Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6β,10β-Cadinene-6,10-diol: a sesquiterpenoid from the rhizome of *Acorus calamus* L.

Wei-Wei Dong,[a] Kai-Bei Yu,[b] Jian-Min Yue[c] and Run-Hua Lu[a]*

[a]Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China, [b]Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China, and [c]Shanghai Institute for Biological Sciences, Chinese Academy of Sciences, Shanghai 201203, People's Republic of China
Correspondence e-mail: lurh@cib.ac.cn

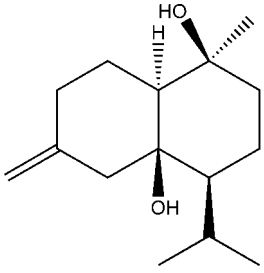
Received 17 July 2007; accepted 19 July 2007

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.090; data-to-parameter ratio = 10.7.

The title compound [systematic name: (1*S*,6*R*,7*S*,10*S*)-7-isopropyl-10-methyl-4-methylenedecahydronaphthalene-6,10-diol], $C_{15}H_{26}O_2$, was isolated from the rhizome of *Acorus calamus* L. The molecule contains two fused six-membered rings, each in a chair conformation. The molecules are linked together by intermolecular O—H···O hydrogen bonding.

Related literature

For general background, see: Robert & Henrey (1983). For a related structure, from which the absolute configuration was assigned for the title compound, see: Yamamura *et al.* (1971).



Experimental

Crystal data

$C_{15}H_{26}O_2$
M_r = 238.36
Orthorhombic, $P2_12_12$
a = 9.8941 (4) Å
b = 15.7201 (7) Å
c = 9.0288 (4) Å
V = 1404.3 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 153 (2) K
0.36 × 0.36 × 0.36 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
13880 measured reflections
1860 independent reflections
1823 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.090
S = 1.00
1860 reflections
174 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O2	0.90 (3)	1.94 (3)	2.7240 (13)	146 (2)
O2—H2*O*···O1^{i}	0.86 (2)	2.10 (2)	2.9452 (13)	167 (2)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1997); software used to prepare material for publication: *SHELXTL*.

Support from the 'Western Light' Joint Research Program of the Chinese Academy of Sciences is acknowledged. The authors are also grateful to the analytical staff of Chengdu Institute of Biology, Chinese Academy of Sciences for measuring the NMR spectra.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2295).

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 doi:10.1107/S1600536807035325

supplementary materials

Acta Cryst. (2007). E**63**, o3579 [**doi:10.1107/S1600536807035325**]

6β,10β-Cadinene-6,10-diol: a sesquiterpenoid from the rhizome of *Acorus calamus* L.

W.-W. Dong, K.-B. Yu, J.-M. Yue and R.-H. Lu

Comment

Acorus calamus L. is a traditional Chinese herb distributed widely in China. It is well known for its medicinal properties such as emetic stomach in dyspepsy, remittet fever, nerve tonic and expectorant (Robert & Henrey, 1983). As part of our investigation of the bioactive constituents of the rhizome of *A. calamus*, we recently isolated the title compound. Its structure was elucidated by spectroscopic analysis including two-dimensional NMR, and was confirmed by single-crystal X-ray diffraction analysis.

The molecule contains two *trans* fused six-membered rings (Fig. 1). In the crystal structure, the molecules are combined by intermolecular hydrogen bonding.

Experimental

The rhizome of *Acorus calamus L*. (15 kg) were collected in the Tibet Autonomous area of China. The ethanol extract (2 kg) was suspended in water (2 l) and partitioned successively with petroleum ether, EtOAc and n-butanol. The EtOAc extract (1 kg) was chromatographed over silica gel (160–200 mesh, 2.3 kg) column with eluents of increasing polarity [petroleum ether-acetone (20:1, 15:1, 10:1, 5:1, 2:1, 1:1)] to afford Fr. 1–7 according to TLC analysis. Fr.5 (200 g) was applied to a silica gel (200–300 mesh, 1.6 kg) column and eluted with petroleum ether-acetone (10:1, 5:1, 2:1, 1:1) to obtain the title compound. The isolated product was recrystallized from a methanol solution to afford single crystals (30 mg).

Refinement

Hydroxy H atoms were located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions with C—H = 0.94–1.00 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C). As no significant anomalous scatters the absolute configuration could not be determined from the X-ray analysis, Friedel pairs were merged. We assigned the conformation by reference to the chiral molecule of known absolute configuration (Yamamura *et al.*, 1971).

Figures

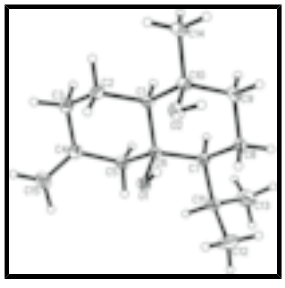
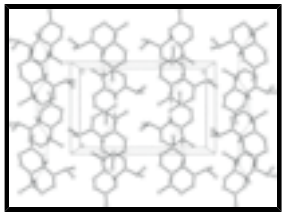


Fig. 1. View of the molecule of (1) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.



(1*S*,6*R*,7*S*,10*S*)-7-isopropyl-10-methyl-4-methylenedecahydronaphthalene-6,10-diol

Crystal data

$C_{15}H_{26}O_2$	D_x = 1.127 Mg m^{-3}
M_r = 238.36	Melting point = 443–444 K
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2 2ab	Cell parameters from 13629 reflections
a = 9.8941 (4) Å	θ = 3.0–27.5º
b = 15.7201 (7) Å	μ = 0.07 mm^{-1}
c = 9.0288 (4) Å	T = 153 (2) K
V = 1404.3 (1) Å^3	Block, colourless
Z = 4	0.36 × 0.36 × 0.36 mm
F_{000} = 528	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	1823 reflections with $I > 2\sigma(I)$
Radiation source: Rotating Anode	R_{int} = 0.021
Monochromator: graphite	θ_{max} = 27.5º
T = 153(2) K	θ_{min} = 3.0º
ω scans	h = −12→12
Absorption correction: none	k = −20→20
13880 measured reflections	l = −11→11
1860 independent 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
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.1876P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.00	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
1860 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

174 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.033 (5)
Secondary atom site location: difference Fourier map	

Special details

Experimental. ^{13}C NMR (600 MHz, $CDCl_3$, δ, p.p.m.): 50.3(C1), 22.8(C2), 34.3(C3), 145.9(C4), 45.6(C5), 76.4(C6), 51.5(C7), 16.5(C8), 41.3(C9), 71.8(C10), 25.5(C11), 18.0(C12), 23.6(C13), 28.1(C14), 111.5(C15).

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.61793 (9)	0.70501 (6)	0.40440 (9)	0.0191 (2)
O2	0.84327 (9)	0.79719 (6)	0.46991 (9)	0.0219 (2)
C1	0.74027 (12)	0.79747 (8)	0.22996 (13)	0.0169 (2)
H1	0.7625	0.8010	0.1221	0.020*
C2	0.64773 (13)	0.87333 (8)	0.26421 (16)	0.0238 (3)
H2A	0.6980	0.9270	0.2480	0.029*
H2B	0.6201	0.8712	0.3695	0.029*
C3	0.52124 (14)	0.87232 (9)	0.16545 (18)	0.0288 (3)
H3A	0.4599	0.9190	0.1955	0.035*
H3B	0.5479	0.8820	0.0611	0.035*
C4	0.44852 (13)	0.78906 (9)	0.17762 (14)	0.0238 (3)
C5	0.53566 (12)	0.71155 (8)	0.15681 (14)	0.0222 (3)
H5A	0.5623	0.7076	0.0513	0.027*
H5B	0.4818	0.6603	0.1809	0.027*
C6	0.66413 (11)	0.71181 (7)	0.25289 (12)	0.0156 (2)
C7	0.75716 (12)	0.63626 (7)	0.21106 (14)	0.0183 (2)
H7	0.7832	0.6457	0.1053	0.022*
C8	0.88917 (13)	0.63991 (8)	0.29947 (14)	0.0211 (3)
H8A	0.8690	0.6338	0.4063	0.025*
H8B	0.9482	0.5920	0.2697	0.025*
C9	0.96268 (12)	0.72341 (8)	0.27323 (15)	0.0213 (3)
H9A	0.9885	0.7273	0.1675	0.026*
H9B	1.0467	0.7242	0.3328	0.026*
C10	0.87654 (12)	0.80067 (8)	0.31411 (13)	0.0187 (2)
C11	0.69087 (14)	0.54676 (8)	0.21637 (16)	0.0251 (3)

H11	0.5945	0.5535	0.1852	0.030*
C12	0.6908 (2)	0.50475 (10)	0.36807 (18)	0.0434 (4)
H12A	0.6393	0.4516	0.3636	0.052*
H12B	0.6491	0.5431	0.4404	0.052*
H12C	0.7840	0.4925	0.3980	0.052*
C13	0.75947 (19)	0.48835 (9)	0.1035 (2)	0.0398 (4)
H13A	0.8562	0.4845	0.1258	0.048*
H13B	0.7472	0.5116	0.0037	0.048*
H13C	0.7190	0.4315	0.1087	0.048*
C14	0.95452 (14)	0.88257 (9)	0.28216 (18)	0.0277 (3)
H14A	0.9036	0.9314	0.3203	0.033*
H14B	0.9669	0.8888	0.1750	0.033*
H14C	1.0430	0.8801	0.3307	0.033*
C15	0.31656 (14)	0.78297 (11)	0.20471 (19)	0.0336 (3)
H15A	0.274 (2)	0.7267 (13)	0.214 (2)	0.040 (5)*
H15B	0.263 (2)	0.8316 (14)	0.223 (2)	0.046 (6)*
H1O	0.688 (3)	0.7229 (15)	0.460 (3)	0.062 (7)*
H2O	0.918 (2)	0.7915 (13)	0.518 (2)	0.045 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0174 (4)	0.0239 (4)	0.0159 (4)	0.0001 (4)	0.0043 (3)	0.0028 (4)
O2	0.0207 (4)	0.0292 (4)	0.0160 (4)	0.0001 (4)	−0.0021 (3)	−0.0034 (4)
C1	0.0172 (5)	0.0171 (5)	0.0165 (5)	−0.0021 (4)	0.0004 (4)	0.0021 (4)
C2	0.0238 (6)	0.0174 (5)	0.0301 (7)	0.0009 (5)	−0.0020 (5)	0.0013 (5)
C3	0.0242 (6)	0.0253 (6)	0.0370 (7)	0.0027 (5)	−0.0041 (6)	0.0092 (6)
C4	0.0201 (6)	0.0293 (6)	0.0220 (6)	0.0001 (5)	−0.0051 (5)	0.0077 (5)
C5	0.0181 (5)	0.0253 (6)	0.0231 (6)	−0.0038 (5)	−0.0047 (5)	0.0024 (5)
C6	0.0152 (5)	0.0173 (5)	0.0144 (5)	−0.0020 (4)	0.0009 (4)	0.0021 (4)
C7	0.0187 (5)	0.0174 (5)	0.0186 (5)	−0.0020 (4)	0.0032 (5)	−0.0007 (4)
C8	0.0194 (5)	0.0208 (5)	0.0232 (6)	0.0036 (5)	0.0015 (5)	−0.0012 (5)
C9	0.0144 (5)	0.0267 (6)	0.0228 (6)	−0.0008 (4)	0.0013 (4)	−0.0032 (5)
C10	0.0173 (5)	0.0214 (5)	0.0173 (5)	−0.0026 (5)	0.0003 (4)	−0.0013 (5)
C11	0.0251 (6)	0.0180 (5)	0.0322 (7)	−0.0036 (5)	0.0052 (6)	−0.0010 (5)
C12	0.0644 (11)	0.0240 (6)	0.0417 (8)	−0.0068 (7)	0.0146 (9)	0.0073 (7)
C13	0.0463 (9)	0.0239 (7)	0.0491 (9)	−0.0067 (6)	0.0125 (8)	−0.0134 (7)
C14	0.0237 (6)	0.0261 (6)	0.0332 (7)	−0.0090 (5)	−0.0006 (6)	−0.0006 (6)
C15	0.0211 (6)	0.0432 (8)	0.0367 (8)	0.0028 (6)	−0.0012 (6)	0.0089 (7)

Geometric parameters (Å, °)

O1—C6	1.4462 (13)	C7—H7	1.0000
O1—H1O	0.90 (3)	C8—C9	1.5193 (16)
O2—C10	1.4457 (14)	C8—H8A	0.9900
O2—H2O	0.86 (2)	C8—H8B	0.9900
C1—C2	1.5350 (17)	C9—C10	1.5290 (17)
C1—C10	1.5484 (16)	C9—H9A	0.9900
C1—C6	1.5568 (15)	C9—H9B	0.9900

C1—H1	1.0000	C10—C14	1.5284 (17)
C2—C3	1.5367 (19)	C11—C12	1.521 (2)
C2—H2A	0.9900	C11—C13	1.530 (2)
C2—H2B	0.9900	C11—H11	1.0000
C3—C4	1.4976 (19)	C12—H12A	0.9800
C3—H3A	0.9900	C12—H12B	0.9800
C3—H3B	0.9900	C12—H12C	0.9800
C4—C15	1.332 (2)	C13—H13A	0.9800
C4—C5	1.5044 (18)	C13—H13B	0.9800
C5—C6	1.5389 (16)	C13—H13C	0.9800
C5—H5A	0.9900	C14—H14A	0.9800
C5—H5B	0.9900	C14—H14B	0.9800
C6—C7	1.5493 (16)	C14—H14C	0.9800
C7—C8	1.5318 (17)	C15—H15A	0.98 (2)
C7—C11	1.5531 (16)	C15—H15B	0.94 (2)
C6—O1—H1O	105.3 (17)	C7—C8—H8A	109.4
C10—O2—H2O	107.7 (14)	C9—C8—H8B	109.4
C2—C1—C10	113.29 (10)	C7—C8—H8B	109.4
C2—C1—C6	110.89 (9)	H8A—C8—H8B	108.0
C10—C1—C6	112.60 (9)	C8—C9—C10	112.45 (9)
C2—C1—H1	106.5	C8—C9—H9A	109.1
C10—C1—H1	106.5	C10—C9—H9A	109.1
C6—C1—H1	106.5	C8—C9—H9B	109.1
C1—C2—C3	111.16 (11)	C10—C9—H9B	109.1
C1—C2—H2A	109.4	H9A—C9—H9B	107.8
C3—C2—H2A	109.4	O2—C10—C14	109.30 (11)
C1—C2—H2B	109.4	O2—C10—C9	109.37 (10)
C3—C2—H2B	109.4	C14—C10—C9	110.01 (10)
H2A—C2—H2B	108.0	O2—C10—C1	106.14 (9)
C4—C3—C2	110.96 (10)	C14—C10—C1	111.99 (10)
C4—C3—H3A	109.4	C9—C10—C1	109.94 (10)
C2—C3—H3A	109.4	C12—C11—C13	109.84 (12)
C4—C3—H3B	109.4	C12—C11—C7	114.92 (12)
C2—C3—H3B	109.4	C13—C11—C7	109.61 (11)
H3A—C3—H3B	108.0	C12—C11—H11	107.4
C15—C4—C3	123.18 (14)	C13—C11—H11	107.4
C15—C4—C5	121.77 (13)	C7—C11—H11	107.4
C3—C4—C5	115.05 (11)	C11—C12—H12A	109.5
C4—C5—C6	113.62 (10)	C11—C12—H12B	109.5
C4—C5—H5A	108.8	H12A—C12—H12B	109.5
C6—C5—H5A	108.8	C11—C12—H12C	109.5
C4—C5—H5B	108.8	H12A—C12—H12C	109.5
C6—C5—H5B	108.8	H12B—C12—H12C	109.5
H5A—C5—H5B	107.7	C11—C13—H13A	109.5
O1—C6—C5	105.78 (9)	C11—C13—H13B	109.5
O1—C6—C7	111.20 (9)	H13A—C13—H13B	109.5
C5—C6—C7	110.57 (9)	C11—C13—H13C	109.5
O1—C6—C1	110.04 (9)	H13A—C13—H13C	109.5
C5—C6—C1	109.09 (9)	H13B—C13—H13C	109.5

C7—C6—C1	110.07 (9)	C10—C14—H14A	109.5
C8—C7—C6	110.54 (9)	C10—C14—H14B	109.5
C8—C7—C11	112.21 (10)	H14A—C14—H14B	109.5
C6—C7—C11	115.85 (9)	C10—C14—H14C	109.5
C8—C7—H7	105.8	H14A—C14—H14C	109.5
C6—C7—H7	105.8	H14B—C14—H14C	109.5
C11—C7—H7	105.8	C4—C15—H15A	119.8 (12)
C9—C8—C7	111.05 (10)	C4—C15—H15B	121.4 (14)
C9—C8—H8A	109.4	H15A—C15—H15B	118.7 (18)
C10—C1—C2—C3	173.71 (10)	O1—C6—C7—C11	62.51 (13)
C6—C1—C2—C3	−58.55 (13)	C5—C6—C7—C11	−54.69 (13)
C1—C2—C3—C4	54.42 (15)	C1—C6—C7—C11	−175.28 (11)
C2—C3—C4—C15	129.47 (15)	C6—C7—C8—C9	−57.77 (13)
C2—C3—C4—C5	−50.57 (16)	C11—C7—C8—C9	171.24 (10)
C15—C4—C5—C6	−129.50 (14)	C7—C8—C9—C10	58.00 (13)
C3—C4—C5—C6	50.54 (15)	C8—C9—C10—O2	61.20 (13)
C4—C5—C6—O1	66.73 (12)	C8—C9—C10—C14	−178.75 (12)
C4—C5—C6—C7	−172.78 (10)	C8—C9—C10—C1	−54.97 (13)
C4—C5—C6—C1	−51.61 (13)	C2—C1—C10—O2	62.15 (13)
C2—C1—C6—O1	−59.63 (12)	C6—C1—C10—O2	−64.69 (12)
C10—C1—C6—O1	68.48 (12)	C2—C1—C10—C14	−57.05 (14)
C2—C1—C6—C5	56.00 (12)	C6—C1—C10—C14	176.11 (10)
C10—C1—C6—C5	−175.89 (9)	C2—C1—C10—C9	−179.67 (10)
C2—C1—C6—C7	177.48 (10)	C6—C1—C10—C9	53.49 (12)
C10—C1—C6—C7	−54.41 (12)	C8—C7—C11—C12	43.20 (16)
O1—C6—C7—C8	−66.54 (12)	C6—C7—C11—C12	−85.04 (15)
C5—C6—C7—C8	176.26 (9)	C8—C7—C11—C13	−81.05 (14)
C1—C6—C7—C8	55.66 (12)	C6—C7—C11—C13	150.71 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O2	0.90 (3)	1.94 (3)	2.7240 (13)	146 (2)
O2—H2O···O1^{i}	0.86 (2)	2.10 (2)	2.9452 (13)	167 (2)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1.

Fig. 1

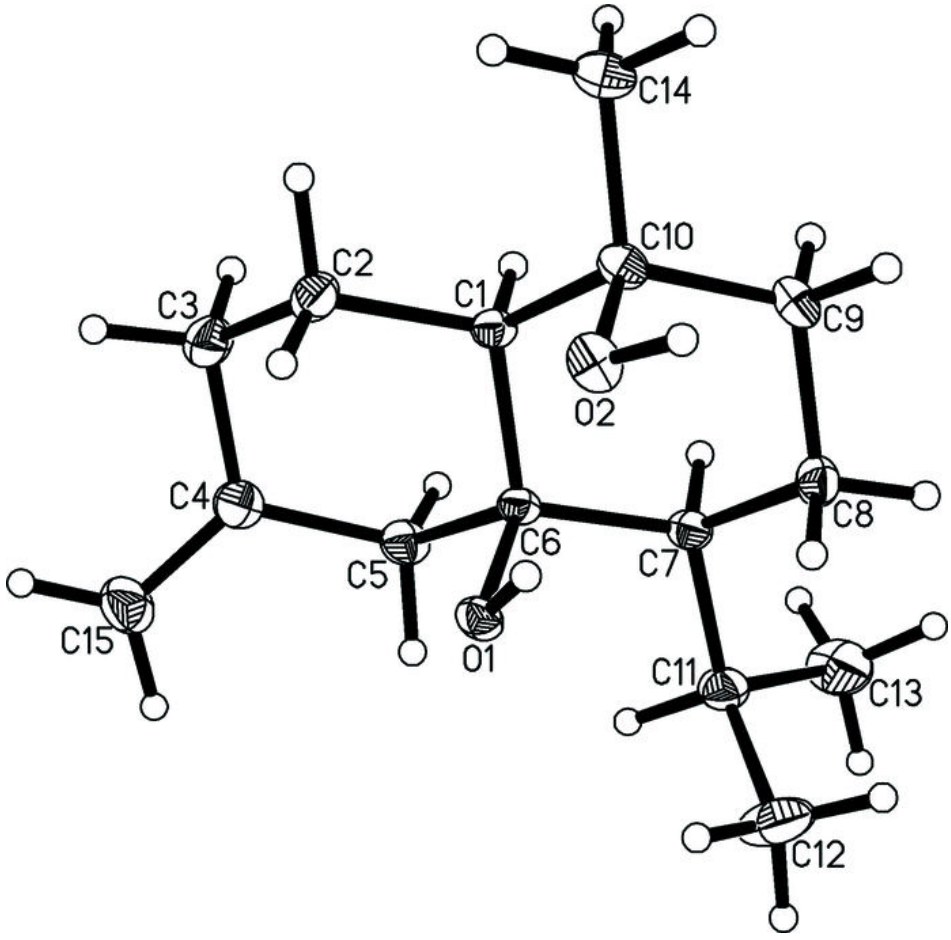


Fig. 2