324 (10) | 0.0355 (10) | 0.0075 (9)   | 0.0057 (8)   | -0.0070 (8) |
| C10  | 0.0338 (10) | 0.0320 (9)  | 0.0299 (9)  | 0.0001 (8)   | 0.0051 (8)   | -0.0013 (7) |
| C11  | 0.0308 (9)  | 0.0213 (8)  | 0.0427 (10) | -0.0030 (7)  | -0.0009 (8)  | 0.0039 (8)  |
| C12  | 0.0364 (10) | 0.0291 (9)  | 0.0448 (11) | -0.0018 (8)  | 0.0051 (8)   | 0.0089 (8)  |
| C13  | 0.0346 (10) | 0.0239 (8)  | 0.0438 (10) | 0.0001 (8)   | -0.0017 (8)  | 0.0043 (8)  |
| C14  | 0.0593 (14) | 0.0448 (12) | 0.0369 (10) | -0.0012 (11) | -0.0121 (10) | -0.0034 (9) |
| C15  | 0.0315 (10) | 0.0373 (10) | 0.0444 (11) | 0.0078 (8)   | -0.0088 (8)  | 0.0016 (8)  |
| C2'  | 0.0320 (10) | 0.0286 (9)  | 0.0395 (10) | 0.0020 (8)   | -0.0002 (8)  | 0.0009 (8)  |
| C3'  | 0.0257 (8)  | 0.0277 (9)  | 0.0364 (9)  | 0.0022 (7)   | -0.0014 (8)  | 0.0005 (7)  |
| C4'  | 0.0334 (10) | 0.0326 (10) | 0.0395 (10) | -0.0022 (8)  | -0.0060 (8)  | 0.0041 (8)  |
| C5'  | 0.0299 (9)  | 0.0312 (9)  | 0.0466 (11) | -0.0040 (8)  | -0.0008 (8)  | 0.0013 (8)  |
| C6'  | 0.0263 (9)  | 0.0292 (9)  | 0.0454 (10) | 0.0008 (8)   | 0.0033 (8)   | 0.0070 (8)  |
| C7'  | 0.0300 (9)  | 0.0350 (10) | 0.0338 (9)  | 0.0037 (8)   | 0.0022 (8)   | 0.0041 (8)  |
| C8'  | 0.0257 (9)  | 0.0290 (9)  | 0.0372 (10) | 0.0047 (7)   | 0.0033 (8)   | -0.0031 (8) |
| C9'  | 0.0335 (10) | 0.0414 (11) | 0.0346 (9)  | -0.0054 (9)  | 0.0026 (8)   | -0.0099 (8) |
| C10' | 0.0389 (11) | 0.0366 (10) | 0.0439 (11) | -0.0079 (9)  | 0.0013 (9)   | -0.0101 (9) |
| C11' | 0.0334 (9)  | 0.0290 (9)  | 0.0415 (10) | -0.0010 (8)  | 0.0005 (8)   | 0.0021 (8)  |

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

|        |           |          |           |
|--------|-----------|----------|-----------|
| N1—C13 | 1.448 (2) | C11—C12  | 1.489 (3) |
| N1—C2' | 1.459 (2) | C11—C13  | 1.503 (3) |
| N1—H1N | 1.01 (3)  | C11—H11  | 1.0000    |
| O1—C5  | 1.433 (2) | C13—H13A | 0.9900    |
| O1—C4  | 1.450 (2) | C13—H13B | 0.9900    |
| O2—C12 | 1.342 (2) | C14—H14A | 0.9800    |
| O2—C6  | 1.451 (2) | C14—H14B | 0.9800    |
| O3—C12 | 1.198 (2) | C14—H14C | 0.9800    |

## supplementary materials

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|            |             |               |             |
|------------|-------------|---------------|-------------|
| C1—C10     | 1.324 (3)   | C15—H15A      | 0.9800      |
| C1—C2      | 1.482 (3)   | C15—H15B      | 0.9800      |
| C1—H1      | 0.9500      | C15—H15C      | 0.9800      |
| C2—C3      | 1.536 (3)   | C2'—C3'       | 1.505 (3)   |
| C2—H2A     | 0.9900      | C2'—C11'      | 1.521 (3)   |
| C2—H2B     | 0.9900      | C2'—H2'       | 1.0000      |
| C3—C4      | 1.494 (3)   | C3'—C8'       | 1.384 (3)   |
| C3—H3A     | 0.9900      | C3'—C4'       | 1.392 (3)   |
| C3—H3B     | 0.9900      | C4'—C5'       | 1.365 (3)   |
| C4—C5      | 1.457 (2)   | C4'—H4'       | 0.9500      |
| C4—C15     | 1.485 (3)   | C5'—C6'       | 1.387 (3)   |
| C5—C6      | 1.481 (2)   | C5'—H5'       | 0.9500      |
| C5—H5      | 1.0000      | C6'—C7'       | 1.374 (3)   |
| C6—C7      | 1.523 (2)   | C6'—H6'       | 0.9500      |
| C6—H6      | 1.0000      | C7'—C8'       | 1.387 (3)   |
| C7—C11     | 1.522 (2)   | C7'—H7'       | 0.9500      |
| C7—C8      | 1.523 (3)   | C8'—C9'       | 1.506 (3)   |
| C7—H7      | 1.0000      | C9'—C10'      | 1.529 (3)   |
| C8—C9      | 1.523 (3)   | C9'—H9'1      | 0.9900      |
| C8—H8A     | 0.9900      | C9'—H9'2      | 0.9900      |
| C8—H8B     | 0.9900      | C10'—C11'     | 1.505 (3)   |
| C9—C10     | 1.489 (3)   | C10'—H10A     | 0.9900      |
| C9—H9A     | 0.9900      | C10'—H10B     | 0.9900      |
| C9—H9B     | 0.9900      | C11'—H11A     | 0.9900      |
| C10—C14    | 1.492 (3)   | C11'—H11B     | 0.9900      |
| C13—N1—C2' | 113.94 (16) | C7—C11—H11    | 108.4       |
| C13—N1—H1N | 113.4 (14)  | O3—C12—O2     | 121.07 (19) |
| C2'—N1—H1N | 113.0 (14)  | O3—C12—C11    | 128.96 (19) |
| C5—O1—C4   | 60.70 (11)  | O2—C12—C11    | 109.95 (16) |
| C12—O2—C6  | 110.53 (15) | N1—C13—C11    | 111.42 (16) |
| C10—C1—C2  | 127.98 (19) | N1—C13—H13A   | 109.3       |
| C10—C1—H1  | 116.0       | C11—C13—H13A  | 109.3       |
| C2—C1—H1   | 116.0       | N1—C13—H13B   | 109.3       |
| C1—C2—C3   | 109.79 (16) | C11—C13—H13B  | 109.3       |
| C1—C2—H2A  | 109.7       | H13A—C13—H13B | 108.0       |
| C3—C2—H2A  | 109.7       | C10—C14—H14A  | 109.5       |
| C1—C2—H2B  | 109.7       | C10—C14—H14B  | 109.5       |
| C3—C2—H2B  | 109.7       | H14A—C14—H14B | 109.5       |
| H2A—C2—H2B | 108.2       | C10—C14—H14C  | 109.5       |
| C4—C3—C2   | 111.31 (15) | H14A—C14—H14C | 109.5       |
| C4—C3—H3A  | 109.4       | H14B—C14—H14C | 109.5       |
| C2—C3—H3A  | 109.4       | C4—C15—H15A   | 109.5       |
| C4—C3—H3B  | 109.4       | C4—C15—H15B   | 109.5       |
| C2—C3—H3B  | 109.4       | H15A—C15—H15B | 109.5       |
| H3A—C3—H3B | 108.0       | C4—C15—H15C   | 109.5       |
| O1—C4—C5   | 59.08 (11)  | H15A—C15—H15C | 109.5       |
| O1—C4—C15  | 113.32 (15) | H15B—C15—H15C | 109.5       |
| C5—C4—C15  | 122.12 (16) | N1—C2'—C3'    | 108.01 (15) |
| O1—C4—C3   | 115.67 (15) | N1—C2'—C11'   | 115.82 (16) |

|              |              |                |              |
|--------------|--------------|----------------|--------------|
| C5—C4—C3     | 116.57 (16)  | C3'—C2'—C11'   | 110.19 (16)  |
| C15—C4—C3    | 116.63 (17)  | N1—C2'—H2'     | 107.5        |
| O1—C5—C4     | 60.22 (11)   | C3'—C2'—H2'    | 107.5        |
| O1—C5—C6     | 118.22 (15)  | C11'—C2'—H2'   | 107.5        |
| C4—C5—C6     | 125.54 (16)  | C8'—C3'—C4'    | 118.90 (17)  |
| O1—C5—H5     | 114.0        | C8'—C3'—C2'    | 122.89 (17)  |
| C4—C5—H5     | 114.0        | C4'—C3'—C2'    | 118.20 (17)  |
| C6—C5—H5     | 114.0        | C5'—C4'—C3'    | 121.92 (19)  |
| O2—C6—C5     | 105.81 (15)  | C5'—C4'—H4'    | 119.0        |
| O2—C6—C7     | 105.04 (14)  | C3'—C4'—H4'    | 119.0        |
| C5—C6—C7     | 114.46 (14)  | C4'—C5'—C6'    | 118.93 (19)  |
| O2—C6—H6     | 110.4        | C4'—C5'—H5'    | 120.5        |
| C5—C6—H6     | 110.4        | C6'—C5'—H5'    | 120.5        |
| C7—C6—H6     | 110.4        | C7'—C6'—C5'    | 119.91 (18)  |
| C11—C7—C6    | 101.64 (14)  | C7'—C6'—H6'    | 120.0        |
| C11—C7—C8    | 112.93 (15)  | C5'—C6'—H6'    | 120.0        |
| C6—C7—C8     | 117.69 (15)  | C6'—C7'—C8'    | 121.19 (18)  |
| C11—C7—H7    | 108.0        | C6'—C7'—H7'    | 119.4        |
| C6—C7—H7     | 108.0        | C8'—C7'—H7'    | 119.4        |
| C8—C7—H7     | 108.0        | C3'—C8'—C7'    | 119.15 (18)  |
| C7—C8—C9     | 115.68 (16)  | C3'—C8'—C9'    | 122.00 (17)  |
| C7—C8—H8A    | 108.4        | C7'—C8'—C9'    | 118.71 (17)  |
| C9—C8—H8A    | 108.4        | C8'—C9'—C10'   | 111.81 (17)  |
| C7—C8—H8B    | 108.4        | C8'—C9'—H9'1   | 109.3        |
| C9—C8—H8B    | 108.4        | C10'—C9'—H9'1  | 109.3        |
| H8A—C8—H8B   | 107.4        | C8'—C9'—H9'2   | 109.3        |
| C10—C9—C8    | 114.03 (16)  | C10'—C9'—H9'2  | 109.3        |
| C10—C9—H9A   | 108.7        | H9'1—C9'—H9'2  | 107.9        |
| C8—C9—H9A    | 108.7        | C11'—C10'—C9'  | 109.47 (16)  |
| C10—C9—H9B   | 108.7        | C11'—C10'—H10A | 109.8        |
| C8—C9—H9B    | 108.7        | C9'—C10'—H10A  | 109.8        |
| H9A—C9—H9B   | 107.6        | C11'—C10'—H10B | 109.8        |
| C1—C10—C9    | 119.88 (18)  | C9'—C10'—H10B  | 109.8        |
| C1—C10—C14   | 125.72 (19)  | H10A—C10'—H10B | 108.2        |
| C9—C10—C14   | 114.40 (17)  | C10'—C11'—C2'  | 111.16 (17)  |
| C12—C11—C13  | 113.08 (17)  | C10'—C11'—H11A | 109.4        |
| C12—C11—C7   | 103.38 (14)  | C2'—C11'—H11A  | 109.4        |
| C13—C11—C7   | 114.96 (16)  | C10'—C11'—H11B | 109.4        |
| C12—C11—H11  | 108.4        | C2'—C11'—H11B  | 109.4        |
| C13—C11—H11  | 108.4        | H11A—C11'—H11B | 108.0        |
| C10—C1—C2—C3 | -109.7 (2)   | C8—C7—C11—C13  | -80.1 (2)    |
| C1—C2—C3—C4  | 45.8 (2)     | C6—O2—C12—O3   | -176.92 (19) |
| C5—O1—C4—C15 | -114.62 (18) | C6—O2—C12—C11  | 2.1 (2)      |
| C5—O1—C4—C3  | 106.90 (17)  | C13—C11—C12—O3 | 33.4 (3)     |
| C2—C3—C4—O1  | -155.15 (16) | C7—C11—C12—O3  | 158.4 (2)    |
| C2—C3—C4—C5  | -88.5 (2)    | C13—C11—C12—O2 | -145.45 (17) |
| C2—C3—C4—C15 | 67.8 (2)     | C7—C11—C12—O2  | -20.5 (2)    |
| C4—O1—C5—C6  | 116.94 (18)  | C2'—N1—C13—C11 | -176.27 (16) |
| C15—C4—C5—O1 | 99.73 (18)   | C12—C11—C13—N1 | -69.3 (2)    |

## supplementary materials

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|               |              |                   |              |
|---------------|--------------|-------------------|--------------|
| C3—C4—C5—O1   | -105.36 (17) | C7—C11—C13—N1     | 172.34 (15)  |
| O1—C4—C5—C6   | -105.11 (19) | C13—N1—C2'—C3'    | 176.64 (16)  |
| C15—C4—C5—C6  | -5.4 (3)     | C13—N1—C2'—C11'   | -59.3 (2)    |
| C3—C4—C5—C6   | 149.53 (17)  | N1—C2'—C3'—C8'    | 108.4 (2)    |
| C12—O2—C6—C5  | 138.80 (16)  | C11'—C2'—C3'—C8'  | -19.0 (2)    |
| C12—O2—C6—C7  | 17.36 (19)   | N1—C2'—C3'—C4'    | -72.8 (2)    |
| O1—C5—C6—O2   | 56.9 (2)     | C11'—C2'—C3'—C4'  | 159.81 (17)  |
| C4—C5—C6—O2   | 128.86 (18)  | C8'—C3'—C4'—C5'   | -0.3 (3)     |
| O1—C5—C6—C7   | 172.03 (14)  | C2'—C3'—C4'—C5'   | -179.21 (18) |
| C4—C5—C6—C7   | -115.99 (19) | C3'—C4'—C5'—C6'   | 0.7 (3)      |
| O2—C6—C7—C11  | -28.52 (17)  | C4'—C5'—C6'—C7'   | -0.6 (3)     |
| C5—C6—C7—C11  | -144.12 (16) | C5'—C6'—C7'—C8'   | 0.3 (3)      |
| O2—C6—C7—C8   | -152.39 (15) | C4'—C3'—C8'—C7'   | -0.1 (3)     |
| C5—C6—C7—C8   | 92.0 (2)     | C2'—C3'—C8'—C7'   | 178.76 (17)  |
| C11—C7—C8—C9  | 145.45 (17)  | C4'—C3'—C8'—C9'   | -175.66 (18) |
| C6—C7—C8—C9   | -96.5 (2)    | C2'—C3'—C8'—C9'   | 3.2 (3)      |
| C7—C8—C9—C10  | 78.9 (2)     | C6'—C7'—C8'—C3'   | 0.1 (3)      |
| C2—C1—C10—C9  | 168.58 (19)  | C6'—C7'—C8'—C9'   | 175.83 (18)  |
| C2—C1—C10—C14 | -10.4 (3)    | C3'—C8'—C9'—C10'  | -17.3 (3)    |
| C8—C9—C10—C1  | -99.0 (2)    | C7'—C8'—C9'—C10'  | 167.10 (17)  |
| C8—C9—C10—C14 | 80.1 (2)     | C8'—C9'—C10'—C11' | 47.4 (2)     |
| C6—C7—C11—C12 | 29.12 (18)   | C9'—C10'—C11'—C2' | -66.1 (2)    |
| C8—C7—C11—C12 | 156.16 (16)  | N1—C2'—C11'—C10'  | -72.9 (2)    |
| C6—C7—C11—C13 | 152.84 (16)  | C3'—C2'—C11'—C10' | 50.1 (2)     |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$      | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|----------|-------------|-------------|---------------|
| N1—H1N $\cdots$ O3 | 1.01 (3) | 2.32 (3)    | 2.992 (2)   | 123.5 (18)    |

Fig. 1

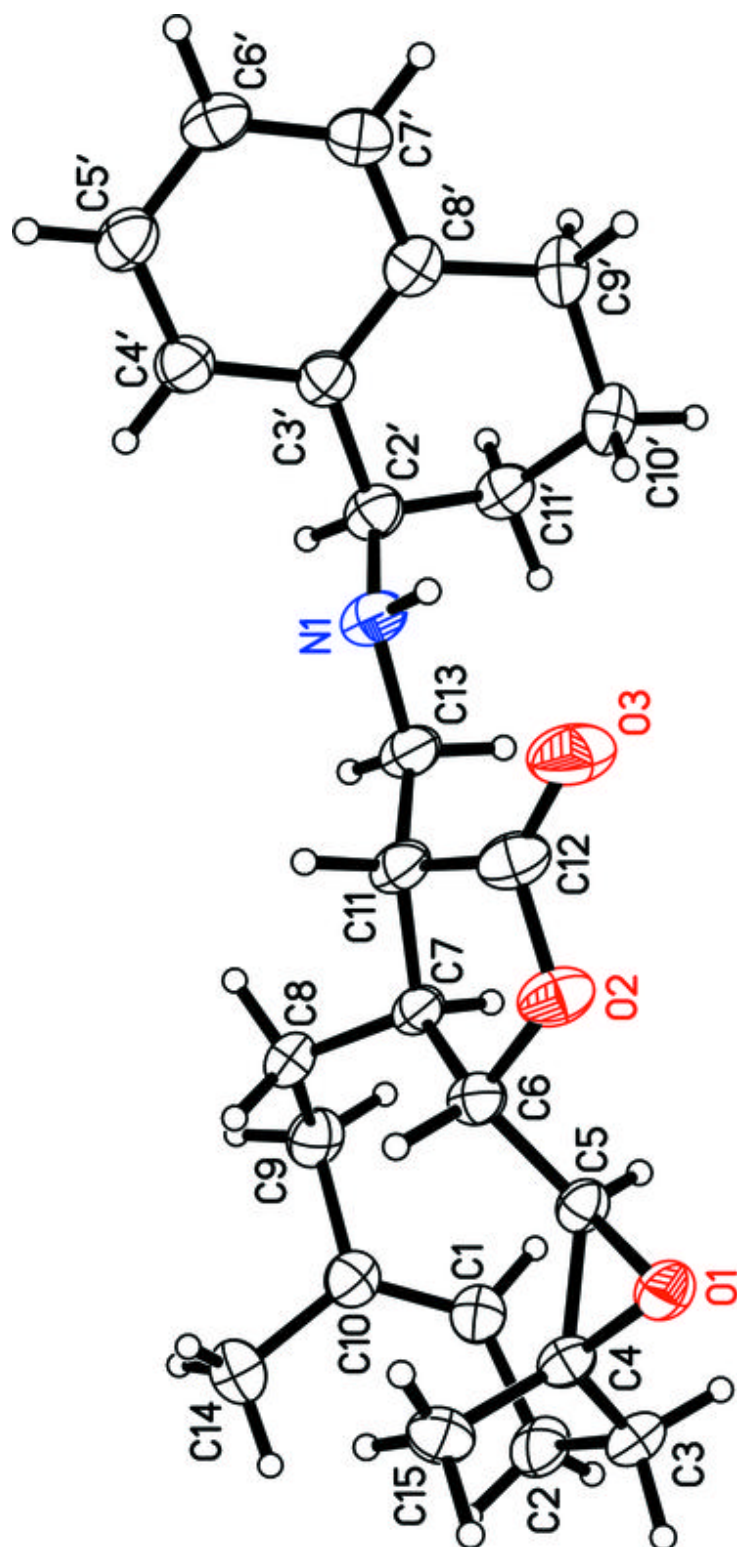


Fig. 2

