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Diethyl 2,5-bis(3,4,5-trimethoxyphenyl)-furan-3,4-dicarboxylate 0.16-hydrate

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Key indicators

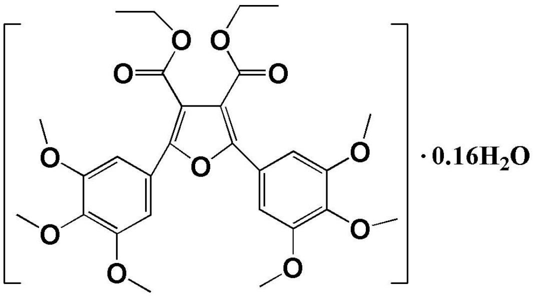
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.064
wR factor = 0.180
Data-to-parameter ratio = 13.5

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

In the title compound, $C_{28}H_{32}O_{11}\cdot0.16H_2O$, the substituted benzene rings are twisted slightly away from the furan ring. The dihedral angle between the two benzene rings is 8.8 (1)°. In the crystal structure, the molecules exist as C—H···O hydrogen-bonded dimers.

Comment

Furan-3,4-dicarboxylic acid and its esters have received much attention due to their versatile applications. They have been used as starting materials in the synthesis of several bioactive natural products, such as argocic acid, aromaticin, biotin and perrilin. They have also been used in the synthesis of several pharmacologically useful molecules, in the preparation of complexes with rare earth metal ions and as potential dienes in Diels–Alder reactions for the synthesis of several novel heterocycles (Deshpande *et al.*, 2002). In this paper, we report the crystal structure of the title compound, (I).



(I)

In compound (I), the two substituted benzene rings are twisted slightly away from the furan ring (Fig. 1). The C1–C6 and C14–C19 benzene rings form dihedral angles of 6.1 (2) and 7.5 (1)°, respectively, with the furan ring. The dihedral angle between the two benzene rings is 8.8 (1)°. The conformations of the two ester groups with respect to the furan ring are different (see Table 1 for torsion angles).

An intramolecular C6—H6···O10 hydrogen bond is observed. The crystal packing shows that the molecules exist as C—H···O hydrogen-bonded (Table 2) dimers. These dimers are interlinked *via* C—H···O hydrogen bonds (Fig. 2).

Experimental

Compound (I) was synthesized according to the literature procedure of Wu *et al.* (1997). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution at room temperature.

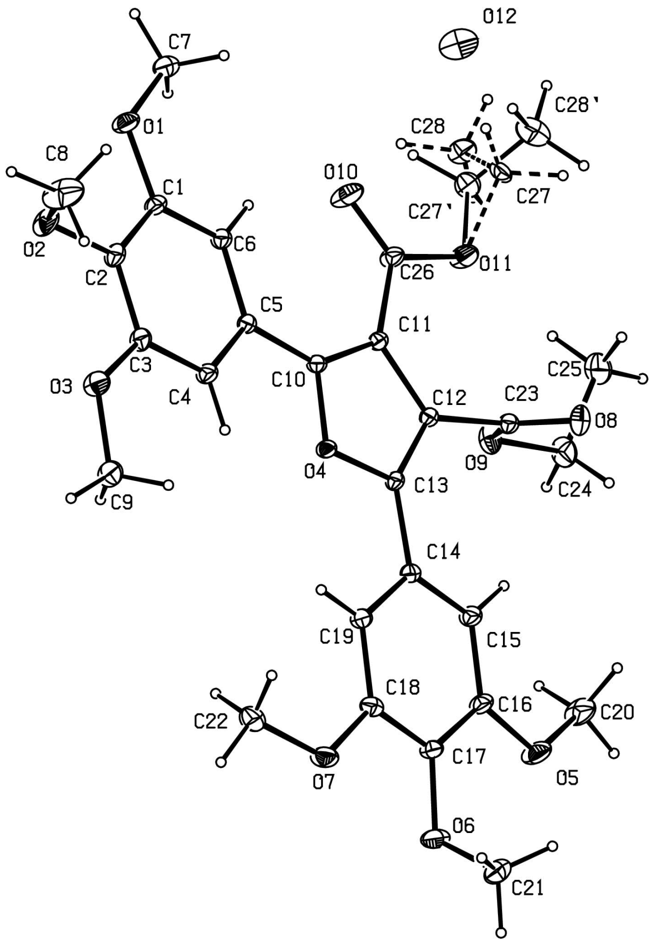


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Both disordered components of the ethyl group are shown.

Crystal data

$C_{28}H_{32}O_{11}\cdot 0.16H_2O$
$M_r = 547.42$
Monoclinic, $P2_1/n$
$a = 8.4402\ (7)$ Å
$b = 22.1502\ (19)$ Å
$c = 14.5043\ (12)$ Å
$\beta = 95.835\ (2)^\circ$
$V = 2697.6\ (4)$ Å^3
$Z = 4$
$D_x = 1.348$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2494 reflections
$\theta = 2.7$–23.8°
$\mu = 0.11$ mm^{-1}
$T = 292\ (2)$ K
Block, colourless
$0.50 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.949$, $T_{\max} = 0.979$
14583 measured reflections
5270 independent reflections
3426 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 26.0^\circ$
$h = -10 \rightarrow 10$
$k = -27 \rightarrow 25$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.180$
$S = 1.06$
5270 reflections
389 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

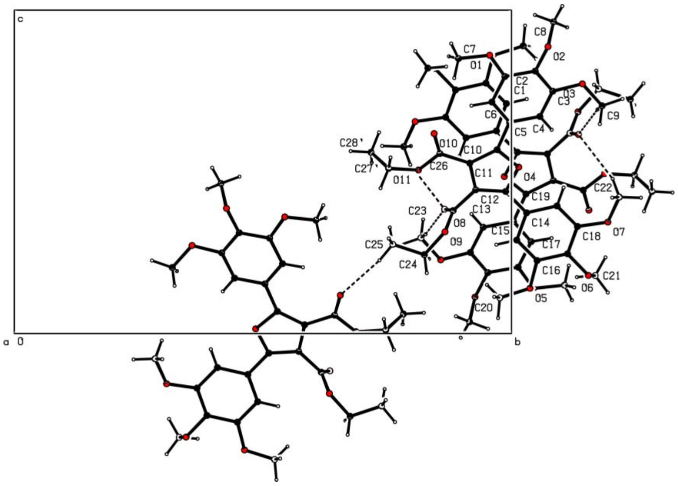


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.367 (3)	C17—O6	1.386 (3)
C2—O2	1.377 (3)	C18—O7	1.368 (3)
C3—O3	1.365 (3)	C20—O5	1.414 (4)
C7—O1	1.406 (4)	C21—O6	1.398 (4)
C8—O2	1.395 (5)	C22—O7	1.421 (4)
C9—O3	1.416 (4)	C23—O8	1.202 (3)
C10—O4	1.363 (3)	C24—O9	1.466 (3)
C10—C11	1.376 (3)	C26—O10	1.193 (3)
C12—C13	1.345 (3)	C26—O11	1.319 (3)
C13—O4	1.372 (3)	C27—O11	1.480 (7)
C16—O5	1.364 (3)		
O8—C23—O9	124.3 (2)	O10—C26—C11	127.0 (2)
O9—C23—C12	111.7 (2)	O11—C26—C11	111.7 (2)
C4—C5—C10—C11	−171.6 (3)	C10—C11—C26—O10	−7.6 (5)
C5—C10—C11—C26	−5.2 (6)	C12—C11—C26—O10	171.6 (3)
C23—C12—C13—C14	−8.3 (5)	C10—C11—C26—O11	172.4 (3)
C12—C13—C14—C19	178.4 (3)	C12—C11—C26—O11	−8.3 (4)
C13—C12—C23—O8	−78.8 (4)	C25—C24—O9—C23	−84.7 (4)
C11—C12—C23—O8	96.2 (4)	C28—C27—O11—C26	−91.5 (7)
C13—C12—C23—O9	97.4 (3)	C28'—C27'—O11—C26	110.9 (6)
C11—C12—C23—O9	−87.6 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O10	0.93	2.13	2.949 (3)	147
C9—H9*C*···O8$^{\rm i}$	0.96	2.57	3.490 (4)	161
C22—H22*A*···O8$^{\rm i}$	0.96	2.59	3.510 (4)	160
C25—H25*B*···O10$^{\rm ii}$	0.96	2.60	3.540 (4)	166

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

One of the ethyl groups is found to be disordered over two orientations. The occupancies of the disordered positions C27/C27′ and C28/C28′ were refined to 0.467 (12)/0.533 (12). Suitable restraints were applied to the O—C and C—C distances involving the disordered atoms. Water atom O12, with a partial occupancy of 0.16 [initially refined to 0.160 (7)], was found at a hydrogen-bonding distance of 2.78 Å from O2. The assignment of this peak to a partially occupied water molecule is tentative, being based only on the crystallographic evidence; the water probably derives from the methanol solvent used for recrystallization. The H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.97–0.97 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C) for others. Each methyl group was allowed to rotate freely about its C—C bond. The H atoms of the water molecule were not located in the difference map.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 2001); software used to prepare material for publication: *SHELXTL*.

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