Marchand, A. P., Huang, C., Kaya, R., Baker, A. D., Jemmis, E. D. & Dixon, D. A. (1987). *J. Am. Chem. Soc.* **109**, 7095–7101.

Marchand, A. P., Suri, S. C., Earlywine, A. D., Powell, D. R. & van der Helm, D. (1984). *J. Org. Chem.* **49**, 670–675.

Marchand, A. P. & Zhao, D. (1989). In preparation.

Nicolet Instrument Corporation (1986). *SHELXTL* for Desktop 30 (Microeclipse). PN-269–1040340, April.

Ōsawa, E. & Kanematsu (1986). *Molecular Structure and Energetics*, Vol. 3, edited by J. F. Liebman & A. Greenberg, pp. 329–369. New York: VCH.

Watson, W. H. & Nagl, A. (1988). *Acta Cryst.* C**44**, 1852–1853.

Watson, W. H., Nagl, A., Marchand, A. P. & Deshpande, M. N. (1989). *Acta Cryst.* C**45**, 1339–1342.

Watson, W. H., Nagl, A., Marchand, A. P., Reddy, G. M. & Reddy, S. P. (1989). *Acta Cryst.* C**45**, 659–661.

Acta Cryst. (1989). C**45**, 1346–1348

Structure of 1-Amino-2-(9-methyl-1,8-dioxo-1,2,3,4,5,6,7,8,9,10-decahydro-10-acridinyl)propenedinitrile

By S. Selladurai, K. Subramanian* and S. Natarajan

Department of Physics, College of Engineering, Anna University, Madras-25, India

(*Received* 6 *October* 1988; *accepted* 18 *January* 1989)

Abstract. $C_{18}H_{18}N_4O_2$, M_r = 322·4, triclinic, $P\bar{1}$, a = 8·5138 (6), b = 9·407 (3), c = 11·838 (1) Å, α = 70·76 (2), β = 79·485 (8), γ = 79·51 (2)°, V = 872·5 Å^3, Z = 2, D_x = 1·23 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 6·36 cm^{-1}, F(000) = 340, T = 296 K, R = 0·049 for 3302 observed reflections with $I > 3\sigma(I)$. The central acridinyl ring and one of the side rings adopt sofa conformations, the other side ring is midway between a half-chair and a sofa conformation. The 9-methyl group adopts an axial position on the acridinyl ring, with the dicyano group equatorial. The structure is stabilized by two N—H···O intermolecular hydrogen bonds N(18)···O(11) and N(18)···O(13) with N···O distances and N—H···O angles 2·84 (3), 2·81 (3) Å and 142 (2), 130 (2)° respectively.

Introduction. The title compound was supplied by Professor V. T. Ramakrishnan, Department of Chemistry, University of Madras, India. Aminoacridine derivatives have general uses in medicine especially as antibacterials for wound therapy (Acheson, 1956). For this reason the crystal structure of the title compound is of considerable interest to our continuing investigations.

Experimental. The crystals were obtained from acetone/ethanol as very small transparent yellow-coloured, rectangular blocks. A specimen with dimensions 0·25 × 0·35 × 0·65 mm was selected. The unit-cell parameters were obtained by measuring the 2θ values of 25 medium angle ($35 < 2\theta < 45°$) reflections (least-squares treatment). Intensity data were collected on an Enraf–Nonius CAD-4 diffractometer. Graphite-crystal-monochromated Cu $K\alpha$ radiation was used to measure 3594 independent reflections with $4 \leq 2\theta \leq 150°$ (h $0 \rightarrow 10$, k $-11 \rightarrow 11$, l $-13 \rightarrow 14$), of which 3302 were considered observed [$I > 3\sigma(I)$, where $\sigma(I)$ was determined from counting statistics]. Lorentz, polarization and absorption (ω scans, $T_{\min}$ = 0·903, $T_{\max}$ = 0·989) corrections were applied.

The structure was determined by direct methods using *SHELX86* (Sheldrick, 1986) and refinement was carried out by full-matrix least-squares method using *SHELX76* (Sheldrick, 1976), with anisotropic temperature factors for non-H atoms. All 18 H atoms in the structure were located in a difference Fourier map and not refined. Subsequent refinement converged to R = 0·049 and wR = 0·059. Final difference density within ±0·17 e Å^{-3}. $w = 8{\cdot}002/[\sigma^2F_o + 0{\cdot}00008|F_o|^2]$. Ratio of final shift to e.s.d. < 0·1. The atomic scattering factors used for all the atoms were as provided in the *SHELX76* program. Computer programs: molecular illustrations drawn using *MOLDRAW* (Ugliengo, Borzani & Viterbo, 1988); *PARST* (Nardelli, 1983) for geometrical calculations.

Discussion. Table 1 contains the positional and equivalent isotropic thermal parameters of the non-H atoms.† Table 2 contains torsion angles and

---

* To whom correspondence should be addressed.

† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles involving non-H atoms, bond lengths and angles involving H atoms, and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51884 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/89/091346-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors, for non-*H *atoms, with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
C(1)	0·4515 (3)	0·5520 (3)	0·6870 (2)	3·9
C(2)	0·5491 (3)	0·4099 (3)	0·6655 (2)	4·8
C(3)	0·5267 (3)	0·2732 (3)	0·7759 (2)	4·6
C(4)	0·3522 (3)	0·2557 (2)	0·8143 (2)	3·6
C(4*a*)	0·2310 (3)	0·3905 (2)	0·7893 (2)	3·1
C(5)	0·0561 (3)	0·3681 (2)	0·8305 (2)	3·8
C(6)	−0·1997 (3)	0·5079 (3)	0·9151 (2)	3·7
C(6*a*)	−0·0432 (2)	0·5145 (2)	0·8393 (2)	3·1
C(7)	−0·3094 (3)	0·6523 (3)	0·9133 (2)	4·3
C(8)	−0·2740 (3)	0·7806 (3)	0·8005 (3)	4·6
C(9)	−0·0960 (3)	0·7994 (3)	0·7798 (2)	4·4
C(9*a*)	0·0081 (2)	0·6523 (2)	0·7818 (2)	3·0
N(10)	0·1670 (2)	0·6606 (2)	0·7238 (2)	3·0
C(10*a*)	0·2794 (2)	0·5296 (2)	0·7346 (2)	2·8
O(11)	−0·2400 (2)	0·3844 (2)	0·9817 (2)	5·5
C(12)	−0·0085 (4)	0·3054 (4)	0·7471 (4)	6·7
O(13)	0·3099 (2)	0·1293 (2)	0·8710 (2)	4·8
C(14)	0·2131 (3)	0·8043 (2)	0·6481 (2)	3·3
C(15)	0·2686 (3)	0·8961 (2)	0·6942 (2)	3·5
C(16)	0·3142 (3)	0·0407 (2)	0·6096 (2)	4·3
N(17)	0·3518 (4)	0·1512 (3)	0·5444 (2)	6·7
N(18)	0·2896 (3)	0·8694 (2)	0·8093 (2)	4·4
C(19)	0·1949 (3)	0·8440 (2)	0·5237 (2)	4·2
N(20)	0·1785 (4)	0·8690 (3)	0·4250 (2)	6·4

Table 2. *Geometrical data*

(*a*) Torsion angles (°) with e.s.d.'s in parentheses

C(10*a*)—C(1)—C(2)—C(3)	−51·0 (3)	C(6*a*)—C(6)—C(7)—C(8)	22·1 (4)
C(1)—C(2)—C(3)—C(4)	54·9 (3)	C(7)—C(8)—C(9)—C(9*a*)	52·9 (3)
C(2)—C(3)—C(4)—C(4*a*)	−28·9 (4)	C(8)—C(9)—C(9*a*)—C(6*a*)	−23·7 (4)
C(3)—C(4)—C(4a)—C(10*a*)	−1·8 (4)	C(9)—C(9*a*)—C(6*a*)—C(6)	−7·8 (4)
C(4)—C(4*a*)—C(10*a*)—C(1)	6·1 (4)	O(11)—C(6)—C(6*a*)—C(5)	8·8 (4)
C(4*a*)—C(10*a*)—C(1)—C(2)	20·8 (4)	C(6)—C(6*a*)—C(5)—C(12)	77·0 (3)
C(10*a*)—C(4*a*)—C(5)—C(6*a*)	−18·1 (4)	C(12)—C(5)—C(4*a*)—C(4)	−76·7 (3)
C(4*a*)—C(5)—C(6*a*)—C(9*a*)	19·4 (4)	C(5)—C(4*a*)—C(4)—O(13)	−2·3 (3)
C(5)—C(6*a*)—C(9*a*)—N(10)	−8·2 (4)	C(2)—C(1)—C(10*a*)—N(10)	−161·0 (2)
C(6*a*)—C(9*a*)—N(10)—C(10*a*)	−6·8 (4)	C(8)—C(9)—C(9*a*)—N(10)	158·2 (2)
C(9*a*)—N(10)—C(10*a*)—C(4*a*)	8·1 (4)	C(9)—C(9*a*)—N(10)—C(14)	−11·4 (4)
N(10)—C(10*a*)—C(4*a*)—C(5)	5·6 (4)	C(1)—C(10*a*)—N(10)—C(14)	12·6 (4)
C(9*a*)—C(6*a*)—C(6)—C(7)	8·9 (4)	C(2)—C(3)—C(4)—O(13)	153·9 (3)
C(6)—C(7)—C(8)—C(9)	−52·4 (3)	C(8)—C(7)—C(6)—O(11)	−159·1 (3)

(*b*) Puckering parameters for the three rings (Å, °)

q_2	q_3	Q	φ_2	θ
0·401	−0·253	0·474	−53·4	122·3
[C(10*a*) C(1) C(2) C(4) C(4*a*)]				
0·176	0·061	0·186	64·3	70·9
[C(4*a*) C(5) C(6*a*) C(9*a*) N(10) C(10*a*)]				
0·427	0·233	0·486	−177·8	61·3
[C(6*a*) C(6) C(7) C(8) C(9) C(9*a*)]				

puckering parameters for the rings. The numbering of atoms and bond lengths are given in Fig. 1(*a*). The bond angles are given in Fig. 1(*b*). Fig. 2 illustrates the molecular packing viewed along **c**. The mean C—N distance is 1·416 (2) Å and the mean C—N—C angle is 120·0 (2)°; these values agree with those in phenothiazine derivatives (Chu & van der Helm, 1976, 1977). The cyclic N atom N(10) is slightly pyramidal, deviating by 0·1 Å from the plane through C(9*a*), C(10*a*), C(14); the sum of the angles at N(10) is 358·8°. In the dicyano group the C—N length 1·137 (3) Å and C—C—N angle 176·7 (2)° agree with those observed in tetracyanoethylene (Becker, Coppens & Ross, 1973).

The double bonds C(4)=O(13) and C(6)=O(11) have the value 1·24 Å and compare with the corresponding values observed in many organic crystals (see *e.g.* Dideberg, Campsteyn & Dupont, 1973). The central acridinyl ring and the side rings (left and right) are respectively sofa, sofa, and midway between a half-chair and a sofa conformation. The 9-methyl group adopts an axial position on the acridinyl ring, with the dicyano group equatorial.

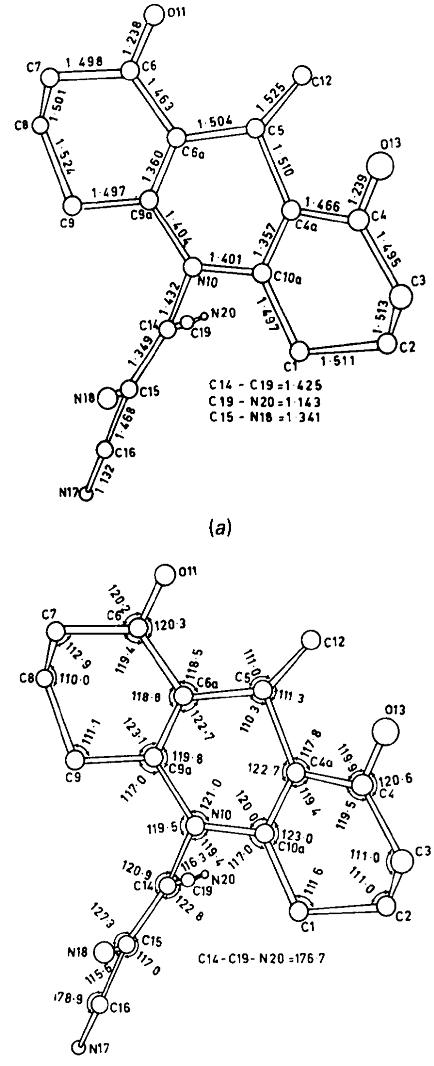

Fig. 1. (*a*) Numbering of atoms and bond lengths (Å) involving non-H atoms. E.s.d.'s from full-matrix least-squares refinement are: C—C, 0·002–0·006; C=O, 0·008; C—N, 0·003–0·004 Å. (*b*) Bond angles (°) involving non-H atoms. E.s.d.'s from full-matrix least-squares refinement are: C—C—C, 0·2–0·3, C—C—O, 0·3 and C—C—N, 0·2–0·3°.

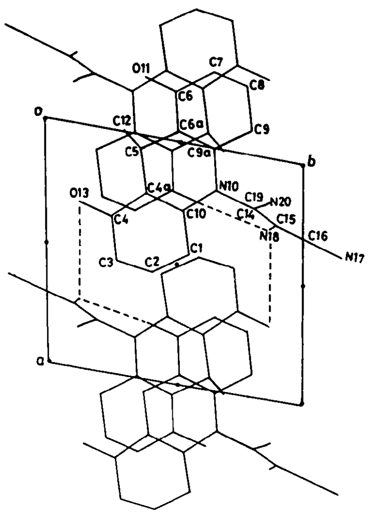

Fig. 2. The packing of the molecule in the projection of the c axis.

The dihedral angle between the least-squares planes of the dicyano group and the acridinyl ring is 92·6 (8)°. N(18) is involved in two hydrogen bonds N(18)···O(11) and N(18)···O(13) with distances and angles 2·84 (3), 2·81 (3) Å and 142 (2), 130 (2)° respectively, and H′(18)···O(11) and H″(18)···O(13) distances 1·93 and 2·00 Å respectively (symmetry: $-x$, $-y+1$, $-z+2$ and x, $y+1$, z). The packing of the molecule is stabilized by the hydrogen-bonding scheme and the van der Waals forces.

We thank Professor Frank R. Fronczek, Department of Chemistry, Louisiana State University and Agricultural and Mechanical College, Louisiana, for helping in data collection.

One of the authors (SS) thanks UGC, New Delhi, India, for the research fellowship.

References

Acheson, R. M. (1956). *Acridines*, 1st edition. London: Arnold.

Becker, P., Coppens, P. & Ross, F. K. (1973). *J. Am. Chem. Soc.* **95**, 7604–7609.

Chu, S. S. C. & van der Helm, D. (1976). *Acta Cryst.* **B32**. 1012–1016.

Chu, S. S. C. & van der Helm, D. (1977). *Acta Cryst.* **B33**, 873–876.

Dideberg, O., Campsteyn, H. & Dupont, L. (1973). *Acta Cryst.* **B29**, 103–112.

Nardelli, M. (1983). *J. Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

Ugliengo, P., Borzani, G. & Viterbo, D. (1988). *J. Appl. Cryst.* **21**, 75.

Acta Cryst. (1989). **C45**, 1348–1350

Crystal Studies of Acridinium Dyes. I. 10-Methyl-9-phenylacridinium Chloride Hydrate

By Kees Goubitz, Celeste A. Reiss and Dick Heijdenrijk

Laboratory for Crystallography, University of Amsterdam, Nieuwe Achtergracht 166, *Amsterdam, The Netherlands*

and Stephan A. Jonker and Jan W. Verhoeven

Laboratory for Organic Chemistry, University of Amsterdam, Nieuwe Achtergracht 166, *Amsterdam, The Netherlands*

(*Received* 6 *October* 1988; *accepted* 28 *April* 1989)

Abstract. $C_{20}H_{16}N^+.Cl^-.H_2O$, $M_r = 323{\cdot}8$, triclinic, $P\bar{1}$, $a = 11{\cdot}0409\ (7)$, $b = 15{\cdot}2215\ (10)$, $c = 5{\cdot}1772\ (4)$ Å, $\alpha = 97{\cdot}599\ (9)$, $\beta = 96{\cdot}633\ (9)$, $\gamma = 108{\cdot}026\ (6)°$, $V = 808{\cdot}7\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}33$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 21{\cdot}2$ cm^{-1}, $F(000) = 340$, room temperature. Final $R = 0{\cdot}058$ for 2340 observed reflections. The angle between the acridinium and the phenyl groups is 68°. The acridinium moiety is planar, the maximum distance from the plane is 0·070 Å for C(9).

Introduction. 10-Methyl-9-phenylacridinium chloride is the basic structure of a group of acridinium dyes with interesting photophysical properties. These properties, as well as their electrochemical behaviour (Koper, Jonker & Verhoeven, 1985) are strongly influenced by the presence of substituents on the phenyl group (Jonker, Ariese & Verhoeven, 1989). This influence is mainly due to the steric effect of the substituents on the twist angle between the planes of the acridinium and the phenyl groups. In order to

0108-2701/89/091348-03$03.00