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(Anthracen-9-ylmethyl)diethylamine at 100 K

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean *σ*(C–C) = 0.001 Å
R factor = 0.048
wR factor = 0.137
Data-to-parameter ratio = 26.2

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

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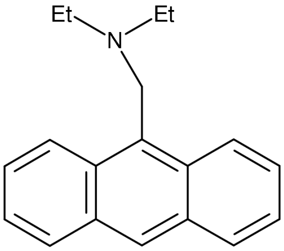
The molecular geometry in the title compound, $C_{19}H_{21}N$, is as expected for a compound of this kind. The molecules are interconnected to form layers by C—H⋯π interactions, some between edge-to-face molecules in the manner characteristic of polycyclic hydrocarbon compounds and others involving H atoms of both methyl groups of the methyldiethylamine substituent.

Comment

Interest in compounds capable of functioning as molecular devices has grown rapidly in the past fifteen years (Desilva *et al.*, 1997). A fundamental requirement of these systems is the ability to communicate information to the user by some appropriate means. Photo-induced electron transfer or PET-based systems, which use fluorescence emission as a method to transmit relevant data, have been employed in a variety of circumstances. Indeed, PET molecules capable of indicating the presence of cations, anions and neutral molecules have been reported (Desilva *et al.*, 1993). The mechanism by which this particular family of sensor molecules functions hinges on a 'box' or modular logic approach (Bissell *et al.*, 1993). We have previously reported the structure of a PET fluorescent saccharide sensor (Barkhuizen *et al.*, 2004) based on a molecule originally synthesized by Shinkai and his group (James *et al.*, 1995). The title compound, (I), presented here is representative of a subgroup of molecules designed to operate as single molecule pH PET sensors based on the same modular logic approach. Compound (I) alters its fluorescence emission intensity as a function of the pH of its environment in aqueous methanol solutions (Desilva & Rupasinghe, 1985).



(I)

The molecule of (I) is shown in Fig. 1. Selected bond lengths and angles, primarily for the methyldiethylamine substituent, are given in Table 1. These, along with C—C distances and internal angles in the anthracene fragment in the ranges, respectively, of 1.3588 (14)–1.4451 (13) Å and 116.86 (8)–121.39 (9)°, are as expected for this type of molecule. The anthracene moiety is essentially planar with an r.m.s. dis-

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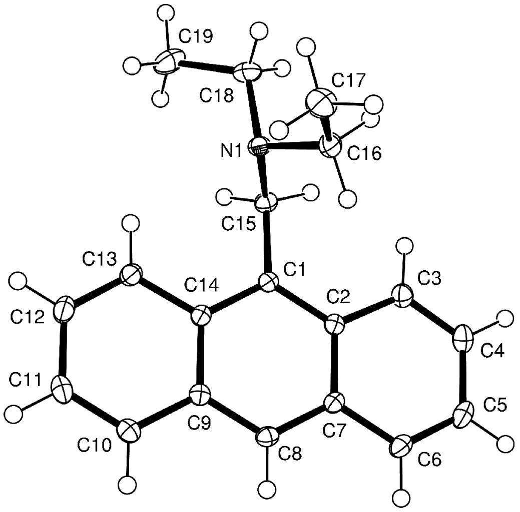

Figure 1
The molecule of (I). Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms as small spheres of arbitrary radii.

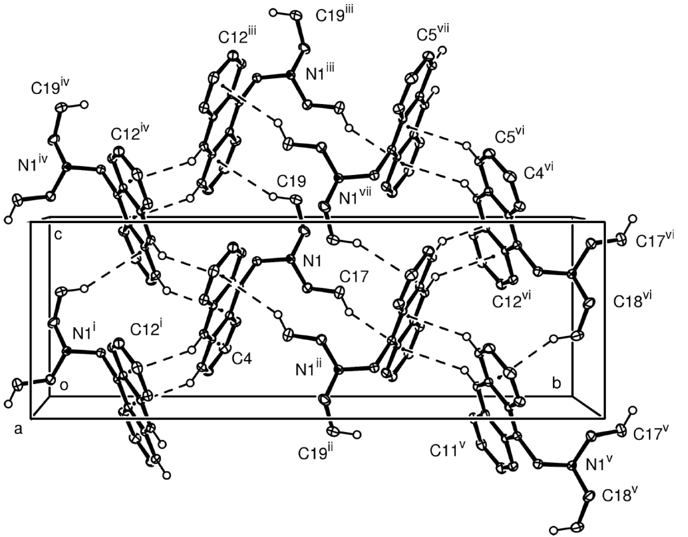

Figure 2
The packing of (I). Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms as small spheres of arbitrary radii. Dashed lines represent C—H$\cdots\pi$ contacts. Selected atoms are labelled. [Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $x, y, 1 + z$; (iv) (iv) $x, \frac{1}{2} - y, z + \frac{1}{2}$; (v) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vi) $1 - x, \frac{1}{2} - y, \frac{3}{2} - z$; (vii) $1 - x, 1 - y, 2 - z$.]

placement for the atoms (C1–C14) which define it of 0.0234 Å. The largest displacement is that of C4 at 0.0380 (8) Å, followed by that of C7 at 0.0355 (8) Å. The displacements from the anthracene least-squares plane of atoms C15, N1, C16, C17, C18 and C19 are −0.0534 (12), 1.2190 (13), 2.2643 (15), 3.6724 (17), 1.0212 (17) and 0.2988 (18) Å, respectively. That is, as expected, atom C15 is more or less in the plane of the ring system and N1 displaced from it. The atoms of the ethyl group defined by C16–C17 are still further displaced, in the same sense as N1, while the ethyl group defined by C18–C19 is directed back towards the plane of the ring system. The torsion angles around the C15—N1 bond given in Table 1 are entirely consistent with this interpretation. The intermolecular C—H$\cdots\pi$ contacts given in Table 2 and shown in Fig. 2 interconnect the molecules to form layers parallel to (100). The layers are related to one another purely by cell translation in the direction of *a*.

Experimental

Compound (I) was synthesized according to the procedure of Atkinson *et al.* (1973). 9-Chloromethylanthracene (0.52 g, 2.3 mmol) and diethylamine (0.34 g, 4.7 mmol) were added to a solution of triethylamine (1.1 g, 11 mmol) in anhydrous dichloromethane (50 ml). The resulting solution was refluxed overnight, allowed to cool and washed with water (3 × 50 ml). The organic layer was retained, dried over anhydrous sodium sulfate and the solvent evaporated under reduced pressure. The residue was chromatographed on silica with an ethyl acetate/hexane mixture (1:3) as eluant. The resulting yellow solid was recrystallized from methanol affording 0.16 g (47%) of the product. Needle-shaped crystals of (I) were grown from methanol in a refrigerator at 283 K (m.p. 360–361 K). ^{1}H NMR (CDCl$_3$, 300 MHz): δ 1.07 (*t*, J = 7.0 Hz, 6H), 2.60 (*q*, J = 7.0 Hz, 4H), 4.48 (*s*, 2H), 7.41–7.51 (*m*, 4H), 7.96 (*AB, JAB* = 8.0 Hz, 2 H), 7.99 (*AB, JAB* = 8.0 Hz, 2H), 8.38 (*s*, 1H), 8.52 (*AB, JAB* = 9.0 Hz, 2H), 8.55 (*AB, JAB* = 8.0 Hz, 2H).

Crystal data

$C_{19}H_{21}N$
$M_r = 263.37$
Monoclinic, $P2_1/c$
$a = 8.523$ (1) Å
$b = 22.678$ (5) Å
$c = 7.769$ (2) Å
$\beta = 93.388$ (15)°
$V = 1499.0$ (5) Å^3
$Z = 4$

$D_x = 1.167$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2017 reflections
$\theta = 4$–32°
$\mu = 0.07$ mm^{-1}
$T = 100$ (2) K
Cut prism, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur2 area-detector diffractometer
ω–2θ scans
Absorption correction: none
14548 measured reflections
4796 independent reflections

3585 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\rm max} = 31.9°$
$h = -12 \rightarrow 12$
$k = -33 \rightarrow 31$
$l = -11 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.137$
$S = 1.06$
4796 reflections
183 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C15	1.4738 (12)	C1—C15	1.5201 (13)
N1—C16	1.4753 (13)	C16—C17	1.5183 (15)
N1—C18	1.4782 (12)	C18—C19	1.5215 (15)
C15—N1—C16	110.27 (7)	C2—C1—C15	121.86 (8)
C15—N1—C18	109.24 (8)	N1—C15—C1	113.06 (7)
C16—N1—C18	110.22 (8)	N1—C16—C17	113.32 (8)
C14—C1—C15	118.57 (8)	N1—C18—C19	113.92 (9)
C2—C1—C15—N1	−107.99 (9)	C1—C15—N1—C18	−172.35 (8)
C14—C1—C15—N1	73.42 (10)	C15—N1—C16—C17	−162.17 (8)
C1—C15—N1—C16	66.38 (10)	C15—N1—C18—C19	76.45 (11)

Table 2
Geometry (Å, °) of C—H···π contacts in (I).

C—H···*Cg*[a]	C—H	H···*Cg*	H_{perp}[b]	γ[c]	C—H···*Cg*	C···*Cg*
C6—H6···$Cg1^{i}$	0.95	2.58	2.56	7	145.56	3.405
C8—H8···$Cg3^{i}$	0.95	2.77	2.74	9	145.74	3.595
C17—H17*B*···$Cg3^{ii}$	0.95	2.78	2.76	8	138.84	3.581
C19—H19*A*···$Cg2^{iii}$	0.95	2.91	2.89	6	150.72	3.795

Notes: (*a*) *Cg*(*n*), $n = 1$ to 3, are the centroids of rings defined by C1–C2/C7–C9/C14, C2–C7 and C9–C14, respectively; (*b*) H_{perp} is the perpendicular distance of the H atom from the mean plane of the ring; (*c*) γ is the angle at hydrogen between H···*Cg* and H_{perp}. Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $x, y, 1 + z$.

In the final stages of refinement, H atoms were introduced in calculated positions, with C—H distances of 0.95, 0.99 and 0.98 Å for aryl, methylene and methyl H atoms, respectively, and refined using a riding model, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $U_{iso}(H) = 1.2U_{eq}(C)$ otherwise. The orientation of the rigid body methyl groups was also refined.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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