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1′-Methyl-4′-(3,4,5-trimethoxyphenyl)dispiro[indene-2,3′-pyrrolidine-2′,3″-indole]-1,2″,3(1″*H*)-trione

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The pyrrolidine ring of the title compound, $C_{29}H_{26}N_2O_6$, adopts a twist conformation. The structure is stabilized by weak intramolecular C—H···O interactions as well as by intermolecular C—H···O, N—H···O and N—H···N interactions.

Key indicators

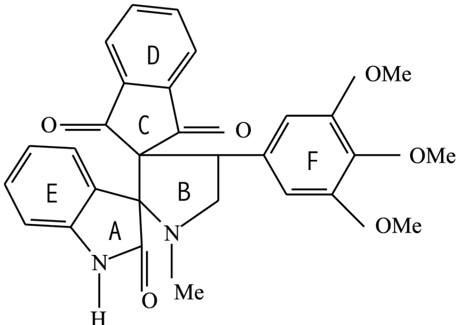
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.059
wR factor = 0.170
Data-to-parameter ratio = 17.7

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

Comment

Several unusual amino acids which contain the pyrrolidine motif have been investigated by Galeazzi *et al.* (1999). Furthermore, pyrrolidine compounds are capable of exhibiting antimicrobial and antifungal activity (Amal Raj *et al.*, 2003). As part of our ongoing studies of spiro-pyrrolidines, the crystal structure analysis of the title compound, (I), has been carried out and the results are presented here.

The C—C bond lengths (Table 1) in pyrrolidine ring *B* are longer than normal, perhaps due to the three bulky substituents. A similar effect has been found in a related structure reported by Jeyabharathi *et al.* (2001).



(I)

The geometry of the oxindole moiety is normal and agrees very well with that in other publications (Govind *et al.*, 2003; Govindasamy *et al.*, 1999; Sethu Sankar *et al.*, 2002). The Cambridge Structural Database (CSD) November 2003 Release (Allen, 2002) contains 20 examples of this geometry.

The bond lengths and angles of the trimethoxyphenyl and indandione moieties are perfectly normal, as evidenced by 252 and 34 examples, respectively, in the CSD.

Ring *A* is in an envelope conformation with lowest asymmetry parameter (Nardelli, 1983) ΔC_S(C4) = 0.009 (1); atom C4 deviates by 0.046 (2) Å from the least-squares plane passing through the remaining four atoms (N1, C2, C5 and C10) of the ring. Pyrrolidine ring *B* adopts a twist conformation, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.441 (2) Å and φ = 17.1 (2)°, and asymmetry parameter ΔC_2(C13) = 0.008 (1). Ring *C* is also in a twist conformation, with puckering parameters q_2 = 0.096 (2) Å and φ = −170.6 (2)°, and asymmetry parameter ΔC_2(C29) = 0.008 (1).

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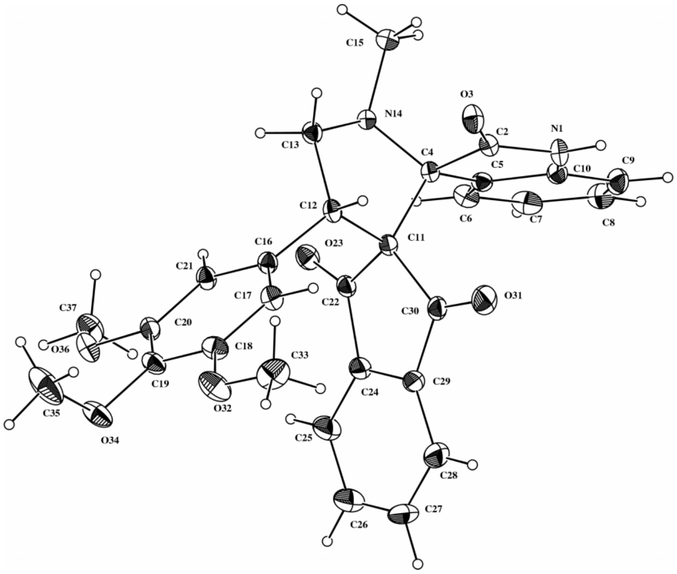

Figure 1
View of (I), with the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

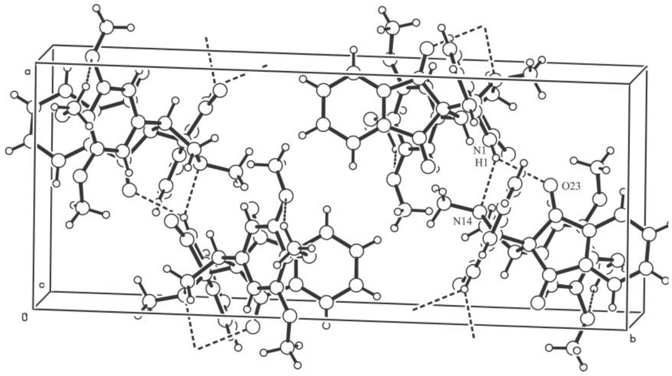

Figure 2
A view of the packing of the molecules in the unit cell; dashed lines indicate hydrogen bonds.

In addition to van der Waals interactions, the crystal structure is stabilized by weak C—H$\cdots$O intramolecular interactions, and also by C—H$\cdots$O, N—H$\cdots$O and N—H$\cdots$N intermolecular interactions.

Experimental

A mixture of 3,4,5-trimethoxybenzylidine (1 mmol), 1,3-indandione (1 mmol) and sarcosine (1 mmol) was refluxed in aqueous methanol. The resulting crude product was filtered off and recrystallized from methanol.

Crystal data

$C_{29}H_{26}N_2O_6$
$M_r = 498.52$
Monoclinic, $P2_1/n$
$a = 10.3884$ (7) Å
$b = 23.2621$ (14) Å
$c = 11.1685$ (7) Å
$\beta = 108.633$ (1)°
$V = 2557.5$ (3) Å^3
$Z = 4$
$D_x = 1.295$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3201 reflections
$\theta = 2.4$–$23.0°$
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.20 × 0.19 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.982$, $T_{\max} = 0.983$
15 973 measured reflections
5915 independent reflections
3919 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 28.0°$
$h = -13 \rightarrow 13$
$k = -30 \rightarrow 26$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.170$
$S = 1.00$
5915 reflections
334 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N14	1.452 (2)	C12—C13	1.537 (3)
C4—C11	1.556 (2)	C13—N14	1.475 (2)
C11—C12	1.563 (2)		
N14—C4—C11	101.0 (1)	N14—C13—C12	106.3 (1)
C4—C11—C12	101.0 (1)	C4—N14—C13	108.5 (1)
C13—C12—C11	102.8 (1)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C6—H6$\cdots$O23	0.93	2.51	3.150 (3)	126
C12—H12$\cdots$O3	0.98	2.41	3.010 (2)	119
C21—H21$\cdots$O23	0.93	2.58	3.207 (3)	125
C35—H35$C\cdots$O32	0.96	2.49	3.050 (4)	117
N1—H1$\cdots$O23$^{\rm i}$	0.86	2.45	3.125 (2)	136
N1—H1$\cdots$N14$^{\rm i}$	0.86	2.27	3.006 (2)	143
C8—H8$\cdots$O3$^{\rm ii}$	0.93	2.47	3.331 (3)	155
C13—H13$A\cdots$O3$^{\rm iii}$	0.97	2.45	3.400 (2)	167
C21—H21$\cdots$O3$^{\rm iii}$	0.93	2.58	3.336 (3)	139

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and N—H = 0.86 Å, and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms, $1.2U_{\rm eq}({\rm C})$ for other C-bound H atoms and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$ for the indole H atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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