

***trans*-3-Acetyl-5-benzoyl-2-methyl-4-phenyl-4,5-dihydrofuran at 150 K**M. Subha Nandhini,^a
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s_natarajan50@yahoo.com**Key indicators**

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.051

wR factor = 0.149

Data-to-parameter ratio = 22.7

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

In the title compound, $\text{C}_{20}\text{H}_{18}\text{O}_3$, the benzoyl and phenyl groups lie on opposite sides of the dihydrofuran ring. The dihydrofuran ring adopts a conformation intermediate between an envelope and a half-chair. The crystal packing is characterized by van der Waals forces and $\text{C}-\text{H}\cdots\text{O}$ interactions.

Comment

Dihydrofurans represent an important class of heterocycles, characterized by highly pronounced insect antifeedant activities (Gebbinck *et al.*, 1999), and they have been characterized by elemental analysis, IR, ^1H , ^{13}C and two-dimensional NMR spectroscopic methods. Dihydrofurans have also been found to possess antifungal (Pour *et al.*, 2003; Kunes *et al.*, 2001) and anti-inflammatory (Kumar *et al.*, 2003) properties. Since the title compound, (I), has several reactive functional groups, it can also serve as a starting material for the synthesis of novel heterocycles. A series of investigations on the synthesis, NMR and crystal structure elucidation of hydrofuran derivatives is being carried out in our laboratory.

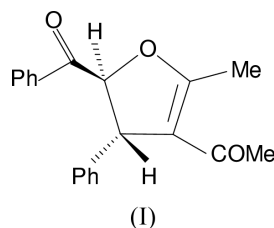


Fig. 1 shows the atom-numbering scheme adopted for (I). The two phenyl rings are planar, while the five-membered dihydrofuran ring adopts a conformation intermediate between an envelope and a half-chair; this seems to be different from the conformations observed in related structures, *viz.* (3*S*,5*S*,1'*S*)-3-benzyl-5-[1'-(*tert*-butoxycarbonylamino)-2'-phenylethyl]-4,5-dihydrofuran-2(3*H*)-one (Sopková *et al.*, 1996), methyl 4-hydroxy-3-[4-methoxy-2-methoxymethylenoxy]phenyl]-2-[[4-methoxy-2-(methoxymethylenoxy)phenyl]methyl]-5-oxo-2,5-dihydrofuran-2-carboxylate (Boehlow *et al.*, 1997) and 5-hydroxy-2,2-dimethyl-5-phenyl-2,5-dihydrofuran-3-carbonitrile (Ferguson *et al.*, 1990). The C—C and C—O distances (Table 1) agree well with those in similar dihydrofuran derivatives.

The carbonyl group $\text{C}8=\text{O}3$ is oriented away from the phenyl ring at C4 to avoid electronic repulsion. The benzoyl group at C3 and the phenyl group at C4 lie on opposite sides of

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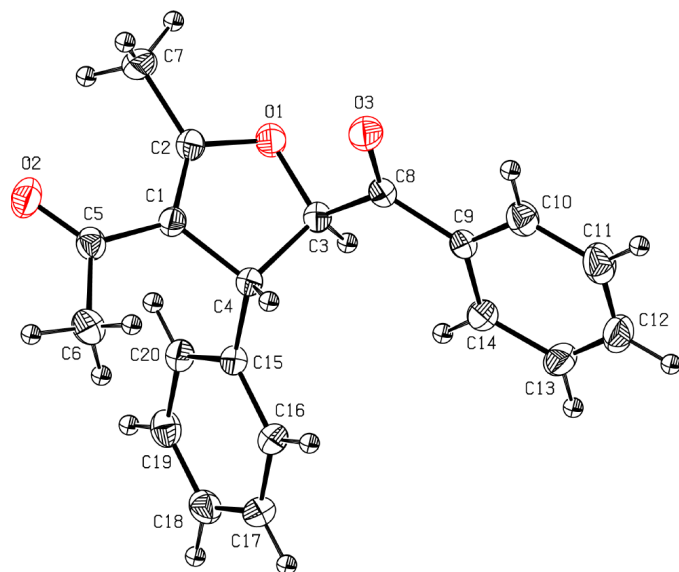


Figure 1
The molecular structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

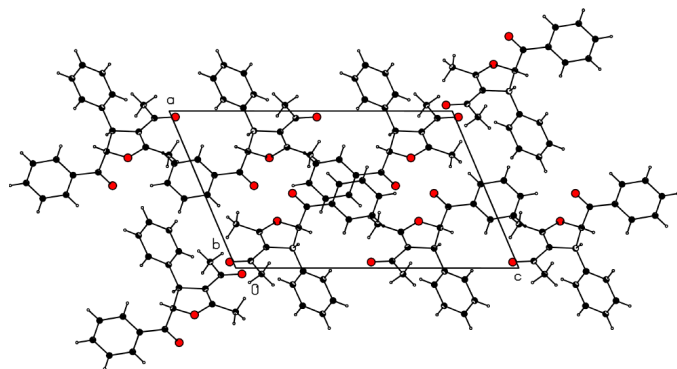


Figure 2
Packing of the molecules of (I), viewed down the *b* axis.

the dihydrofuran ring. This is evident from the torsion angle H4—C4—C3—C8 value of 9.9 (1)°; this value, close to zero, indicates that atoms H4 and C8 are eclipsed. This configuration, to avoid steric interactions between the two phenyl rings, is probably established in the cyclization step of the formation of the compound. Fig. 2 shows the arrangement of molecules as stacked layers, viewed down the *b* axis. Apart from the C—H···O hydrogen-bond interactions listed in Table 2, there are no other notable intermolecular contacts.

Experimental

The title dihydrofuran was obtained by the reaction of acetylacetone (100 mg, 0.001 mol) with 2,2'-sulfonylbis(1,3-diphenylprop-2-en-1-one) (478 mg, 0.001 mol) in the presence of sodium ethoxide (0.002 mol) in dimethylformamide (10 ml). Colourless single crystals of the compound were obtained as transparent plates from its saturated solution in methanol, by slow evaporation at room temperature.

Crystal data

$C_{20}H_{18}O_3$
 $M_r = 306.34$
Monoclinic, $P2_1/c$
 $a = 9.846$ (2) Å
 $b = 10.564$ (2) Å
 $c = 16.340$ (3) Å
 $\beta = 113.023$ (2)°
 $V = 1564.2$ (5) Å³
 $Z = 4$

$D_x = 1.301$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
 $\theta = 2.2$ – 26.3 °
 $\mu = 0.09$ mm⁻¹
 $T = 150$ (2) K
Plate, colourless
0.28 × 0.22 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{min} = 0.95$, $T_{max} = 0.99$
21162 measured reflections

4750 independent reflections
3623 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.036$
 $\theta_{max} = 30.5$ °
 $h = -14 \rightarrow 14$
 $k = -15 \rightarrow 15$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.149$
 $S = 1.08$
4750 reflections
209 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.5171P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.45$ e Å⁻³
 $\Delta\rho_{min} = -0.42$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0025 (19)

Table 1

Selected geometric parameters (Å, °).

O1—C2	1.360 (2)	C1—C5	1.465 (2)
O1—C3	1.446 (2)	C1—C4	1.524 (2)
O2—C5	1.232 (2)	C2—C7	1.487 (2)
O3—C8	1.222 (2)	C3—C8	1.530 (2)
C1—C2	1.351 (2)	C3—C4	1.571 (2)
C2—O1—C3	108.0 (1)	O1—C3—C4	106.8 (1)
C2—C1—C5	125.1 (1)	C8—C3—C4	109.7 (1)
C2—C1—C4	109.5 (1)	C4—C3—H3	110.8
C5—C1—C4	125.4 (1)	C15—C4—C3	113.7 (1)
C1—C2—O1	114.1 (1)	C3—C4—H4	109.2
C1—C2—C7	132.1 (1)	C16—C15—C20	118.9 (1)
O1—C2—C7	113.9 (1)	C20—C15—C4	121.1 (1)
O1—C3—C8	107.9 (1)	C2—C1—C4—C3	7.9 (1)
C5—C1—C2—O1	179.7 (1)	C5—C1—C4—C3	-172.5 (1)
C4—C1—C2—O1	-0.7 (2)	O1—C3—C4—C15	111.1 (1)
C5—C1—C2—C7	0.5 (2)	C8—C3—C4—C15	-132.2 (1)
C4—C1—C2—C7	-179.9 (1)	O1—C3—C4—C1	-12.1 (1)
C3—O1—C2—C1	-7.8 (2)	C8—C3—C4—C1	104.6 (1)
C3—O1—C2—C7	171.6 (1)	C2—C1—C5—O2	10.4 (2)
C2—O1—C3—C8	-105.3 (1)	C4—C1—C5—O2	-169.2 (1)
C2—O1—C3—C4	12.6 (1)	O1—C3—C8—O3	30.3 (2)
C2—C1—C4—C15	-114.3 (1)	C4—C3—C8—O3	-85.7 (2)
C5—C1—C4—C15	65.3 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C20—H20···O2 ⁱ	0.95	2.62	3.552 (2)	166
C16—H16···O2 ⁱⁱ	0.95	2.63	3.426 (2)	141
C12—H12···O3 ⁱⁱ	0.95	2.66	3.558 (2)	157

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

The H atoms were placed in calculated positions and were allowed to ride on their respective carrier atoms. The C—H lengths are 0.98 Å for methyl H atoms, 1.00 Å for other Csp^3 —H and 0.95 Å for Csp^2 —

H. For methyl H atoms, U_{iso} values were set equal to $1.2U_{\text{eq}}$ of the parent atom.

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

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