

## 9,10-Dihydro-9-methylacridine

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(Received 3 October 1989; accepted 12 December 1990)

**Abstract.**  $C_{14}H_{13}N$ ,  $M_r = 195.27$ , triclinic,  $P\bar{1}$ ,  $a = 10.354$  (1),  $b = 10.332$  (2),  $c = 10.452$  (1) Å,  $\alpha = 79.51$  (1),  $\beta = 81.06$  (1),  $\gamma = 81.76$  (1)°,  $V = 1078.4$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.203$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.65$  cm<sup>-1</sup>,  $F(000) = 416$ ,  $T = 293$  K, final  $R = 0.036$  for 1858 observed reflections. The phenyl rings of the acridine ring system are planar. The angles between the normals to the least-squares planes of the phenyl rings are 156° in one molecule and 160° in the other; the angle between the normals to the least-squares planes of the independent molecules is 99°. The two independent molecules are held by a weak hydrogen bond of 2.49 Å at an angle of 170°.

**Experimental.** The synthetic material was prepared by the slow addition of an ethereal solution of methylithium to a solution of acridine in diethyl ether, stirred at 273 K followed by hydrolysis with dilute hydrochloric acid and evaporation of the organic layer. A colorless prism (dimensions 0.63 × 0.20 × 0.09 mm) obtained from ethanol (95%) was used for data collection on an Enraf-Nonius CAD-4 diffractometer, using *SDP* (Frenz, 1978), with graphite-monochromated Mo  $K\alpha$  radiation at 293 (1) K using the  $\omega$ - $2\theta$  scan technique ( $\omega$  varied from 2–20° min<sup>-1</sup>) to a  $2\theta$  maximum of 45°. 2822 unique reflections were measured within the range  $-10 \leq h \leq 11$ ,  $-10 \leq k \leq 11$ ,  $0 \leq l \leq 11$  of which 1858 with  $I > 3.0\sigma$  were used for all calculations. Three standard reflections were measured every 97 reflections, no crystal decay was detected. Cell constants were refined from 25 reflections in the range  $15 < 2\theta < 19^\circ$ . Lorentz and polarization corrections were applied to the data; no corrections were made for absorption. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and the remaining atoms were located in succeeding difference Fourier synthesis. H atoms were located and their positions and isotropic thermal parameters were refined. The structure was refined in full-matrix least squares using *SHELXL76* (Sheldrick, 1976) where the function minimized was  $\sum w(|F_o| - |F_c|)^2$  and the weight  $w$  is defined by the Killean & Lawrence (1969) method with terms of 0.020 and 1.0.

Atomic scattering factors and the values for  $f'$  and  $f''$  were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). Anomalous-dispersion effects were included in  $F_c$  (Ibers & Hamilton, 1964). The final cycle of refinement included 375 variable parameters and converged with an unweighted agreement factor  $R$  of 0.036, a weighted agreement factor  $wR$  of 0.041, and  $(\Delta/\sigma)_{\text{max}}$  of 0.003.

The standard deviation of an observation of unit weight was 0.97. There was one correlation coefficient greater than 0.50 which was between parameters 5 and 315. The final difference Fourier map showed no significant residual electron density and the highest peak had a maximum  $\rho$  of 0.11 e Å<sup>-3</sup> with an e.s.d. based on a  $\Delta F$  of 0.03 (Cruickshank, 1949). Plots of  $\sum w(|F_o| - |F_c|)^2$  versus  $|F_o|$ , reflection order in data collection,  $\sin \theta/\lambda$ , and various classes of indices showed no unusual trends.

**Discussion.** Final positional parameters for the non-H atoms for 9,10-dihydro-9-methylacridine are listed in Table 1.\* Bond lengths are listed in Table 2. An *ORTEPII* (Johnson, 1976) thermal ellipsoid drawing of the two independent molecules *A* and *B* (50% probability of non-H atoms) depicting the numbering scheme is shown in Fig. 1; a molecular packing diagram is shown in Fig. 2. The crystal structure of 9,10-dihydro-9-methylacridine compares very closely with the crystal structures of 9,10-dihydro-9-isopropylacridine (Chu, Rosenstein & Napoleone, 1980) and 9-*tert*-butyl-9,10-dihydroacridine (Chu & Rosenstein, 1979) with the methyl substituent in an axial conformation as expected because of the *peri* interactions of the neighboring ring H atoms, H(1) and H(8). The folding angles between the least-squares planes of the two benzene rings in the 9-alkyl-9,10-dihydroacridines are 160.4

\* Lists of structure factors, anisotropic thermal parameters, torsion angles, bond angles, least-squares planes, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53822 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates of non-H atoms and equivalent isotropic temperature factors

Molecule A	x	y	z	B(Å <sup>2</sup> )
N10	0.1630 (2)	0.6717 (2)	0.6675 (2)	4.19 (5)
C1	0.4614 (2)	0.7834 (3)	0.4684 (3)	5.47 (7)
C2	0.4195 (3)	0.8273 (3)	0.3477 (3)	5.81 (7)
C3	0.2920 (3)	0.8178 (2)	0.3321 (2)	5.04 (6)
C4	0.2072 (2)	0.7660 (2)	0.4380 (2)	4.30 (6)
C4a	0.2496 (2)	0.7235 (2)	0.5593 (2)	3.71 (5)
C5	0.0888 (2)	0.6589 (2)	0.8997 (2)	4.57 (6)
C6	0.1159 (3)	0.6487 (3)	1.0263 (2)	5.37 (7)
C7	0.2425 (3)	0.6484 (2)	1.0501 (2)	5.36 (7)
C8	0.3424 (2)	0.6581 (2)	0.9460 (3)	5.05 (6)
C8a	0.3185 (2)	0.6676 (2)	0.8172 (2)	3.97 (5)
C9	0.4282 (2)	0.6682 (2)	0.7033 (2)	4.70 (6)
C9a	0.3791 (2)	0.7290 (2)	0.5762 (2)	4.07 (6)
C10a	0.1892 (2)	0.6678 (2)	0.7953 (2)	3.63 (5)
C11	0.4940 (3)	0.5254 (3)	0.6968 (3)	6.92 (8)
Molecule B				
N10	0.1686 (2)	0.3419 (2)	0.7210 (2)	4.12 (4)
C1	0.1795 (3)	0.1187 (2)	0.4847 (3)	5.25 (6)
C2	0.1737 (3)	0.2058 (3)	0.3692 (2)	5.79 (7)
C3	0.1638 (2)	0.3399 (3)	0.3705 (2)	5.14 (6)
C4	0.1602 (2)	0.3855 (2)	0.4869 (2)	4.39 (6)
C4a	0.1678 (2)	0.2973 (2)	0.6030 (2)	3.62 (5)
C5	0.2638 (2)	0.3114 (2)	0.9227 (2)	4.66 (6)
C6	0.3184 (3)	0.2294 (3)	1.0235 (3)	5.53 (7)
C7	0.3335 (3)	0.0939 (3)	1.0284 (3)	6.25 (8)
C8	0.2903 (3)	0.0420 (3)	0.9326 (3)	5.67 (7)
C8a	0.2334 (2)	0.1218 (2)	0.8302 (2)	4.15 (6)
C9	0.1764 (2)	0.0662 (2)	0.7308 (2)	4.66 (6)
C9a	0.1758 (2)	0.1611 (2)	0.6037 (2)	4.00 (5)
C10a	0.2224 (2)	0.2588 (2)	0.8247 (2)	3.72 (5)
C11	0.0374 (3)	0.0317 (3)	0.7858 (3)	6.31 (7)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:  $\frac{1}{3}(a\beta_{11} + b\beta_{22} + c\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha)$ .

Table 2. Bond lengths (Å), with e.s.d.'s in parentheses

		A	B			A	B
N10	C4a	1.405 (3)	1.394 (3)	C5	H5	0.98 (2)	0.98 (2)
N10	C10a	1.396 (3)	1.391 (3)	C6	C7	1.371 (4)	1.378 (4)
N10	H10	0.87 (2)	0.87 (2)	C6	H6	0.94 (2)	1.02 (3)
C1	C2	1.375 (4)	1.372 (3)	C7	C8	1.379 (3)	1.376 (5)
C1	C9a	1.385 (3)	1.385 (4)	C7	H7	1.02 (2)	0.95 (2)
C1	H1	0.96 (3)	0.97 (2)	C8	C8a	1.390 (4)	1.382 (3)
C2	C3	1.375 (4)	1.377 (4)	C8	H8	0.98 (2)	0.94 (2)
C2	H2	0.99 (2)	0.98 (3)	C8a	C9	1.512 (3)	1.504 (4)
C3	C4	1.378 (3)	1.376 (4)	C8a	C10a	1.393 (3)	1.395 (3)
C3	H3	1.06 (2)	0.99 (2)	C9	C11	1.542 (4)	1.532 (4)
C4	C4a	1.381 (3)	1.384 (3)	C9	C9a	1.495 (3)	1.501 (3)
C4	H4	0.95 (2)	0.98 (2)	C9	H9	0.98 (3)	0.97 (2)
C4a	C9a	1.390 (3)	1.397 (3)	C11	H111	0.97 (2)	0.98 (3)
C5	C6	1.377 (4)	1.368 (3)	C11	H112	1.05 (3)	1.03 (2)
C5	C10a	1.385 (3)	1.390 (4)	C11	H113	1.10 (4)	1.07 (3)

Table 3. Cremer &amp; Pople ring puckering parameters for 9-alkyl-9,10-dihydroacridines

The coordinates were translated and arranged such that the parameters are in the same octant for comparison.

9-Substituent	$q_2$	$q_3$	$Q$	$\varphi_2$ (°)	$\theta$ (°)
Methyl A	0.326	0.040	0.329	184.0	83.0
Methyl B	0.334	0.048	0.337	181.5	81.9
Isopropyl	0.322	0.060	0.327	180.4	79.5
tert-Butyl	0.396	0.072	0.403	180.4	79.7

(methyl A), 155.8 (methyl B), 156.1 (isopropyl) and 150.2° (tert-butyl). The Cremer & Pople (1975) ring puckering parameters for the central rings of these three 9-alkyl-9,10-dihydroacridines are listed in Table 3. In all of these structures, the conformation

of the central ring is intermediate between a boat ( $\theta = 90^\circ$ ) and a half-boat ( $\theta = 45^\circ$ ). These boat-half-boat conformations are barely twisted (non-twisted  $\varphi = 0^\circ$  or  $180^\circ$ , twisted  $\varphi_2 = 90^\circ$ ), although in the two molecules of the 9-methyl derivative, one molecule is slightly more twisted than the other. Unlike the other two 9-alkyl-9,10-dihydroacridines cited above, however, the crystal packing does possess hydrogen bonding. The two independent molecules (A and B) of the 9-methyl derivative are held together by a weak hydrogen bond in which molecule A is the hydrogen acceptor and molecule B is the hydrogen donor with a bond length (N10<sub>A</sub>...H10<sub>B</sub>) of 2.49 (3) Å and an angle (N10<sub>A</sub>...H10<sub>B</sub>—N10<sub>B</sub>) of 170 (2)°. The angle between the normals to the least-squares planes of the two molecules is 98.8 (5)°.

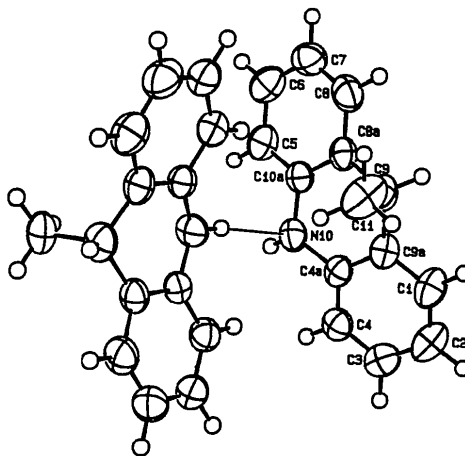


Fig. 1. Thermal ellipsoid (50% probability of non-H atoms) depiction of molecule A (right) and molecule B (left) showing the numbering scheme (thin line denotes hydrogen bond).

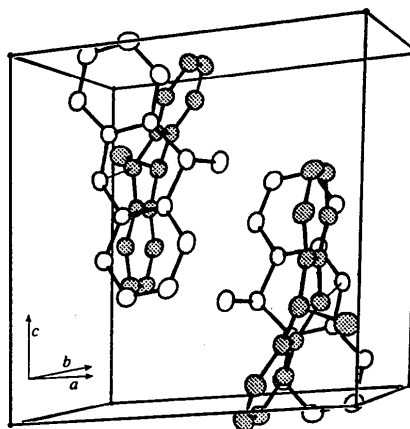


Fig. 2. Molecular packing diagram showing hydrogen bond (thin line) between molecule A (white ellipsoids) and molecule B (shaded ellipsoids).

The authors wish to thank Dr P. E. Fanwick, Department of Chemistry, Purdue University, for his assistance, the use of the Enraf-Nonius CAD-4 diffractometer, and the ORTEPII plots.

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*Acta Cryst.* (1991). **C47**, 1637–1640

## 9-Phenylacridine and 9-Phenylacridine Hydrochloride

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(Received 3 October 1989; accepted 11 December 1990)

**Abstract.** 9-Phenylacridine,  $C_{19}H_{13}N$ ,  $M_r = 255.32$ , monoclinic,  $P2_1/n$ ,  $a = 8.574$  (2),  $b = 17.862$  (2),  $c = 9.126$  (2) Å,  $\beta = 107.723$  (8)°,  $V = 1331.2$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.274$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.69$  cm<sup>-1</sup>,  $F(000) = 536$ ,  $T = 293$  K,  $R = 0.033$  for 1300 observed reflections. 9-Phenylacridine hydrochloride,  $C_{19}H_{14}N^+Cl^-$ ,  $M_r = 291.78$ , monoclinic,  $C2/c$ ,  $a = 14.728$  (3),  $b = 10.443$  (9),  $c = 18.885$  (3) Å,  $\beta = 90.36$  (1)°,  $V = 2904$  (1) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.334$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 2.52$  cm<sup>-1</sup>,  $F(000) = 1216$ ,  $T = 293$  K, final  $R = 0.033$  for 1642 observed reflections. The acridine rings and phenyl rings are planar in both compounds. The angle between the normal to the least-squares plane of the acridine ring and the normal to the least-squares plane of the phenyl ring is 76° in the free-base compound and 65° in the hydrochloride salt.

**Experimental.** 9-Phenylacridine. The synthetic material was prepared following a procedure by Albert (1966). A pale yellow crystal (dimensions 0.82 × 0.30 × 0.26 mm) obtained from diethyl ether was used for data collection on an Enraf-Nonius CAD-4 diffractometer, using SDP (Frenz, 1978), with graphite-monochromated Mo  $K\alpha$  radiation at 293 (1) K using the  $\omega$ -2 $\theta$  scan technique ( $\omega$  varied from 2–20° min<sup>-1</sup>) to a 2 $\theta$  maximum of 45°. 1838 unique reflections were measured within the range  $-9 \leq h \leq 8$ ,  $0 \leq k \leq 19$ ,  $0 \leq l \leq 9$  of which 1300 with  $I > 3.0\sigma I$  were used for all calculations. Three stand-

ard reflections were measured every 97 reflections, no crystal decay was detected. Cell constants were refined from 25 reflections in the range  $19 < 2\theta < 23^\circ$ . Lorentz and polarization corrections were applied to the data; no corrections were made for absorption. The structure was solved by direct methods using SHELXS86 (Sheldrick, 1986) and the remaining atoms were located in a succeeding difference Fourier synthesis. H atoms were located and their positions and isotropic thermal parameters were refined. The structure was refined in full-matrix least squares using SHELX76 (Sheldrick, 1976) where the function minimized was  $\sum w(|F_o| - |F_c|)^2$  and the weight  $w$  is defined by the Killean & Lawrence (1969) method with terms of 0.030 and 1.0.

Atomic scattering factors and the values for  $f'$  and  $f''$  were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). Anomalous-dispersion effects were included in  $F_c$  (Ibers & Hamilton, 1964). The final cycle of refinement included 233 variable parameters and converged with an unweighted agreement factor  $R$  of 0.033, a weighted agreement factor  $wR$  of 0.040, and  $(\Delta/\sigma)_{\text{max}}$  of 0.001.

The standard deviation of an observation of unit weight was 1.25. There were no correlation coefficients greater than 0.50. The final difference Fourier map showed no significant residual electron density and the highest peak had a maximum  $\rho$  of 0.10 e Å<sup>-3</sup> with an e.s.d. based on a  $\Delta F$  of 0.03