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3-Acetyl-3-benzyl-5-hydroxy-6,7-dimethoxy-4-methylbenzofuran-2(3*H*)-one

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.144
Data-to-parameter ratio = 15.0

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

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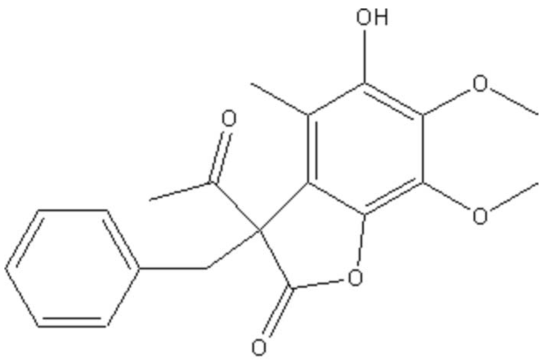
In the title compond $C_{20}H_{20}O_6$, the benzofuran ring system is nearly planar. The crystal structure is stabilized by O—H···O hydrogen bonds.

Comment

The title compound, (I), 3-acetyl-3-benzyl-5-hydroxy-6,7-dimethoxy-4-methylbenzofuran-2(3*H*)-one is the unexpected product of the synthesis of ethyl 2-(2,5-bis(benzyloxy)-3,4-dimethoxy-6-methylphenyl)-3-oxobutanoate, an important medicinal intermediate, by the procedures reported by Kucklander *et al.* (1988) and Kadota *et al.* (1999). In (I) (Fig. 1), the 1-benzofuran ring system is almost planar with an r.m.s. deviation of 0.029 Å from the mean plane. The acetyl group is nearly orthogonal to the benzofuran ring system with a dihedral angle of 88.5 (2)°. The two methoxy groups lie *anti* to each other, on either side of the benzofuran ring plane [torsion angles C8—O2—C2—C3 = −79.05, C9—O3—C3—C2 = −90.85].



(I)

In the crystal structure, the molecules are linked by intermolecular O—H···O hydrogen-bonding interactions into alternating rows along the *a* axis (Fig. 2, Table 1).

Experimental

Ethyl acetoacetate (1.4 ml, 11 mmol) was added dropwise to a suspension of NaH (0.3 g, 12 mmol) in THF (5 ml) at 273 K–278 K. After 40 min, a solution of 2,3-dimethoxy-5-methylcyclohexa-2,5-diene-1,4-dione (1 g, 5.5 mmol) in THF (15 ml) was added dropwise at room temperature. After stirring for 3 h, benzyl bromide (1.3 ml, 11 mmol) was added dropwise at 323 K. After a further 5 h stirring, the reaction mixture was filtered and the crude residue purified by flash column chromatography on silica gel. Single crystals suitable for X-ray analysis were obtained from ethylacetate–petroleum ether (1:4).

Crystal data

$C_{20}H_{20}O_6$
$M_r = 356.36$
Monoclinic, $P2_1/n$
$a = 9.586$ (6) Å
$b = 17.189$ (11) Å
$c = 10.948$ (7) Å
$\beta = 95.133$ (13)°
$V = 1797$ (2) Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.30 × 0.28 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker,1996)
$T_{\min} = 0.971$, $T_{\max} = 0.981$
9892 measured reflections
3626 independent reflections
1699 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.059$
$\theta_{\max} = 26.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.144$
$S = 1.01$
3626 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0170 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5$^{\rm i}$	0.82	2.19	2.963 (3)	157
O1—H1···O2	0.82	2.25	2.697 (3)	114

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

All H atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, $U_{\rm iso}$=1.2$U_{\rm eq}$ (C) for aromatic; 0.97 Å, $U_{\rm iso}$ = 1.2$U_{\rm eq}$ (C) for CH$_2$; 0.96 Å, $U_{\rm iso}$ = 1.5$U_{\rm eq}$ (C) for CH$_3$ atoms.

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

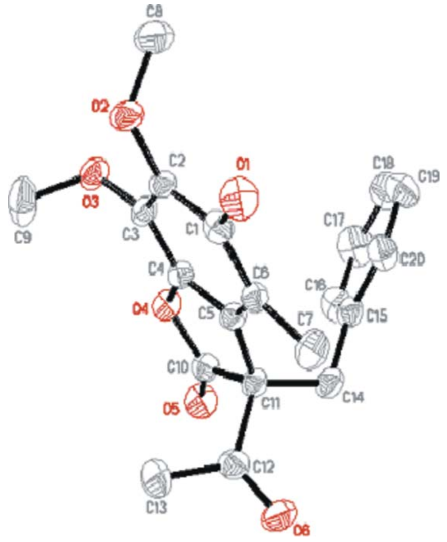


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity.

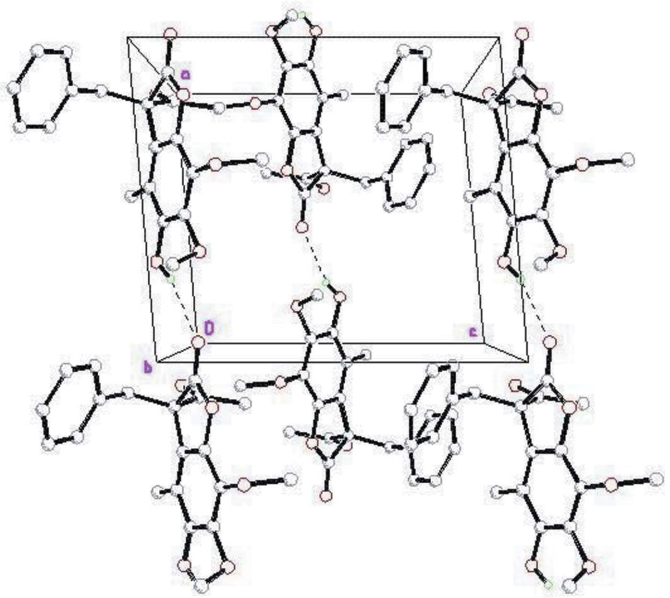


Figure 2
Part of the crystal packing in (I), with hydrogen bonds shown as dashed lines. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

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