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5,5′-Dimethoxy-3,3′-(3-fluorophenylmethanediyl)bis(1*H*-indole)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.179
Data-to-parameter ratio = 14.9

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

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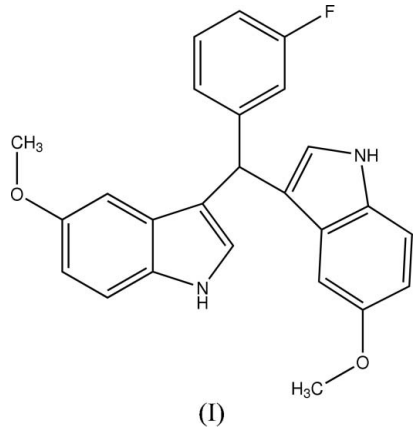
The title compound, $C_{25}H_{21}FN_2O_2$, was synthesized by the reaction of 5-methoxy-1*H*-indole and 3-fluorobenzaldehyde in ethanol, using $CuBr_2$ as a catalyst under microwave irradiation. In the crystal structure, there are two intermolecular hydrogen bonds, one N—H···O and one C—H···F, also one intermolecular N—H···π(arene) contact.

Comment

Development of bis(indolyl)alkane synthesis has been of considerable interest because of the wide occurrence of bis(indolyl)alkanes in various natural products possessing biological activity (Bell *et al.*, 1994) and their usefulness for drug design. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of compound (I) is shown in Fig. 1. Bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Ring *A* comprises atoms C20–C25, ring *B* atoms C5/N1/C6/C7/C8 and ring *C* atoms C14/N2/C15/C16/C17. The various dihedral angles are *A*/*C* = 109.4 (2)°, *B*/*C* = 94.4 (1)° and *A*/*B* = 80.0 (2)°.

The crystal structure of (I) is stabilized by two intermolecular N—H···O and C—H···F hydrogen bonds and one intermolecular N–H···π(arene) contact (Fig. 2 and Table 1; *Cg*1 is the centroid of atoms C11–C14/C17/C18). The N—H···O hydrogen bond and N—H···π(arene) contact are also present in a very similar compound, in which the fluoro group is replaced by a nitro group (Guo *et al.*, 2006).

Experimental

Compound (I) was prepared by the reaction of 5-methoxy-1*H*-indole (20 mmol) with 3-fluorobenzaldehyde (10 mmol) in ethanol (5 ml), using $CuBr_2$ (0.446 g) as catalyst under microwave irradiation

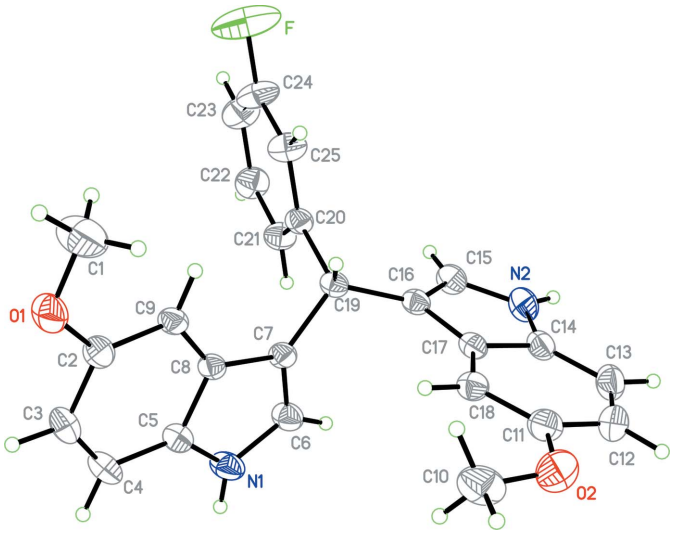


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

(280 W) for 10 min. After completion, the reaction mixture was quenched wtih H_2O (10 ml) and extracted with EtOAc (3 × 10 ml). The combined organic layers were dried over Na_2SO_4, concentrated, and purified by column chromatography on SiO_2 (ethyl acetate–petroleum ether 1:3 *v*/*v*) to afford the pure product, (I) (m.p. 472–475 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{25}H_{21}FN_2O_2$
M_r = 400.44
Monoclinic, $P2_1/n$
a = 9.1910 (18) Å
b = 10.838 (2) Å
c = 20.879 (4) Å
β = 94.92 (3)°
V = 2072.1 (7) Å^3
Z = 4
D_x = 1.284 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.964, T_{max} = 0.976
4322 measured reflections
4062 independent reflections
2600 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.179
S = 1.04
4062 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 0.9173P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.023
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.025 (2)

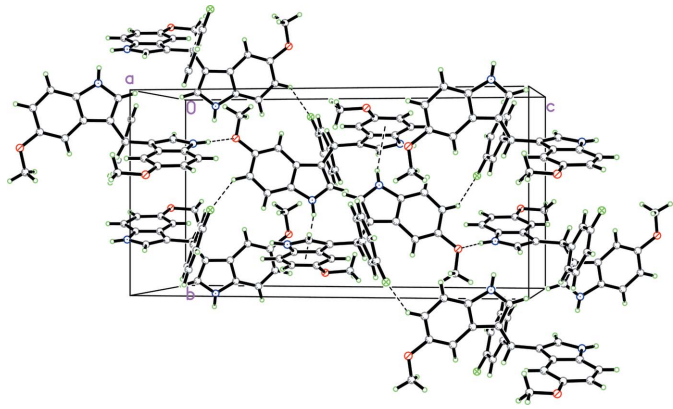


Figure 2
The crystal structure of (I). Dashed lines indicate N—H···O, C—H···F and N—H···π contacts.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.86	2.16	2.946 (3)	152
C3—H3A···F^{ii}	0.93	2.51	3.264 (4)	138
N1—H1A···Cg1	0.86	2.46	3.277 (2)	159

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

All H atoms were placed in idealized positions and refined as riding, with C—H = 0.93–0.98 Å and N—H = 0.86 Å, and with U_{iso}(H) = xU_{eq}(parent atom), where x = 1.5 for methyl H and 1.2 for all other H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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