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## 9-Phenylacridine and 9-Phenylacridine Hydrochloride

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**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

(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.

**9-Phenylacridine hydrochloride.** The synthetic material was prepared following a procedure by Albert (1966). Titration of an ethanolic 9-phenylacridine solution with concentrated hydrochloric acid afforded a black precipitate of the hydrochloride salt. A purple chunk (dimensions  $0.98 \times 0.41 \times 0.29$  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  $2$ – $20^\circ \text{ min}^{-1}$ ) to a  $2\theta$  maximum of  $45^\circ$ . 2042 unique reflections were measured within the range  $-15 \leq h \leq 15$ ,  $0 \leq k \leq 11$ ,  $0 \leq l \leq 20$  of which 1642 with  $I > 3.0\sigma I$  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  $20 < 2\theta < 23^\circ$ . 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 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 246 variable parameters and converged with an unweighted agreement factor  $R$  of 0.033, a weighted agreement factor  $wR$  of 0.049, and  $(\Delta/\sigma)_{\text{max}}$  of 0.11.

The standard deviation of an observation of unit weight was 1.30. There were three correlation coefficients greater than 0.50, the highest of which was 0.54 between parameters 1 and 5. The final difference Fourier map showed no significant residual electron density and the highest peak had a maximum  $\rho$  of  $0.18 \text{ e } \text{\AA}^{-3}$  with an e.s.d. based on a  $\Delta F$  of 0.04 (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.** This communication constitutes the first report of a crystal structure of a 9-arylacridine (free

Table 1. Positional parameters of non-H atoms and equivalent isotropic thermal parameters

	x	y	z	B(Å <sup>2</sup> )
Free base				
N10	-0.2116 (2)	0.13927 (8)	0.5307 (2)	4.22 (3)
C1	0.2233 (2)	0.0821 (1)	0.6368 (2)	4.40 (4)
C2	0.2353 (2)	0.0700 (1)	0.4937 (2)	5.07 (5)
C3	0.0984 (2)	0.0802 (1)	0.3625 (2)	5.26 (5)
C4	-0.0458 (2)	0.1026 (1)	0.3771 (2)	4.85 (5)
C4a	-0.0645 (2)	0.1166 (1)	0.5244 (2)	3.80 (4)
C5	0.3814 (2)	0.1802 (1)	0.6765 (2)	4.81 (5)
C6	-0.4038 (2)	0.1991 (1)	0.8118 (2)	5.40 (5)
C7	-0.2750 (2)	0.1919 (1)	0.9508 (2)	5.24 (5)
C8	-0.1269 (2)	0.1654 (1)	0.9508 (2)	4.42 (4)
C8a	-0.0964 (2)	0.14577 (9)	0.8104 (2)	3.61 (4)
C9	0.0548 (2)	0.11957 (9)	0.8026 (2)	3.46 (4)
C9a	0.0735 (2)	0.10573 (9)	0.6575 (2)	3.63 (4)
C10a	-0.2274 (2)	0.15390 (9)	0.6703 (2)	3.90 (4)
C11	0.1944 (2)	0.1063 (1)	0.9449 (2)	3.56 (4)
C12	0.2847 (2)	0.1651 (1)	1.0264 (2)	4.39 (4)
C13	0.4147 (2)	0.1517 (1)	1.1574 (2)	5.08 (5)
C14	0.4561 (2)	0.0797 (1)	1.2070 (2)	5.00 (5)
C15	0.3671 (2)	0.0210 (1)	1.1271 (2)	5.01 (5)
C16	0.2365 (2)	0.0340 (1)	0.9973 (2)	4.44 (4)
HCl salt				
C1	-0.28814 (3)	0.45138 (5)	0.52651 (3)	4.66 (1)
N10	-0.1007 (1)	0.5217 (2)	0.57272 (8)	3.31 (3)
C1	0.1015 (1)	0.3287 (2)	0.6106 (1)	3.73 (4)
C2	0.0762 (1)	0.2164 (2)	0.5810 (1)	4.14 (4)
C3	-0.0107 (1)	0.2030 (2)	0.5493 (1)	4.05 (4)
C4	-0.0691 (1)	0.3025 (2)	0.5467 (1)	3.58 (4)
C4a	-0.0435 (1)	0.4212 (2)	0.57634 (9)	3.16 (4)
C5	-0.1442 (1)	0.7387 (2)	0.5905 (1)	4.14 (4)
C6	-0.1236 (1)	0.8565 (2)	0.6155 (1)	4.67 (5)
C7	-0.0399 (2)	0.8798 (2)	0.6498 (1)	4.77 (5)
C8	0.0216 (1)	0.7851 (2)	0.6586 (1)	4.13 (4)
C8a	0.0043 (1)	0.6598 (2)	0.63255 (9)	3.29 (4)
C9	0.0651 (1)	0.5567 (2)	0.63936 (9)	3.13 (4)
C9a	0.0426 (1)	0.4371 (2)	0.61006 (9)	3.09 (4)
C10a	-0.0805 (1)	0.6387 (2)	0.59807 (9)	3.29 (4)
C11	0.1527 (1)	0.5731 (2)	0.6784 (1)	3.25 (4)
C12	0.2201 (1)	0.6538 (2)	0.6536 (1)	3.85 (4)
C13	0.3005 (1)	0.6661 (2)	0.6914 (1)	4.60 (5)
C14	0.3142 (1)	0.5999 (2)	0.7526 (1)	4.77 (5)
C15	0.2483 (2)	0.5196 (2)	0.7779 (1)	4.78 (5)
C16	0.1675 (1)	0.5055 (2)	0.7411 (1)	4.04 (4)

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

base or salt form). Final positional parameters for the non-H atoms of both 9-phenylacridine and its hydrochloric salt are listed in Table 1.\* Bond lengths are listed in Table 2. ORTEPII (Johnson, 1976) drawings of the two structures showing thermal ellipsoids of non-H scaled to 50% probability level are shown in Fig. 1. Molecular packing diagrams of the two structures are shown in Fig. 2. Least-squares planes of the acridine and the phenyl atoms of the two structures indicate that the maximum distance from the plane for the free base is 0.064 (acridine ring) and 0.0045 Å (phenyl ring) with an angle of  $75.7^\circ$  between the normals to the planes and for the hydrochloride salt 0.052 (acridine ring) and 0.0013 Å

\* 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 53820 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

		Free base	HCl salt			Free base	HCl salt
N10	C4a	1.342 (2)	1.349 (2)	C7	H7	0.98 (2)	0.93 (2)
N10	C10a	1.347 (2)	1.345 (2)	C8	C8a	1.427 (3)	1.420 (3)
N10	H10		0.93 (2)	C8	H8	0.96 (1)	1.00 (2)
C1	C2	1.358 (3)	1.349 (3)	C8a	C9	1.400 (2)	1.405 (3)
C1	C9a	1.419 (3)	1.426 (3)	C8a	C10a	1.430 (2)	1.423 (2)
C1	H1	0.98 (1)	0.95 (2)	C9	C9a	1.403 (3)	1.405 (3)
C2	C3	1.410 (2)	1.417 (3)	C9	C11	1.493 (2)	1.491 (3)
C2	H2	1.00 (2)	0.97 (2)	C11	C12	1.380 (2)	1.387 (3)
C3	C4	1.344 (3)	1.350 (3)	C11	C16	1.386 (2)	1.394 (3)
C3	H3	0.99 (2)	0.96 (2)	C12	C13	1.385 (2)	1.385 (3)
C4	C4a	1.424 (3)	1.411 (3)	C12	H12	0.99 (2)	0.90 (2)
C4	H4	0.99 (1)	0.93 (2)	C13	C14	1.373 (3)	1.362 (3)
C4a	C9a	1.427 (2)	1.425 (2)	C13	H13	0.99 (2)	0.97 (2)
C5	C6	1.349 (3)	1.352 (3)	C14	C15	1.369 (3)	1.371 (3)
C5	C10a	1.419 (3)	1.409 (3)	C14	C14	1.01 (1)	0.94 (2)
C5	H5	1.01 (1)	0.96 (2)	C15	C16	1.380 (2)	1.383 (3)
C6	C7	1.412 (2)	1.410 (3)	C15	H15	1.01 (2)	0.95 (2)
C6	H6	0.98 (2)	0.92 (2)	C16	H16	0.95 (2)	0.97 (2)
C7	C8	1.355 (3)	1.351 (3)				

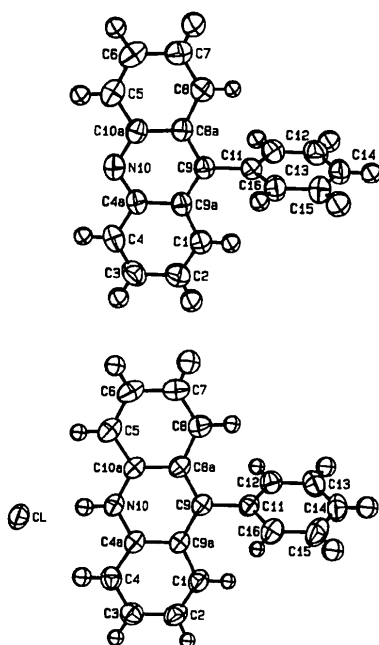


Fig. 1. Thermal ellipsoid (50% probability) depiction of the free base (top) and the HCl salt (bottom) showing the numbering scheme.

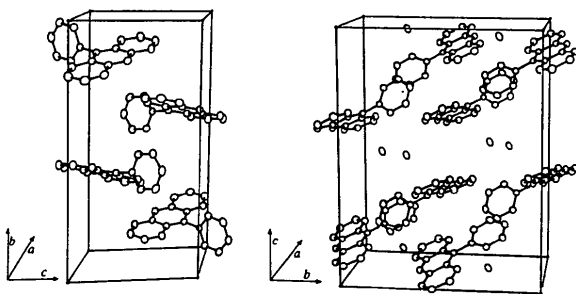


Fig. 2. Molecular packing of the free base (left) and the HCl salt (right).

(phenyl ring) with an angle of  $65.3^\circ$  between the normals to the planes. These results are similar to those found in a series of 10-methyl-9-(alkylphenyl)acridinium chloride or perchlorate salts reported recently [(I): Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven (1989); (II) and (IV) Reiss, Goubitz & Heijdenrijk (1989a,b); (III): Kronenburg, Goubitz, Reiss & Heijdenrijk (1989); (V) and (VIII): Goubitz, Reiss & Heijdenrijk (1989a,b); (VII): Kronenburg, Reiss, Goubitz & Heijdenrijk (1989); (IX): Reiss, Goubitz, Zoutberg & Heijdenrijk (1989)]. The first report in this series discusses the structure of 10-methyl-9-phenylacridinium chloride monohydrate which has a maximum distance from the least-squares plane of 0.070 (acridinium ring) and 0.014 Å (phenyl ring) with an angle of  $68^\circ$  between the normals to the two planes – similar to the 9-phenylacridine hydrochloride reported herein. In the entire series mentioned above, the angle between the normal to the least-squares plane of the acridinium ring atoms and the normal to the least-squares plane of the phenyl ring atoms ranges from  $63^\circ$  for 10-methyl-9-(4-hydroxyphenyl)acridinium chloride to  $76^\circ$  for 10-methyl-9-(2-methylphenyl)acridinium perchlorate in the structures bearing small substituents [(I) through (IV)] and from  $57^\circ$  for 10-methyl-9-[4-(1,4,7,10-tetraoxa-13-aza-13-cyclopentadecyl)phenyl]acridinium perchlorate to  $78^\circ$  for 10-methyl-9-[2-methyl-4-(1,4,7,10-tetraoxa-13-aza-13-cyclopentadecyl)phenyl]acridinium perchlorate in the compounds bearing large substituents [(V) through (VIII)]. The angles between the normals to the least-squares planes of other related structures include  $67^\circ$  for 9,10-diphenylanthracene (Roszak & Duax, 1987),  $73$ ,  $74$  and  $80^\circ$  for the three independent molecules of 9-acetoxy-10-phenylanthracene (Roszak & Borowiak, 1987), and  $84^\circ$  for 9-acetoxy-10-(4-acetoxyphenyl)anthracene (Roszak & Duax, 1987). It appears then that the twist angle between the phenyl ring and acridine ring is more a function of crystal packing energies than the phenyl ring substituents.

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## Structure of *trans*-Bis(4-dimethylaminonaphthalene-1-azo)-4,4'-stilbene

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**Abstract.**  $C_{38}H_{34}N_6$ ,  $M_r = 574.73$ , monoclinic,  $P2_1/c$ ,  $a = 9.286$  (1),  $b = 9.375$  (1),  $c = 35.250$  (4) Å,  $\beta = 92.681$  (5)°,  $V = 3065.4$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.25$  (2),  $D_x = 1.245$  (1) Mg m<sup>-3</sup>,  $Cu K\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 0.508$  mm<sup>-1</sup>,  $F(000) = 1216$ ,  $T = 298$  K,  $R = 0.053$ , 3819 unique observed reflections. All distances and angles lie within the range of values found for compounds having a similar composition to that of the title compound. The molecule can be described as quasi centrosymmetric and nearly planar. Intermolecular bonding is by van der Waals interaction.

**Introduction.** Azo and anthraquinone derivatives are the most frequently used dyes for liquid crystalline guest–host systems. The azo compounds show in most cases a sufficient solubility and have higher order parameters and greater extinction coefficients than the anthraquinone dyes. A serious problem in their use is the poor stability to illumination due to *cis*–*trans* isomerization in liquid crystalline solution. New azo derivatives with a more extended molecular shape have been developed that have significantly higher stabilities (Seki, Shishido, Uchida & Wada, 1982).

**Experimental.** The title compound was synthesized using 4,4'-diaminostilbene dihydrochloride and 1-dimethylaminonaphthalene (Aftergut & Cole, 1979). There resulted a mixture of more than ten different dyes and so column chromatography was used for purification of the main product which was additionally characterized by VIS, NMR, and mass spectroscopy, yield about 60%. Reddish brown crystals in the form of sheets could be obtained by slow evaporation of a solution with chloroform. The main problem with the structure analysis was the availability of single crystals, because by recrystallization from different solvents twins were nearly always obtained. The crystal density was determined using a pycnometer. A crystal of size  $0.04 \times 0.35 \times 1.4$  mm with well developed faces (001), (010) and (10 $\bar{1}$ ) was used for the X-ray measurements with a Stoe Stadi IV diffractometer with graphite monochromator. Lattice parameters were obtained by least-squares fit of 50 reflections,  $25 \leq 2\theta \leq 62^\circ$ ; intensity data were collected using  $\omega$ – $2\theta$  scan, absorption was ignored ( $\mu R = 0.102$ ); max.  $\sin\theta/\lambda = 0.58$  Å<sup>-1</sup>;  $h$ :  $-10 \rightarrow 10$ ,  $k$ :  $0 \rightarrow 10$ ,  $l$ :  $0 \rightarrow 39$ ; three standard reflections measured after every 50 reflections showed no significant intensity variations. 4878 reflections were measured of which 4203 were unique and 3819 with  $F_o > 2\sigma(F_o)$  were subsequently used in the refinement ( $R_{int} = 0.0386$ ). The structure was solved by the direct methods;  $F$  magnitudes were used in full-matrix least-squares refinement; all atoms (H atoms inclu-

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