Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(4a*S*,6c*S*)-8-Hydroxy-9-isopropyl-4,4,6c-trimethyl-1,2,3,4,4a,5,6,6c-octahydrophenanthren-3-one

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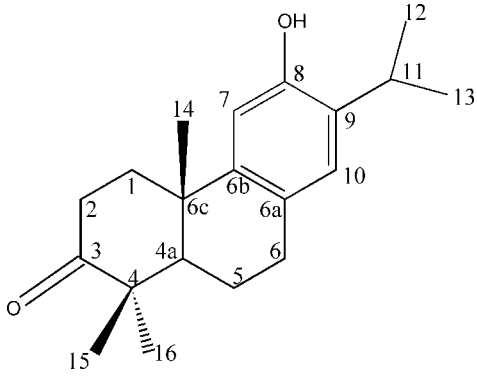
Received 26 April 2007; accepted 5 May 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 15.5.

The title compound, $C_{20}H_{28}O_2$, is a diterpenoid isolated from the wood of the *Tetraclinis articulata* plant. The molecule contains three fused rings which exhibit different conformations. The non-aromatic oxo-substituted ring has a chair conformation, while the central ring has an envelope conformation. The crystal structure is stabilized by O—H···O hydrogen bonds.

Related literature

For related literature, see: Abbas *et al.* (2006); Atta-ur-Rahman & Choudary (1999); Azucena & Mobashery (2001); Cremer & Pople (1975); He *et al.* (1999); Panter *et al.* (2002); Ulusu *et al.* (2002).



Experimental

Crystal data

$C_{20}H_{28}O_2$
M_r = 300.42
Orthorhombic, $P2_12_12_1$
a = 7.5554 (3) Å
b = 13.3987 (6) Å
c = 16.7825 (6) Å
V = 1698.94 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.5 × 0.4 × 0.3 mm

Data collection

Bruker X8 APEX CCD area-detector diffractometer
Absorption correction: none
26366 measured reflections
2891 independent reflections
2669 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.07
2891 reflections
187 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	2.02	2.7969 (13)	158

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX-W2K-NT* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; 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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Professor J. C. Daran for fruitful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2021).

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 doi:10.1107/S1600536807022258

supplementary materials

Acta Cryst. (2007). E**63**, o2915 [**doi:10.1107/S1600536807022258**]

(4a*S*,6c*S*)-8-Hydroxy-9-isopropyl-4,4,6c-trimethyl-1,2,3,4,4a,5,6,6c-octahydrophenanthren-3-one

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Comment

Diterpenoids producing by plants exhibit well known pharmacological activities (Atta-ur-Rahman & Choudary, 1999; Panter *et al.*, 2002; Azucena & Mobashery, 2001; He *et al.*, 1999; Ulusu *et al.*, 2002). In order to isolate similar compounds, we have undertaken this work. Thus, hexane extraction of wood from the Tetraclinis articulata plant (Abbas *et al.*, 2006), followed by chromatography on a silica gel column, gave the diterpene compound (I) as the sole product in good yield.

The molecule (I) is composed of three fused six-membered rings. The non aromatic six membered ring C4a/C6C/C1/C2/C3/C4 has a chair conformation, as indicated by the total puckering amplitude QT = 0.48 (2)Å and spherical polar angle θ =167.65 (1)° with φ = 163.52 (2)°. The six membered ring C5/C6/C6a/C6b/C6c/C4a displays an envelope conformation with QT = 0.54 (1) Å, θ =52.01 (1)° (Cremer & Pople, 1975). Molecules are linked by intermolecular O—H···O hydrogen bonds (Table 1, Figure 2) involving the carbonyl and the hydroxyl groups. The hydrogen bonds system results in the formation of chains parallel to the *b* axis.

Experimental

The first study was undertaken on a hexane extract. Subsequent studies were carried out by continuous hexane extraction of thuya wood with a soxhlet apparatus, over a 48 h period. The residue obtained, after evaporation of hexane, was chromatographed on a silica gel column with hexane–ethyl acetate (96:4 *v*/*v*) as the eluant. This allowed the isolation of compound (I) in 85% yield, as the sole product. Suitable crystals of compound (I) were obtained by evaporation of a dichloromethane solution; m.p. 464–465 K. Spectroscopic analysis: ^{1}H NMR (300 MHz, $CDCl_3$, δ, p.p.m.): 6.55 (H-7,s), 6.80 (H-10, s), 5.20 (OH, s), 1.28 (H-12, d, J = 10 Hz), 1.29 (H-13, d, J = 10 Hz), 1.15 (H-14, s), 1.12 (H-15, s), 1.13 (H-16, s); ^{13} C NMR (75 MHz, $CDCl_3$, δ, p.p.m.): 34.6 (C1), 37.6 (C2), 218.2 (C3), 47.6 (C4), 50.8 (C4a), 20.4 (C5), 29.3 (C6), 128.4 (C6a), 145.5 (C6b), 34.8 (C6c), 111.9 (C7), 151.7 (C8), 132.9 (C9), 126.8 (C10), 22.5 (C11), 24.6 (C12), 24.7 (C13), 26.7 (C14), 21.0 (C15), 21.3 (C16).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.96Å (methyl), 0.97 Å (methylene), 0.98Å (methine) and O—H= 0.82 Å with U_{iso}(H) = 1.2U_{eq}(aromatic, methylene, methine and OH) or U_{iso}(H) = 1.5U_{eq}(methyl). In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined. The Friedel pairs were merged and any references to the Flack parameter was removed.

Figures

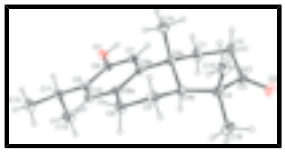

Fig. 1. Molecular structure of compound I, showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

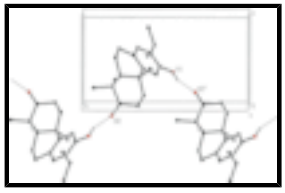

Fig. 2. Partial packing diagram, showing the O—H···O interactions (dashed lines) and the formation of a chain parallel to the *b* axis. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) −*x*, *y* − 1/2, 1/2 − *z*.]

(4aS,6cS)-8-Hydroxy-9-isopropyl-4,4,6c-trimethyl-1,2,3,4,4a,5,6,6c- octahydrophenanthren-3-one

Crystal data

$C_{20}H_{28}O_2$	F_{000} = 656
M_r = 300.42	D_x = 1.175 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 7546 reflections
a = 7.5554 (3) Å	θ = 2.9–30.5º
b = 13.3987 (6) Å	μ = 0.07 mm^{-1}
c = 16.7825 (6) Å	*T* = 298 (2) K
V = 1698.94 (12) Å^3	Prism, colourless
Z = 4	0.5 × 0.4 × 0.3 mm

Data collection

Bruker X8 APEX CCD area-detector diffractometer	2669 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.029
Monochromator: graphite	θ_{max} = 30.5º
T = 298(2) K	θ_{min} = 3.0º
φ and ω scans	*h* = −10→10
Absorption correction: none	*k* = −18→19
26366 measured reflections	*l* = −21→23
2891 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.4523P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.105	$\Delta\rho_{max}$ = 0.51 e Å^{-3}

S = 1.07
2891 reflections
187 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0911 (2)	0.63566 (12)	0.27798 (10)	0.023
H13A	0.0367	0.6657	0.2315	0.028*
H13B	0.0788	0.5639	0.2731	0.028*
C2	−0.0064 (2)	0.66929 (13)	0.35037 (12)	0.030
H18A	0.0365	0.6321	0.3961	0.037*
H18B	−0.1310	0.6539	0.3439	0.037*
C3	0.0129 (2)	0.77835 (11)	0.36676 (9)	0.0193 (3)
C4	0.1986 (2)	0.82219 (11)	0.36287 (9)	0.0176 (3)
C4A	0.3067 (2)	0.77609 (10)	0.29306 (8)	0.0145 (2)
H2	0.2598	0.8076	0.2448	0.017*
C5	0.5023 (2)	0.80471 (11)	0.29528 (9)	0.0202 (3)
H12A	0.5135	0.8749	0.3084	0.024*
H12B	0.5619	0.7665	0.3364	0.024*
C6	0.5902 (2)	0.78463 (11)	0.21496 (9)	0.0201 (3)
H9A	0.5797	0.8441	0.1824	0.024*
H9B	0.7153	0.7727	0.2238	0.024*
C6A	0.5147 (2)	0.69726 (10)	0.16901 (8)	0.0154 (3)
C6B	0.37190 (19)	0.64076 (10)	0.19642 (8)	0.0146 (2)
C6C	0.28920 (19)	0.66197 (10)	0.27846 (9)	0.0153 (3)
C7	0.3095 (2)	0.56252 (10)	0.14895 (8)	0.0160 (3)
H5	0.2158	0.5236	0.1671	0.019*
C8	0.3845 (2)	0.54175 (10)	0.07531 (8)	0.0168 (3)
C9	0.5259 (2)	0.59822 (10)	0.04603 (8)	0.0168 (3)
C10	0.5878 (2)	0.67493 (11)	0.09440 (8)	0.0173 (3)
H7	0.6823	0.7132	0.0764	0.021*
C11	0.6034 (2)	0.57533 (11)	−0.03525 (9)	0.0221 (3)
H17	0.6046	0.5026	−0.0413	0.026*
C12	0.4833 (3)	0.61746 (14)	−0.10046 (9)	0.0291 (4)
H15A	0.5331	0.6027	−0.1518	0.044*
H15B	0.4733	0.6884	−0.0942	0.044*
H15C	0.3681	0.5876	−0.0964	0.044*
C13	0.7933 (3)	0.61207 (15)	−0.04667 (11)	0.0322 (4)
H20A	0.8333	0.5949	−0.0992	0.048*
H20B	0.8687	0.5812	−0.0078	0.048*
H20C	0.7970	0.6832	−0.0402	0.048*
C14	0.3857 (3)	0.59435 (12)	0.33870 (9)	0.0249 (3)
H16A	0.3380	0.6052	0.3910	0.037*

H16B	0.5097	0.6100	0.3387	0.037*
H16C	0.3694	0.5257	0.3239	0.037*
C15	0.2800 (2)	0.80433 (13)	0.44633 (9)	0.0261 (3)
H19A	0.3979	0.8309	0.4477	0.039*
H19B	0.2835	0.7340	0.4571	0.039*
H19C	0.2089	0.8370	0.4859	0.039*
C16	0.1831 (2)	0.93550 (12)	0.34921 (10)	0.0246 (3)
H14A	0.2993	0.9643	0.3465	0.037*
H14B	0.1186	0.9651	0.3925	0.037*
H14C	0.1217	0.9478	0.3001	0.037*
O1	0.32359 (17)	0.46427 (8)	0.02913 (7)	0.0228 (2)
H1	0.2418	0.4362	0.0521	0.034*
O2	−0.11497 (17)	0.82758 (9)	0.38691 (7)	0.0265 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.022	0.021	0.028	−0.006	0.008	−0.009
C2	0.021	0.023	0.047	−0.003	0.011	−0.009
C3	0.0204 (7)	0.0205 (6)	0.0170 (6)	0.0031 (6)	0.0006 (5)	−0.0014 (5)
C4	0.0194 (7)	0.0163 (6)	0.0170 (6)	0.0027 (5)	−0.0025 (5)	−0.0032 (5)
C4A	0.0157 (6)	0.0122 (5)	0.0155 (6)	0.0006 (5)	−0.0019 (5)	−0.0009 (4)
C5	0.0179 (7)	0.0196 (6)	0.0232 (7)	−0.0041 (5)	−0.0015 (6)	−0.0038 (5)
C6	0.0193 (7)	0.0180 (6)	0.0229 (6)	−0.0058 (5)	0.0011 (6)	−0.0005 (5)
C6A	0.0149 (6)	0.0126 (5)	0.0188 (6)	−0.0012 (5)	0.0002 (5)	0.0010 (4)
C6B	0.0146 (6)	0.0122 (5)	0.0170 (6)	0.0001 (5)	0.0009 (5)	0.0009 (4)
C6C	0.0159 (7)	0.0119 (5)	0.0180 (6)	0.0000 (5)	0.0022 (5)	0.0000 (5)
C7	0.0173 (6)	0.0129 (5)	0.0178 (6)	−0.0024 (5)	0.0024 (5)	0.0003 (5)
C8	0.0199 (7)	0.0127 (6)	0.0179 (6)	0.0004 (5)	0.0013 (5)	−0.0004 (5)
C9	0.0191 (7)	0.0140 (6)	0.0174 (6)	0.0011 (5)	0.0032 (5)	0.0019 (5)
C10	0.0170 (7)	0.0144 (6)	0.0207 (6)	−0.0016 (5)	0.0027 (5)	0.0033 (5)
C11	0.0278 (8)	0.0174 (6)	0.0210 (6)	0.0004 (6)	0.0088 (6)	0.0011 (5)
C12	0.0381 (10)	0.0311 (8)	0.0180 (6)	−0.0031 (8)	0.0024 (7)	0.0009 (6)
C13	0.0291 (9)	0.0392 (9)	0.0283 (8)	−0.0005 (8)	0.0124 (7)	0.0037 (7)
C14	0.0356 (9)	0.0185 (7)	0.0206 (6)	0.0084 (7)	0.0030 (7)	0.0043 (5)
C15	0.0298 (9)	0.0322 (8)	0.0162 (6)	0.0053 (7)	−0.0048 (6)	−0.0047 (6)
C16	0.0261 (8)	0.0167 (6)	0.0309 (8)	0.0028 (6)	−0.0039 (7)	−0.0066 (6)
O1	0.0295 (6)	0.0186 (5)	0.0202 (5)	−0.0076 (5)	0.0060 (5)	−0.0047 (4)
O2	0.0216 (6)	0.0288 (6)	0.0291 (6)	0.0078 (5)	0.0021 (5)	−0.0042 (5)

Geometric parameters (Å, °)

C1—C2	1.490 (2)	C7—C8	1.3877 (19)
C1—C6C	1.538 (2)	C7—H5	0.9300
C1—H13A	0.9700	C8—O1	1.3748 (17)
C1—H13B	0.9700	C8—C9	1.398 (2)
C2—C3	1.494 (2)	C9—C10	1.391 (2)
C2—H18A	0.9700	C9—C11	1.516 (2)
C2—H18B	0.9700	C10—H7	0.9300

C3—O2	1.2177 (19)	C11—C13	1.529 (2)
C3—C4	1.522 (2)	C11—C12	1.530 (2)
C4—C16	1.540 (2)	C11—H17	0.9800
C4—C15	1.548 (2)	C12—H15A	0.9600
C4—C4A	1.556 (2)	C12—H15B	0.9600
C4A—C5	1.527 (2)	C12—H15C	0.9600
C4A—C6C	1.5541 (19)	C13—H20A	0.9600
C4A—H2	0.9800	C13—H20B	0.9600
C5—C6	1.526 (2)	C13—H20C	0.9600
C5—H12A	0.9700	C14—H16A	0.9600
C5—H12B	0.9700	C14—H16B	0.9600
C6—C6A	1.513 (2)	C14—H16C	0.9600
C6—H9A	0.9700	C15—H19A	0.9600
C6—H9B	0.9700	C15—H19B	0.9600
C6A—C6B	1.396 (2)	C15—H19C	0.9600
C6A—C10	1.401 (2)	C16—H14A	0.9600
C6B—C7	1.3986 (19)	C16—H14B	0.9600
C6B—C6C	1.5384 (19)	C16—H14C	0.9600
C6C—C14	1.541 (2)	O1—H1	0.8200
C2—C1—C6C	114.05 (14)	C14—C6C—C4A	115.77 (12)
C2—C1—H13A	108.7	C8—C7—C6B	121.33 (13)
C6C—C1—H13A	108.7	C8—C7—H5	119.3
C2—C1—H13B	108.7	C6B—C7—H5	119.3
C6C—C1—H13B	108.7	O1—C8—C7	121.11 (13)
H13A—C1—H13B	107.6	O1—C8—C9	117.79 (13)
C1—C2—C3	113.42 (15)	C7—C8—C9	121.10 (13)
C1—C2—H18A	108.9	C10—C9—C8	116.86 (13)
C3—C2—H18A	108.9	C10—C9—C11	123.02 (13)
C1—C2—H18B	108.9	C8—C9—C11	120.12 (13)
C3—C2—H18B	108.9	C9—C10—C6A	123.15 (13)
H18A—C2—H18B	107.7	C9—C10—H7	118.4
O2—C3—C2	120.23 (16)	C6A—C10—H7	118.4
O2—C3—C4	122.28 (13)	C9—C11—C13	114.20 (14)
C2—C3—C4	117.35 (14)	C9—C11—C12	109.89 (13)
C3—C4—C16	108.46 (13)	C13—C11—C12	110.37 (13)
C3—C4—C15	105.51 (13)	C9—C11—H17	107.4
C16—C4—C15	108.51 (13)	C13—C11—H17	107.4
C3—C4—C4A	111.28 (11)	C12—C11—H17	107.4
C16—C4—C4A	108.62 (13)	C11—C12—H15A	109.5
C15—C4—C4A	114.28 (12)	C11—C12—H15B	109.5
C5—C4A—C6C	109.47 (12)	H15A—C12—H15B	109.5
C5—C4A—C4	112.95 (12)	C11—C12—H15C	109.5
C6C—C4A—C4	117.68 (12)	H15A—C12—H15C	109.5
C5—C4A—H2	105.2	H15B—C12—H15C	109.5
C6C—C4A—H2	105.2	C11—C13—H20A	109.5
C4—C4A—H2	105.2	C11—C13—H20B	109.5
C6—C5—C4A	110.79 (12)	H20A—C13—H20B	109.5
C6—C5—H12A	109.5	C11—C13—H20C	109.5
C4A—C5—H12A	109.5	H20A—C13—H20C	109.5

C6—C5—H12B	109.5	H20B—C13—H20C	109.5
C4A—C5—H12B	109.5	C6C—C14—H16A	109.5
H12A—C5—H12B	108.1	C6C—C14—H16B	109.5
C6A—C6—C5	115.00 (12)	H16A—C14—H16B	109.5
C6A—C6—H9A	108.5	C6C—C14—H16C	109.5
C5—C6—H9A	108.5	H16A—C14—H16C	109.5
C6A—C6—H9B	108.5	H16B—C14—H16C	109.5
C5—C6—H9B	108.5	C4—C15—H19A	109.5
H9A—C6—H9B	107.5	C4—C15—H19B	109.5
C6B—C6A—C10	118.92 (13)	H19A—C15—H19B	109.5
C6B—C6A—C6	122.87 (13)	C4—C15—H19C	109.5
C10—C6A—C6	118.17 (13)	H19A—C15—H19C	109.5
C6A—C6B—C7	118.64 (13)	H19B—C15—H19C	109.5
C6A—C6B—C6C	120.57 (12)	C4—C16—H14A	109.5
C7—C6B—C6C	120.76 (12)	C4—C16—H14B	109.5
C1—C6C—C6B	110.39 (12)	H14A—C16—H14B	109.5
C1—C6C—C14	109.21 (13)	C4—C16—H14C	109.5
C6B—C6C—C14	106.64 (12)	H14A—C16—H14C	109.5
C1—C6C—C4A	108.02 (12)	H14B—C16—H14C	109.5
C6B—C6C—C4A	106.76 (11)	C8—O1—H1	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	2.02	2.7969 (13)	158

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

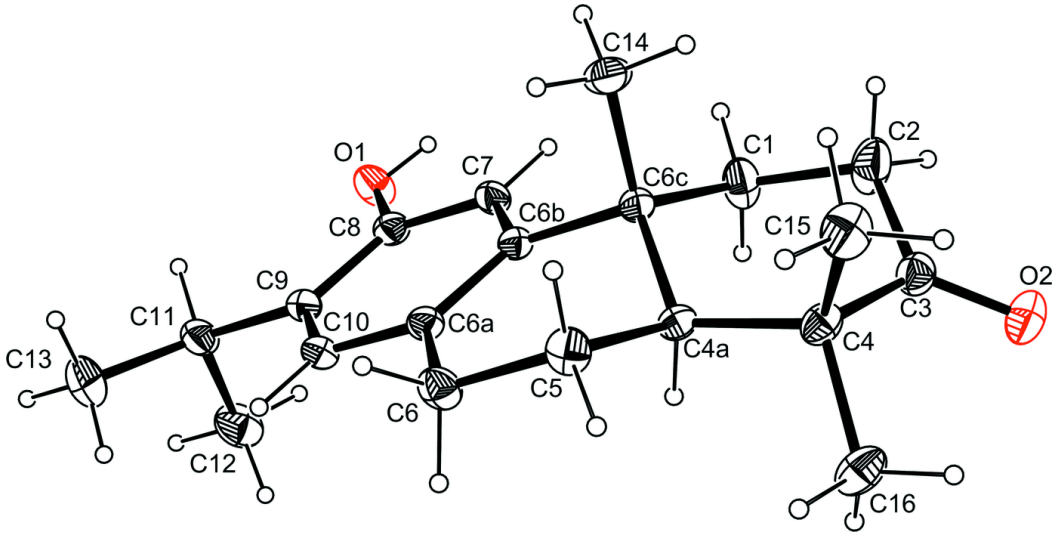
C14
O1
C7
C6c
C1
C2
C8
C6b
C15
O2
C9
C3
C11
C10
C6a
C4a
C4
C13
C5
C6
C12
C16

Fig. 2