

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(4a*S*^{*},10a*R*^{*})-7-Hydroxy-8-isopropyl-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-2-oneAbdellah Zeroual,^a Nouredine Mazoir,^a Moha Berraho,^{b*} Aziz Auhmani^a and Ahmed Benharref^a^aLaboratoire de Chimie des Substances Naturelles, Faculté des Sciences Semlalia, BP 2390, Boulevard My Abdellah, 40000 Marrakech, Morocco, and ^bLaboratoire de Chimie de Coordination, Unité Matériaux, Faculté des Sciences Semlalia, BP 2390, Boulevard My Abdellah, 40000 Marrakech, Morocco
Correspondence e-mail: mberraho@yahoo.fr

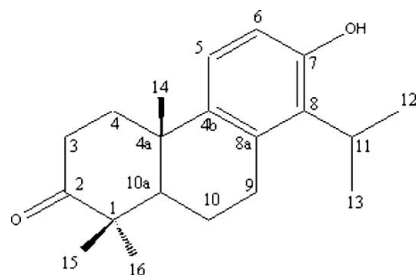
Received 1 June 2007; accepted 10 July 2007

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.125; data-to-parameter ratio = 12.3.

The title compound, $\text{C}_{20}\text{H}_{28}\text{O}_2$, is a new totarolone which was isolated from *Tetraclinis articulata* wood. The molecule forms an extended sheet of three fused rings which exhibit different conformations. The non-aromatic oxo-substituted ring has a screw boat conformation, while the central ring has a half-chair conformation. Molecules are linked to each other by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds involving carbonyl and hydroxyl groups.

Related literature

For related literature, see: Atta-Ur-Rahman & Choudary (1999); Barrero *et al.* (2003); Bellakhdar (1997); Comte *et al.* (1995); Cremer & Pople (1975); Evidente *et al.* (1997); Lawsen *et al.* (1988); Macias *et al.* (2000); Panter *et al.* (2002); Vlietinck (1987); Zeroual *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{20}\text{H}_{28}\text{O}_2$
 $M_r = 300.42$
Monoclinic, $P2_1$
 $a = 7.1873$ (4) Å $b = 14.2273$ (9) Å
 $c = 8.3860$ (4) Å
 $\beta = 96.952$ (4)°
 $V = 851.21$ (8) Å³ $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹ $T = 298$ (2) K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: none
10680 measured reflections2537 independent reflections
2329 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.126$
 $S = 1.09$
2537 reflections
207 parameters
1 restraintH atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}1-\text{H}\cdots\text{O}2^i$	0.81 (4)	2.06 (4)	2.799 (3)	152 (4)

Symmetry code: (i) $x - 1, y, z + 1$.

Data collection: COLLECT (Bruker–Nonius, 2006); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON (Spek, 2003).

The authors thank Professor Michel Giorgi for collecting the data, and Professor Nouzha Bouhmaida and Professor J.-C. Daran for fruitful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WW2088).

References

- Atta-Ur-Rahman & Choudary, M. I. (1999). *Nat. Prod. Rep.* **16**, 619–635.
Barrero, A. F., Quílez del Moral, J. F., Lucas, R., Payá, M., Akssira, M., Akkad, S. & Mellouki, F. (2003). *J. Nat. Prod.* **66**, 844–850.
Bellakhdar, J. (1997). *La Pharmacopée Marocaine Traditionnelle*, pp. 272–274. Paris: Edition Ibis Press.
Bruker–Nonius (2006). COLLECT. Bruker–Nonius BV, Delft, The Netherlands.
Comte, G., Allais, D. P., Simon, A., Es-Saady, D., Chulia, A. J., Delage, C. & Saux, M. (1995). *J. Nat. Prod.* **58**, 239–243.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Evidente, A., Sparapano, L., Fierro, O., Bruno, G., Giordano, F. & Motta, A. (1997). *Phytochemistry*, **45**, 705–713.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Lawsen, D. R., Daneshmandi, D. A., Shilling, D. G., Menetrez, M. L. & Spurr, H. W. (1988). *Am. Chem. Soc. Symp. Ser.* **380**, 363–377.
Macias, F. A., Simonet, A. M., Pacheco, P. C., Berrero, A. F., Cabera, E. & Jiménez-Gonzalez, D. (2000). *J. Agric. Food Chem.* **48**, 3003–3007.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

- Panter, K. E., Manners, G. D., Stegelmeier, B. L., Lee, S., Gardner, D. R., Ralphs, M. H., Pfister, J. A. & James, L. F. (2002). *Biochem. Syst. Ecol.* **30**, 113–128.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Vlietinck, A. (1987). *Biologically Active Natural Products*, edited by K. Hostettmann & P. J. Lea, pp. 33–47. Oxford University Press.
- Zeroual, A., Mazoir, N., Maya, C. M., Berraho, M., Auhmani, A. & Benharref, A. (2007). *Acta Cryst.* **E63**, o2915.

supplementary materials

Acta Cryst. (2007). E63, o3497–o3498 [doi:10.1107/S1600536807033594]

(4a*S,10a*R**)-7-Hydroxy-8-isopropyl-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-2-one**

A. Zeroual, N. Mazoir, M. Berraho, A. Auhmani and A. Benharref

Comment

Natural products, through evolution of producing organisms have many biological activities that are manifested through an array of different molecular target sites. Thus, diterpenoids isolated from plants have good pharmacological activities (Atta-Ur-Rahman & Choudary, 1999, Panter *et al.*, 2002). The divatriene-diols from tobacco leaf surfaces are phytotoxics to serious weed *Echinochloa crus-galli* (Lawsen *et al.*, 1988). Some of them have been suggested as herbicides (Macias *et al.*, 2000). In our study, we are interested to the aromatic and medicinal plant: *Tetraclinis articulata* which was subsequently used in traditional medicine (Bellakhdar, 1997). Some of its oxygenated compounds are effective as an antibiotic (Vlietinck, 1987), antifongic (Evidente *et al.*, 1997), cytotoxic (Comte *et al.*, 1995) and inhibit various human leukocyte functions (Barrero *et al.*, 2003). In order to isolate similar compounds, we have studied the hexanic extract of *Tetraclinis articulata* wood. Thus, extraction with hexane using a soxhlet apparatus allow us to isolate compound (I), new diterpenic isomer of (4a*S*,6*cS*)-8-Hydroxy-9-isopropyl-4,4,6*c*-trimethyl-1,2,3,4,4a,5,6, 6*c*-octahydrophenanthren-3-one (Zeroual *et al.*, 2007). The structure of (I) was established by ¹H and ¹³C NMR and confirmed by its single-crystal X-ray structure.

The molecule (I) is built up from three fused six-membered rings. The non aromatic oxo-substituted ring has a screw boat conformation, as indicated by the total puckering amplitude QT = 0.63 (2) Å and spherical polar angle $\theta = -82.16$ (2)° with $\varphi = 76.89$ (2)°. While the central ring has a half chair conformation with QT = 0.53 (2) Å, $\theta = 127.66$ (2)°, $\varphi = 151.53$ (2)° (Cremer & Pople, 1975). Molecules are linked by intermolecular O—H...O hydrogen bonds (Table 1, Figure 2) involving the carbonyl and the hydroxyl groups and propagate in chain parallel to the (101) plane.

Experimental

Continuous extraction with hexane employing a soxhlet apparatus during 48 h of *Tetraclinis articulata* wood let us, after evaporation of hexane and chromatography using silica gel column with hexane/ethyl acetate (97/3) as an eluent, to isolate compound (I) in 75% yield. Suitable crystals of (I) were obtained by evaporation of a dichloromethane solution at 277 K. m.p. = 444–445 K (dichloromethane); Spectroscopic analysis: ¹H NMR (300 MHz, CDCl₃, δ , p.p.m.): 2.21 (2*H*, H3, m), 2.04 (2*H*, H4, m), 6.90 (1*H*, H5, d, J = 8.4 Hz), 6.50 (1*H*, H6, d, J = 8.4 Hz), 4.85 (OH, s), 2.85 (2*H*, H9, m), 1.56 (2*H*, H10, m), 1.78 (1*H*, H10*a*, dd, J₁ = 10.7 Hz, J₂ = 1.9 Hz), 3.10 (1*H*, H11, m), 1.28 (3*H*, H12, d, J = 10 Hz), 1.30 (3*H*, H13, d, J = 10 Hz), 1.42 (3*H*, H14, s), 1.11 (3*H*, H15, s), 1.08 (3*H*, H16, s); ¹³C NMR (75 MHz, CDCl₃, δ , p.p.m.): 47.5 (C1), 218.5 (C2), 38.3 (C3), 34.8 (C4), 37.5 (C4a), 131.3 (C4b), 124.0 (C5), 115.0 (C6), 152.9 (C7), 159.6 (C8), 140.1 (C8a), 28.9 (C9), 21.0 (C10), 49.8 (C10*a*), 26.8 (C11), 20.1 (C12), 20.2 (C13), 22.4 (C14), 24.8 (C15), 21.2 (C16).

Refinement

Except H11 and O—H hydrogen atom, which were placed by Fourier synthesis C11—H11=0.99 (4) Å; O—H = 0.81 (4) Å, all H atoms attached to carbon atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic),

supplementary materials

0.96 Å (methyl), 0.97 Å (methylene), and 0.98 Å (methine) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic, methylene, methine and OH})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$.

In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined and thus the Friedel pairs were merged and any references to the Flack parameter were removed.

Figures

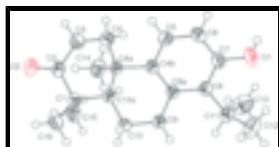


Fig. 1. : Molecular structure of compound (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

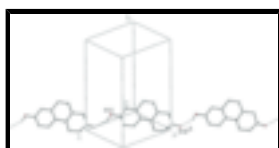


Fig. 2. : Partial packing view showing the O—H...O interactions (dashed lines) and the formation of a chain parallel to the (101) plane. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $-1 + x, y, 1 + z$]

(4aS*,10aR*)-7-Hydroxy-8-isopropyl-1,1,4a-trimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-2-one

Crystal data

$\text{C}_{20}\text{H}_{28}\text{O}_2$

$M_r = 300.42$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 7.1873$ (4) Å

$b = 14.2273$ (9) Å

$c = 8.3860$ (4) Å

$\beta = 96.952$ (4)°

$V = 851.21$ (8) Å³

$Z = 2$

$F_{000} = 328$

$D_x = 1.172$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2550 reflections

$\theta = 3.5\text{--}30.0^\circ$

$\mu = 0.07$ mm⁻¹

$T = 298$ (2) K

Prism, colourless

$0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: none

10680 measured reflections

2537 independent reflections

2329 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\text{max}} = 30.0^\circ$

$\theta_{\text{min}} = 2.9^\circ$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 20$

$l = -11 \rightarrow 11$

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
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.0844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.09$	$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
2537 reflections	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
207 parameters	Extinction correction: none
1 restraint	Absolute structure: unknown - see Supplementary Materials - Refinement section
Primary atom site location: structure-invariant direct methods	Flack parameter:
Secondary atom site location: difference Fourier map	Rogers parameter:

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
H	0.542 (5)	0.581 (3)	0.585 (4)	0.069 (10)*
H11	0.665 (4)	0.334 (3)	0.265 (4)	0.068 (9)*
C1	1.2274 (3)	0.52902 (14)	-0.1588 (2)	0.0380 (4)
C2	1.3486 (3)	0.61648 (15)	-0.1271 (2)	0.0398 (4)
C3	1.3735 (3)	0.6564 (2)	0.0381 (3)	0.0542 (6)
H3A	1.4179	0.7205	0.0311	0.065*
H3B	1.4722	0.6212	0.1009	0.065*
C4	1.2054 (3)	0.65802 (17)	0.1310 (3)	0.0471 (5)
H4A	1.1733	0.7230	0.1499	0.057*
H4B	1.2404	0.6289	0.2349	0.057*
C4A	1.0296 (2)	0.60751 (12)	0.0471 (2)	0.0330 (3)
C4B	0.8971 (2)	0.58501 (12)	0.1734 (2)	0.0332 (3)
C5	0.8517 (3)	0.65705 (14)	0.2743 (2)	0.0411 (4)
H5	0.8987	0.7170	0.2608	0.049*
C6	0.7385 (3)	0.64173 (15)	0.3939 (2)	0.0448 (4)
H6	0.7078	0.6913	0.4582	0.054*
C7	0.6712 (3)	0.55241 (15)	0.4172 (2)	0.0428 (4)
C8	0.7087 (3)	0.47748 (13)	0.3179 (2)	0.0371 (4)
C8A	0.8209 (2)	0.49546 (13)	0.1932 (2)	0.0330 (3)

supplementary materials

C9	0.8596 (3)	0.41598 (13)	0.0819 (2)	0.0394 (4)
H9A	0.9486	0.3730	0.1394	0.047*
H9B	0.7442	0.3816	0.0514	0.047*
C10	0.9367 (3)	0.44883 (15)	-0.0687 (2)	0.0412 (4)
H10A	0.8390	0.4804	-0.1389	0.049*
H10B	0.9793	0.3952	-0.1258	0.049*
C10A	1.0988 (2)	0.51580 (12)	-0.02329 (19)	0.0333 (3)
H10	1.1775	0.4853	0.0654	0.040*
C11	0.6362 (3)	0.37951 (16)	0.3478 (3)	0.0441 (4)
C12	0.4239 (4)	0.3730 (2)	0.3432 (4)	0.0662 (7)
H12A	0.3892	0.3092	0.3627	0.099*
H12B	0.3653	0.3926	0.2396	0.099*
H12C	0.3836	0.4131	0.4245	0.099*
C13	0.7334 (5)	0.3358 (2)	0.5020 (4)	0.0752 (9)
H13A	0.6838	0.2741	0.5154	0.113*
H13B	0.7120	0.3744	0.5920	0.113*
H13C	0.8656	0.3316	0.4956	0.113*
C14	0.9273 (3)	0.67265 (15)	-0.0801 (3)	0.0469 (5)
H14A	0.8182	0.6413	-0.1323	0.070*
H14B	1.0095	0.6884	-0.1582	0.070*
H14C	0.8902	0.7290	-0.0296	0.070*
C15	1.3668 (3)	0.44607 (19)	-0.1482 (3)	0.0574 (6)
H15A	1.2991	0.3882	-0.1670	0.086*
H15B	1.4370	0.4447	-0.0433	0.086*
H15C	1.4511	0.4537	-0.2277	0.086*
C16	1.1232 (3)	0.5328 (2)	-0.3292 (2)	0.0567 (6)
H16A	1.0469	0.4776	-0.3484	0.085*
H16B	1.2124	0.5354	-0.4055	0.085*
H16C	1.0450	0.5877	-0.3403	0.085*
O1	0.5674 (3)	0.53377 (15)	0.5392 (2)	0.0646 (5)
O2	1.4340 (3)	0.65004 (15)	-0.2306 (2)	0.0618 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0377 (8)	0.0433 (9)	0.0349 (7)	-0.0009 (7)	0.0115 (6)	-0.0001 (7)
C2	0.0321 (8)	0.0469 (10)	0.0419 (9)	-0.0007 (7)	0.0105 (7)	0.0037 (8)
C3	0.0420 (10)	0.0700 (15)	0.0520 (11)	-0.0190 (11)	0.0122 (8)	-0.0117 (11)
C4	0.0409 (9)	0.0522 (11)	0.0501 (10)	-0.0126 (9)	0.0134 (8)	-0.0140 (9)
C4A	0.0323 (7)	0.0311 (8)	0.0374 (8)	-0.0010 (6)	0.0109 (6)	-0.0013 (6)
C4B	0.0318 (7)	0.0329 (8)	0.0364 (8)	0.0007 (6)	0.0096 (6)	-0.0019 (6)
C5	0.0442 (9)	0.0332 (8)	0.0485 (10)	-0.0014 (8)	0.0158 (7)	-0.0053 (8)
C6	0.0502 (10)	0.0429 (10)	0.0441 (9)	0.0036 (8)	0.0174 (8)	-0.0090 (8)
C7	0.0456 (10)	0.0486 (11)	0.0371 (9)	0.0021 (8)	0.0166 (7)	-0.0007 (8)
C8	0.0405 (9)	0.0358 (9)	0.0365 (8)	-0.0020 (7)	0.0111 (7)	0.0016 (7)
C8A	0.0325 (7)	0.0332 (8)	0.0343 (7)	0.0013 (6)	0.0084 (6)	-0.0006 (6)
C9	0.0464 (9)	0.0308 (8)	0.0441 (9)	-0.0048 (7)	0.0177 (7)	-0.0041 (7)
C10	0.0459 (9)	0.0392 (9)	0.0410 (9)	-0.0073 (8)	0.0163 (7)	-0.0085 (7)

C10A	0.0341 (7)	0.0334 (8)	0.0340 (7)	0.0004 (6)	0.0105 (6)	-0.0009 (6)
C11	0.0513 (11)	0.0418 (10)	0.0414 (9)	-0.0049 (8)	0.0141 (8)	0.0063 (8)
C12	0.0557 (13)	0.0660 (16)	0.0774 (17)	-0.0132 (12)	0.0109 (12)	0.0178 (13)
C13	0.089 (2)	0.0552 (15)	0.0755 (17)	-0.0051 (14)	-0.0143 (15)	0.0209 (14)
C14	0.0488 (10)	0.0403 (10)	0.0529 (11)	0.0029 (8)	0.0117 (9)	0.0112 (8)
C15	0.0601 (13)	0.0531 (12)	0.0656 (14)	0.0122 (11)	0.0343 (11)	0.0053 (11)
C16	0.0524 (11)	0.0832 (17)	0.0358 (9)	-0.0141 (12)	0.0105 (8)	-0.0057 (10)
O1	0.0843 (13)	0.0630 (11)	0.0551 (9)	-0.0062 (10)	0.0440 (9)	-0.0062 (9)
O2	0.0670 (10)	0.0661 (11)	0.0576 (9)	-0.0202 (9)	0.0293 (8)	-0.0013 (8)

Geometric parameters (Å, °)

C1—C2	1.524 (3)	C9—C10	1.513 (3)
C1—C16	1.532 (3)	C9—H9A	0.9700
C1—C15	1.544 (3)	C9—H9B	0.9700
C1—C10A	1.560 (2)	C10—C10A	1.518 (2)
C2—O2	1.219 (2)	C10—H10A	0.9700
C2—C3	1.488 (3)	C10—H10B	0.9700
C3—C4	1.515 (3)	C10A—H10	0.9800
C3—H3A	0.9700	C11—C12	1.525 (3)
C3—H3B	0.9700	C11—C13	1.526 (3)
C4—C4A	1.547 (3)	C11—H11	0.99 (4)
C4—H4A	0.9700	C12—H12A	0.9600
C4—H4B	0.9700	C12—H12B	0.9600
C4A—C14	1.532 (3)	C12—H12C	0.9600
C4A—C10A	1.539 (2)	C13—H13A	0.9600
C4A—C4B	1.541 (2)	C13—H13B	0.9600
C4B—C5	1.392 (2)	C13—H13C	0.9600
C4B—C8A	1.405 (2)	C14—H14A	0.9600
C5—C6	1.383 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—C7	1.382 (3)	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
C7—O1	1.363 (2)	C15—H15C	0.9600
C7—C8	1.399 (3)	C16—H16A	0.9600
C8—C8A	1.419 (2)	C16—H16B	0.9600
C8—C11	1.519 (3)	C16—H16C	0.9600
C8A—C9	1.513 (2)	O1—H	0.81 (4)
C2—C1—C16	109.82 (18)	H9A—C9—H9B	107.7
C2—C1—C15	105.10 (17)	C9—C10—C10A	109.39 (15)
C16—C1—C15	108.72 (19)	C9—C10—H10A	109.8
C2—C1—C10A	110.36 (14)	C10A—C10—H10A	109.8
C16—C1—C10A	114.69 (15)	C9—C10—H10B	109.8
C15—C1—C10A	107.67 (15)	C10A—C10—H10B	109.8
O2—C2—C3	119.9 (2)	H10A—C10—H10B	108.2
O2—C2—C1	121.45 (19)	C10—C10A—C4A	110.77 (14)
C3—C2—C1	118.43 (17)	C10—C10A—C1	113.42 (14)
C2—C3—C4	118.09 (17)	C4A—C10A—C1	115.10 (14)
C2—C3—H3A	107.8	C10—C10A—H10	105.5

supplementary materials

C4—C3—H3A	107.8	C4A—C10A—H10	105.5
C2—C3—H3B	107.8	C1—C10A—H10	105.5
C4—C3—H3B	107.8	C8—C11—C12	114.4 (2)
H3A—C3—H3B	107.1	C8—C11—C13	112.5 (2)
C3—C4—C4A	114.68 (16)	C12—C11—C13	110.5 (2)
C3—C4—H4A	108.6	C8—C11—H11	112 (2)
C4A—C4—H4A	108.6	C12—C11—H11	103.9 (17)
C3—C4—H4B	108.6	C13—C11—H11	102.5 (18)
C4A—C4—H4B	108.6	C11—C12—H12A	109.5
H4A—C4—H4B	107.6	C11—C12—H12B	109.5
C14—C4A—C10A	113.31 (15)	H12A—C12—H12B	109.5
C14—C4A—C4B	108.67 (14)	C11—C12—H12C	109.5
C10A—C4A—C4B	110.03 (13)	H12A—C12—H12C	109.5
C14—C4A—C4	109.42 (16)	H12B—C12—H12C	109.5
C10A—C4A—C4	106.51 (15)	C11—C13—H13A	109.5
C4B—C4A—C4	108.80 (14)	C11—C13—H13B	109.5
C5—C4B—C8A	118.19 (15)	H13A—C13—H13B	109.5
C5—C4B—C4A	118.32 (15)	C11—C13—H13C	109.5
C8A—C4B—C4A	123.49 (14)	H13A—C13—H13C	109.5
C6—C5—C4B	121.75 (18)	H13B—C13—H13C	109.5
C6—C5—H5	119.1	C4A—C14—H14A	109.5
C4B—C5—H5	119.1	C4A—C14—H14B	109.5
C7—C6—C5	119.62 (18)	H14A—C14—H14B	109.5
C7—C6—H6	120.2	C4A—C14—H14C	109.5
C5—C6—H6	120.2	H14A—C14—H14C	109.5
O1—C7—C6	121.19 (19)	H14B—C14—H14C	109.5
O1—C7—C8	117.43 (19)	C1—C15—H15A	109.5
C6—C7—C8	121.37 (16)	C1—C15—H15B	109.5
C7—C8—C8A	117.96 (16)	H15A—C15—H15B	109.5
C7—C8—C11	120.44 (16)	C1—C15—H15C	109.5
C8A—C8—C11	121.56 (16)	H15A—C15—H15C	109.5
C4B—C8A—C8	120.99 (15)	H15B—C15—H15C	109.5
C4B—C8A—C9	120.11 (15)	C1—C16—H16A	109.5
C8—C8A—C9	118.89 (15)	C1—C16—H16B	109.5
C10—C9—C8A	113.39 (16)	H16A—C16—H16B	109.5
C10—C9—H9A	108.9	C1—C16—H16C	109.5
C8A—C9—H9A	108.9	H16A—C16—H16C	109.5
C10—C9—H9B	108.9	H16B—C16—H16C	109.5
C8A—C9—H9B	108.9	C7—O1—H	112 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H\cdots O2^i$	0.81 (4)	2.06 (4)	2.799 (3)	152 (4)

Symmetry codes: (i) $x-1, y, z+1$.

Fig. 1

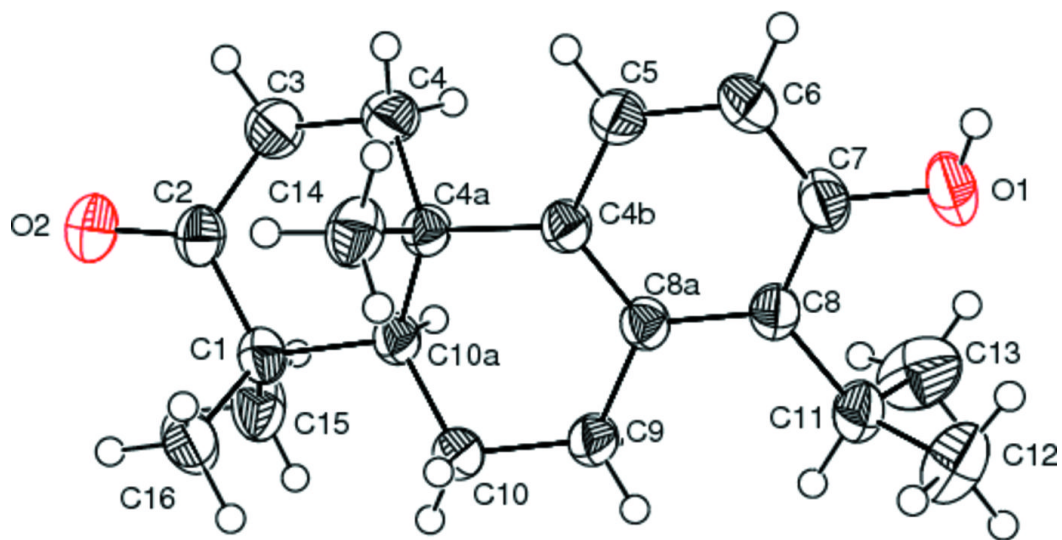


Fig. 2

