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1-(1,8-Dihydroxy-6-methoxy-3-methylnaphthalen-2-yl)ethanone

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.066
wR factor = 0.148
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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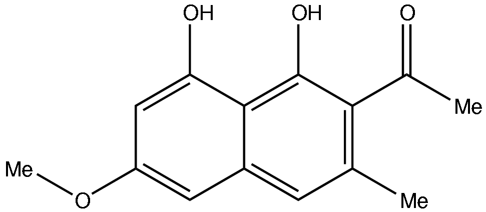
The molecule of the title compound, $C_{14}H_{14}O_4$, is planar and the structural dimensions are in the normal ranges. There are two intramolecular O—H···O hydrogen bonds, but no intermolecular interactions were observed.

Comment

Several naphthalene derivatives, such as torachrysone and torachrysone-8-*O*-*β*-D-*gluco*pyranoside, have been isolated from Rumex (Demirezer *et al.*, 2001; Suri *et al.*, 1978) and Rhubarb (Tsuboi *et al.*, 1977) plant species in Turkey and Japan, respectively. The title compound, (I), extracted from the *Cassia alata* plant found in West Malaysia, is an analogue of torachrysone but with a methyl group at C1 on the naphthalene fragment (Fig. 1). Genus *Cassia* is known to possess some medicinal properties and is a rich source of anthraquinones and flavonoids (Gupta & Singh, 1991). Therefore, *Cassia alata* is likely to be only the second species, in addition to *C. tora* (Shibata *et al.*, 1969), found to contain compound (I).



(I)

The molecule is planar apart from methyl H atoms, with a maximum deviation from the mean plane of −0.025 (4) Å for atom C14 from the mean plane. The bond lengths and angles (Table 1) are in the normal ranges (Allen *et al.*, 1987) and are comparable to those in chemically synthesized 2-bromo-1-(1-hydroxynaphthalen-2-yl)ethanone (Köysal *et al.*, 2004). However, in (I), the C9—O3 bond [1.335 (3) Å] is slightly shorter than C7—O2 [1.356 (3) Å], which in turn is comparable to the length of 1.366 (7) Å observed in 2-bromo-1-(1-hydroxynaphthalen-2-yl)ethanone. There are two intramolecular hydrogen bonds, *viz*. O2—H2*A*···O3 and O3—H3*A*···O4 (Table 2), resulting in the formation of two pseudo-six-membered rings (C9—C8—C7—O2—H2*A*···O3 and C13—C10—C9—O3···H). In contrast to 2-bromo-1-(1-hydroxynaphthalen-2-yl)ethanone, there are no intermolecular interactions observed in this structure.

Experimental

Cassia alata was collected from Section 16 Shah Alam, Selangor, in September 2002. The specimen (SK996-04) was deposited at the

Laboratory of Natural Products Mini Herbarium, Institute of Bioscience, Universiti Putra Malaysia. The bark of *C. alata* (1.0 kg) was extracted with dichloromethane at room temperature for 2 d. Removal of solvent gave 12 g of crude extract which was fractionated on a silica gel column, eluting with dichloromethane containing increasing percentages of acetone. Fractions 4 to 9, which eluted at 90:10 dichloromethane–acetone, were rechromatographed to give 25 mg of (I) (long yellow needles; m.p. 483–487 K) after evaporation. ^{1}H NMR (CDCl$_3$, 300 MHz): δ 2.64 (3H, *s*, CH$_3$), 2.75 (3H, *s*, CH$_3$), 3.90 (3H, *s*, OCH$_3$), 6.85 (1H, *s*), 6.51 (1H, *dd*, J_m = 2.4 Hz), 6.48 (1H, *dd*, J_m = 2.4 Hz), 10.44 (1H, *s*, OH); ^{13}C NMR (CDCl$_3$, 75 MHz): δ 203.6 (C═O), 169.7 (C1), 134.3 (C2), 139.9 (C3), 121.0 (C4), 99.2 (C5), 163.6 (C6), 100.8 (C7), 160.2 (C8), 108.8 (C9), 112.3 (C10), 25.4 (CH$_3$), 31.8 (CH$_3$) and 55.4 (OCH$_3$). EI–MS *m/z*: 246 (M^+), 231 (100).

Crystal data

$C_{14}H_{14}O_4$
$M_r = 246.25$
Triclinic, $P\bar{1}$
$a = 6.993\ (4)$ Å
$b = 7.543\ (4)$ Å
$c = 11.058\ (6)$ Å
$\alpha = 83.530\ (9)^\circ$
$\beta = 85.171\ (9)^\circ$
$\gamma = 79.017\ (9)^\circ$
$V = 567.7\ (5)$ Å^3
$Z = 2$
$D_x = 1.441$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 518 reflections
$\theta = 1.8$–25.0°
$\mu = 0.11$ mm^{-1}
$T = 273\ (2)$ K
Slab, yellow
$0.50 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.94$, $T_{\max} = 0.98$
5437 measured reflections
1981 independent reflections
1368 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 25.0^\circ$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.148$
$S = 1.14$
1981 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.2434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.365 (3)	O3—C9	1.335 (3)
O1—C12	1.428 (3)	O4—C13	1.259 (3)
O2—C7	1.356 (3)		
C12—O1—C5—C4	−1.7 (4)	C9—C10—C13—O4	0.8 (4)
C12—O1—C5—C6	177.9 (3)	C1—C10—C13—O4	−178.5 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3	0.82	1.85	2.570 (3)	145
O3—H3*A*···O4	0.82	1.65	2.400 (3)	150

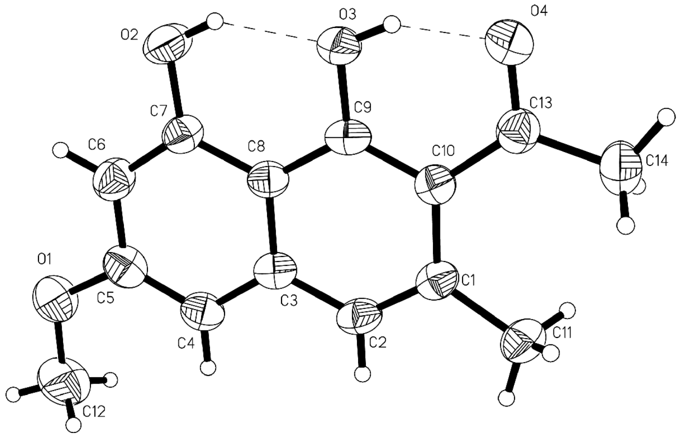


Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids and H atoms as spheres of arbitrary radii. The dashed lines indicate hydrogen bonds.

H atoms were located in a difference map, idealized and refined as riding atoms, with C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and O—H distances of 0.82 Å, and with $U_{\rm iso}$(H) = $1.2_{\rm eq}$(C) for CH or $1.5U_{\rm eq}$(C,O) for CH$_3$ and OH.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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