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3-(Anthracen-9-ylmethylene)-2,4-dimethyl-3*H*-benzo[1,5]diazepine

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.123
Data-to-parameter ratio = 10.4

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

The title compound, $C_{26}H_{20}N_2$, crystallizes with two almost identical molecules in the asymmetric unit. The diazepine ring adopts a boat conformation.

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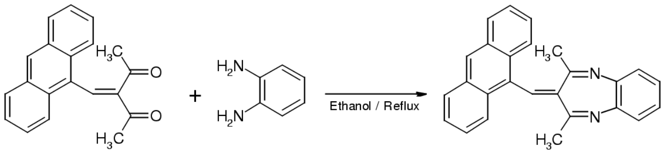
Comment

Charge-transfer processes are fundamental in photosynthesis. They are important in the solar field, as well as in biochemical reactions. We synthesized a new donor–acceptor system based on anthracene connected to the acceptor by a double bond (Elazami *et al.*, 1999; Senhaji *et al.*, 2001), as is the case with 1,4-benzodiazepine (Tucker & Count, 1996). Our focus is the study of different intramolecular interactions in this system, using the techniques of absorption and fluorescence spectroscopy. The synthesis of the title compound, (I), was performed by reacting 3-(anthracen-9-ylmethylene)pentane-2,4-dione and 1,2-diaminobenzene in ethanol (see Scheme).



(I)

Compound (I) has a high stability at room temperature, and its structure has been determined by IR, MS and NMR (^{1}H and ^{13}C) spectroscopy. Since these techniques did not provide sufficient information about the conformation of the reaction product, we have carried out the X-ray structure analysis. (I) crystallizes with two almost identical molecules in the asymmetric unit. A least-squares fit of all non-H atoms of the two molecules gives an r.m.s. deviation of 0.075 Å. The diazepine ring adopts a boat conformation, with the two N atoms and atoms C1 and C8 coplanar. Atom C9 is the bow atom and C2 and C7 are the stern atoms.

Experimental

Equimolar quantities of 3-(anthracen-9-ylmethylene)pentane-2,4-dione (2.88 g, 10 mmol) and benzene-1,2-diamine (1.1 g, 10 mmol) in 30 ml of ethanol were added to a 100 ml three-necked flask fitted with a reflux condenser. The mixture was stirred and refluxed for 12 h. The solution was filtered and extracted with diethyl ether (50 ml). The solvent was removed using a rotary evaporator. The residue was recrystallized from methanol, leading to yellow crystals of the title compound, (I).

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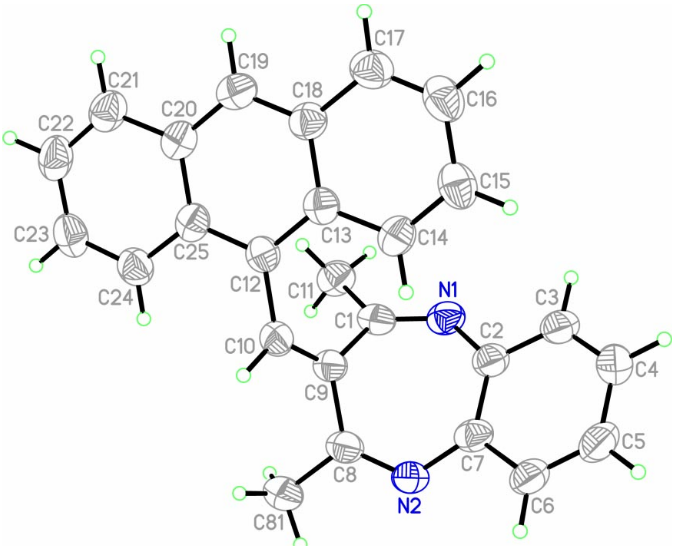

Figure 1
Perspective view of molecule 1 of the asymmetric unit, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

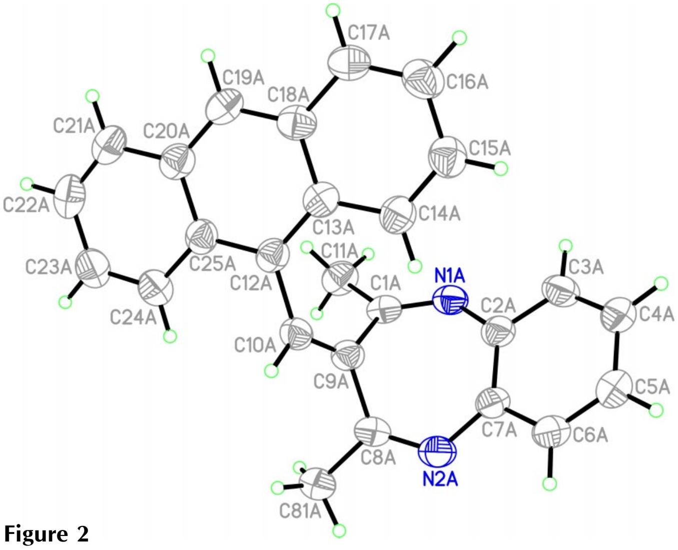

Figure 2
Perspective view of molecule 2 of the asymmetric unit, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

Crystal data

$C_{26}H_{20}N_2$
$M_r = 360.44$
Orthorhombic, $Pca2_1$
$a = 12.8014$ (9) Å
$b = 10.4027$ (8) Å
$c = 28.5030$ (19) Å
$V = 3795.7$ (5) Å^3
$Z = 8$
$D_x = 1.261$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24219 reflections
$\theta = 2.3$–29.5°
$\mu = 0.07$ mm^{-1}
$T = 173$ (2) K
Block, yellow
0.48 × 0.42 × 0.36 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: none
31816 measured reflections
5318 independent reflections
3581 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.072$
$\theta_{\rm max} = 29.5°$
$h = -17 \rightarrow 17$
$k = -14 \rightarrow 14$
$l = -39 \rightarrow 39$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.123$
$S = 0.94$
5318 reflections
509 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.266 (4)	C1*A*—C9*A*	1.497 (4)
N1—C2	1.417 (4)	N1*A*—C2*A*	1.409 (4)
C2—C7	1.411 (4)	C2*A*—C7*A*	1.417 (4)
C7—N2	1.416 (4)	C7*A*—N2*A*	1.417 (4)
N2—C8	1.291 (4)	N2*A*—C8*A*	1.267 (4)
C8—C9	1.491 (4)	C8*A*—C9*A*	1.504 (4)
C1*A*—N1*A*	1.282 (4)		
C1—N1—C2—C7	38.7 (5)	C1*A*—N1*A*—C2*A*—C7*A*	38.0 (4)
C2—C7—N2—C8	−39.3 (5)	C2*A*—C7*A*—N2*A*—C8*A*	−40.7 (5)
N1—C1—C9—C8	−62.4 (4)	N1*A*—C1*A*—C9*A*—C8*A*	−61.5 (4)
N2—C8—C9—C1	62.1 (4)	N2*A*—C8*A*—C9*A*—C1*A*	61.9 (4)

H atoms were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm C_{methyl}})$] using a riding model, with C—H = 0.95 Å or methyl C—H = 0.98 Å. The methyl groups were allowed to rotate but not to tip. (I) crystallized in the polar space group $Pna2_1$; however, in the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Accordingly, Friedel pairs were merged.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

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