C(3)	0.429 (1)	0.754 (1)	0.2515 (9)	0.055 (2)
C(4)	0.229 (1)	0.507 (1)	0.2987 (9)	0.066 (2)
C(5)	0.249 (2)	0.353 (1)	0.344 (1)	0.071 (3)
C(6)	0.421 (1)	0.287 (1)	0.384 (1)	0.070 (3)
C(7)	0.588 (1)	0.362 (1)	0.3823 (8)	0.053 (2)
C(8)	0.570 (1)	0.516 (1)	0.3370 (9)	0.049 (2)
C(9)	0.399 (1)	0.585 (1)	0.2953 (8)	0.050 (2)
C(10)	0.693 (1)	0.737 (2)	0.1438 (7)	0.041 (2)
N(11)	0.704 (2)	0.862 (1)	0.0743 (9)	0.059 (3)
C(12)	0.735 (2)	0.813 (1)	−0.045 (1)	0.055 (3)
C(13)	0.757 (2)	0.634 (1)	−0.026 (1)	0.046 (2)
N(14)	0.719 (1)	0.611 (1)	0.087 (1)	0.051 (3)
C(15)	0.743 (2)	0.886 (2)	0.337 (1)	0.089 (3)
C(16)	0.957 (2)	0.893 (1)	0.359 (1)	0.091 (3)

Table 2. *Selected geometric parameters* (Å, °)

C(10)—N(14)	1.28 (2)	C(9)—C(3)	1.55 (2)
C(13)—N(14)	1.40 (2)	C(5)—C(4)	1.40 (2)
H(N14)—N(14)	0.94 (6)	C(6)—C(5)	1.34 (2)
C(10)—N(11)	1.34 (2)	C(7)—C(6)	1.36 (2)
C(12)—N(11)	1.49 (2)	C(7)—C(8)	1.40 (1)
H(N11)—N(11)	0.94 (4)	C(2)—C(3)	1.55 (1)
C(8)—O(1)	1.36 (2)	C(15)—C(2)	1.51 (3)
C(2)—O(1)	1.51 (2)	C(10)—C(2)	1.45 (1)
C(4)—C(5)	1.40 (2)	C(16)—C(15)	1.51 (3)
C(8)—C(9)	1.35 (1)	C(13)—C(12)	1.53 (2)
Br···H(N14)	2.54 (5)	Br···H(N11^{i})	2.25 (4)
O(1)···H(N14)	2.24 (7)		
C(10)—N(14)—C(13)	116 (1)	O(1)—C(8)—C(7)	122 (1)
C(10)—N(14)—H(N14)	110 (4)	C(9)—C(8)—C(7)	122 (1)
C(13)—N(14)—H(N14)	133 (4)	C(9)—C(3)—C(2)	100 (1)
C(10)—N(11)—C(12)	111 (1)	O(1)—C(2)—C(3)	108 (1)
C(10)—N(11)—H(N11)	134 (5)	O(1)—C(2)—C(15)	107 (1)
C(12)—N(11)—H(N11)	115 (5)	O(1)—C(2)—C(10)	105 (1)
C(2)—O(1)—C(8)	106 (1)	C(3)—C(2)—C(15)	110 (1)
C(4)—C(9)—C(8)	121 (1)	C(3)—C(2)—C(10)	112 (1)
C(4)—C(9)—C(3)	129 (1)	C(15)—C(2)—C(10)	115 (1)
C(8)—C(9)—C(3)	109 (1)	C(2)—C(15)—C(16)	116 (2)
C(9)—C(4)—C(5)	116 (1)	N(14)—C(10)—N(11)	109 (1)
C(4)—C(5)—C(6)	122 (1)	N(14)—C(10)—C(2)	128 (2)
C(5)—C(6)—C(7)	123 (1)	N(11)—C(10)—C(2)	123 (2)
C(6)—C(7)—C(8)	116 (1)	N(11)—C(12)—C(13)	100 (1)
O(1)—C(8)—C(9)	116 (1)	N(14)—C(13)—C(12)	103 (1)
Br···H(N14)—O(1)	116 (3)	Br···H(N14)—N(14)	133 (5)
O(1)···H(N14)—N(14)	110 (5)	Br···H(N11^{i})—N(11^{i})	170 (7)

Symmetry code: (i) $x, y - 1, z$.

Enraf–Nonius (1989) *CAD*-4 *Software* was used for data collection and reduction. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refinement was carried out using *SHELX*76 (Sheldrick, 1976) with anisotropic displacement parameters for Br, ethyl C, and N atoms. H atoms were included as fixed atoms in idealized positions with C_{sp^2}—H = 0.97 and C_{sp^3}—H = 0.98 Å. An attempt to freely refine H atoms attached to N atoms was not satisfactory; these H atoms could be fairly well positioned by constraining the N—H bond length to 0.94 Å, a value which has been found in histamine (Bonnet & Ibers, 1973) and in protonated histamine (Veidis, Palenik, Schaffin & Trotter, 1969). The ellipsoid plot was drawn using *ORTEP*II (Johnson, 1976); the packing diagram was obtained using *ATOMS* (Dowty, 1993). All computations were carried out on a Pentium 90 computer.

The authors thank Mr Serge Brunel for technical assistance.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1181). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,2,3,4,6,7-Hexahydro-10-chloronaphtho[3,2-*c*]acridine

Jayanta Kumar Ray and Manash Haldar

Department of Chemistry, Indian Institute of Technology, Kharagpur 721 302, India

Gur Dayal Nigam

Department of Physics and Meteorology, Indian Institute of Technology, Kharagpur 721 302, India

Kandasamy Sivakumar† and Hoong-Kun Fun*

X-ray Crystallography Laboratory, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

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Abstract

The title molecule, $C_{21}H_{18}ClN$, shows conformational flexibility; each of the two unsaturated rings present in the structure adopts two conformations, a major

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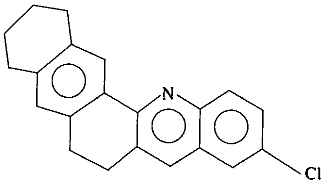
† On leave from: Department of Physics, Anna University, Madras 600 025, India.

component and a minor component. The molecule as a whole is almost planar. The crystal structure is stabilized by van der Waals interactions.

Comment

Monoaza polycyclic azaarenes are very important molecules for molecular recognition because of their unique structural features that help to hold the planar hosts in the cavity by weak van der Waals forces or hydrogen bonds. These molecules are also important for studying the role of the presence of 'bay regions' in a molecule towards carcinogenic activity (Kumar, 1985). We have synthesized one novel chloronaphthoacridine moiety, (I), belonging to this category. Since the geometry of the unsaturated 'cyclohexene' rings can be used to predict the site of action, the crystal structure was determined to evaluate the conformational features of the molecule.



(I)

The molecule as a whole is almost planar, with a tilt of 10.95 (3)° between the segments connected by the cyclohexene ring *C* comprising atoms C5, C6, C7, C8, C17 and C18. Both the cyclohexene rings present in the structure undergo conformational changes, as evidenced by the disorder present in the structure. In ring *C*, both the major and minor conformers are in half-chair conformations, but in opposite orientations. In the case of ring *E* (C10–C15), the major conformation is between sofa and half-chair [the asymmetry parameters (Nardelli, 1983*a*) are $\Delta C_S(C10) = 0.050\,(2)$ and $\Delta C_2(C10–C15) = 0.069\,(7)$], whereas the minor conformation is half-chair [$\Delta C_2(C10–C15) = 0.013\,(7)$]. The bond lengths and angles in the molecule are normal. The crystal structure is stabilized by van der Waals interactions.

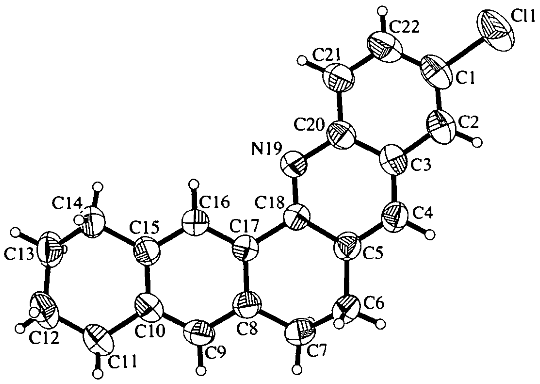


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids with the numbering scheme. For clarity, the minor-conformer positions are not shown.

Experimental

The compound was synthesized by the condensation of the chloroaldehyde, obtained from 1-keto-1,2,3,4,5,6,7,8-octahydroanthracene, with 4-chloroaniline, followed by regioselective thermal cyclization of the resulting aniline (Ramesh, Kar, Chatterjee & Ray, 1988). The single crystals were obtained by slow cooling from a chloroform and petroleum ether solvent system (333–353 K).

Crystal data

$C_{21}H_{18}ClN$
$M_r = 319.81$
Monoclinic
$P2_1/c$
$a = 12.119\,(1)$ Å
$b = 12.984\,(1)$ Å
$c = 10.335\,(1)$ Å
$\beta = 90.67\,(1)°$
$V = 1626.1\,(2)$ Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–25°$
$\mu = 0.234$ mm^{-1}
$T = 293\,(2)$ K
Transparent block
0.56 × 0.48 × 0.40 mm
Colourless

Data collection

Siemens *P*4 four-circle diffractometer
$\theta–2\theta$ scans
Absorption correction: none
4628 measured reflections
3675 independent reflections
2462 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0183$
$\theta_{\rm max} = 27.49°$
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 1$
$l = -1 \rightarrow 12$
3 standard reflections monitored every 100 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0454$
$wR(F^2) = 0.1278$
$S = 1.113$
3675 reflections
334 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.001$
$\Delta\rho_{\rm max} = 0.163$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.214$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl1	−0.02003 (4)	0.68458 (5)	0.68867 (6)	0.1009 (3)
C1	0.09996 (14)	0.66175 (14)	0.6012 (2)	0.0656 (5)
C2	0.19874 (15)	0.69122 (13)	0.6502 (2)	0.0617 (4)

C3	0.29594 (12)	0.67048 (11)	0.58012 (15)	0.0498 (3)
C4	0.40263 (14)	0.69693 (12)	0.6254 (2)	0.0552 (4)
C5	0.49339 (12)	0.67207 (11)	0.55540 (14)	0.0490 (3)
C6†	0.6107 (2)	0.6855 (2)	0.6059 (2)	0.0557 (7)
C6′†	0.5975 (6)	0.7370 (9)	0.564 (2)	0.086 (8)
C7†	0.6852 (3)	0.7177 (2)	0.4968 (3)	0.0589 (8)
C7′†	0.6940 (14)	0.6696 (17)	0.5276 (8)	0.072 (7)
C8	0.67700 (12)	0.64083 (12)	0.38620 (14)	0.0506 (4)
C9	0.76635 (12)	0.61859 (13)	0.3085 (2)	0.0565 (4)
C10	0.75800 (12)	0.55594 (12)	0.2000 (2)	0.0529 (4)
C11	0.85832 (15)	0.5370 (2)	0.1170 (2)	0.0703 (5)
C12†	0.8423 (3)	0.4524 (3)	0.0154 (3)	0.0780 (13)
C12′†	0.8329 (10)	0.5194 (16)	−0.0267 (4)	0.089 (5)
C13†	0.7288 (3)	0.4583 (4)	−0.0466 (5)	0.0697 (13)
C13′†	0.756 (2)	0.4277 (12)	−0.0214 (41)	0.114 (12)
C14†	0.6419 (3)	0.4371 (3)	0.0560 (3)	0.0578 (11)
C14′†	0.6476 (18)	0.4699 (16)	0.0306 (9)	0.069 (6)
C15	0.65560 (12)	0.51308 (12)	0.16795 (15)	0.0507 (4)
C16	0.56543 (12)	0.53566 (12)	0.24476 (14)	0.0481 (3)
C17	0.57431 (11)	0.59818 (10)	0.35363 (13)	0.0415 (3)
C18	0.47665 (11)	0.62270 (10)	0.43320 (13)	0.0417 (3)
N19	0.37841 (10)	0.59756 (10)	0.38691 (11)	0.0483 (3)
C20	0.28819 (12)	0.61943 (12)	0.46024 (15)	0.0493 (3)
C21	0.18240 (13)	0.5913 (2)	0.4122 (2)	0.0656 (5)
C22	0.09013 (15)	0.6119 (2)	0.4817 (2)	0.0710 (5)

† Partial occupancy (see below).

Table 2. *Selected geometric parameters* (Å, °)

Cl1—C1	1.747 (2)	C10—C15	1.396 (2)
C1—C2	1.350 (3)	C10—C11	1.516 (2)
C1—C22	1.398 (3)	C11—C12′	1.530 (4)
C2—C3	1.416 (2)	C11—C12	1.531 (4)
C3—C20	1.407 (2)	C12—C13	1.512 (4)
C3—C4	1.412 (2)	C12′—C13′	1.512 (5)
C4—C5	1.362 (2)	C13—C14	1.528 (5)
C5—C18	1.429 (2)	C13′—C14′	1.528 (5)
C5—C6	1.519 (2)	C14—C15	1.528 (3)
C5—C6′	1.519 (3)	C14′—C15	1.528 (3)
C6—C7	1.511 (3)	C15—C16	1.389 (2)
C6′—C7′	1.511 (3)	C16—C17	1.391 (2)
C7—C8	1.520 (3)	C17—C18	1.484 (2)
C7′—C8	1.520 (3)	C18—N19	1.319 (2)
C8—C9	1.386 (2)	N19—C20	1.367 (2)
C8—C17	1.400 (2)	C20—C21	1.417 (2)
C9—C10	1.388 (2)	C21—C22	1.362 (2)
C2—C1—C22	121.9 (2)	C10—C11—C12′	114.8 (5)
C2—C1—Cl1	119.8 (2)	C10—C11—C12	114.1 (2)
C22—C1—Cl1	118.22 (15)	C13—C12—C11	111.2 (3)
C1—C2—C3	119.5 (2)	C13′—C12′—C11	102 (2)
C20—C3—C4	117.22 (13)	C12—C13—C14	109.1 (4)
C20—C3—C2	119.52 (15)	C12′—C13′—C14′	105.3 (15)
C4—C3—C2	123.2 (2)	C13—C14—C15	109.9 (3)
C5—C4—C3	120.57 (14)	C13′—C14′—C15	114.4 (19)
C4—C5—C18	117.93 (14)	C16—C15—C10	118.89 (14)
C4—C5—C6	123.3 (2)	C16—C15—C14	119.3 (2)
C18—C5—C6	118.53 (15)	C10—C15—C14	121.7 (2)
C4—C5—C6′	120.9 (5)	C16—C15—C14′	124.5 (8)
C18—C5—C6′	114.1 (7)	C10—C15—C14′	114.4 (9)
C7—C6—C5	109.9 (2)	C15—C16—C17	122.11 (14)
C7′—C6′—C5	108.0 (11)	C16—C17—C8	119.06 (13)
C6—C7—C8	110.2 (2)	C16—C17—C18	121.27 (12)
C6′—C7′—C8	106.5 (9)	C8—C17—C18	119.64 (12)
C9—C8—C17	118.48 (14)	N19—C18—C5	123.36 (13)
C9—C8—C7	121.9 (2)	N19—C18—C17	117.93 (12)
C17—C8—C7	119.4 (2)	C5—C18—C17	118.71 (12)
C9—C8—C7′	120.7 (6)	C18—N19—C20	118.15 (12)
C17—C8—C7′	116.0 (8)	N19—C20—C3	122.69 (13)
C8—C9—C10	122.69 (14)	N19—C20—C21	118.58 (14)
C9—C10—C15	118.77 (14)	C3—C20—C21	118.72 (14)
C9—C10—C11	120.05 (15)	C22—C21—C20	120.6 (2)
C15—C10—C11	121.16 (15)	C21—C22—C1	119.6 (2)
C18—C5—C6—C7	41.6 (3)	C15—C10—C11—C12′	29.7 (9)
C18—C5—C6′—C7′	−54.7 (16)	C10—C11—C12—C13	40.4 (4)
C5—C6—C7—C8	−55.3 (4)	C10—C11—C12′—C13′	−57.8 (17)
C5—C6′—C7′—C8	65 (2)	C11—C12—C13—C14	−64.3 (6)
C6—C7—C8—C17	39.0 (3)	C11—C12′—C13′—C14′	73 (3)
C6′—C7′—C8—C17	−52.6 (18)	C12—C13—C14—C15	56.6 (6)
C7—C8—C17—C18	−4.4 (2)	C12′—C13′—C14′—C15	−63 (3)
C7′—C8—C17—C18	25.9 (8)	C11—C10—C15—C14	5.9 (3)
C6—C5—C18—C17	−7.4 (2)	C11—C10—C15—C14′	−14.0 (9)
C6′—C5—C18—C17	27.0 (7)	C13—C14—C15—C10	−28.5 (5)
C8—C17—C18—C5	−12.4 (2)	C13′—C14′—C15—C10	31.1 (19)
C15—C10—C11—C12	−11.4 (3)		

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from difference Fourier maps, but their positional parameters could not be refined because of non-convergence. The *R* value at this stage was 0.059 (*wR* = 0.198) and a difference Fourier map showed peaks of 0.5 e Å^{-3} near C6, C7, C12, C13 and C14. They were first interpreted as the disordered H-atom positions, but refinement based on this did not proceed well. They were then considered as the disordered positions of C atoms, due to conformational flexibility of the cyclohexene rings. Refinement with this model resulted in good convergence with a final *R* value of 0.046 (*wR* = 0.128). This positional disorder was considered for the atoms in each ring, *i.e.* C6 (C6′) and C7 (C7′) [86% (14%)] and C12 (C12′), C13 (C13′) and C14 (C14′) [81% (19%)]. The final difference Fourier map was featureless, with a maximum peak of 0.16 e Å^{-3}. The H atoms were considered only for the major positions and not for the minor positions, and all of them were refined isotropically.

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983*b*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: KH1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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