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(3*E*)-5-Hydroxy-3-[5-(hydroxymethyl)-furan-2-ylmethylene]-1-benzofuran-2(3*H*)-one

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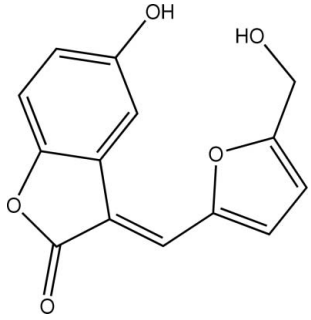
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.164; data-to-parameter ratio = 12.8.

The title compound, $C_{14}H_{10}O_5$, was isolated from *Senecio cannabifolius* Less var. *integrifolius* aqueous extract. The structure was elucidated on the basis of spectroscopic data, including MS, ^{1}H NMR and ^{13}C NMR, and the relative configuration was confirmed by X-ray crystallographic analysis. The benzofuran ring is almost coplanar with the furan ring [dihedral angle = 10.47 (15)°]. A chain is formed through supramolecular $R_2^2(10)$ synthons and three-centre hydrogen bonds.

Related literature

The title compound was first isolated by Wu *et al.* (2002).



Experimental

Crystal data

$C_{14}H_{10}O_5$
M_r = 258.22
Triclinic, $P\overline{1}$
a = 7.544 (3) Å
b = 8.974 (2) Å
c = 9.291 (3) Å
α = 107.509 (7)°
β = 97.844 (6)°
γ = 100.735 (7)°
V = 576.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.48 × 0.24 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.968, T_{max} = 0.984
3184 measured reflections
2273 independent reflections
1496 reflections with $I > 2\sigma(I)$
R_{int} = 0.138

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.164
S = 0.97
2273 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O3^{i}	0.82	1.90	2.682 (3)	160
O3—H3···O3^{i}	0.82	1.96	2.717 (3)	154
C9—H9···O1^{ii}	0.93	2.56	3.414 (3)	153

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $-x+2, -y+2, -z+1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; 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: RK2041).

References

Bruker (2000). *SADABS*, *SAINT*, *SHELXTL* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wu, B., Wu, L.-J. & Wang, L.-Q. (2002). *J. Asian Nat. Prod. Res.* **4**, 315–317.

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supplementary materials

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(3*E*)-5-Hydroxy-3-[5-(hydroxymethyl)furan-2-ylmethylene]-1-benzofuran-2(3*H*)-one

H.-Y. Ma, J. Shi, C.-H. Wang and Z.-T. Wang

Comment

The title compound, (*E*)-5-hydroxy-3-((5-(hydroxymethyl)furan-2-yl)methylene) benzofuran-2(3*H*)-one, cannabifolactone was first obtained from *Senecio cannabifolius* by Wu *et al.* (2002). Its structure was identified by NMR spectroscopy. But the (*Z*) or (*E*) configuration of it was not determined in the literature. We have now isolated the compound from *Senecio cannabifolius* Less var *integrifolius*. Here, the molecular structure of (I) is reported (Fig. 1).

The molecule contains a benzyl ring, a fused five-membered lactone ring and a furan ring connected by a congjugated double bonds. Three above rings are almost planar.

On Fig. 2, two molecules at the invert position are linked *via* supramolecular synthons $R_2^2(10)$ including paired hydrogen bonds C9—H9···O1^{ii} and C9^{ii}—H9^{ii}···O1 (symmetry code ii: 2 − *x*, 2 − *y*, 1 − *z*). The dimer interacted with the other one through tricentered hydrogen bonds including O3—H3···O3^{i} and O5—H5A···O3^{i} (symmetry code i: *x*, 2 − *y*, *z*). A chain was formed through altering of the supramolecular synthons $R_2^2(10)$ and tricentered hydrogen bonds.

Experimental

Air-dried aerial parts (35 kg) of *Senecio cannabifolius* Less var *integrifolius* were powdered and extracted three times with H_2O. The extract was concentrated and acidified with 36% HCl to pH 3 and extracted with *Et*O*Ac* three times. The *Et*O*Ac* fraction was chromatographed on silica gel column, using a gradient mixture of $CHCl_3$–CH_3OH as eluant. The $CHCl_3$–CH_3OH (100:3 *v*/*v*) fraction, subjected to repeated chromatography over silica gel and Sephadex LH–20 columns, gave compound (I) (yield 0.000057%, m.p. 497–499 K).

Yellow–orange needles crystals suitable for *X*–ray studies were grown from ethylacetate–methanol by slow evaporation at room temperature. ESI-MS: m/*z* 259 [*M*+H]+; ^{1}H NMR (*DMSO*, 500 MHz, p.p.m.): 4.70(2*H*, s, H-15), 5.70(1*H*, s, 15-OH), 6.65(1*H*, d, *J* = 3.4 Hz, H-13), 6.79(1*H*, dd, *J* = 2.6, 8.6 Hz, H-7), 7.04 (1*H*, d, *J* = 8.6 Hz, H-8), 7.31 (1*H*, d, *J* = 3.4 Hz, H-12), 7.41 (1*H*, s, H-10), 7.80 (1*H*, d, *J* = 2.6 Hz, H-5), 9.50(1*H*, s, 6-OH). ^{13}C NMR ($CDCl_3$, 125 MHz, p.p.m.): 57.8 (C-15), 111.8 (C-8), 112.3 (C-13), 112.6 (C-5), 118.2 (C-3), 118.3 (C-7), 124.0 (C-4), 124.0 (C-10), 124.3 (C-12), 148.9 (C-9), 152.1 (C-11), 155.0 (C-6), 161.7 (C-14), 172.3 (C-2).

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å, C—H = 0.98 Å (methine H), 0.97 Å (methylene H), 0.96 Å (methyl H) and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where *x* = 1.2 for methine and methylene H and *x* = 1.5 for all other H.

Figures

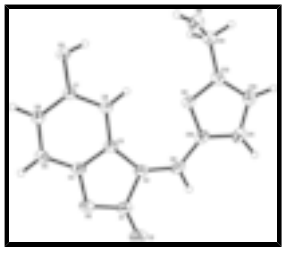

Fig. 1. A drawing of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a spheres with arbitrary radius.

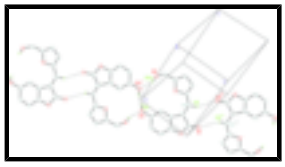

Fig. 2. A packing diagram of (I) showing the formation of a chain *via* hydrogen bonds.

(3E)-5-Hydroxy-3-[5-(hydroxymethyl)furan-2-ylmethylene]-1-benzofuran-2(3*H*)-one

Crystal data

$C_{14}H_{10}O_5$	$Z = 2$
$M_r = 258.22$	$F_{000} = 268$
Triclinic, $P\overline{1}$	$D_x = 1.487$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.544$ (3) Å	Cell parameters from 993 reflections
$b = 8.974$ (2) Å	$\theta = 4.7$–48.1°
$c = 9.291$ (3) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 107.509$ (7)°	$T = 293$ (2) K
$\beta = 97.844$ (6)°	Needle, yellow
$\gamma = 100.735$ (7)°	0.48 × 0.24 × 0.15 mm
$V = 576.8$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2273 independent reflections
Radiation source: fine–focus sealed tube	1496 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.138$
$T = 293(2)$ K	$\theta_{max} = 26.0^\circ$
φ and ω scans	$\theta_{min} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -9 \rightarrow 9$
$T_{min} = 0.968$, $T_{max} = 0.984$	$k = -11 \rightarrow 6$
3184 measured reflections	$l = -10 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.064$	H-atom parameters constrained
$wR(F^2) = 0.164$	$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} = 0.040$
2235 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
174 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

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 $F^2 > 2\sigma(F^2)$ 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	1.0229 (2)	1.1646 (3)	0.3963 (2)	0.0690 (7)
O2	0.8629 (2)	1.2740 (2)	0.2522 (2)	0.0562 (5)
O3	0.1254 (2)	1.1304 (2)	0.0008 (2)	0.0559 (5)
H3	0.0779	1.0385	−0.0045	0.084*
O4	0.3470 (2)	0.82265 (19)	0.29896 (18)	0.0419 (5)
O5	−0.0173 (3)	0.6380 (2)	0.0900 (2)	0.0612 (6)
H5A	−0.0237	0.7203	0.0705	0.092*
C1	0.8761 (3)	1.1658 (3)	0.3283 (3)	0.0484 (7)
C2	0.6891 (3)	1.0621 (3)	0.3073 (3)	0.0400 (6)
C3	0.5654 (3)	1.1226 (3)	0.2156 (3)	0.0374 (6)
C4	0.6769 (3)	1.2464 (3)	0.1850 (3)	0.0433 (6)
C5	0.6139 (3)	1.3299 (3)	0.0957 (3)	0.0520 (7)
H5	0.6933	1.4105	0.0758	0.062*
C6	0.4262 (3)	1.2883 (3)	0.0363 (3)	0.0475 (7)
H6	0.3774	1.3424	−0.0245	0.057*
C7	0.3106 (3)	1.1680 (3)	0.0659 (3)	0.0397 (6)
C8	0.3757 (3)	1.0833 (3)	0.1552 (3)	0.0390 (6)
H8	0.2959	1.0027	0.1746	0.047*

C9	0.6754 (3)	0.9416 (3)	0.3666 (3)	0.0431 (6)
H9	0.7862	0.9379	0.4215	0.052*
C10	0.5221 (3)	0.8192 (3)	0.3603 (3)	0.0428 (6)
C11	0.5126 (4)	0.6904 (4)	0.4072 (3)	0.0577 (8)
H11	0.6112	0.6619	0.4551	0.069*
C12	0.3258 (4)	0.6066 (3)	0.3705 (3)	0.0588 (8)
H12	0.2783	0.5112	0.3870	0.071*
C13	0.2293 (3)	0.6913 (3)	0.3069 (3)	0.0436 (6)
C14	0.0307 (3)	0.6752 (3)	0.2518 (3)	0.0524 (7)
H14A	−0.0409	0.5911	0.2804	0.063*
H14B	−0.0009	0.7752	0.3026	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0256 (10)	0.0971 (18)	0.0869 (15)	0.0081 (10)	−0.0017 (9)	0.0446 (13)
O2	0.0245 (9)	0.0618 (13)	0.0823 (13)	0.0000 (8)	0.0010 (8)	0.0348 (11)
O3	0.0269 (9)	0.0512 (12)	0.0931 (14)	0.0015 (8)	−0.0062 (8)	0.0419 (11)
O4	0.0319 (9)	0.0408 (10)	0.0556 (11)	0.0072 (7)	0.0019 (7)	0.0236 (8)
O5	0.0495 (11)	0.0631 (13)	0.0757 (14)	0.0095 (10)	−0.0044 (9)	0.0401 (11)
C1	0.0290 (13)	0.0597 (18)	0.0572 (16)	0.0077 (11)	0.0028 (11)	0.0249 (14)
C2	0.0252 (12)	0.0488 (15)	0.0460 (14)	0.0083 (10)	0.0035 (10)	0.0182 (11)
C3	0.0280 (11)	0.0384 (13)	0.0449 (13)	0.0071 (10)	0.0053 (9)	0.0140 (11)
C4	0.0259 (12)	0.0415 (14)	0.0584 (16)	0.0022 (10)	0.0013 (10)	0.0176 (12)
C5	0.0340 (13)	0.0443 (16)	0.0788 (19)	−0.0013 (11)	0.0058 (12)	0.0309 (14)
C6	0.0357 (13)	0.0399 (15)	0.0717 (18)	0.0061 (11)	0.0037 (12)	0.0308 (13)
C7	0.0270 (12)	0.0360 (13)	0.0558 (15)	0.0041 (10)	0.0019 (10)	0.0199 (11)
C8	0.0265 (11)	0.0398 (14)	0.0530 (15)	0.0054 (10)	0.0045 (10)	0.0222 (11)
C9	0.0285 (12)	0.0535 (16)	0.0503 (15)	0.0149 (11)	0.0038 (10)	0.0207 (12)
C10	0.0373 (13)	0.0472 (15)	0.0478 (14)	0.0163 (11)	0.0021 (10)	0.0206 (12)
C11	0.0500 (16)	0.0614 (19)	0.0741 (19)	0.0226 (14)	0.0044 (14)	0.0386 (16)
C12	0.0574 (17)	0.0461 (16)	0.079 (2)	0.0089 (13)	0.0034 (14)	0.0360 (15)
C13	0.0414 (13)	0.0366 (14)	0.0520 (15)	0.0030 (11)	0.0061 (11)	0.0189 (11)
C14	0.0419 (14)	0.0464 (16)	0.0674 (18)	0.0021 (12)	0.0052 (12)	0.0245 (14)

Geometric parameters (Å, °)

O1—C1	1.201 (3)	C5—H5	0.9300
O2—C1	1.373 (3)	C6—C7	1.377 (3)
O2—C4	1.400 (3)	C6—H6	0.9300
O3—C7	1.380 (3)	C7—C8	1.383 (3)
O3—H3	0.8200	C8—H8	0.9300
O4—C13	1.365 (3)	C9—C10	1.420 (4)
O4—C10	1.375 (3)	C9—H9	0.9300
O5—C14	1.417 (3)	C10—C11	1.346 (4)
O5—H5A	0.8200	C11—C12	1.410 (4)
C1—C2	1.490 (3)	C11—H11	0.9300
C2—C9	1.348 (4)	C12—C13	1.348 (4)
C2—C3	1.464 (3)	C12—H12	0.9300

C3—C4	1.389 (3)	C13—C14	1.481 (3)
C3—C8	1.400 (3)	C14—H14A	0.9700
C4—C5	1.370 (3)	C14—H14B	0.9700
C5—C6	1.384 (3)		
C1—O2—C4	107.42 (19)	C7—C8—C3	118.1 (2)
C7—O3—H3	109.5	C7—C8—H8	121.0
C13—O4—C10	107.05 (18)	C3—C8—H8	121.0
C14—O5—H5A	109.5	C2—C9—C10	131.6 (2)
O1—C1—O2	120.6 (2)	C2—C9—H9	114.2
O1—C1—C2	130.4 (3)	C10—C9—H9	114.2
O2—C1—C2	109.03 (19)	C11—C10—O4	109.0 (2)
C9—C2—C3	137.1 (2)	C11—C10—C9	131.1 (2)
C9—C2—C1	117.8 (2)	O4—C10—C9	119.9 (2)
C3—C2—C1	105.0 (2)	C10—C11—C12	107.4 (2)
C4—C3—C8	118.2 (2)	C10—C11—H11	126.3
C4—C3—C2	105.99 (19)	C12—C11—H11	126.3
C8—C3—C2	135.8 (2)	C13—C12—C11	106.8 (2)
C5—C4—C3	124.1 (2)	C13—C12—H12	126.6
C5—C4—O2	123.3 (2)	C11—C12—H12	126.6
C3—C4—O2	112.5 (2)	C12—C13—O4	109.6 (2)
C4—C5—C6	116.7 (2)	C12—C13—C14	134.4 (2)
C4—C5—H5	121.7	O4—C13—C14	116.0 (2)
C6—C5—H5	121.7	O5—C14—C13	112.6 (2)
C7—C6—C5	120.9 (2)	O5—C14—H14A	109.1
C7—C6—H6	119.5	C13—C14—H14A	109.1
C5—C6—H6	119.5	O5—C14—H14B	109.1
C6—C7—O3	118.0 (2)	C13—C14—H14B	109.1
C6—C7—C8	122.0 (2)	H14A—C14—H14B	107.8
O3—C7—C8	120.0 (2)		
C4—O2—C1—O1	−179.9 (2)	C5—C6—C7—C8	−0.2 (4)
C4—O2—C1—C2	0.7 (3)	C6—C7—C8—C3	−0.3 (4)
O1—C1—C2—C9	−3.0 (4)	O3—C7—C8—C3	178.3 (2)
O2—C1—C2—C9	176.3 (2)	C4—C3—C8—C7	1.3 (3)
O1—C1—C2—C3	179.2 (3)	C2—C3—C8—C7	−177.2 (3)
O2—C1—C2—C3	−1.4 (3)	C3—C2—C9—C10	0.2 (5)
C9—C2—C3—C4	−175.4 (3)	C1—C2—C9—C10	−176.6 (2)
C1—C2—C3—C4	1.6 (3)	C13—O4—C10—C11	−0.9 (3)
C9—C2—C3—C8	3.2 (5)	C13—O4—C10—C9	179.5 (2)
C1—C2—C3—C8	−179.8 (3)	C2—C9—C10—C11	172.7 (3)
C8—C3—C4—C5	−2.0 (4)	C2—C9—C10—O4	−7.8 (4)
C2—C3—C4—C5	176.9 (3)	O4—C10—C11—C12	1.7 (3)
C8—C3—C4—O2	179.79 (19)	C9—C10—C11—C12	−178.9 (3)
C2—C3—C4—O2	−1.3 (3)	C10—C11—C12—C13	−1.8 (3)
C1—O2—C4—C5	−177.9 (3)	C11—C12—C13—O4	1.3 (3)
C1—O2—C4—C3	0.4 (3)	C11—C12—C13—C14	−176.1 (3)
C3—C4—C5—C6	1.5 (4)	C10—O4—C13—C12	−0.2 (3)
O2—C4—C5—C6	179.6 (2)	C10—O4—C13—C14	177.7 (2)
C4—C5—C6—C7	−0.4 (4)	C12—C13—C14—O5	−113.2 (3)

C5—C6—C7—O3	−178.8 (2)	O4—C13—C14—O5	69.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5A···O3^{i}	0.82	1.90	2.682 (3)	160
O3—H3···O3^{i}	0.82	1.96	2.717 (3)	154
C9—H9···O1^{ii}	0.93	2.56	3.414 (3)	153

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

Fig. 1

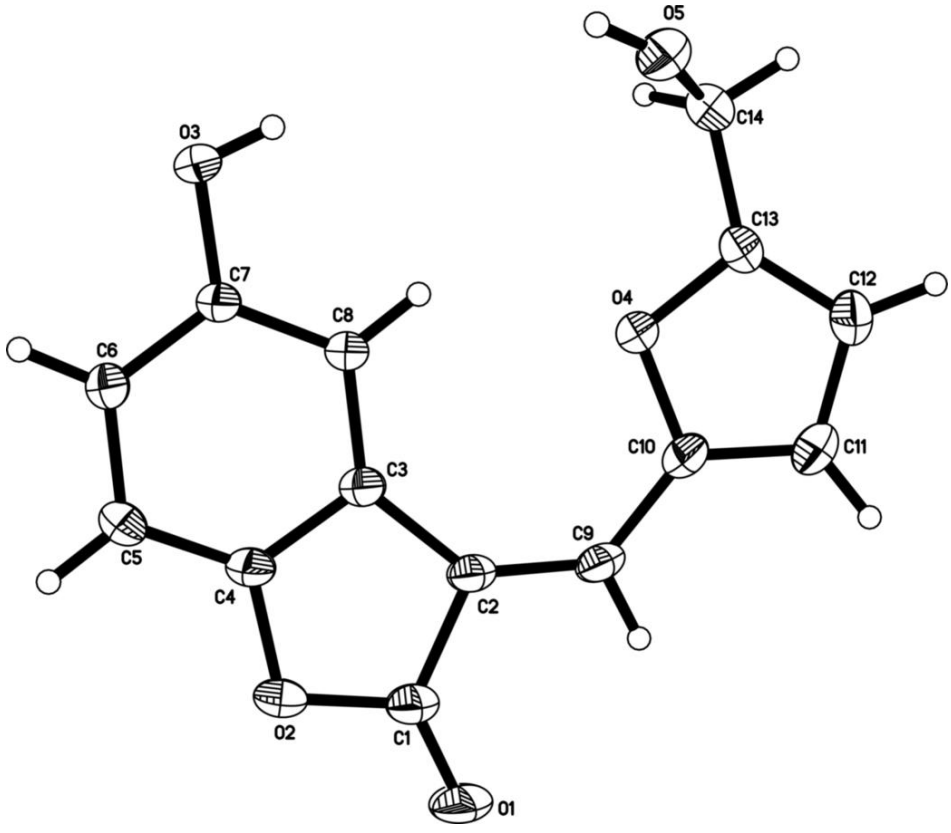

Fig. 2