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3-(3-Methoxybenzoyl)-1′,6,6-trimethyl-4,5,6,7-tetrahydrospiro[benzo[*b*]furan-2(3*H*),3′(2′*H*)-1*H*-indole]-4,2′-dione

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Key indicators

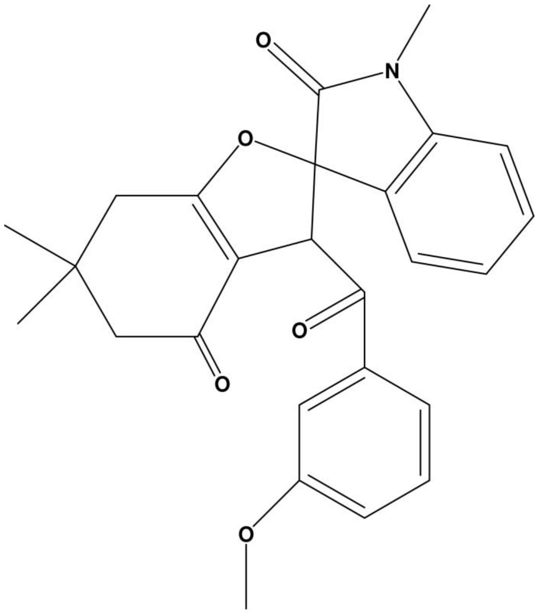
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.123
Data-to-parameter ratio = 17.3

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

In the title compound, $C_{26}H_{25}NO_5$, the dihydrofuran ring is non-planar, with the spiro C atom next to the O atom deviating by 0.089 (1) Å from the plane of the other atoms in the ring; the six-membered ring fused with the dihydrofuran ring is also non-planar. The molecular structure is stabilized by weak intramolecular C—H···O interactions. The crystal packing is stabilized by four intermolecular C—H···O interactions, generating $R_2^2(10)$, $R_2^2(16)$, $R_2^2(14)$ and $R_2^2(16)$ ring motif.

Comment

Tetrahydrofuran is a common motif which can be found in numerous natural products (*e.g.* polyether antibiotics, nucleosides, lignans) (Garzino *et al.*, 2000). The construction of polyfunctionalized furans, spiro-furans and furan-fused cycloalkanes are important from the standpoint of the synthesis of biologically active natural products, such as aflatoxin, asteltoxin, monensin, panacene and so on (Schoop *et al.*, 2000). In particular, 4,5-dihydrofurans are convenient synthetic starting materials since dihydrofurans can be easily transformed by dehydrogenation or reduction into the corresponding furans or tetrahydrofurans.



(I)

In the molecular structure of the title compound, (I) (Fig. 1), the bond lengths and angles (Table 1) are comparable with those in a similar structure reported previously (Gayathri *et*

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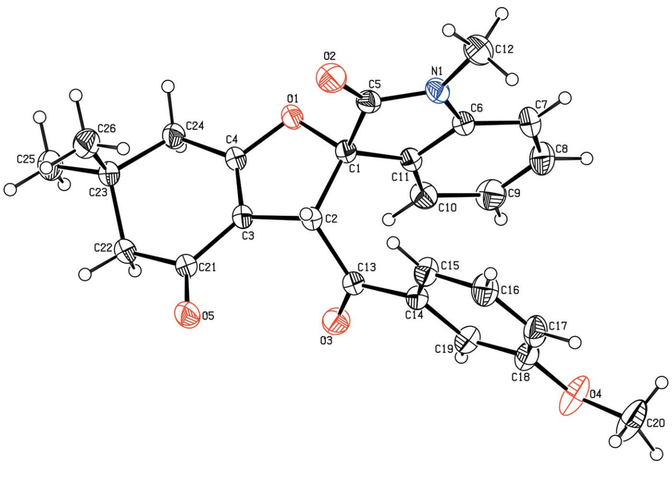

Figure 1

The molecular structure of title compound (I), showing 30% probability displacement ellipsoids.

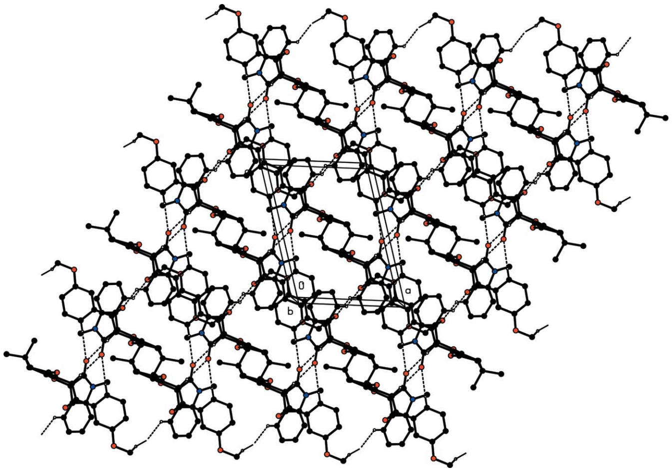

Figure 2

The molecular packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dsahed lines. For clarity, H atoms which are not involved in hydrogen bonding have been omitted.

al., 2006), except for the O1—C4 bond length in the dihydrofuran ring. The torsion angles C11—C1—C2—C13 [1.9 (1)°] and C5—C1—C2—C13 [−116.1 (1)°] deviate from the values observed in the similar structure. The sum of the angles at atom N1 (360.0°) indicates sp^2 hybridization. Atoms C12 and O2 deviate by 0.074 (2) and −0.083 (1) Å, respectively, from the plane of the five-membered ring (C1/C11/C6/N1/C5). The five- and six-membered rings in the indole unit are coplanar, making a dihedral angle of 1.3 (1)°; the dihedral angle between the two benzene rings is 41.1 (1)°. The methoxy group at C18 is twisted slightly away from the C14–C19 benzene ring [torsion angle C19—C18—O4—C20 = −172.2 (2)°], slightly different from that in the similar structure (Gayathri *et al.*, 2006). The dihydrofuran ring in the structure is non-planar, with atom C1 deviating by 0.089 (1) Å from the plane of the other atoms in the ring. The six-membered ring (C4/C3/C21–C24) is non-planar, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.353 (2), q_3 = 0.296 (2), Q_T = 0.461 (2) Å and φ = 50.0 (2)°.

The molecular structure is stabilized by a weak intramolecular C—H···O interaction. The crystal packing is stabilized by intermolecular C—H···O interactions. Atoms C2 and C15 act as donors to O2 ($2 - x, 2 - y, 1 - z$), generating centrosymmetric dimers of $R_2^2(10)$ and $R_2^2(16)$ rings centered at ($1, 1, \frac{1}{2}$). Atom C10 acts as a donor to O3($1 - x, 1 - y, -z$), generating a centrosymmetric dimer with an $R_2^2(14)$ ring centered at ($\frac{1}{2}, \frac{1}{2}, 0$). These three dimers run linearly along the *c* axis. Adjacent dimers running along the *a* axis are linked by another C—H···O intermolecular interaction, wherein atom C20 acts as donor to O3($2 - x, 1 - y, -z$), generating a centrosymmetric dimer with an $R_2^2(16)$ ring centered at ($1, \frac{1}{2}, 0$). Thus, the crystal packing is highly stabilized by four C—H···O dimers.

Experimental

To a stirred mixture of 3-[(3-methoxyphenyl)-2-oxoethylidene]-1-methyloxindole (1.02 mmol, 0.3 g, 1 equivalent), dimedone (1.02 mmol, 0.143 g, 1 equivalent) and $NaHCO_3$ (3.07 mmol, 0.258 g, 3 equivalents) in acetonitrile (10 ml), ceric ammonium nitrate (2.56 mmol, 1.4 g, 2.5 equivalanets) dissolved in acetonitrile (5 ml) was added dropwise at 273 K under an N_2 atmosphere. The reaction mixture was further stirred until the completion of the reaction, as monitored by thin-layer chromatography. Water was added to the mixture and the product was extracted into ethyl acetate (2 × 20 ml); it was then dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure gave a crude product, which was further purified by column chromatography on silica gel (100–200 mesh), with ethylacetate–hexane (4:6) as eluent, affording the pure product (yield 0.33 g, 75%) as a white crystalline solid. Single crystals of the product, (I), were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{26}H_{25}NO_5$
M_r = 431.47
Triclinic, $P\overline{1}$
a = 9.4557 (8) Å
b = 11.750 (1) Å
c = 12.3946 (10) Å
α = 116.168 (1)°
β = 93.005 (1)°
γ = 111.925 (1)°

V = 1106.90 (16) Å^3
Z = 2
D_x = 1.295 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, white
0.26 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
12751 measured reflections

5046 independent reflections
4353 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017
$\theta_{\rm max}$ = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
S = 1.03
5046 reflections
291 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.2028P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.462 (1)	C4—C24	1.483 (2)
C1—C11	1.499 (2)	C5—O2	1.207 (2)
C1—C5	1.543 (2)	C5—N1	1.356 (2)
C1—C2	1.571 (2)	C6—N1	1.406 (2)
C2—C3	1.497 (2)	C12—N1	1.451 (2)
C2—C13	1.522 (2)	C13—O3	1.206 (1)
C3—C4	1.332 (2)	C18—O4	1.363 (2)
C3—C21	1.448 (2)	C20—O4	1.425 (2)
C4—O1	1.358 (1)	C21—O5	1.221 (2)
C5—N1—C6	111.3 (1)	C4—O1—C1	107.9 (1)
C5—N1—C12	123.8 (1)	C18—O4—C20	117.2 (1)
C6—N1—C12	124.9 (1)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O2	0.96	2.52	2.896 (2)	103
C2—H2···O2^{i}	0.98	2.46	3.434 (2)	177
C10—H10···O3^{ii}	0.93	2.43	3.201 (2)	140
C15—H15···O2^{i}	0.93	2.44	3.320 (2)	158
C20—H20*C*···O3^{iii}	0.96	2.58	3.483 (3)	157

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x+1, -y+1, -z$; (iii) $-x+2, -y+1, -z$.

All H atoms were refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for aromatic, C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for CH, C—H = 0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for CH$_2$ and C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for CH$_3$ H atoms.

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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