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9-(*tert*-Butylamino)acridine

Joanna Meszko,[a] Karol Krzymiński,[a] Antoni Konitz[b] and Jerzy Błażejowski[a]*

[a]University of Gdańsk, Faculty of Chemistry, J. Sobieskiego 18, 80-952 Gdańsk, Poland, and [b]Technical University of Gdańsk, Department of Inorganic Chemistry, G. Narutowicza 11/12, 80-952 Gdańsk, Poland

Correspondence e-mail: bla@chemik.chem.univ.gda.pl

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.129
Data-to-parameter ratio = 14.4

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

The crystal structure of 9-(*tert*-butylamino)acridine, $C_{17}H_{18}N_2$, is composed of molecules of the amino tautomeric form. The acridine moiety is slightly folded along the C···N line passing through opposite atoms of the central ring, and the orientation of the *tert*-butylamino group makes the conjugation between the lone pair of the N atom and the acridine π-system almost negligible. An intermolecular hydrogen bond, involving the exocyclic NH group as donor and the endocyclic N atom as acceptor, links the molecules into infinite chains stretching along the *b* axis of the crystal.

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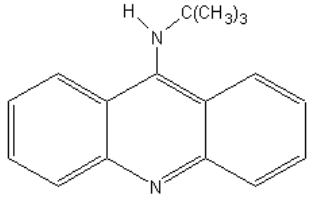
Comment

9-Aminoacridine in the gaseous and liquid phases can potentially exist in amino or imino tautomeric forms (Rak *et al.*, 1997), even though it has been established that only the amino tautomer is present in the crystalline phase (Chaudhuri, 1983). Theory also predicts the co-existence of tautomers in the case of 9-aminoacridines substituted at the exocyclic N atom (Rak *et al.*, 1998). It is thus interesting to determine which of the two forms exists in the crystal for any particular derivative. Among simple 9-aminoacridines, only the structure of 9-(phenylamino)acridine has been determined. Its crystals were found to be composed of molecules of the amino tautomeric form (Leardini *et al.*, 1998). We have determined the structures of 9-(dimethylamino)acridine (blocked amino form) and 9-(methylimino)-10-methylacridine (blocked imino form), which prove that 9-aminoacridine derivatives can be obtained, by synthesis, in both tautomeric forms, and both kinds of tautomers can indeed exist in the crystalline phase (Rak *et al.*, 1998). The aim of the present investigation was to check how far bulky substituents, such as *tert*-butyl, at the exocyclic N atom, affect the ability of 9-aminoacridines to exist and crystallize in either the amino or the imino form. 9-Aminoacridines are capable of specific interactions with surrounding molecules, and in some cases show distinctive biological activities, which most probably depend on the tautomeric form (Barbe *et al.*, 1996; Rak *et al.*, 1997). These considerations form the background to our investigations.



(I)

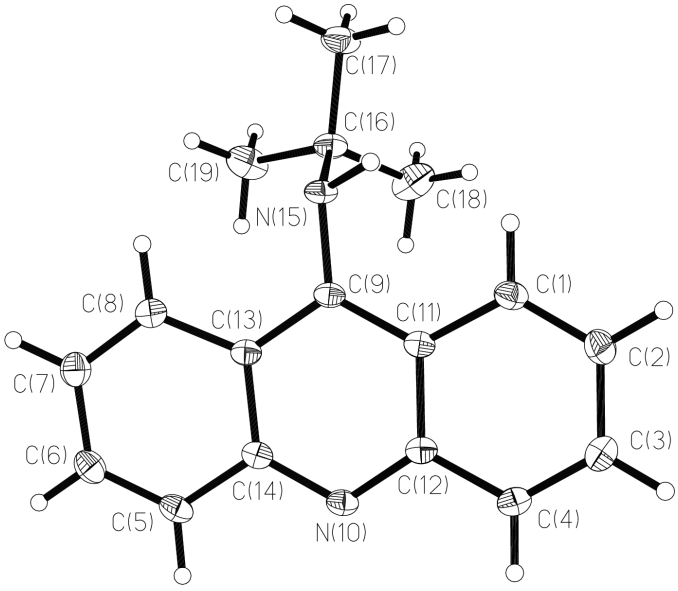


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

The present study has shown that the crystals of 9-(*tert*-butylamino)acridine, (I), are built of molecules in representing the amino tautomeric form (Fig. 1 and Table 1). The acridine moiety in (I) is slightly folded along the C9···N10 line, the dihedral angle between the mean planes of the benzene rings being 4.6 (1)°. The exocyclic C9—N15 bond is directed slightly towards the concave side of the acridine nucleus; the N10···C9—N15 angle is 175.9 (2)°, and the dihedral angle between the plane of C9, N15 and the nitrogen lone pair, and the mean plane of the acridine nucleus is 37°, which means that conjugation between the nitrogen lone pair and the acridine π-system is almost negligible.

There is one independent intermolecular hydrogen bond in the crystal, *viz*. N15—H15*A*···N10^{i} [symmetry code: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, z], which is responsible for the formation of infinite chains stretching along the *b* axis (Fig. 2 and Table 2); the dihedral angle formed by the acridine planes of two neighbouring molecules within the chain is 66.2 (2)°.

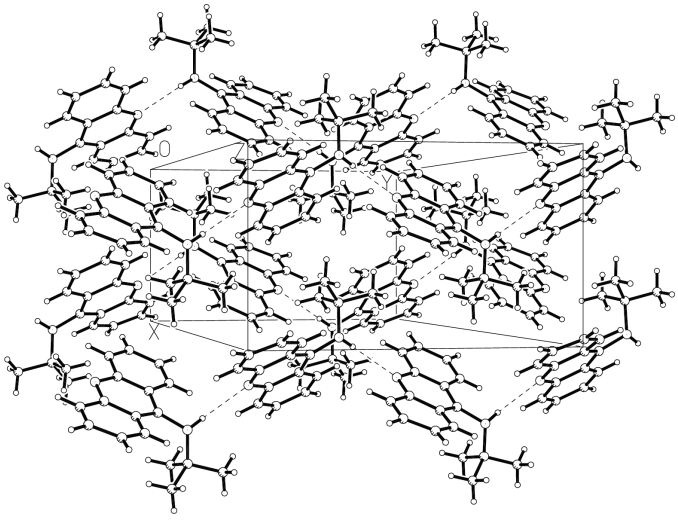


Figure 2
Packing diagram of (I), viewed along the *c* axis. Hydrogen bonds are represented by dashed lines.

Experimental

9-(*tert*-Butylamino)acridine was obtained by heating (2 h at 433 K) a mixture of 9-chloroacridine and *tert*-butylamine dissolved in phenol (Drozdov & Cherntzov, 1935*a*,*b*; Dupre & Robinson, 1945). The product was purified chromatographically and crystals suitable for an X-ray study were grown from cyclohexane.

Crystal data

$C_{17}H_{18}N_2$
M_r = 250.33
Orthorhombic, *Pbca*
a = 7.623 (3) Å
b = 12.762 (4) Å
c = 28.155 (7) Å
V = 2739.1 (15) Å^3
Z = 8
D_x = 1.214 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 50 reflections
θ = 1.4–25.5°
μ = 0.07 mm^{-1}
T = 293 (2) K
Block, yellow
0.6 × 0.4 × 0.3 mm

Data collection

Kuma KM-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
18864 measured reflections
2569 independent reflections
1817 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 25.6°
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 15$
$l = -34 \rightarrow 34$
3 standard reflections
every 150 reflections
intensity decay: 26.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.09
2569 reflections
179 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.3081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C9—C11	1.410 (3)	C11—C12	1.438 (3)
C9—C13	1.410 (3)	C13—C14	1.432 (3)
C9—N15	1.412 (2)	N15—H15*A*	0.93 (2)
N10—C12	1.346 (2)	N15—C16	1.506 (3)
N10—C14	1.346 (3)		
C9—N15—H15*A*	109.2 (14)	C11—C9—N15	122.89 (17)
C9—N15—C16	118.89 (15)	C13—C9—N15	119.26 (17)
C11—C9—C13	117.80 (17)	C12—N10—C14	117.62 (16)
C9—N15—H15*A*—C16	128.3 (7)	C11—C9—N15—C16	95.3 (2)
C11—C9—C13—C14	−8.7 (3)	C12—N10—C14—C13	1.7 (3)
C11—C9—N15—H15*A*	−24.6 (14)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N15—H15A···N10^{i}	0.93 (2)	2.38 (2)	3.244 (3)	154 (2)

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

The atom H15*A* (bonded to N15) was refined in the isotropic approximation; all other H atoms were riding on the carrier atoms, with their displacement parameters set to $1.2U_{eq}$ of the corresponding carrier atom ($1.5U_{eq}$ in the case of the methyl H atoms).

Data collection: *KM*-4 *Software* (Kuma Diffraction, 1989); cell refinement: *KM*-4 *Software*; data reduction: *KM*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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