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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.061
 wR factor = 0.192
Data-to-parameter ratio = 14.1

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

5,5'-Dimethoxy-3,3'-(3-nitrophenylmethanediyl)- bis(1*H*-indole)

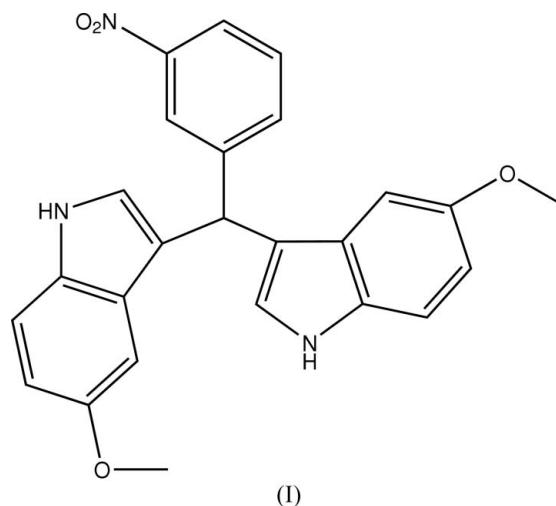
The title compound, $\text{C}_{25}\text{H}_{21}\text{N}_3\text{O}_4$, was synthesized by the reaction of 5-methoxy-1*H*-indole and 3-nitrobenzaldehyde in ethanol using CuBr_2 as a catalyst under microwave irradiation. In the crystal structure, there is one intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond and one intermolecular $\text{N}-\text{H}\cdots\pi$ contact.

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Comment

Development of bis(indolyl)alkane synthesis has been of considerable interest in organic synthesis 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.



The molecular structure of the title compound, (I), is shown in Fig. 1. Geometric parameters are unexceptional. The nitro group and one of the methoxy groups are almost coplanar with the aromatic ring to which they are attached, while the other methoxy group is significantly twisted out of the plane of the aromatic ring (Table 1). The crystal structure is stabilized by one intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond and one intermolecular $\text{N}-\text{H}\cdots\pi$ contact (Table 2).

Experimental

The title compound was prepared by the reaction of 5-methoxy-1*H*-indole (20 mmol) with 3-nitrobenzaldehyde (10 mmol) in ethanol (5 ml), using CuBr_2 (0.446 g) as a catalyst under microwave irradiation for 10 min. After completion, the reaction mixture was quenched with H_2O (10 ml) and extracted with EtOAc (3×10 ml). The combined organic layers were then dried over Na_2SO_4 , concentrated, and purified by column chromatography on SiO_2 (ethyl acetate–petroleum, 1:3) to afford the pure product (m.p. 452–453 K). Crystals

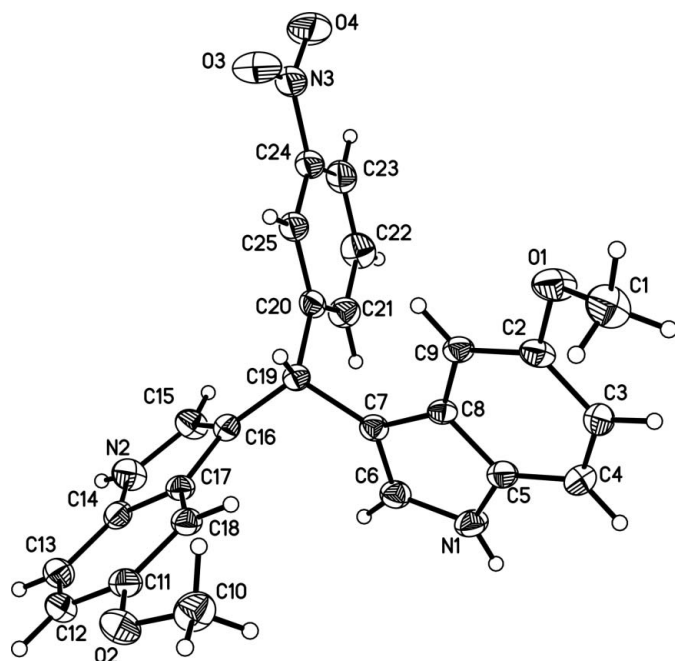


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

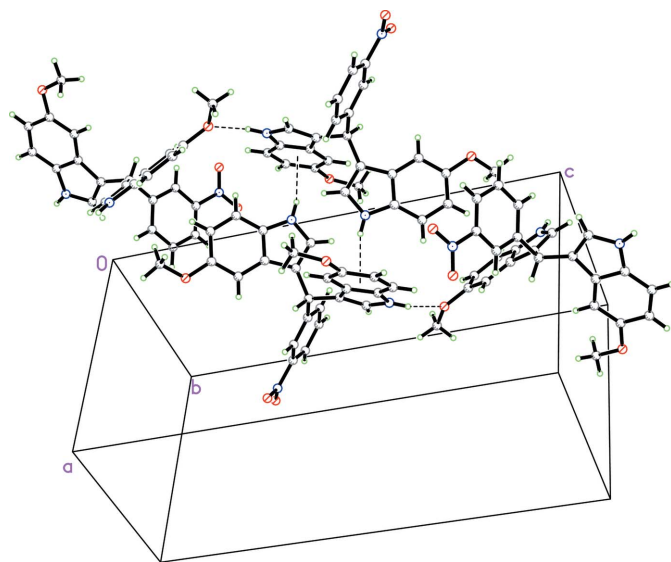


Figure 2
Partial packing diagram of the title compound. Dashed lines indicate N—H...O and N—H... π contacts.

of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{25}H_{21}N_3O_4$
 $M_r = 427.45$
Monoclinic, $P2_1/c$
 $a = 10.631$ (2) Å
 $b = 9.3570$ (19) Å
 $c = 21.072$ (4) Å
 $\beta = 92.09$ (3)°
 $V = 2094.7$ (7) Å³

$Z = 4$
 $D_x = 1.355$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
Block, yellow
0.40 × 0.30 × 0.10 mm

Data collection

Enraf-Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.964$, $T_{\max} = 0.991$
4324 measured reflections

4095 independent reflections
2615 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\max} = 26.0^\circ$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.192$
 $S = 1.04$
4095 reflections
290 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 1.7P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0103 (17)

Table 1

Selected torsion angles (°).

C1—O1—C2—C9	145.9 (3)	O4—N3—C24—C23	−3.2 (5)
C10—O2—C11—C12	173.5 (3)	O3—N3—C24—C25	−4.5 (5)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A...O1 ⁱ	0.86	2.12	2.900 (4)	151
N1—H1A...Cg1 ⁱⁱ	0.86	2.46	3.277 (2)	160

Symmetry codes: (i) $x, -y + \frac{5}{2}, z + \frac{1}{2}$; (ii) $-x, -y + 2, -z + 1$. Cg1 is the centroid of the C11–14, C17, C18 ring.

All H atoms were placed in idealized positions and refined as riding, with C—H = 0.93–0.98 Å, N—H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$.

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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