8-(4-Methoxyphenyl)-3,5-bis[(*E*)-1-(4-methoxy-phenyl)methylidene]-1,2,3,5,6,7-hexahydro-dicyclopenta[*b*,e]pyridine

Jonathan D. Crane* and John Crompton

Department of Chemistry, University of Hull, Cottingham Road, Kingston-upon-Hull HU6 7RX, England

Correspondence e-mail: j.d.crane@hull.ac.uk

Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.055
*wR* factor = 0.144
Data-to-parameter ratio = 25.6

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

At 150 K, the tricyclic core of the title compound, $C_{34}H_{31}NO_3$, is close to planar. One of the three 4-methoxyphenyl substituents is twisted out of the plane of the tricyclic core due to steric congestion.

Comment

The title compound, (I) (Fig. 1), is observed to be mildly fluorescent in hydrocarbon solvents due to the extended conjugation of the pyridine and 4-methoxyphenyl groups.

(I)
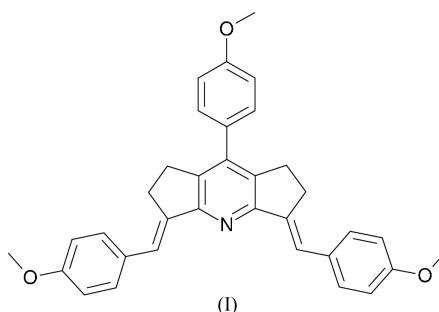
In the solid state, the central tricyclic core is close to planar, with a maximum deviation from the least squares plane of 0.1454 (19) Å for the methylene atom C10. The angles between the least-squares planes of the pyridine ring and the three 4-methoxyphenyl groups are 43.65 (5), 12.56 (5) and 18.24 (5)°, respectively. Thus, steric congestion causes a substantial twist of the central unique 4-methoxyphenyl group, but packing effects also bend this group out of the plane of the
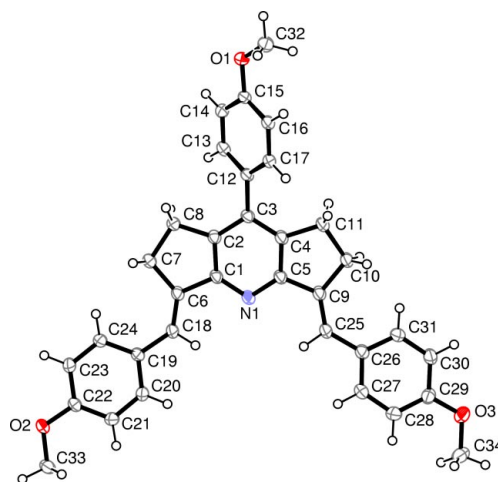

Figure 1
View of the molecule of (I), showing the atom-labelling scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitary size.

molecule (Fig. 2). The molecules pack in layers (Fig. 3) and these out-of-plane 4-methoxyphenyl groups occupy the gaps in the neighbouring layers.

Experimental

The title compound, (I), was prepared by the method of Kneeland *et al.* (1993). Suitable crystals were grown by recrystallization from toluene. Spectroscopic analysis, IR (KBr disk, ν cm^{-1}): 2835 (*w*), 1605 (*m*), 1510 (*s*), 1291 (*m*), 1251 (*s*), 1238 (*m*), 1176 (*m*), 1035 (*m*), 822 (*w*), 807 (*w*), 590 (*w*), 528 (*m*); ^{1}H NMR (CDCl$_3$, p.p.m.): 7.64 (*br s*, 2H), 7.54 (*d*, 4H), 7.31 (*d*, 2H), 6.99 (*d*, 2H), 6.94 (*d*, 4H), 3.87 (*s*, 3H), 3.84 (*s*, 6H), 3.12 (*m*, 4H), 2.96 (*m*, 4H); ^{13}C NMR (CDCl$_3$, p.p.m.): 161.0, 159.3, 158.4, 143.3, 139.5, 136.3, 131.0, 130.4, 129.5, 121.4, 113.9, 55.3, 29.4, 27.9; analysis calculated for C$_{34}$H$_{31}$NO$_3$: C 81.41, H 6.23, N 2.79%; found: C 81.15, H 6.31, N 2.87%.

Crystal data

C$_{34}$H$_{31}$NO$_3$	$Z = 2$
$M_r = 501.60$	$D_x = 1.320$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 5.8866$ (7) Å	Cell parameters from 13469
$b = 10.8186$ (12) Å	reflections
$c = 20.898$ (2) Å	$\theta = 2.0$–32.3°
$\alpha = 75.096$ (9)°	$\mu = 0.08$ mm^{-1}
$\beta = 85.572$ (10)°	$T = 150$ (2) K
$\gamma = 78.964$ (10)°	Needle, yellow
$V = 1261.8$ (2) Å^3	$0.60 \times 0.20 \times 0.20$ mm

Data collection

Stoe IPDS–II area-detector	$R_{\mathrm{int}} = 0.086$
diffractometer	$\theta_{\max} = 32.3°$
ω scans	$h = -7 \rightarrow 8$
27 584 measured reflections	$k = -16 \rightarrow 16$
8874 independent reflections	$l = -31 \rightarrow 31$
4469 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.055$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.144$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 0.92$	$\Delta\rho_{\max} = 0.33$ e Å^{-3}
8874 reflections	$\Delta\rho_{\min} = -0.28$ e Å^{-3}
347 parameters	Extinction correction: *SHELXL*97
H-atom parameters constrained	Extinction coefficient: 0.025 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.343 (2)	C4—C5	1.401 (2)
N1—C1	1.344 (2)	C5—C9	1.478 (2)
C1—C2	1.400 (2)	C6—C18	1.336 (2)
C1—C6	1.481 (2)	C9—C25	1.339 (3)
C2—C3	1.405 (2)	C18—C19	1.470 (2)
C3—C4	1.401 (2)	C25—C26	1.467 (2)
C3—C12	1.490 (2)		
C5—N1—C1	114.00 (14)	N1—C5—C4	124.88 (15)
N1—C1—C2	125.69 (14)	N1—C5—C9	124.14 (15)
N1—C1—C6	123.32 (15)	C18—C6—C1	121.67 (15)
C1—C2—C3	119.65 (15)	C25—C9—C5	122.91 (14)
C4—C3—C2	114.97 (14)	C6—C18—C19	132.16 (15)
C5—C4—C3	120.56 (14)	C9—C25—C26	130.15 (15)
C1—C6—C18—C19	173.69 (17)	C5—C9—C25—C26	176.12 (17)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—
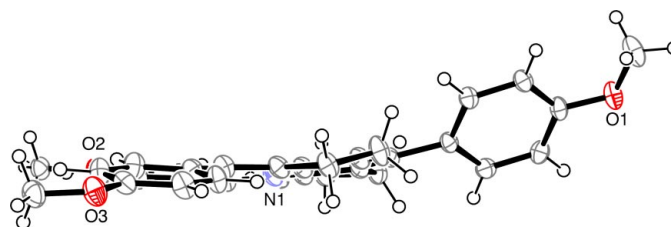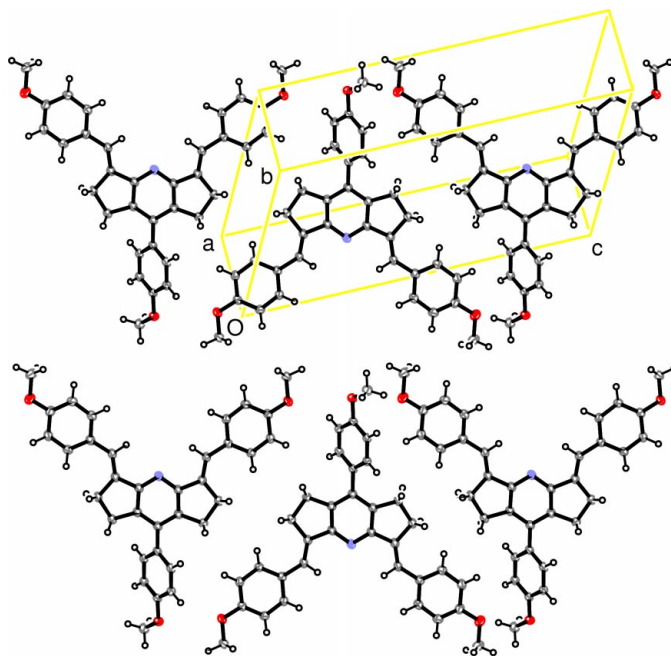

Figure 2
Side view of (I).

Figure 3
The molecular packing of one layer of (I).

H distances of 0.98 Å, but each group was allowed to rotate freely about its X—C bond. All other H atoms were placed in geometrically idealized positions, with C—H distances of 0.95–0.9 Å. U_{iso}(H) was set to $1.2U_{\mathrm{eq}}$(C) for all H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *X-STEP*32 (Stoe & Cie, 2001) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *WinGX* (Farrugia, 1999) and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX*.

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kneeland, D. M., Ariga, K., Lynch, V. M., Huang, C. Y. & Anslyn, E. V. (1993). *J. Am. Chem. Soc.* **115**, 10042–10055.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Stoe & Cie (2001). *X-AREA*, *X-RED* and *XSTEP*32. Stoe and Cie GmbH, Darmstadt, Germany.