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14-*n*-Butyldibenz[*a,h*]acridine

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Key indicators

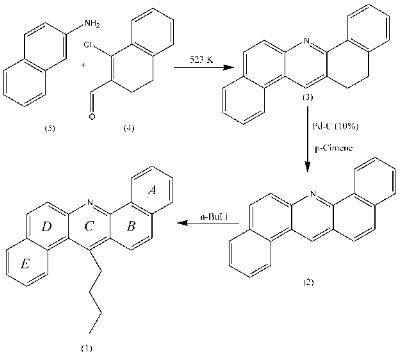
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.167
Data-to-parameter ratio = 19.2

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

The crystal structure of the monoclinic form of the title compound, $C_{25}H_{21}N$, is influenced by the presence of the alkyl chain, whose *trans* conformation leads to distortion of the aromatic moiety from planarity, the dihedral angle between the two outer rings being *ca* 20°. The C—C—C ring angles and C—C ring distances nearest to the *n*-butyl substituent are perturbed from typical values to minimize the steric effect.

Comment

The use of alkylated acridine derivatives in molecular recognition has made enormous strides in recent years (Pan *et al.*, 2000). Regioselective alkylation of polycyclic benz- and dibenzacridines not only improves the solubility of these compounds in common organic solvents (Ray *et al.*, 1996), but also provides a simple path for the synthesis of alkylated molecular hosts (Haldar *et al.*, 1997).



Molecule (I) is composed of five (*A*, *B*, *C*, *D* and *E*) fused six-membered aromatic rings with one *n*-butyl chain attached to the central ring at the position *para* to the N atom (see *Scheme*). The conformation adopted by (I) in the solid state is affected by the presence of the alkyl chain at C3 (Fig. 1). C—C distances in the environment of the alkyl chain (C2—C3, C3—C4 and C4—C9) are rather similar to those found in monoclinic dibenz[*a,h*]anthracene (Robertson & White, 1956). On the other hand, the C2—C14 distance [1.470 (2) Å] is 0.08 Å larger than the equivalent distance in dibenz[*a,h*]anthracene. All ring angles are reasonably close to ideal geometry, but the presence of the alkyl chain increases the C3—C2—C14 angle [to 126.74 (15)°].

Rings *A* and *B* are reasonably planar, with mean deviations from planarity of 0.0024 and 0.0075 Å, respectively. On the other hand, rings *C*, *D* and *E* are less planar; the corresponding mean deviations are 0.0283, 0.0396 and 0.0278 Å, respectively, the main deviation from planarity taking place at the *D* ring.

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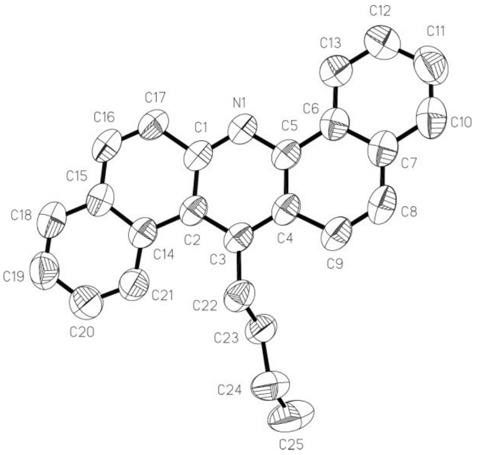

Figure 1
View of the title molecule, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

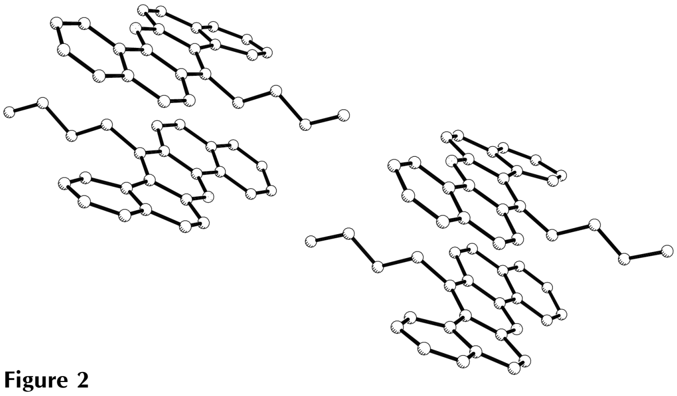

Figure 2
Reduced packing diagram of the title compound, showing the orientation of the alkyl chain. H atoms are omitted.

These values and the C1—C2—C3—C4 and C1—C2—C14—C15 torsion angles (Table 1) reflect the distortion of the molecule in order to minimize the steric interaction between ring *E* and the alkyl chain. As expected on basic chemical grounds and as obtained using molecular mechanics calculations (*GAUSSIAN*98 suite of programs, UFF force field; Frisch *et al.*, 1998), the parent compound, without the alkyl chain, should be planar, whereas in this case the dihedral angle between rings *A* and *E* is 19.67 (10)°. The value of the C22—C23—C24—C25 torsion angle [−178.93 (18)°] implies a *trans* conformation for the alkyl chain. The steric interaction between the *n*-butyl substituent and the *E* ring results in a C3—C22—C23—C24 torsion angle of 169.42 (16)°.

Like dibenz[*a,h*]anthracene, the title compound has a monoclinic crystal structure. However, the presence of the alkyl chain completely changes the molecular packing, leading to a different space group and cell parameters (except for a similar *c* axis), with 8 instead of 2 molecules per unit cell. Within the three-dimensional network, it is noticeable that the alkyl chain of the one molecule is directed towards the N atom of the next molecule in the same layer (Fig. 2), thus making the packing more effective.

Experimental

In an attempt to synthesize monoalkylated polyazaarenes, we heated a mixture of 1-chloro-3,4-dihydronaphthalene-2-carbaldehyde, (4), with 3-naphthylamine, (5), to obtain dihydrodibenzacridine, which on dehydrogenation with Pd–C (10%) in *p*-cymene produced dihydrodibenz[*a,h*]acridine (Haldar *et al.*, 1989). The direct alkylation of dibenz[*a,h*]acridine with three equivalents of *n*-butyllithium produced the title compound in 70% yield (see *Scheme*). Single crystals were obtained by slow evaporation from a chloroform–petroleum ether solution.

Crystal data

$C_{25}H_{21}N$
$M_r = 335.43$
Monoclinic, $C2/c$
$a = 19.5507$ (11) Å
$b = 12.7195$ (7) Å
$c = 14.7732$ (8) Å
$\beta = 93.322$ (1)°
$V = 3667.6$ (4) Å^3
$Z = 8$

$D_x = 1.215$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10111 reflections
$\theta = 1.4$–28.4°
$\mu = 0.07$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.50 × 0.25 × 0.25 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.966$, $T_{max} = 0.983$
12785 measured reflections

4539 independent reflections
2208 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.3°$
$h = -25 \rightarrow 26$
$k = -14 \rightarrow 16$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.167$
$S = 0.99$
4539 reflections
236 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0845P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C3	1.421 (2)	C3—C4	1.412 (2)
C2—C14	1.470 (2)	C4—C9	1.451 (2)
N1—C1—C2	124.98 (14)	C21—C14—C15	115.86 (16)
C3—C2—C1	115.69 (15)	C21—C14—C2	125.33 (15)
C3—C2—C14	126.74 (15)	C3—C22—C23	115.11 (13)
C5—N1—C1—C2	−3.5 (2)	C3—C2—C14—C15	163.67 (14)
C5—N1—C1—C17	173.30 (13)	C1—C2—C14—C15	−11.4 (2)
N1—C1—C2—C3	8.1 (2)	C4—C3—C22—C23	−81.85 (18)
C17—C1—C2—C3	−168.54 (14)	C2—C3—C22—C23	103.20 (18)
C1—C2—C3—C22	168.36 (13)	C3—C22—C23—C24	169.42 (16)
C3—C2—C14—C21	−17.9 (2)	C22—C23—C24—C25	−178.93 (18)
C1—C2—C14—C21	166.99 (15)		

All H atoms were placed in geometrically calculated positions and refined as riding.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1997)); software used to prepare material for publication: *SHELXTL*.

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References

Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Zakrzewski, V. G., Montgomery, J. A., Stratmann, R. E., Burant, J. C., Dapprich, S., Millam, J. M., Daniels, A. D., Kudin, K. N., Strain, M. C., Farkas, O. *et al.* (1998). *GAUSSIAN*98. Gaussian Inc., Pittsburgh, PA, USA.

Haldar, M. K., Kar, G. K. & Ray, J. K. (1997). *Synlett*, pp. 1057–1058.

Haldar, M. K., Karmakar, A. C. & Ray, J. K. (1989). *Tetrahedron Lett.* **30**, 223–224.

Pan, D., Roy, B. C., Kar, G. K. & Ray, J. K. (2000). *J. Chem. Soc. Perkin Trans* 1, pp. 2171–2173.

Ray, J. K., Roy, B. C. & Kar, G. K. (1996). *J. Org. Chem.* **61**, 1863–1866.

Robertson, J. M. & White, J. G. (1956). *J. Chem. Soc.* pp. 925.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS*97, *SHELXL*97 and *SHELXTL*.. University of Göttingen, Germany.

Siemens (1995). *SMART* (Version 5.505) and *SAINT* (Version 4.0). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.