Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[4-(3,5-Dimethoxy-2-methylphenoxy)-2,6-dihydroxy-3-methylphenyl]ethanone

Chatchanok Karalai,[a] Nisakorn Saewon,[a] Kan Chantrapromma,[b] Suchada Chantrapromma,[a]* Hoong-Kun Fun[c] and Ibrahim Abdul Razak[c]

[a]Department of Chemistry, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, [b]Institute of Science, Walailak University, Thasala, Nakhon Si Thammarat 80160, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: suchada@ratree.psu.ac.th

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C—C) = 0.004 Å
R factor = 0.084
wR factor = 0.228
Data-to-parameter ratio = 13.1

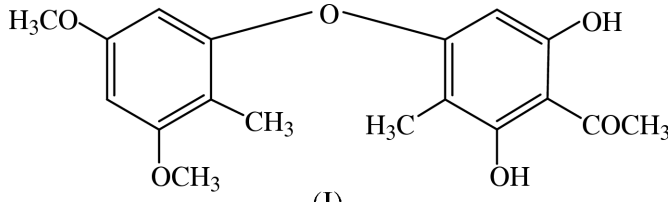
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 November 2000
Accepted 20 November 2000
Online 1 December 2000

The benzene rings of the title compound, $C_{18}H_{20}O_6$, are nearly perpendicular to each other [dihedral angle 75.2 (1)°]. The two methoxy groups and the acetyl group are almost coplanar to their attached benzene rings. One hydroxyl group is involved in an intramolecular O—H···O hydrogen bond with the adjacent acetyl O atom. The crystal structure is stabilized by intermolecular O—H···O contacts.

Comment

It has been reported that *p*-depsides could be readily converted into the corresponding diphenyl ether *via* an intramolecular Smiles rearrangement (Elix *et al.*, 1984). A number of depsides have been prepared as intermediate compounds in the synthesis of corresponding diphenyl ether molecules. Suitable reaction conditions for an intramolecular Smiles rearrangement of the prepared depsides have also been studied extensively (Elix & Jenie, 1989; Elix *et al.*, 1990). The crystal structure determination of the title compound, (I), was undertaken as part of structural studies on diphenyl ether derivatives. Knowledge of the three-dimensional structure of the title molecule could be useful for the understanding of this synthesis. The NMR spectrum of the obtained product did not show the signal ranging from 11 to 14 p.p.m., an indication of the predicted intramolecular hydrogen bonding between the hydroxyl and acetyl groups, whereas the X-ray data did clearly show this hydrogen bonding.



(I)

This structure is similar to 2-(4-acetyl-3,5-dihydroxy-2-methylphenoxy)-4,6-dimethoxy-3-methylbenzoic acid (Chantrapromma *et al.*, 2000). A displacement ellipsoid plot with the numbering scheme is shown in Fig. 1. The bond lengths and angles observed in the structure are normal and agree reasonably with the reported values (Elix *et al.*, 1978; Allen *et al.*, 1987; Chantrapromma *et al.*, 1998, 2000). The benzene rings are nearly perpendicular to each other [dihedral angle 75.2 (1)°]. The two methoxy groups and the acetyl group are nearly coplanar with the benzene rings [C18—O6—C11—C10 172.8 (3)°, C17—O5—C9—C10 5.4 (4)° and C2—C3—C13—C14 −0.2 (5)°]. There are two hydroxyl groups in a molecule involved in hydrogen bonding: one hydroxyl group, O4—H4*A*,

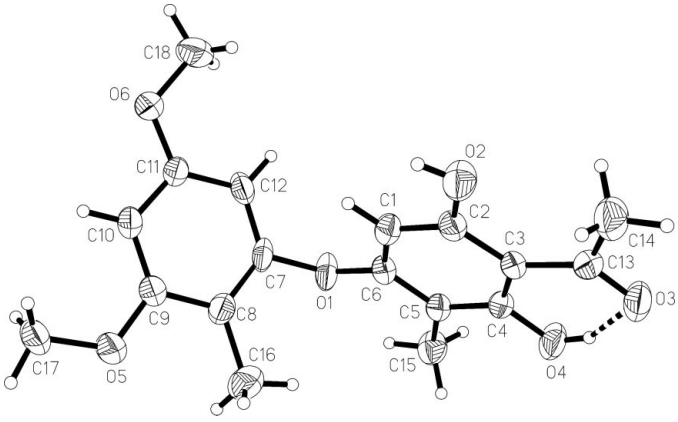


Figure 1
The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

involved as a donor in an intramolecular hydrogen bond with the O3 acetyl group acting as acceptor, the other, O2—H2*A*, involved in an intermolecular hydrogen bond with the O6 methoxy group of an adjacent molecule.

Experimental

To a solution of 2-(3,5-dihydroxy-4-acetyl-2-methylphenoxy)-4,6-dimethoxy-3-methylbenzoic acid (0.16 mmol) in anhydrous THF was added methyl lithium (1.95 mmol). The mixture was kept and stirred at 273 K for 3 h and at room temperature for an additional 6 h. The reaction mixture was acidified with cold diluted saturated ammonium chloride (30 ml) and extracted with ether. The residue was purified by preparative thin-layer chromatography with 20% hexane/chloroform as eluent to give colorless solids of (I) (m.p. 462–463 K). Crystals were recrystallized from chloroform–ethyl acetate. It was difficult to get a good quality crystal since the title compound is air sensitive.

Crystal data

$C_{18}H_{20}O_6$	$Z = 2$
$M_r = 332.34$	$D_x = 1.338$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.4228$ (2) Å	Cell parameters from 2684 reflections
$b = 10.1460$ (1) Å	$\theta = 1.8$–$28.3°$
$c = 11.3559$ (3) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 102.474$ (1)°	$T = 293$ (2) K
$\beta = 90.989$ (2)°	Slab, light yellow
$\gamma = 98.544$ (2)°	0.44 × 0.22 × 0.18 mm
$V = 824.70$ (3) Å^3	

Data collection

Siemens SMART CCD area-detector	$R_{int} = 0.063$
ω scans	$\theta_{max} = 25.0°$
4715 measured reflections	$h = -4 \rightarrow 8$
2864 independent reflections	$k = -12 \rightarrow 12$
1615 reflections with $I > 2\sigma(I)$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1109P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.084$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.228$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.43$ e Å^{-3}
2864 reflections	Extinction correction: *SHELXL*97
218 parameters	Extinction coefficient: 0.040 (9)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.379 (3)	O5—C9	1.373 (4)
O1—C7	1.414 (3)	O5—C17	1.428 (3)
O2—C2	1.366 (3)	O6—C11	1.374 (3)
O3—C13	1.250 (3)	O6—C18	1.432 (4)
O4—C4	1.351 (3)		
C6—O1—C7	118.6 (2)	O1—C6—C5	115.8 (2)
C9—O5—C17	118.1 (2)	O5—C9—C10	124.5 (3)
C11—O6—C18	118.2 (2)	O5—C9—C8	113.5 (3)
O2—C2—C1	119.5 (2)	O6—C11—C12	123.6 (3)
O2—C2—C3	119.0 (2)	O6—C11—C10	115.7 (2)
O4—C4—C5	115.9 (2)	O3—C13—C3	120.2 (3)
O4—C4—C3	120.4 (3)	O3—C13—C14	117.2 (3)
O1—C6—C1	121.7 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O6^{i}	0.82	2.02	2.837 (3)	179
O4—H4*A*···O3	0.82	1.74	2.482 (3)	149

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

Crystal decay was monitored by *SAINT* (Siemens, 1996) and was found to be negligible. After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms. Due to large fraction of weak data at higher angles, the 2θ maximum is limited to 50°.

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* and *PLATON* (Spek, 1990).

This work has received partial support from Prince of Songkla university and National Science and Technology Development Agency, Thailand. The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610942.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Chantrapromma, K., Sortiruk, R., Chantrapromma, S., Ponglimanont, C., Fun, H.-K. & Chinnakali, K. (1998). *Acta Cryst.* C**54**, 1494–1496.
Chantrapromma, S., Fun, H.-K., Razak, I. A., Saewon, N., Karalai, C. & Chantrapromma, K. (2000). *Acta Cryst.* C**56**, e598–599.
Elix, J. A., Engkaninan, U., Jones, A. J., Raston, C. L., Sargen, M. V. & White A. L. (1978). *Aust. J. Chem.* **31**, 2057–2068.
Elix, J. A. & Jenie, U. A. (1989). *Aust. J. Chem.* **42**, 987–994.
Elix, J. A., Jiang, H. & Wardlaw, J. H. (1990). *Aust. J. Chem.* **43**, 1745–1758.
Elix, J. A., Jones, A. J., Lajide, L., Coppins, B. J. & James, P. W. (1984). *Aust. J. Chem.* **37**, 2349–2364.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.