Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4,6-dimethoxyphenyl)prop-2-en-1-one

Shan Gao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.102
Data-to-parameter ratio = 9.1

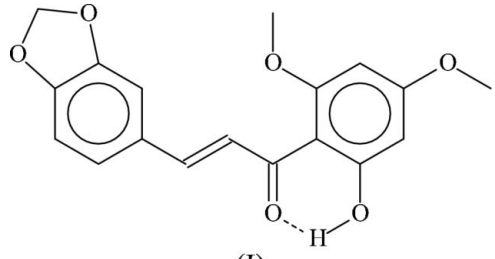
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 July 2006
Accepted 19 July 2006

In the title compound, $C_{18}H_{16}O_6$, a herbicide, the dioxole ring adopts a flattened envelope conformation. The two aromatic rings at either end of the propenone linkage are almost coplanar with it. The hydroxy group is involved in an intramolecular O—H···O hydrogen bond.

Comment

The piperonylidenyl $CH_2O_2C_6H_3—C{=}CH—$ unit is found in a number of naturally occurring compounds, as well as in synthetic chemicals that possess biological activity. The crystal structures of a number of such compounds have been determined (Cambridge Structural Database, Version 5.27; Allen, 2002), for example (+)-bornyl piperate (Rukachaisirikul *et al.*, 2004), calocedrin (Wang *et al.*, 1987*b*), savinin (Shieh *et al.*, 1990; Wang *et al.*, 1987*a*), stiripentol (Lisgarten & Palmer, 1988; Toffoli *et al.*, 1988), methysticin (Engel & Nowacki, 1972) and the alkaloid piperine (Grynpas & Lindley, 1975). In continuation of our structural studies of herbicides (Gao & Ng, 2005*a*,*b*), we report here the structure of the title compound, (I).



(I)

Compound (I) has the $CH_2O_2C_6H_3—C{=}CH—$ unit attached to a ketonic function. The other substituent of the ketone is the 2-hydroxy-4,6-dimethoxyphenyl group, the hydroxy group of which is involved in an intramolecular O—H···O hydrogen bond (Fig. 1 and Table 1). The dioxole ring adopts a flattened envelope conformation with C1 as the flap atom. The two aromatic rings at either end of the propenone linkage are almost coplanar; the C2–C7 and C11–C16 planes form dihedral angles of 5.3 (2) and 2.1 (2)°, respectively, with the C8/C9/C10/O3 plane.

Experimental

The title compound was purchased from Tianjian Bodi Chemical Reagent Company and recrystallized from ethanol.

Crystal data

$C_{18}H_{16}O_6$
M_r = 328.31
Orthorhombic, $P2_12_12_1$
a = 4.0062 (2) Å
b = 13.6947 (9) Å
c = 27.530 (2) Å
V = 1510.4 (2) Å^3
Z = 4
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 295 (2) K
Block, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.724, T_{max} = 1.00
13868 measured reflections
2031 independent reflections
1877 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.05
2031 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.1168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4*o*···O3	0.87 (1)	1.62 (1)	2.451 (2)	161 (3)
C9—H9···O6	0.93	2.12	2.771 (2)	126
C13—H13···O2^{i}	0.93	2.53	3.443 (2)	166

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

In the absence of significant anomalous scattering, Friedel pairs were averaged. Carbon-bound H atoms were positioned geometrically [C—H = 0.93–0.97 Å] and were included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms. The methyl groups were rotated to fit the electron density. The hydroxy H atom was located in a difference Fourier map, and was refined with a distance restraint of O—H = 0.85 (1) Å.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

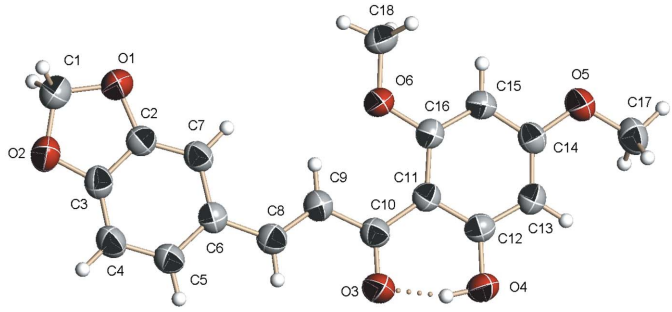


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii. The dotted line indicates the intramolecular O—H···O hydrogen bond.

The authors thank the National Natural Science Foundation of China (grant No. 20101003), the Scientific Fund for Remarkable Teachers of Heilongjiang Province (grant No. 1054 G036) and the University of Malaya for supporting this study.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Engel, P. & Nowacki, W. (1972). *Z. Kristallogr.* **136**, 437–452.
Gao, S. & Ng, S. W. (2005*a*). *Acta Cryst.* E**61**, o3761–o3762.
Gao, S. & Ng, S. W. (2005*b*). *Acta Cryst.* E**61**, o3763–o3764.
Grynpas, M. & Lindley, P. F. (1975). *Acta Cryst.* B**31**, 2663–2667.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lisgarten, J. N. & Palmer, R. A. (1988). *Acta Cryst.* C**44**, 1992–1994.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Rukachaisirikul, T., Prabpal, S., Kongsaeree, P. & Suksamram, A. (2004). *Chem. Pharm. Bull.* **52**, 760–761.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Shieh, H.-L., Cordell, G. A., Lankin, D. C. & Lotter, H. (1990). *J. Org. Chem.* **55**, 5139–5145.
Toffoli, P., Rouland, J.-C., Rodier, N., Ceolin, R., Lepage, F. & Astoin, J. (1988). *Acta Cryst.* C**44**, 2212–2214.
Wang, Y., Cheng, M. C., Jan, S. T. & Cheng, S. C. (1987*a*). *Acta Cryst.* C**43**, 1005–1006.
Wang, Y., Cheng, M. C., Jan, S. T. & Cheng, S. C. (1987*b*). *Acta Cryst.* C**43**, 1006–1008.