

2-(4-Bromophenyl)-1-methyl-1*H*-phenanthro-
[9,10-*d*]imidazoleChristian B. Nielsen,^{a*}
Michael Pittelkow^b and
Henning O. Sørensen^c^aThe Danish Polymer Center, Risø National Laboratory, PO Box 49, DK-4000 Roskilde, Denmark, ^bUniversity of Copenhagen, Department of Chemistry, Universitetsparken 5, 2100 Copenhagen, Denmark, and ^cCenter for Fundamental Research: Metal Structures in Four Dimensions, Risø National Laboratory, PO Box 49, DK-4000 Roskilde, DenmarkCorrespondence e-mail:
christian.nielsen@risoe.dk

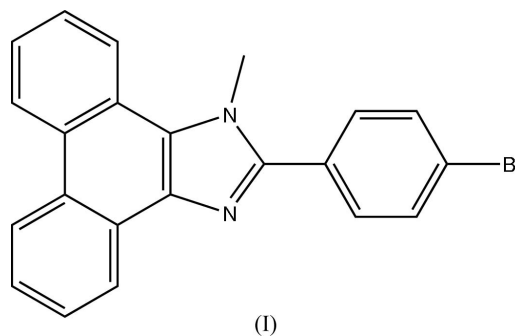
Key indicators

Single-crystal X-ray study
 $T = 122$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.024
 wR factor = 0.057
Data-to-parameter ratio = 22.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{22}\text{H}_{15}\text{BrN}_2$, the phenanthrene moiety is slightly skewed. The dihedral angle between the phenanthro[9,10-*d*]imidazole mean plane and the benzene ring is 37.78 (6°). The crystal packing is stabilized mainly by aromatic interactions, though a weak intermolecular $\text{C}-\text{H}\cdots\text{N}$ hydrogen bond is also observed.

Comment

The title compound, (I) (Fig. 1), was prepared for use as a building block in the syntheses of oligo-phenylene vinylenes for non-linear optical studies. In (I), the imidazole ring is slightly bent from the skewed phenanthrene ring, with atom C16 located 0.092 (2) Å out of the least-squares plane of the phenanthro[9,10-*d*]imidazole system. The dihedral angle between the phenanthro[9,10-*d*]imidazole mean plane and the benzene ring is 37.78 (6°). This rotation is most probably due to the adjacent *N*-methyl group.



The crystal packing (Fig. 2) is stabilized mainly by aromatic interactions. A herring-bone pattern is formed by the phenanthrene rings at $y = \frac{1}{4}$ and $\frac{3}{4}$, along with stacking of bromobenzene rings in the planes at $y = 0$ and $\frac{1}{2}$. A weak intermolecular $\text{C}-\text{H}\cdots\text{N}$ hydrogen bond (Table 1) is observed in the crystal structure.

Experimental

The title compound was prepared in accordance with a known procedure (Krebs & Jørgensen, 2001).

Crystal data

 $\text{C}_{22}\text{H}_{15}\text{BrN}_2$
 $M_r = 387.27$
Orthorhombic, $Pna2_1$
 $a = 6.0163$ (4) Å
 $b = 29.332$ (3) Å
 $c = 9.1726$ (8) Å
 $V = 1618.7$ (2) Å³
 $Z = 4$
 $D_x = 1.589$ Mg m⁻³Mo $K\alpha$ radiation
Cell parameters from 26555 reflections
 $\theta = 2.3$ – 31.0°
 $\mu = 2.55$ mm⁻¹
 $T = 122$ (1) K
Plate, pale yellow
 $0.55 \times 0.46 \times 0.08$ mm

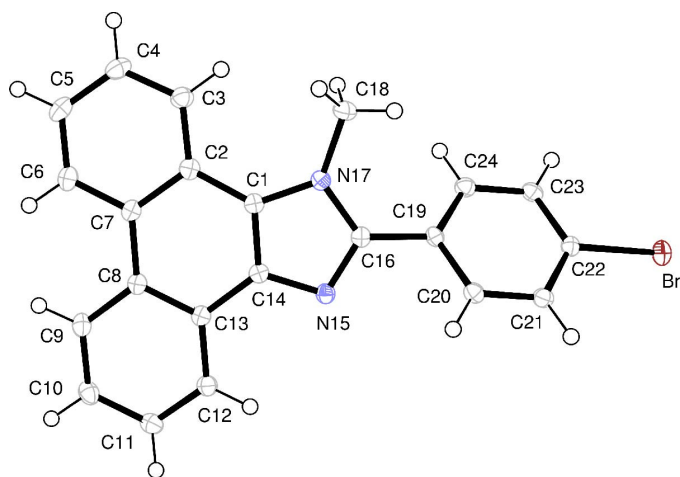


Figure 1
View of (I), with displacement ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

Data collection

Nonius KappaCCD diffractometer ω and φ scans	4771 reflections with $I > 2\sigma(I)$
Absorption correction: Gaussian integration (Coppens, 1970)	$R_{\text{int}} = 0.046$
$T_{\text{min}} = 0.365$, $T_{\text{max}} = 0.903$	$\theta_{\text{max}} = 31.0^\circ$
36851 measured reflections	$h = -8 \rightarrow 8$
5137 independent reflections	$k = -42 \rightarrow 42$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 0.9474P]$
$R[F^2 > 2\sigma(F^2)] = 0.024$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.057$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.45 \text{ e } \text{\AA}^{-3}$
5137 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e } \text{\AA}^{-3}$
226 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	2414 Friedel reflections
	Flack parameter = $-0.009(6)$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C23—H23 \cdots N15 ⁱ	0.95	2.54	3.419 (2)	154

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

All H atoms were located in a difference Fourier map and refined using a riding model, with fixed individual displacement parameters set at 1.2–1.5 times U_{eq} of the parent atom ($C-H = 0.95\text{--}0.98 \text{ \AA}$).

Data collection: COLLECT (Nonius, 1999); cell refinement: DIRAX (Duisenberg, 1992); data reduction: EvalCCD (Duisenberg *et al.*, 2003); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: SHELXL97.

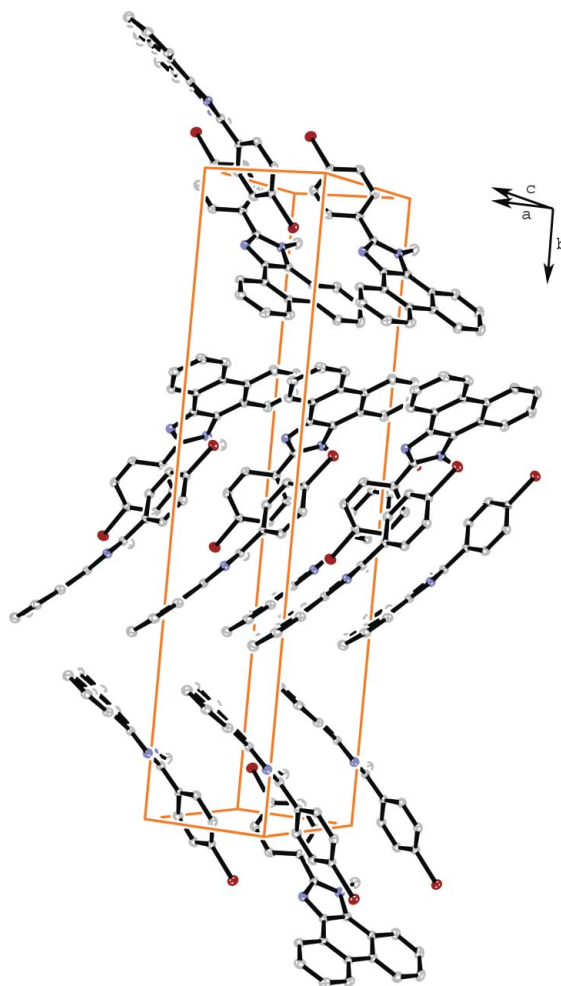


Figure 2
The crystal packing of (I). H atoms have been omitted for clarity.

The authors thank Flemming Hansen for collecting the diffraction data and the Centre for Crystallographic Studies for the use of their equipment. The work has been supported by the Danish National Research Foundation.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Coppens, P. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall and C. P. Huber, pp. 255–270. Copenhagen: Munksgaard.

Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.

Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.

Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.

Krebs, F. C. & Jørgensen, M. (2001). *J. Org. Chem.* **66**, 6169–6173.

Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.