

## (1*S*,3*S*,8*R*,9*S*,10*R*)-9,10-Epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodecane

**Abdoullah Bimoussa,<sup>a</sup> Aziz Auhmani,<sup>a</sup> My Youssef Ait Itto,<sup>\*,a</sup> Jean-Claude Daran<sup>b</sup> and Abdelwahed Auhmani<sup>a</sup>**

<sup>a</sup>Laboratoire de Synthèse Organique et Physico-Chimie Moléculaire, Département de Chimie, Faculté des Sciences Semlalia, BP 2390 Marrakech 40000, Morocco, and

<sup>b</sup>Laboratoire de Chimie de Coordination, 205 route de Narbonne, 31077 Toulouse Cedex 04, France

Correspondence e-mail: aititto@uca.ma

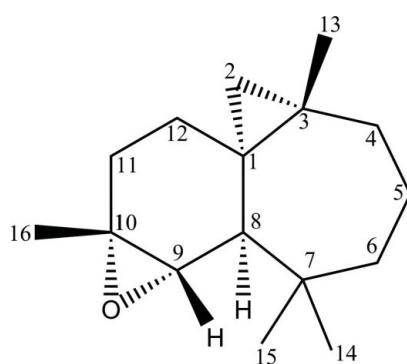
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Key indicators: single-crystal X-ray study;  $T = 180\text{ K}$ ; mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$ ;  $R$  factor = 0.054;  $wR$  factor = 0.105; data-to-parameter ratio = 18.3.

The title compound,  $C_{16}H_{26}O$ , was synthesized by treating (1*S*,3*S*,8*R*)-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodec-9-ene with metachloroperbenzoic acid. The molecule is built up from two fused six- and seven-membered rings. The six-membered ring has a half-chair conformation, whereas the seven-membered ring displays a boat conformation. In the crystal, there are no significant intermolecular interactions present.

## Related literature

For the use of epoxydes in organic synthesis, see: Mori (1989); Paddon-Jones *et al.* (1997); Taylor *et al.* (1991). For their biological activity, see: Kupchan *et al.* (1989); Trost *et al.* (1983); Vollhardt & Schore (1996); Yang (2004). For structural discussion, see: Cremer & Pople (1975); Flack (1983); Flack & Bernardinelli (2000); Spek (2009); Boessenkool & Boyens (1980); Benharref *et al.* (2010). For the synthesis, see: Auhmani *et al.* (2001).



## Experimental

### Crystal data

$C_{16}H_{26}O$	$V = 710.52 (12)\text{ \AA}^3$
$M_r = 234.37$	$Z = 2$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 10.5563 (10)\text{ \AA}$	$\mu = 0.07\text{ mm}^{-1}$
$b = 5.7548 (5)\text{ \AA}$	$T = 180\text{ K}$
$c = 11.7096 (13)\text{ \AA}$	$0.31 \times 0.31 \times 0.25\text{ mm}$
$\beta = 92.777 (8)^\circ$	

### Data collection

Agilent Xcalibur Eos Gemini ultra diffractometer	8241 measured reflections
Absorption correction: multi-scan ( <i>CrysAlis PRO</i> ; Agilent, 2012)	2899 independent reflections
$T_{\min} = 0.767$ , $T_{\max} = 1.0$	2150 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.055$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$	1 restraint
$wR(F^2) = 0.105$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.14\text{ e \AA}^{-3}$
2899 reflections	$\Delta\rho_{\text{min}} = -0.21\text{ e \AA}^{-3}$
158 parameters	

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL2013*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5778).

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# supporting information

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## (1S,3S,8R,9S,10R)-9,10-Epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodecane

**Abdoullah Bimoussa, Aziz Auhmani, My Youssef Ait Itto, Jean-Claude Daran and Abdelwahed Auhmani**

### S1. Comment

Epoxides are important synthetic intermediates that are widely used in organic synthesis. They are described in the synthesis of Antifungal products (Taylor *et al.*, 1991) and different pheromones (Mori, 1989, Paddon-Jones *et al.*, 1997). Besides, many natural products possess this functional group as an essential structural moiety for their biological activities (Yang, 2004; Vollhardt *et al.*, 1996; Trost *et al.*, 1983; Kupchan *et al.*, 1974). Because of their widespread occurrence and synthetic utility, the development of methods for the direct asymmetric synthesis of epoxides has grown significantly. In order to prepare new epoxides with natural products, we synthesized (1S,3S,8R,9S,10R)-9,10-epoxy-3,7,7,10-tetramethyltricyclo [6.4.0.0<sup>1,3</sup>]dodecane in three stages from  $\beta$ -himachalene. The title compound was prepared by treating (1S,3S,8R)-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodec-9-ene (Auhmani *et al.*, 2001) by meta-chloroperbenzoic acid.

The title compound is built up from two fused six and seven-membered rings (Fig. 1). The six-membered-ring has an half chair conformation with puckering parameters:  $Q = 0.447$  (3) Å,  $\theta = 128.5$  (4) $^\circ$  and  $\varphi = 171.6$  (6) $^\circ$  (Cremer & Pople, 1975), whereas the seven-membered ring displays a boat conformation with puckering amplitudes:  $Q_2 = 1.142$  (4) and  $Q_3 = 0.036$  (4) (Boessenkool & Boyens, 1980). Although the absolute configuration is different, this structure is closely related to the (1S,3S,8R,9S,10R)-2,2-Dichloro-3,7,7,10-tetramethyl-9,10-epoxytricyclo [6.4.0.0<sup>1,3</sup>]dodecane (Benharref *et al.*, 2010) however the seven-membered ring displays a chair conformation.

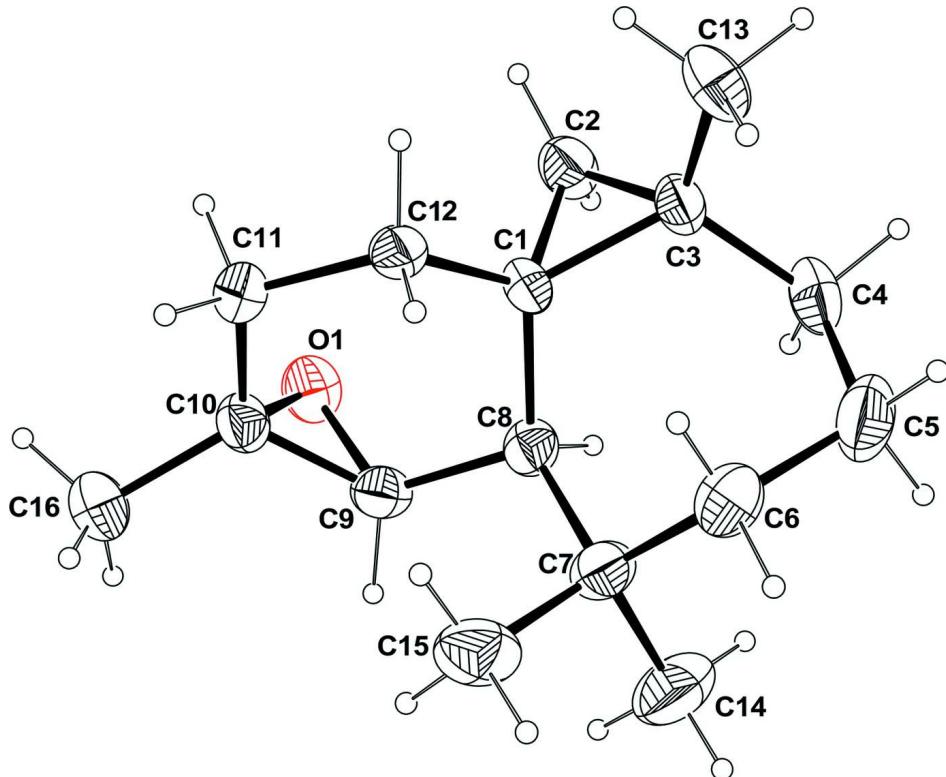
The absolute configuration (1S,3S,8R,9S,10R) is deduced from the chemical pathway. The refinement of the Flack's parameter (-0.2 (10)) (Flack, 1983; Flack & Bernardinelli, 2000) as well as the Hooft's parameter ((Spek, 2009) do not allow to define reliably the absolute configuration.

### S2. Experimental

In 100 mL flask containing (0.220 g, 1.009 mmol) of (1S,3S,8R)-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodec-9-ene in 20 ml of dichloromethane was added a stoichiometric quantity of *m*-chloroperbenzoic acid (*m*-CPBA). The reaction mixture was stirred at room temperature for 2 h and then treated with 10% solution of sodium hydrogencarbonate. The reaction mixture was extracted with dichloromethane (3x 20 mL) and the organic layer were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude product was purified by chromatography on silica gel (230–400 mesh) with Hexane/Ethyl acetate (97:3) as eluent to give the title compound (1S,3S,8R,9S,10R)-9,10-epoxy-3,7,7,10-tetramethyltricyclo [6.4.0.0<sup>1,3</sup>]dodecane in 72% yield. X-ray quality crystals were obtained by slow evaporation from a petroleum ether solution of the title compound.

**S3. Refinement**

All H atoms were fixed geometrically and treated as riding with C—H = 0.98 Å (methyl), 0.99 Å (methylene) and 1.00 Å (methine). In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined and any references to the Flack parameter were removed.

**Figure 1**

Molecular view of the title compound with the atom labeling scheme. Ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

**(1*S*,3*S*,8*R*,9*S*,10*R*)-9,10-Epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0<sup>1,3</sup>]dodecane***Crystal data*

$C_{16}H_{26}O$   
 $M_r = 234.37$   
Monoclinic,  $P2_1$   
Hall symbol: P 2yb  
 $a = 10.5563 (10)$  Å  
 $b = 5.7548 (5)$  Å  
 $c = 11.7096 (13)$  Å  
 $\beta = 92.777 (8)^\circ$   
 $V = 710.52 (12)$  Å<sup>3</sup>  
 $Z = 2$

$F(000) = 260$   
 $D_x = 1.095 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
Cell parameters from 1871 reflections  
 $\theta = 3.9\text{--}26.5^\circ$   
 $\mu = 0.07 \text{ mm}^{-1}$   
 $T = 180 \text{ K}$   
Box, colourless  
 $0.31 \times 0.31 \times 0.25 \text{ mm}$

*Data collection*

Agilent Xcalibur Eos Gemini ultra  
diffractometer

Detector resolution: 16.1978 pixels mm<sup>-1</sup>

Radiation source: Enhance (Mo) X-ray Source  
Graphite monochromator

$\omega$  scans  
Absorption correction: multi-scan  
(CrysAlis PRO; Agilent, 2012)

$T_{\min} = 0.767$ ,  $T_{\max} = 1.0$   
 8241 measured reflections  
 2899 independent reflections  
 2150 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.055$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 3.5^\circ$   
 $h = -13 \rightarrow 13$   
 $k = -7 \rightarrow 7$   
 $l = -14 \rightarrow 14$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.105$   
 $S = 1.05$   
 2899 reflections  
 158 parameters  
 1 restraint

Hydrogen site location: inferred from neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0267P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

#### 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.

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2747 (3)	0.7760 (5)	0.3324 (3)	0.0241 (7)
C2	0.2251 (3)	0.6047 (6)	0.4168 (3)	0.0334 (8)
H2A	0.2213	0.4390	0.3941	0.040*
H2B	0.2461	0.6322	0.4990	0.040*
C3	0.1336 (3)	0.7699 (5)	0.3563 (3)	0.0276 (7)
C4	0.0457 (3)	0.6751 (6)	0.2618 (3)	0.0392 (9)
H4A	-0.0380	0.6418	0.2929	0.047*
H4B	0.0805	0.5271	0.2338	0.047*
C5	0.0281 (3)	0.8450 (7)	0.1615 (3)	0.0493 (11)
H5A	-0.0017	0.7571	0.0927	0.059*
H5B	-0.0391	0.9578	0.1791	0.059*
C6	0.1479 (3)	0.9792 (7)	0.1341 (3)	0.0429 (9)
H6A	0.1271	1.0768	0.0661	0.051*
H6B	0.1680	1.0860	0.1987	0.051*
C7	0.2684 (3)	0.8433 (6)	0.1112 (3)	0.0335 (8)
C8	0.3116 (3)	0.6867 (5)	0.2160 (3)	0.0265 (8)
H8	0.2675	0.5342	0.2043	0.032*
C9	0.4518 (3)	0.6354 (5)	0.2177 (3)	0.0295 (8)
H9	0.4857	0.5925	0.1423	0.035*
C10	0.5427 (3)	0.7440 (6)	0.2993 (3)	0.0289 (8)
C11	0.4987 (3)	0.9110 (5)	0.3879 (3)	0.0296 (8)
H11A	0.5481	1.0566	0.3826	0.036*
H11B	0.5182	0.8436	0.4645	0.036*
C12	0.3575 (3)	0.9715 (5)	0.3781 (3)	0.0274 (7)
H12A	0.3296	1.0162	0.4546	0.033*

H12B	0.3454	1.1078	0.3272	0.033*
C13	0.0746 (3)	0.9619 (6)	0.4244 (3)	0.0381 (9)
H13A	0.0582	1.0970	0.3750	0.057*
H13B	0.1328	1.0060	0.4885	0.057*
H13C	-0.0054	0.9069	0.4539	0.057*
C14	0.2449 (4)	0.6836 (8)	0.0066 (3)	0.0551 (11)
H14A	0.1789	0.5702	0.0227	0.083*
H14B	0.3235	0.6018	-0.0094	0.083*
H14C	0.2174	0.7774	-0.0599	0.083*
C15	0.3718 (3)	1.0198 (6)	0.0830 (3)	0.0460 (10)
H15A	0.3407	1.1195	0.0198	0.069*
H15B	0.4478	0.9367	0.0608	0.069*
H15C	0.3928	1.1157	0.1505	0.069*
C16	0.6798 (3)	0.7660 (7)	0.2706 (3)	0.0460 (10)
H16A	0.7340	0.7499	0.3406	0.069*
H16B	0.6939	0.9186	0.2363	0.069*
H16C	0.7007	0.6438	0.2164	0.069*
O1	0.50267 (19)	0.5041 (4)	0.31494 (19)	0.0350 (6)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0183 (15)	0.0264 (17)	0.0278 (18)	-0.0012 (14)	0.0043 (13)	-0.0014 (15)
C2	0.0307 (19)	0.0345 (19)	0.035 (2)	0.0008 (16)	0.0051 (15)	0.0065 (16)
C3	0.0200 (16)	0.0298 (18)	0.0331 (19)	-0.0018 (15)	0.0035 (14)	0.0032 (16)
C4	0.0207 (18)	0.044 (2)	0.053 (3)	-0.0058 (16)	0.0012 (16)	-0.0024 (19)
C5	0.032 (2)	0.063 (3)	0.051 (3)	0.006 (2)	-0.0125 (18)	-0.001 (2)
C6	0.039 (2)	0.052 (2)	0.036 (2)	0.008 (2)	-0.0060 (16)	0.012 (2)
C7	0.036 (2)	0.040 (2)	0.0245 (19)	0.0031 (17)	-0.0014 (15)	0.0003 (16)
C8	0.0259 (18)	0.0267 (18)	0.0267 (19)	-0.0016 (14)	0.0000 (14)	-0.0050 (14)
C9	0.0309 (19)	0.0325 (19)	0.0254 (19)	0.0047 (16)	0.0049 (14)	0.0018 (16)
C10	0.0229 (17)	0.0339 (19)	0.0299 (19)	0.0008 (16)	0.0008 (14)	0.0046 (16)
C11	0.0251 (17)	0.034 (2)	0.0295 (19)	-0.0038 (14)	-0.0014 (14)	-0.0024 (15)
C12	0.0281 (18)	0.0302 (17)	0.0242 (18)	-0.0042 (16)	0.0028 (13)	-0.0060 (15)
C13	0.0273 (19)	0.036 (2)	0.052 (2)	0.0061 (16)	0.0123 (16)	-0.0024 (18)
C14	0.065 (3)	0.069 (3)	0.030 (2)	0.006 (2)	-0.0101 (19)	-0.010 (2)
C15	0.055 (2)	0.046 (2)	0.037 (2)	0.002 (2)	0.0087 (17)	0.0144 (18)
C16	0.0251 (19)	0.071 (3)	0.043 (2)	0.000 (2)	0.0038 (16)	-0.001 (2)
O1	0.0302 (13)	0.0314 (12)	0.0433 (15)	0.0050 (12)	0.0011 (10)	0.0018 (12)

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

C1—C12	1.507 (4)	C9—O1	1.448 (4)
C1—C2	1.508 (4)	C9—C10	1.462 (4)
C1—C8	1.525 (4)	C9—H9	1.0000
C1—C3	1.529 (4)	C10—O1	1.458 (4)
C2—C3	1.507 (4)	C10—C11	1.504 (4)
C2—H2A	0.9900	C10—C16	1.507 (4)

C2—H2B	0.9900	C11—C12	1.530 (4)
C3—C4	1.511 (5)	C11—H11A	0.9900
C3—C13	1.514 (4)	C11—H11B	0.9900
C4—C5	1.533 (5)	C12—H12A	0.9900
C4—H4A	0.9900	C12—H12B	0.9900
C4—H4B	0.9900	C13—H13A	0.9800
C5—C6	1.529 (5)	C13—H13B	0.9800
C5—H5A	0.9900	C13—H13C	0.9800
C5—H5B	0.9900	C14—H14A	0.9800
C6—C7	1.528 (4)	C14—H14B	0.9800
C6—H6A	0.9900	C14—H14C	0.9800
C6—H6B	0.9900	C15—H15A	0.9800
C7—C15	1.538 (5)	C15—H15B	0.9800
C7—C14	1.541 (5)	C15—H15C	0.9800
C7—C8	1.573 (4)	C16—H16A	0.9800
C8—C9	1.508 (4)	C16—H16B	0.9800
C8—H8	1.0000	C16—H16C	0.9800
C12—C1—C2	118.0 (3)	O1—C9—C8	116.1 (2)
C12—C1—C8	113.6 (2)	C10—C9—C8	122.5 (3)
C2—C1—C8	118.5 (3)	O1—C9—H9	115.5
C12—C1—C3	120.4 (2)	C10—C9—H9	115.5
C2—C1—C3	59.52 (19)	C8—C9—H9	115.5
C8—C1—C3	116.7 (3)	O1—C10—C9	59.45 (19)
C3—C2—C1	60.9 (2)	O1—C10—C11	114.7 (2)
C3—C2—H2A	117.7	C9—C10—C11	120.6 (3)
C1—C2—H2A	117.7	O1—C10—C16	113.3 (3)
C3—C2—H2B	117.7	C9—C10—C16	119.9 (3)
C1—C2—H2B	117.7	C11—C10—C16	115.6 (3)
H2A—C2—H2B	114.8	C10—C11—C12	115.2 (3)
C2—C3—C4	118.3 (3)	C10—C11—H11A	108.5
C2—C3—C13	118.9 (3)	C12—C11—H11A	108.5
C4—C3—C13	113.3 (3)	C10—C11—H11B	108.5
C2—C3—C1	59.5 (2)	C12—C11—H11B	108.5
C4—C3—C1	116.3 (3)	H11A—C11—H11B	107.5
C13—C3—C1	120.6 (3)	C1—C12—C11	113.8 (2)
C3—C4—C5	112.2 (3)	C1—C12—H12A	108.8
C3—C4—H4A	109.2	C11—C12—H12A	108.8
C5—C4—H4A	109.2	C1—C12—H12B	108.8
C3—C4—H4B	109.2	C11—C12—H12B	108.8
C5—C4—H4B	109.2	H12A—C12—H12B	107.7
H4A—C4—H4B	107.9	C3—C13—H13A	109.5
C6—C5—C4	114.3 (3)	C3—C13—H13B	109.5
C6—C5—H5A	108.7	H13A—C13—H13B	109.5
C4—C5—H5A	108.7	C3—C13—H13C	109.5
C6—C5—H5B	108.7	H13A—C13—H13C	109.5
C4—C5—H5B	108.7	H13B—C13—H13C	109.5
H5A—C5—H5B	107.6	C7—C14—H14A	109.5

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C7—C6—C5	118.9 (3)	C7—C14—H14B	109.5
C7—C6—H6A	107.6	H14A—C14—H14B	109.5
C5—C6—H6A	107.6	C7—C14—H14C	109.5
C7—C6—H6B	107.6	H14A—C14—H14C	109.5
C5—C6—H6B	107.6	H14B—C14—H14C	109.5
H6A—C6—H6B	107.0	C7—C15—H15A	109.5
C6—C7—C15	107.8 (3)	C7—C15—H15B	109.5
C6—C7—C14	110.0 (3)	H15A—C15—H15B	109.5
C15—C7—C14	108.2 (3)	C7—C15—H15C	109.5
C6—C7—C8	111.6 (3)	H15A—C15—H15C	109.5
C15—C7—C8	111.3 (3)	H15B—C15—H15C	109.5
C14—C7—C8	107.9 (3)	C10—C16—H16A	109.5
C9—C8—C1	110.3 (3)	C10—C16—H16B	109.5
C9—C8—C7	111.7 (2)	H16A—C16—H16B	109.5
C1—C8—C7	115.3 (2)	C10—C16—H16C	109.5
C9—C8—H8	106.3	H16A—C16—H16C	109.5
C1—C8—H8	106.3	H16B—C16—H16C	109.5
C7—C8—H8	106.3	C9—O1—C10	60.41 (19)
O1—C9—C10	60.14 (19)		

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