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1-Hydroxy-2,3,4,7-tetramethoxyxanthone from *Swertia Chirayita*

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.165
Data-to-parameter ratio = 12.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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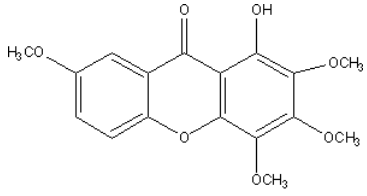
The title compound, 1-hydroxy-2,3,4,7-tetramethoxy-9*H*-xanthen-9-one, $C_{17}H_{16}O_7$, was isolated from *Swertia Chirayita* and is found to be planar with only two of the methoxy substituents lying out of the plane of the molecule.

Comment

Swertia Chirayita (Buch.-Ham.) what does this mean grows abundantly in the temperate regions of the Himalayas. It is used in Tibetan folk medicine as a traditional remedy for chronic fever, anaemia, asthma, liver disorders, hepatitis and stomach ache. Recently, the hexane extract of the plant has been reported to possess hypoglycemic activity. Earlier chemical investigations on the plant resulted in the isolation of a number of xanthones (Rakesh *et al.*, 1991), *e.g.* 1-hydroxy-3,5,8-trimethoxy xanthone, 1-hydroxy-3,7,8-trimethoxy-xanthone, 1,8-dihydroxy-3,5-dimethoxyxanthone, 1,8-dihydroxy-3,5-dimethoxyxanthone, 1,8-dihydroxy-3,7-dimethoxyxanthone, 1,3,6,7-tetrahydroxyxanthone-C-2-β-D-glucoside,1,3,8-trihydroxy-5-methoxyxanthone, 1,3,5,8-tetrahydroxyxanthone and 1,3,7,8-tetrahydroxyxanthone. Most xanthones have phenolic functional groups on an extended tricyclic system, and they often exhibit a wide range of biological and pharmacological activities, *e.g.* cytotoxic, anti-inflammatory, antimicrobial and antifungal (Jiang *et al.*, 2003; Chen *et al.*, 2002; Sun & Ding, 1983; Gales *et al.*, 2001). Xanthones are thought to have antioxidant activity, *e.g.* scavenging free radicals and superoxide anion, and to inhibit lipid peroxidation (Hiroyuki *et al.*, 1994; Eiba *et al.*, 1988).



(I)

We report here the isolation and characterization of 1-hydroxy-2,3,4,7-tetramethoxyxanthone, (I), 1-hydroxy-2,3,4,5-tetramethoxyxanthone, (II), 1-hydroxy-3,5-dimethoxy-xanthone, (III), 1,8-dihydroxy-3,5-dimethoxyxanthone, (IV), and 1,5,8-trihydroxy-3-methoxyxanthone, (V).

The molecular structure of the title compound, (I), isolated from the ethyl acetate extract of *S. Chirayita*, is shown in Fig. 1. Selected bond lengths and angles are listed in Table 1. The overall structure is nearly planar with an intramolecular hydrogen bond O4—H4*A*···O3 with an O4···O3 distance of 2.582 (4) Å (Table 2). Most of the substituents on the xanthone ring are very close to the mean plane of the molecule

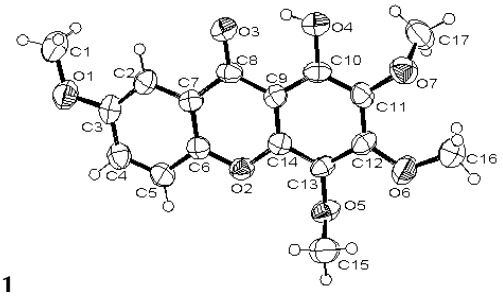


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

(Fig. 1). This is shown by the torsion angles C1—O1—C3—C4 = −179.6 (4)°, C16—O6—C12—C13 = 171.4 (5)°, C15—O5—C13—C14 = −96.4 (5)°, O4—C10—C11—O7 = 3.7 (6)° and O6—C12—C13—C14 = 179.7 (2)°.

In the crystal structure of (I), molecules are linked in pairs by intermolecular C—H···O hydrogen bonds (Table 2). These centrosymmetric dimers stack along the *a* axis (Fig. 2).

Experimental

The plant material *Swertia Chirayita* (whole plant) was bought from the Tibetan hospital of Huangzhong of Qinghai province in October, 2001. It was identified by Professor Rong-Fu Huang in the Northwest Plateau Institute of Biology, Chinese Academy of Sciences, Xining, China.

Extraction and isolation: *Swertia chirayita* (4.0 kg) was milled and extracted by maceration in EtOH for one week; the extract was concentrated *in vacuo* to a syrup, diluted with H_2O and partitioned with $CHCl_3$. The $CHCl_3$ layer was evaporated *in vacuo* to afford a residue (86 g), which was subjected to column chromatography over silica gel, eluted with $CHCl_3$–Me_2CO, to afford 16 fractions (S1–S16). Fractions S2–S9 were repeatedly chromatographed over silica gel and then purified with $CHCl_3$–Me_2CO (12:1, 6:1 and 3:1) to yield (I) (134 mg), (II) (62 mg), and (III) (89 mg), respectively. Fractions S12–S15 were repeatedly chromatographed over silica gel and then purified with $CHCl_3$–MeOH (12:1 and 6:1) and recrystallized (MeOH–Me_2CO) to afford (IV) (102 mg), and (V) (162 mg). Yellow needle-like crystals of (I) were obtained by slow evaporation of a solution in MeOH–Me_2CO (7:3) [m.p: 386–387 K (crystallized from $CHCl_3$–Me_2CO)]. IR (KBr, cm^{-1}): 3442, 1649, 1604, 1589, 1485, 1428, 1393, 1269, 1208, 1058. EIMS *m*/*z* (%): 332 (M^+), 317 (100), 302 (32), 289 (17), 287 (30), 274 (13), 259 (8), 245 (6), 203 (3), 175 (5). ^{1}H NMR ($CDCl_3$): δ 12.67 (*s*, 1H, OH-1), 7.51 (*d*, *J* = 9.2 Hz, 1H, H-5), 7.35 (*dd*, *J* = 9.2, 3.2 Hz, 1H, H-6), 7.61 (*d*, *J* = 3.2 Hz, 1H, H-8), 4.15 (*s*, 3H, OMe), 3.96 (*s*, 3H, OMe), 3.95 (*s*, 3H, OMe), 3.92 (*s*, 3H, OMe). ^{13}C NMR ($CDCl_3$): δ 150.8 (C-1), 135.3 (C-2), 154.2 (C-3), 132.4 (C-4), 146.1 (C-4a), 151.0 (C-4b), 119.3 (C-5), 125.3 (C-6), 156.3 (C-7), 105.2 (C-8), 120.5 (C-8a), 105.0 (C-8*b*), 181.5 (C-9), 56.0 (OMe), 61.3 (OMe), 61.7 (OMe), 62.1 (OMe).

Crystal data

$C_{17}H_{16}O_7$
M_r = 332.30
Monoclinic, $P2_1/n$
a = 7.1510 (10) Å
b = 13.229 (3) Å
c = 16.044 (3) Å
β = 95.18 (3)°
V = 1511.6 (5) Å^3
Z = 4
D_x = 1.460 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 25 reflections
θ = 10–20°
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, colourless
0.4 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2885 measured reflections
2661 independent reflections
923 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.0°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 15$
$l = -19 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.165
S = 0.98
2661 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0024 (9)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.368 (5)	C2—C7	1.406 (6)
O1—C1	1.421 (5)	C3—C4	1.400 (6)
O2—C14	1.365 (5)	C4—C5	1.368 (6)
O2—C6	1.372 (5)	C5—C6	1.391 (6)
O3—C8	1.247 (5)	C6—C7	1.394 (5)
O4—C10	1.357 (5)	C7—C8	1.444 (6)
O5—C13	1.379 (5)	C8—C9	1.442 (6)
O5—C15	1.426 (5)	C9—C14	1.389 (5)
O6—C12	1.354 (5)	C9—C10	1.404 (6)
O6—C16	1.392 (5)	C10—C11	1.391 (6)
O7—C11	1.373 (5)	C11—C12	1.389 (6)
O7—C17	1.434 (5)	C12—C13	1.397 (6)
C2—C3	1.368 (6)	C13—C14	1.375 (6)
C3—O1—C1	116.8 (4)	C9—C8—C7	115.7 (4)
C14—O2—C6	119.3 (3)	C14—C9—C10	118.3 (4)
C13—O5—C15	114.9 (3)	C14—C9—C8	120.7 (4)
C12—O6—C16	124.9 (4)	C10—C9—C8	121.0 (4)
C11—O7—C17	114.8 (4)	O4—C10—C11	117.9 (4)
C3—C2—C7	120.7 (5)	O4—C10—C9	121.1 (4)
C2—C3—O1	125.2 (5)	C11—C10—C9	121.0 (4)
C2—C3—C4	119.6 (5)	O7—C11—C12	121.5 (4)
O1—C3—C4	115.2 (5)	O7—C11—C10	119.3 (4)
C5—C4—C3	121.1 (5)	C12—C11—C10	119.1 (4)
C4—C5—C6	119.1 (5)	O6—C12—C11	125.7 (5)
O2—C6—C5	116.8 (4)	O6—C12—C13	113.9 (4)
O2—C6—C7	122.1 (4)	C11—C12—C13	120.5 (4)
C5—C6—C7	121.0 (4)	C14—C13—O5	119.9 (4)
C6—C7—C2	118.5 (4)	C14—C13—C12	119.5 (4)
C6—C7—C8	120.1 (4)	O5—C13—C12	120.5 (4)
C2—C7—C8	121.4 (4)	O2—C14—C13	116.4 (4)
O3—C8—C9	122.5 (4)	O2—C14—C9	122.0 (4)
O3—C8—C7	121.8 (4)	C13—C14—C9	121.6 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···O3	0.85	1.82	2.582 (4)	148
C17—H17*A*···O7^{i}	0.96	2.46	3.302 (6)	146

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

The crystal did not diffract significantly beyond 20° in θ and only 35% of the data can be considered to be observed. All H atoms were positioned geometrically and treated as riding atoms: O—H = 0.85 Å, C—H = 0.96 Å and U_{iso}(H) = 0.08 Å^2.

Data collection: *CAD*-4 *SDP*/*VAX* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *SDP*/*VAX*; data reduction: *MolEN*/PC (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Siemens, 1994); software used to prepare material for publication: *SHELXTL/PC*.

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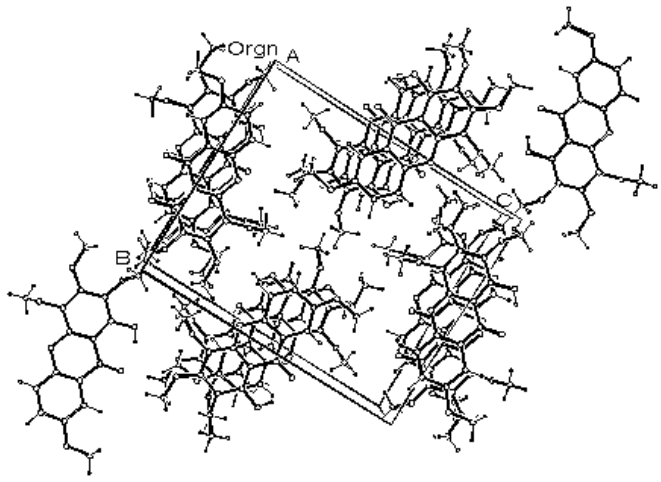

Figure 2
Packing diagram of the crystal structure of (I), viewed down the *a* axis.

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