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Butyl 3-[2-(3,4-dimethoxyphenyl)-7-methoxy-1-benzofuran-5-yl]-2-propenoate

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All non-H atoms in the title compound, $C_{24}H_{26}O_6$, are almost coplanar, except for the two terminal C atoms of the butyl chain. An $R_2^2(32)$ motif is formed by paired C—H···O hydrogen bonds. A chain along the *c* axis is formed by propagation of the C—H···O hydrogen bonds. The motif interacts with others *via* C—H···π interactions and C—H···O hydrogen bonds running along the *a* axis. The combination of these short contacts generates a sheet in the *bc* plane.

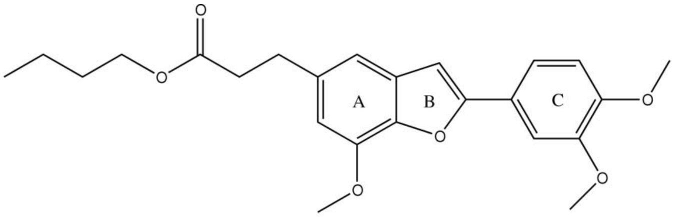
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.075
wR factor = 0.259
Data-to-parameter ratio = 15.1

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

Comment

Homoegonol has been isolated from the seeds of the *Styrax* species and its antifungal and antibacterial activities reported (Pauletti *et al.*, 2000). Several methods have been developed for its synthesis (Schreiber & Stevenson, 1976; Mali & Massey, 1998). We have already prepared a series of derivatives of this nor-lignan, based upon the method of benzofuran construction introduced by Castro *et al.* (1966). The title compound, 5-[2-(butoxycarbonyl)-*trans*-ethenyl]-7-methoxy-2-(3′,4′-dimethoxyphenyl)benzofuran, (I), was prepared as part of our ongoing studies of the natural product homoegonol, (II).



(I)

The molecular structure and atom-numbering scheme of (I) are shown in Fig. 1. Benzene ring *A* (C3–C8) is coplanar with the fused furan ring *B* (O1/C1–C3/C8), as indicated by the mean deviation of 0.0129 (4) Å from the least-squares plane containing atoms O1/C1–C8. The C16—O2—C7—C6 torsion angle of 1.4 (7)° indicates that the methoxy group is coplanar with ring A. Torsion angles C23—O5—C19—C18 = 13.6 (6)° and C24—O6—C20—C21 = −0.3 (6)° suggest that both methoxy groups are nearly coplanar with benzene ring *C* (C17–C22) but pointing away from each other, to relieve steric hindrance. Ring *C* is coplanar with the benzofuran ring system, as suggested by their dihedral angle of 4.4 (2)°.

Atom C24 acts as a donor, *via* atom H24*C*, to atom O4 at (−*x*, −*y*, −*z*), and an $R_2^2(32)$ motif is formed by paired C24—H24*C*···O4 hydrogen bonds around the inversion centre. These motifs interact with each other *via* a C23—H23*A*···O2 hydrogen bond, forming a zigzag chain down the *c* axis (Fig. 2). Fig. 3 shows the C—H···π interaction of (I). The $R_2^2(32)$

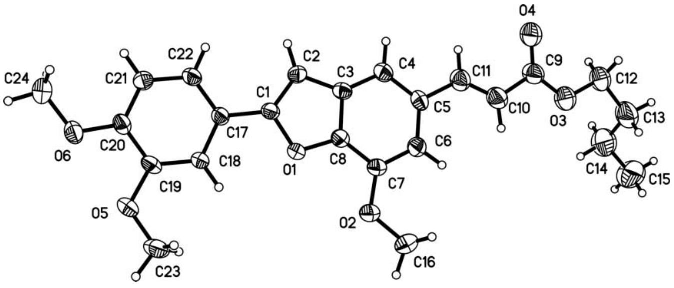


Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

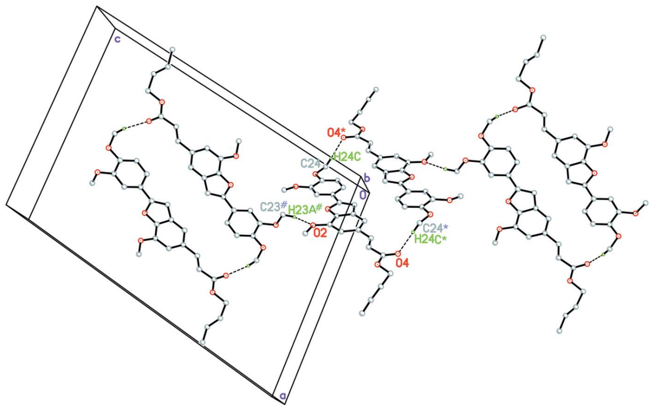


Figure 2
Part of the crystal structure of (I), showing the formation of the zigzag chain *via* hydrogen bonding. Atoms marked with an asterisk (*) or hash (#) are at the symmetry positions ($-x, -y, -z$) and ($\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$), respectively. For clarity, some H atoms have been omitted.

motifs are linked along the *a* axis *via* C—H$\cdots\pi$ interactions (C24—H24*B*$\cdots$*Cg*; *Cg* is the centroid of ring *C*), with C24$\cdots$*Cg* and H24*B*$\cdots$*Cg* distances of 3.521 (6) and 2.689 Å, respectively, and a C24—H24*B*$\cdots$*Cg* angle of 144°. The chains formed *via* these hydrogen bonds and C—H$\cdots\pi$ interactions combine to form a sheet parallel to the *bc* plane of (I).

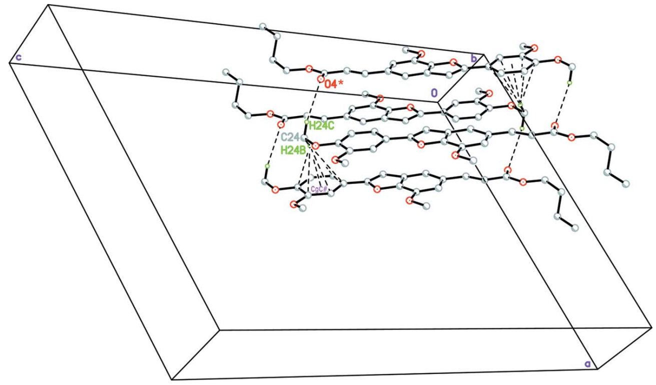


Figure 3
Part of the crystal structure of (I), showing the formation of the C—H$\cdots\pi$ interactions. Atoms marked with an asterisk (*) or hash (#) are at the symmetry positions ($-x, -y, -z$) and ($x, y - 1, z$), respectively. For clarity, some H atoms have been omitted.

Experimental

The title compound was prepared according to a nine-step route, using vanillin and 3,4-dimethoxyacetophenone as starting materials (Yang *et al.*, 1992). A few drops of concentrated H_2SO_4 were added to a solution of 5-(2-carboxy-*trans*-ethenyl)-7-methoxy-2-(3′,4′-dimethoxyphenyl)benzofuran (0.37 g, 1 mmol) in *n*-butyl alcohol (30 ml), and the mixture was stirred under reflux for 6 h. The reaction was monitored by thin-layer chromatography and, when complete, the mixture was diluted with water. The residue was chromatographed on a silica-gel column using an ethyl acetate–petroleum ether mixture (1:8 *v*/*v*) as eluent. Compound (I) was obtained as colourless needles by recrystallizing the crude sample from petroleum ether and ethyl acetate (4:1 *v*/*v*). Crystals of (I) suitable for diffraction analysis were obtained by slow evaporation of a solution in petroleum ether and ethyl acetate (4:1 *v*/*v*) at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 300 MHz, δ, p.p.m.): 0.98 (*t*, CH_3), 1.46 (*m*, 3″-CH_2), 1.71 (*m*, 2″-CH_2), 3.94 (*s*, 3′-OCH_3), 3.99 (*s*, 4′-OCH_3), 4.07 (*s*, 7-OCH_3), 4.22 (*t*, OCH_2), 6.42 (*d*, *J* = 16 Hz, α-vinyl H), 6.89 (*s*, H-3), 6.94 (*d*, *J* = 8.4 Hz, phenyl 5′-H), 6.97 (*d*, *J* = 1.3 Hz, 6-H), 7.33 (*d*, *J* = 1.3 Hz, 4-H), 7.37 (*d*, *J* = 2.0 Hz, phenyl 2′-H), 7.46 (*dd*, *J* = 2.0 and 8.4 Hz, phenyl 6′-H), 7.75 (*d*, *J* = 16 Hz, β-vinyl H).

Crystal data

$C_{24}H_{26}O_6$
$M_r = 410.45$
Monoclinic, $P2_1/n$
$a = 18.381$ (5) Å
$b = 4.8068$ (13) Å
$c = 27.575$ (7) Å
$\beta = 103.655$ (4)°
$V = 2367.4$ (11) Å^3
$Z = 4$

$D_x = 1.151$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1279 reflections
$\theta = 2.4$–20.0°
$\mu = 0.08$ mm^{-1}
$T = 298$ (2) K
Needle, colourless
0.52 × 0.23 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.958$, $T_{max} = 0.985$
11615 measured reflections

4096 independent reflections
1678 reflections with $I > 2\sigma(I)$
$R_{int} = 0.076$
$\theta_{max} = 25.0°$
$h = -15 \rightarrow 21$
$k = -5 \rightarrow 5$
$l = -32 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.259$
$S = 1.00$
4096 reflections
271 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected torsion angles (°).

C12—O3—C9—O4	0.5 (9)	C2—C1—C17—C18	176.3 (4)
C12—O3—C9—C10	179.0 (5)	O1—C1—C17—C18	−3.6 (5)
O4—C9—C10—C11	0.9 (9)	C23—O5—C19—C18	13.6 (6)
O3—C9—C10—C11	−177.7 (5)	C23—O5—C19—C20	−165.6 (4)
C9—C10—C11—C5	178.2 (5)	C24—O6—C20—C21	−0.3 (6)
C2—C1—C17—C22	−2.5 (7)	C24—O6—C20—C19	178.9 (4)
O1—C1—C17—C22	177.6 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C18—H18$\cdots$O1	0.93	2.46	2.790 (5)	101
C23—H23$A\cdots$O2$^{\rm i}$	0.96	2.50	3.244 (5)	134
C24—H24$C\cdots$O4$^{\rm ii}$	0.96	2.68	3.624 (7)	167

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

H atoms were placed in calculated positions and refined in riding mode, with C—H distances in the range 0.93–0.96 Å and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C), or $1.5U_{\rm eq}$(C) for methyl H.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *WinGX* (Version 1.7; Farrugia, 1999).

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