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{3-Hydroxymethyl-1-[2-(3-methoxyphenyl)ethyl]-7-oxabicyclo[2.2.1]hept-5-en-2-yl}methanol

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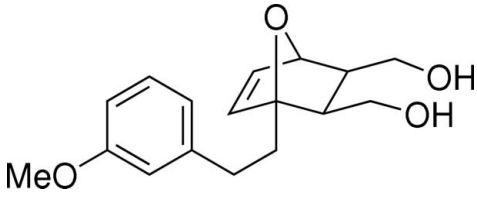
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.083; data-to-parameter ratio = 9.9.

The title compound, $C_{17}H_{22}O_4$, is an oxabicyclo[2.2.1]hept-5-ene with two *exo*-oriented hydroxymethyl groups which are not parallel to each other. The molecules are linked to each other by hydrogen bonds, resulting in a supramolecular network. Intermolecular O—H···O hydrogen bonding is observed between the hydroxyl groups.

Related literature

For related literature, see: Balaban *et al.* (2004); Chiu & Lautens (1997); Hudlicky *et al.* (1996); Wei (2004); Woodward (1940); Buser & Vasella (2005).



Experimental

Crystal data

$C_{17}H_{22}O_4$
M_r = 290.35
Orthorhombic, $Pca2_1$
a = 11.8025 (4) Å
b = 14.9842 (4) Å
c = 8.7569 (3) Å
V = 1548.67 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.43 × 0.27 × 0.24 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: none
9219 measured reflections
1915 independent reflections
1719 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.084
S = 1.08
1915 reflections
193 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2^{i}	0.82	1.93	2.7409 (19)	173

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2310).

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  doi:10.1107/S1600536807050234

supplementary materials

Acta Cryst. (2007). E**63**, o4352 [**doi:10.1107/S1600536807050234**]

{3-Hydroxymethyl-1-[2-(3-methoxyphenyl)ethyl]-7-oxabicyclo[2.2.1]hept-5-en-2-yl}methanol

Y.-W. Wang and Y. Peng

Comment

Furans are generally much less reactive dienes in Diels-Alder cycloaddition reaction due to their inherent aromaticity (Balaban *et al.*, 2004) and usually facile retro- Diels-Alder reaction of the resulting adducts (Woodward, 1940). In view of the importance and great potential of Diels-Alder cycloadducts of furano derivatives as key intermediates for synthesizing structurally complex targets (Hudlicky *et al.*, 1996) or achieving highly region-stereocontrolled reactions *via* the corresponding oxabicyclic adducts (Chiu & Lautens, 1997), a great deal of effort has been devoted to the development of chemical and physical means to promote the frequently difficult [4 + 2] cycloaddition of cyclic furano dienes. For example, Ultrasonic irradiation effectively promoted the Diels-Alder reaction of substituted furans with dimethyl maleate in very high *exo* selectivity (Wei, 2004) and the resulting product stereochemistry was unambiguously confirmed a single-crystal X-ray diffraction analysis (Fig. 1) of the corresponding reduction derivative diol (I). The oxidol bridge is on the same side as two hydroxymethyls, which are not parallel each other.

The molecules are linked by O···O hydrogen bonds to form a two-dimensional supramolecular network structure (Fig. 2). Inter-molecular O—H···O hydrogen bonding is observed between the O(3) and O(2) hydroxyl groups.

Experimental

The title compound was prepared according to literature method (Wei, 2004). Single crystals suitable for X-ray determination were obtained by slow evaporation of a AcOEt solution over a period of several days. IR (film): 3361, 2934, 2837, 1730, 1601, 1587, 1490, 1257, 1155, 1038 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$) delta 2.14–2.47 (m, 2H), 2.67–2.90 (m, 2H), 3.25–3.40 (m, 2H), 3.61–3.79 (m, 2H), 3.78 (m, 2H), 3.81 (s, 3H), 4.86 (d, J = 4.4 Hz, 1H), 6.15 (d, J = 5.8 Hz, 1H), 6.29 (dd, J = 3.8, 5.8 Hz, 1H), 6.75–6.85 (m, 3H), 7.19 (dd, J = 7.4, 7.6 Hz, 1H) p.p.m.; ^{13}C NMR (75 MHz, $CDCl_3$) delta 31.0, 33.8, 46.9, 48.5, 55.1, 60.7, 61.0, 79.2, 90.5, 111.2, 114.0, 120.6, 129.3, 135.2, 136.6, 143.5, 159.6 p.p.m.; LRMS (EI) m/*z* 290 (M^+, 0.04%), 272 (0.1), 254 (0.1), 202 (22), 121 (42), 81 (100).

Refinement

All H atoms were placed geometrically (C—H values were set to 0.98, 0.97, 0.96, 0.93 and 0.82 A° for atoms CH, CH_2, CH_3, CH (phenyl) and OH, respectively) and refined with a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C), or 1.5 U_{eq}(O). In the absence of significant anomalous dispersion effects, 1530 Freidel pairs were merged before refinement.

Figures

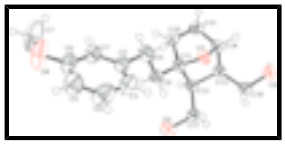

Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

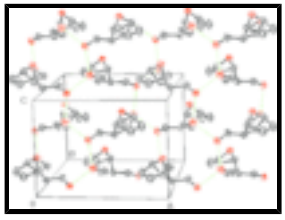

Fig. 2. The 2-D network supramolecular structure of (I). H atoms and 2-(3- methoxy-phenyl)-ethyl have been omitted for clarity. Dashed lines indicate hydrogen-bonding interactions.

{3-Hydroxymethyl-1-[2-(3-methoxyphenyl)ethyl]-7-oxabicyclo[2.2.1]hept-5-en-2- yl}methanol

Crystal data

$C_{17}H_{22}O_4$	F_{000} = 624
M_r = 290.35	D_x = 1.245 Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 3969 reflections
a = 11.8025 (4) Å	θ = 2.7–26.5º
b = 14.9842 (4) Å	μ = 0.09 mm^{-1}
c = 8.7569 (3) Å	T = 294 (2) K
V = 1548.67 (9) Å^3	Block, colorless
Z = 4	0.43 × 0.27 × 0.24 mm

Data collection

Bruker APEX CCD area-detector diffractometer	1719 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.019
Monochromator: graphite	θ_{max} = 27.6º
T = 294(2) K	θ_{min} = 2.2º
φ and ω scans	h = −15→12
Absorption correction: none	k = −19→19
9219 measured reflections	l = −11→11
1915 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.1224P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.084$

$S = 1.08$

1915 reflections

193 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.15$ e Å^{-3}

$\Delta\rho_{min} = -0.16$ e Å^{-3}

Extinction correction: none

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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	1.09490 (11)	0.61849 (8)	0.76469 (15)	0.0394 (3)
C16	1.06391 (15)	0.44022 (12)	0.5973 (2)	0.0395 (4)
H16A	1.0072	0.4506	0.6755	0.047*
H16B	1.0262	0.4142	0.5095	0.047*
C2	0.93740 (19)	0.87373 (13)	0.8725 (3)	0.0521 (5)
C12	1.02937 (13)	0.60627 (11)	0.5159 (2)	0.0345 (4)
H12	1.0448	0.6306	0.4141	0.041*
C17	0.90595 (14)	0.57645 (12)	0.5238 (2)	0.0399 (4)
H17A	0.8970	0.5218	0.4658	0.048*
H17B	0.8865	0.5637	0.6292	0.048*
C9	0.97705 (16)	0.74553 (13)	0.6911 (3)	0.0469 (5)
H9A	0.9098	0.7149	0.7271	0.056*
H9B	0.9555	0.7822	0.6046	0.056*
C11	1.06362 (15)	0.67730 (11)	0.6400 (2)	0.0371 (4)
C15	1.25209 (18)	0.64819 (12)	0.6179 (3)	0.0510 (5)
H15	1.3298	0.6493	0.6004	0.061*
C14	1.18359 (14)	0.57231 (12)	0.6848 (2)	0.0396 (4)
H14	1.2271	0.5301	0.7471	0.047*
C1	0.9513 (2)	0.96320 (13)	0.8366 (3)	0.0540 (5)
H1	1.0123	0.9811	0.7767	0.065*
C10	1.17892 (15)	0.71219 (13)	0.5895 (3)	0.0482 (5)
H10	1.1945	0.7678	0.5471	0.058*
C13	1.11664 (13)	0.52951 (11)	0.5509 (2)	0.0347 (4)
H13	1.1669	0.5209	0.4631	0.042*
O4	0.8824 (2)	1.11539 (11)	0.8606 (3)	0.1037 (8)
C8	1.0225 (2)	0.80543 (15)	0.8182 (4)	0.0675 (7)
H8A	1.0449	0.7685	0.9039	0.081*
H8B	1.0895	0.8362	0.7815	0.081*

C6	0.8749 (2)	1.02585 (14)	0.8895 (3)	0.0633 (6)
C5	0.7847 (2)	1.00053 (18)	0.9786 (4)	0.0775 (8)
H5	0.7338	1.0429	1.0148	0.093*
C3	0.8458 (2)	0.84890 (16)	0.9595 (4)	0.0721 (7)
H3	0.8343	0.7890	0.9824	0.087*
C4	0.7704 (3)	0.91245 (18)	1.0134 (4)	0.0867 (9)
H4	0.7096	0.8949	1.0738	0.104*
C7	0.9686 (4)	1.1454 (2)	0.7638 (5)	0.1168 (15)
H7A	1.0408	1.1281	0.8046	0.175*
H7B	0.9652	1.2092	0.7559	0.175*
H7C	0.9590	1.1195	0.6644	0.175*
O3	1.14629 (12)	0.37876 (8)	0.65379 (16)	0.0444 (3)
H3A	1.1970	0.3728	0.5906	0.067*
O2	0.82975 (11)	0.64249 (10)	0.46535 (16)	0.0453 (3)
H2	0.8367	0.6457	0.3723	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0426 (6)	0.0421 (6)	0.0334 (6)	0.0039 (5)	−0.0046 (5)	−0.0004 (5)
C16	0.0389 (9)	0.0394 (8)	0.0401 (10)	0.0001 (7)	0.0008 (8)	−0.0001 (8)
C2	0.0563 (11)	0.0425 (10)	0.0576 (13)	0.0038 (9)	−0.0086 (10)	−0.0140 (9)
C12	0.0327 (8)	0.0400 (8)	0.0309 (8)	0.0024 (7)	−0.0001 (7)	0.0031 (7)
C17	0.0344 (8)	0.0467 (10)	0.0387 (9)	0.0008 (7)	−0.0029 (8)	0.0012 (8)
C9	0.0439 (10)	0.0438 (9)	0.0529 (12)	0.0080 (8)	−0.0075 (9)	−0.0081 (9)
C11	0.0369 (8)	0.0365 (8)	0.0380 (10)	0.0006 (7)	−0.0036 (7)	0.0011 (7)
C15	0.0333 (8)	0.0505 (9)	0.0692 (15)	−0.0072 (9)	−0.0008 (10)	0.0006 (10)
C14	0.0320 (8)	0.0411 (9)	0.0456 (11)	0.0032 (7)	−0.0063 (8)	0.0016 (8)
C1	0.0625 (12)	0.0495 (10)	0.0498 (12)	−0.0005 (9)	0.0107 (10)	−0.0126 (10)
C10	0.0426 (10)	0.0398 (9)	0.0621 (13)	−0.0073 (8)	−0.0022 (10)	0.0062 (10)
C13	0.0316 (7)	0.0397 (8)	0.0328 (9)	0.0014 (7)	0.0041 (7)	0.0017 (7)
O4	0.156 (2)	0.0442 (9)	0.1105 (19)	0.0152 (11)	0.0508 (17)	−0.0004 (11)
C8	0.0644 (14)	0.0529 (12)	0.0852 (18)	0.0172 (11)	−0.0225 (14)	−0.0283 (13)
C6	0.0859 (16)	0.0431 (10)	0.0610 (14)	0.0087 (11)	0.0140 (14)	−0.0082 (10)
C5	0.0764 (16)	0.0672 (14)	0.089 (2)	0.0094 (12)	0.0282 (16)	−0.0190 (15)
C3	0.0869 (18)	0.0480 (12)	0.0814 (19)	−0.0108 (12)	0.0080 (16)	−0.0010 (13)
C4	0.0826 (18)	0.0800 (16)	0.097 (2)	−0.0172 (15)	0.0362 (19)	−0.0135 (18)
C7	0.196 (4)	0.0599 (16)	0.094 (3)	−0.013 (2)	0.037 (3)	0.0105 (18)
O3	0.0483 (7)	0.0418 (7)	0.0431 (7)	0.0082 (6)	0.0041 (6)	0.0046 (6)
O2	0.0367 (6)	0.0586 (8)	0.0406 (7)	0.0091 (6)	−0.0030 (6)	0.0016 (7)

Geometric parameters (Å, °)

O1—C14	1.437 (2)	C15—H15	0.9300
O1—C11	1.451 (2)	C14—C13	1.552 (3)
C16—O3	1.428 (2)	C14—H14	0.9800
C16—C13	1.530 (2)	C1—C6	1.382 (3)
C16—H16A	0.9700	C1—H1	0.9300
C16—H16B	0.9700	C10—H10	0.9300

C2—C3	1.373 (3)	C13—H13	0.9800
C2—C1	1.387 (3)	O4—C6	1.368 (3)
C2—C8	1.511 (3)	O4—C7	1.399 (4)
C12—C17	1.525 (2)	C8—H8A	0.9700
C12—C11	1.574 (2)	C8—H8B	0.9700
C12—C13	1.574 (2)	C6—C5	1.373 (4)
C12—H12	0.9800	C5—C4	1.365 (4)
C17—O2	1.432 (2)	C5—H5	0.9300
C17—H17A	0.9700	C3—C4	1.386 (4)
C17—H17B	0.9700	C3—H3	0.9300
C9—C11	1.513 (2)	C4—H4	0.9300
C9—C8	1.527 (3)	C7—H7A	0.9600
C9—H9A	0.9700	C7—H7B	0.9600
C9—H9B	0.9700	C7—H7C	0.9600
C11—C10	1.523 (3)	O3—H3A	0.8200
C15—C10	1.314 (3)	O2—H2	0.8200
C15—C14	1.513 (3)		
C14—O1—C11	96.39 (13)	C15—C14—H14	114.9
O3—C16—C13	112.27 (14)	C13—C14—H14	114.9
O3—C16—H16A	109.2	C6—C1—C2	120.2 (2)
C13—C16—H16A	109.2	C6—C1—H1	119.9
O3—C16—H16B	109.2	C2—C1—H1	119.9
C13—C16—H16B	109.2	C15—C10—C11	106.37 (17)
H16A—C16—H16B	107.9	C15—C10—H10	126.8
C3—C2—C1	118.8 (2)	C11—C10—H10	126.8
C3—C2—C8	120.9 (2)	C16—C13—C14	111.59 (15)
C1—C2—C8	120.3 (2)	C16—C13—C12	115.11 (13)
C17—C12—C11	114.31 (15)	C14—C13—C12	100.28 (13)
C17—C12—C13	113.70 (14)	C16—C13—H13	109.8
C11—C12—C13	101.03 (13)	C14—C13—H13	109.8
C17—C12—H12	109.2	C12—C13—H13	109.8
C11—C12—H12	109.2	C6—O4—C7	118.3 (2)
C13—C12—H12	109.2	C2—C8—C9	113.20 (18)
O2—C17—C12	112.39 (15)	C2—C8—H8A	108.9
O2—C17—H17A	109.1	C9—C8—H8A	108.9
C12—C17—H17A	109.1	C2—C8—H8B	108.9
O2—C17—H17B	109.1	C9—C8—H8B	108.9
C12—C17—H17B	109.1	H8A—C8—H8B	107.8
H17A—C17—H17B	107.9	O4—C6—C5	115.2 (2)
C11—C9—C8	112.05 (16)	O4—C6—C1	124.2 (2)
C11—C9—H9A	109.2	C5—C6—C1	120.6 (2)
C8—C9—H9A	109.2	C4—C5—C6	119.3 (2)
C11—C9—H9B	109.2	C4—C5—H5	120.4
C8—C9—H9B	109.2	C6—C5—H5	120.4
H9A—C9—H9B	107.9	C2—C3—C4	120.5 (2)
O1—C11—C9	111.07 (15)	C2—C3—H3	119.7
O1—C11—C10	101.51 (14)	C4—C3—H3	119.7
C9—C11—C10	117.20 (15)	C5—C4—C3	120.6 (3)
O1—C11—C12	100.04 (12)	C5—C4—H4	119.7

C9—C11—C12	119.19 (15)	C3—C4—H4	119.7
C10—C11—C12	105.14 (15)	O4—C7—H7A	109.5
C10—C15—C14	105.69 (18)	O4—C7—H7B	109.5
C10—C15—H15	127.2	H7A—C7—H7B	109.5
C14—C15—H15	127.2	O4—C7—H7C	109.5
O1—C14—C15	102.48 (14)	H7A—C7—H7C	109.5
O1—C14—C13	101.29 (13)	H7B—C7—H7C	109.5
C15—C14—C13	106.85 (17)	C16—O3—H3A	109.5
O1—C14—H14	114.9	C17—O2—H2	109.5
C11—C12—C17—O2	75.2 (2)	O3—C16—C13—C14	−56.0 (2)
C13—C12—C17—O2	−169.53 (15)	O3—C16—C13—C12	−169.41 (15)
C14—O1—C11—C9	−173.54 (15)	O1—C14—C13—C16	−85.19 (15)
C14—O1—C11—C10	−48.20 (15)	C15—C14—C13—C16	167.91 (14)
C14—O1—C11—C12	59.66 (14)	O1—C14—C13—C12	37.20 (15)
C8—C9—C11—O1	61.3 (2)	C15—C14—C13—C12	−69.70 (16)
C8—C9—C11—C10	−54.7 (3)	C17—C12—C13—C16	−3.9 (2)
C8—C9—C11—C12	176.68 (19)	C11—C12—C13—C16	119.04 (16)
C17—C12—C11—O1	87.36 (16)	C17—C12—C13—C14	−123.77 (16)
C13—C12—C11—O1	−35.14 (15)	C11—C12—C13—C14	−0.84 (16)
C17—C12—C11—C9	−33.8 (2)	C3—C2—C8—C9	72.7 (3)
C13—C12—C11—C9	−156.28 (16)	C1—C2—C8—C9	−108.0 (3)
C17—C12—C11—C10	−167.70 (15)	C11—C9—C8—C2	−179.5 (2)
C13—C12—C11—C10	69.80 (16)	C7—O4—C6—C5	−176.5 (3)
C11—O1—C14—C15	49.05 (16)	C7—O4—C6—C1	4.1 (5)
C11—O1—C14—C13	−61.25 (13)	C2—C1—C6—O4	179.6 (3)
C10—C15—C14—O1	−32.1 (2)	C2—C1—C6—C5	0.3 (4)
C10—C15—C14—C13	74.0 (2)	O4—C6—C5—C4	−180.0 (3)
C3—C2—C1—C6	0.8 (4)	C1—C6—C5—C4	−0.6 (5)
C8—C2—C1—C6	−178.4 (2)	C1—C2—C3—C4	−1.6 (4)
C14—C15—C10—C11	0.5 (2)	C8—C2—C3—C4	177.7 (3)
O1—C11—C10—C15	30.7 (2)	C6—C5—C4—C3	−0.1 (5)
C9—C11—C10—C15	151.8 (2)	C2—C3—C4—C5	1.2 (5)
C12—C11—C10—C15	−73.2 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O2^{i}	0.82	1.93	2.7409 (19)	173

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

Fig. 1

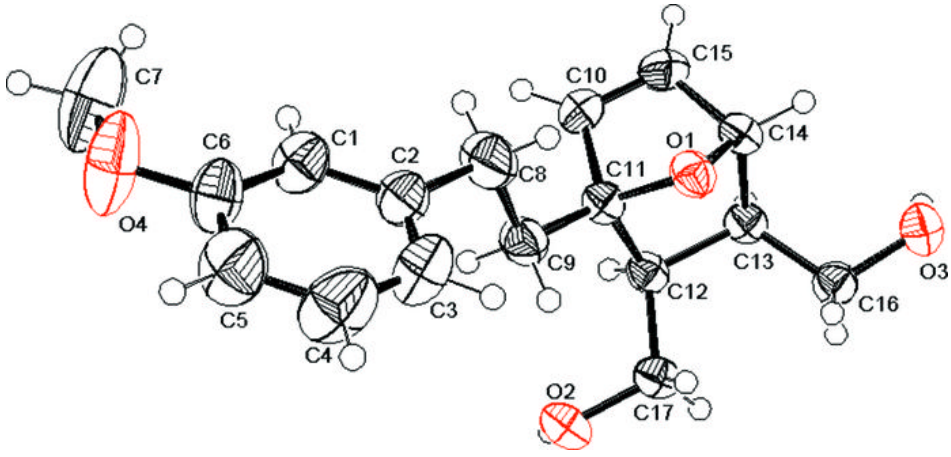
C7
C6
C1
C2
C8
C10
C15
C14
O1
C11
O4
C9
C13
O3
C12
C16
C5
C3
C4
O2
C17

Fig. 2