2,6-Dimethyl-3,7-diphenylbenzo[1,2-b:4,5-b']-difuran

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.009 Å
R factor = 0.084
wR factor = 0.249
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.

In the title compound, $C_{24}H_{18}O_2$, the benzodifuran ring system is planar. The two phenyl rings make dihedral angles of 37.4 (2) and 40.8 (2)° with the benzodifuran ring system.

Comment

The benzo[b]furan nucleus is present in numerous examples of natural products and the chemistry of benzo[b]furan compounds has been extensively studied (Cagniant & Cagniant, 1975). However, benzodifuran derivatives, which have one more furan ring fused to the benzene ring of the benzo[b]furan nucleus, have received limited attention, although they are known to exhibit interesting chemical and physiological properties (Murthy et al., 2002; Rene et al., 1977; Takahashi et al., 1993; Chambers et al., 2001).
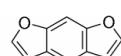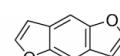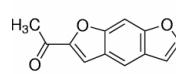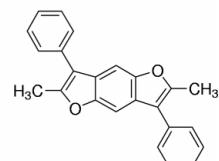
Several structural isomers of the benzodifuran system are possible and two of them are shown above [(I): benzo[1,2-b:5,4-b']difuran; (II): benzo[1,2-b:4,5-b']difuran]. Few benzodifuran compounds have been studied by X-ray diffraction methods (Takahashi & Kobayashi, 2000; Plenkiewicz et al., 2000; Harding et al., 1986). The crystal structure of 6-acetylbenzo[1,2-b:5,4-b']difuran, (III), has been reported and the ring system is almost planar (Bideau et al., 1978). With our continued interest in the syntheses and structures of benzo[b]furan (Park et al., 2000, 2001) and benzodifuran derivatives (Park, Lim et al., 2002; Park & Lim, 2002; Park et al., 2004), we determined the crystal structure of the title compound, (IV), which has a benzodifuran nucleus isomeric with (III).

The benzodifuran ring system in (IV) (Fig. 1) is essentially planar. The dihedral angles between the benzene ring and the furan rings are 0.6 (4) and 0.8 (4)°. These angles are similar to that of a furan-fused TCNQ compound (2.1°; Takahashi & Kobayashi, 2000). The two phenyl rings make dihedral angles of 37.4 (2) and 40.8 (2)° with the benzodifuran ring system, due to steric hindrance with the methyl groups on the furan
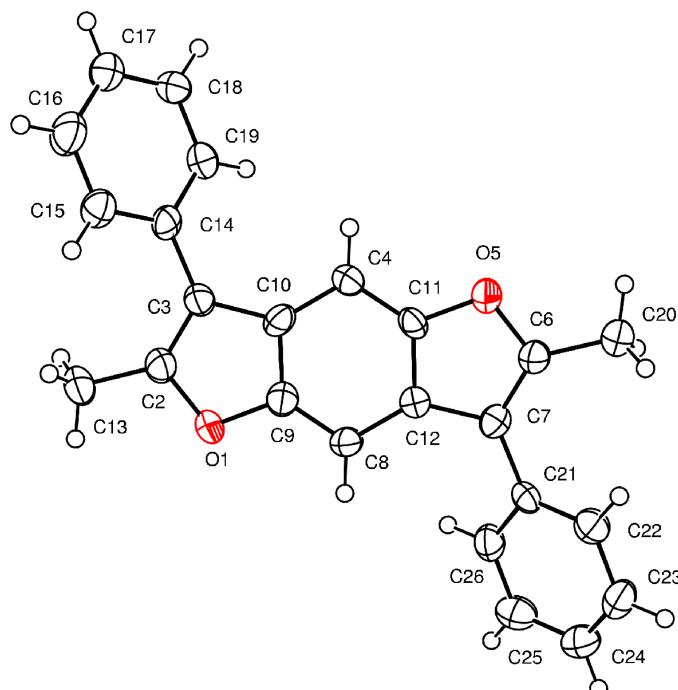
Figure 1
The molecular structure of (IV), showing the atom-numbering scheme and 30% probability displacement ellipsoids.
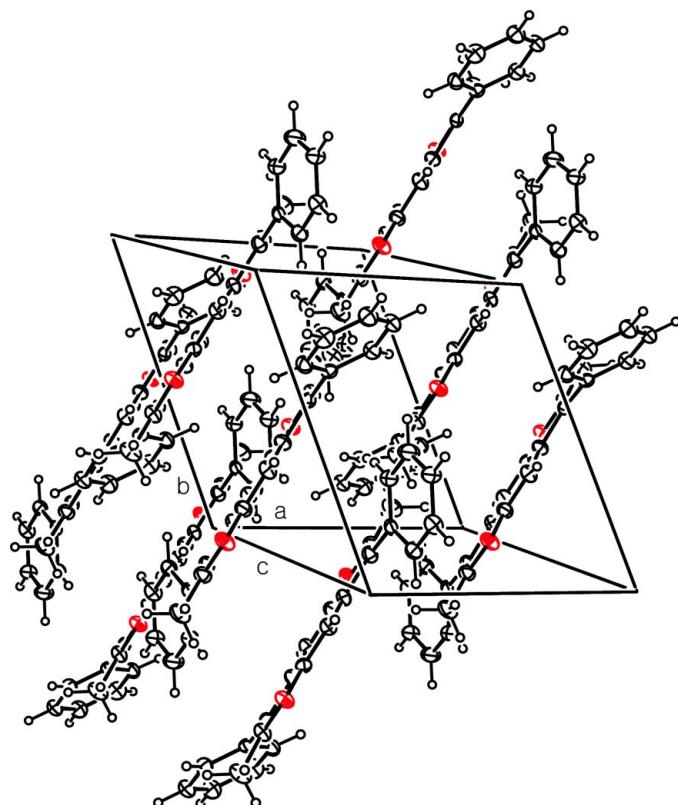
Figure 2
Packing diagram for (IV).

rings. Table 1 lists selected parameters for the fused ring system. The C—O bond distances [1.372 (6)–1.382 (7) Å] are within the normal range. The C2—C3 and C6—C7 bond

distances are 1.363 (8) and 1.340 (8) Å, respectively, and these are much shorter than the other C—C distances [1.409 (9)–1.451 (7) Å] in furan rings. Clearly, these bonds have double-bond character.

Experimental

The title compound, (IV), was prepared from *p*-dimethoxybenzene utilizing photocyclization and photo-Fries rearrangement reactions (Park *et al.*, 2004). Crystals of (IV) suitable for X-ray analysis were obtained by slow evaporation of a chloroform solution. The compound was characterized by NMR and elemental analysis data (Park *et al.*, 2004), ^{1}H NMR (400 MHz, 313 K): δ 2.54 (6H, *s*), 7.37 (2H, *t*, *J* = 7 Hz), 7.49 (4H, *t*, *J* = 8 Hz), 7.54 (4H, *d*, *J* = 8 Hz), 7.55 (2H, *s*). ^{13}C NMR (100 MHz, 313 K): δ 13.12, 100.01, 117.03, 126.19, 126.91, 128.73, 128.86, 133.05, 151.21, 151.68. Analysis calculated for $C_{24}H_{18}O_2$: C 85.18, H 5.36%; found: C 85.39, H 5.61%.

Crystal data

$C_{24}H_{18}O_2$	$Z = 2$
$M_r = 338.38$	$D_x = 1.318$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.6867$ (19) Å	Cell parameters from 23
$b = 9.7435$ (19) Å	reflections
$c = 10.723$ (2) Å	$\theta = 8.3$–15.8°
$\alpha = 116.90$ (3)°	$\mu = 0.08$ mm^{-1}
$\beta = 100.35$ (3)°	$T = 293$ (2) K
$\gamma = 99.36$ (3)°	Block, colorless
$V = 852.8$ (4) Å^3	$0.26 \times 0.20 \times 0.17$ mm

Data collection

Enraf–Nonius CAD-4	$\theta_{max} = 25.3°$
diffractometer	$h = 0 \rightarrow 11$
Non-profiled $\omega/2\theta$ scans	$k = -11 \rightarrow 11$
Absorption correction: none	$l = -12 \rightarrow 12$
3278 measured reflections	3 standard reflections
3080 independent reflections	every 400 reflections
1234 reflections with $I > 2\sigma(I)$	intensity decay: 2%
$R_{int} = 0.051$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.1043P)^2]$
$wR(F^2) = 0.249$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} < 0.001$
3080 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.382 (7)	O5—C6	1.377 (7)
O1—C2	1.387 (7)	C6—C7	1.340 (8)
C2—C3	1.363 (8)	C7—C12	1.451 (7)
C2—C13	1.485 (8)	C8—C9	1.361 (8)
C3—C10	1.445 (8)	C8—C12	1.404 (8)
C4—C11	1.362 (8)	C9—C10	1.409 (9)
C4—C10	1.402 (8)	C11—C12	1.419 (8)
O5—C11	1.372 (6)		
C9—O1—C2	106.6 (5)	C8—C9—C10	126.1 (6)
C3—C2—O1	111.5 (5)	O1—C9—C10	109.5 (5)
C2—C3—C10	106.2 (5)	C4—C10—C9	119.0 (5)
C11—C4—C10	115.4 (5)	C4—C10—C3	134.8 (6)
C11—O5—C6	106.8 (4)	C4—C11—C12	125.4 (5)
C7—C6—O5	112.0 (5)	O5—C11—C12	109.4 (5)
C6—C7—C12	106.8 (5)	C8—C12—C11	119.1 (5)
C9—C8—C12	115.0 (5)	C11—C12—C7	105.0 (5)
C8—C9—O1	124.4 (5)		

H atoms were positioned geometrically and constrained to ride on their attached atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ [$1.5U_{eq}(C)$ for methyl H atoms].

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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