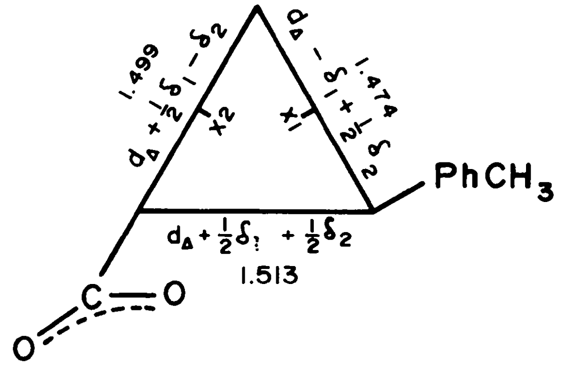
The carboxylic and *p*-tolyl groups both adopt bisected conformations with torsion angles *X*(1)—C(1)—C(4)—O(1) and *X*(2)—C(2)—C(5)—C(6) of −5·1 and −175·3° respectively, where *X*(1) and *X*(2) are the midpoints of the relevant distal bonds.

The C═O bonds of the carboxylate group are equal to within 2σ [C(4)—O(1) = 1·262 (6) and C(4)—O(2) = 1·253 (7) Å], a feature indicating complete electron delocalization. The tolyl group angles and bond distances are in the expected range.

The bond lengths observed for the cyclopropyl ring (I) can be explained by assuming additivity of bond length asymmetries, a principle found to be applicable for pure acceptor substitution (Allen, 1980). The mean C—C distance in the three-membered ring d_Δ is 1·496 (8) Å. The distal bond shortening due to the *p*-tolyl substituent [δ_1 in (I)] is −0·009 Å and δ_2 for the carboxylic group is −0·026 Å. The latter value agrees with the reported mean value for carbonyl groups, δ(C═O) = −0·026 (5) Å (Allen, 1980), but the δ_1 value obtained for the *p*-tolyl substituent is somewhat smaller than that reported for phenyl groups [−0·018 (2) Å]. The precision obtained for the present structure is not high enough to establish firmly that δ(tolyl) is smaller than δ(phenyl) and further work is required to examine this possibility. We note that *E*-2-*p*-nitrophenylcyclopropyl methyl ketone (Bordner, Jones & Johnson, 1972), to our knowledge the only compound so far reported with a similar asymmetry pattern of cyclopropyl bond lengths (1·51, 1·49 and 1·48 Å), yields δ(C═O) = −0·026 Å and $\delta(\mathrm{PhNO_2})$ = −0·017 Å. The latter is larger than our δ_1 value, and both values are similar to the reported values for δ(C═O) and δ(Ph) (Allen, 1980).



The authors are grateful to the CDCHT-ULA and CONICIT-Venezuela (grants C-81-77, C-364, S1-1863 and S1-1878) for financial support and to the CEDEC-ULA for the allocated time.

References

Allen, F. H. (1980). *Acta Cryst.* **B36**, 81–96.
Allen, F. H. (1981). *Acta Cryst.* **B37**, 890–900.
Bordner, J., Jones, L. A. & Johnson, R. L. (1972). *Cryst. Struct. Commun.* **1**, 389–391.
Charton, M. (1970). In *The Chemistry of Alkenes*, edited by J. Zabicky, Vol. II, pp. 511–610. London: Interscience.
Deno, N. C., Richey, H. G. Jr, Liu, J. S., Lincoln, D. N. & Turner, J. O. (1965). *J. Am. Chem. Soc.* **87**, 4533–4538.
Hoffmann, R. (1970). *Tetrahedron Lett.* pp. 2907–2909.
Hoffmann, R. & Stohrer, W. D. (1971). *J. Am. Chem. Soc.* **93**, 6941–6948.
International Tables for X-ray Crystallography (1974). Vol IV. 2nd edition. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Schleyer, P. v. R. & Buss, V. (1969). *J. Am. Chem. Soc.* **91**, 5880–5882.
Sheldrick, G. M. (1977). *SHELX77*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). C**46**, 1079–1081

Crystal Studies of Acridinium Dyes. XII. 9-(4-Dimethylaminophenyl)-10-methylacridinium Chloride

By C. H. Stam

Laboratory for Crystallography, University of Amsterdam, Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands

(*Received* 15 *May* 1989; *accepted* 6 *September* 1989)

Abstract. $\mathrm{C_{22}H_{21}N_2^+.Cl^-}$, $M_r = 348{\cdot}9$, monoclinic, $P2/a$, $a = 11{\cdot}977\,(11)$, $b = 14{\cdot}574\,(10)$, $c = 10{\cdot}461\,(10)$ Å, $\beta = 103{\cdot}79\,(1)°$, $V = 1773\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}31$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 19{\cdot}5$ cm^{-1}, $F(000) = 736$, $T = 295$ K, $R = 0{\cdot}063$ for 2356 observed reflections. There are two independent half molecules in the asymmetric unit. Angles between the plane of the acridinium moiety and phenyl ring are 65·1 and 63·8°.

Introduction. The present investigation is the twelfth in a series of structure determinations of 9-phenylacridinium derivatives (I: Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven, 1989; II, IV: Reiss,

0108-2701/90/061079-03$03.00

Goubitz & Heijdenrijk, 1989*a*,*b*; III, VII: Kronenburg, Goubitz, Reiss & Heijdenrijk, 1989*a*,*b*; V, VIII: Goubitz, Reiss & Heijdenrijk, 1989*a*,*b*; VI: Zoutberg, Reiss, Goubitz & Heijdenrijk, 1989; IX: Reiss, Goubitz, Zoutberg & Heijdenrijk, 1989; X: Häming, Reiss, Goubitz & Heijdenrijk, 1990) undertaken to assist in a study of the effect of various substituents, in this case dimethylamino, on the colour behaviour of these compounds (Jonker, Ariese & Verhoeven, 1989).

Experimental. A black crystal of dimensions 0·08 × 0·2 × 0·6 mm was used to collect 3504 intensities on a Nonius CAD-4 diffractometer employing graphite-monochromated Cu *Kα* radiation and ω–2θ scans. $2{\cdot}5 < \theta < 70°$. h −14, to 14, k 0 to 17, l 0 to 12.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} (Å²)
Cl	0·47401 (12)	0·28937 (7)	0·25839 (12)	0·0837 (7)
Molecule *A*				
C(1)	0·9496 (3)	0·1234 (2)	0·1362 (3)	0·052 (2)
C(2)	1·0433 (3)	0·0743 (3)	0·2042 (4)	0·058 (2)
C(3)	1·0372 (3)	−0·0210 (3)	0·2057 (4)	0·062 (2)
C(4)	0·9419 (3)	−0·0687 (3)	0·1403 (4)	0·056 (2)
C(4*a*)	0·8462 (3)	−0·0190 (2)	0·0677 (3)	0·047 (2)
C(9)	0·7500	0·1285 (3)	0·0000	0·045 (2)
C(9*a*)	0·8484 (3)	0·0777 (2)	0·0661 (3)	0·045 (2)
N(10)	0·7500	−0·0660 (3)	0·0000	0·052 (2)
C(11)	0·7500	−0·1682 (4)	0·0000	0·082 (4)
C(1′)	0·7500	0·2294 (3)	0·0000	0·044 (2)
C(2′)	0·6751 (3)	0·2800 (2)	0·0578 (3)	0·048 (2)
C(3′)	0·6764 (3)	0·3736 (2)	0·0601 (3)	0·048 (2)
C(4′)	0·7500	0·4252 (3)	0·0000	0·046 (2)
N(7′)	0·7500	0·5181 (3)	0·0000	0·057 (3)
C(8′)	0·6730 (4)	0·5715 (3)	0·0613 (5)	0·081 (3)
Molecule *B*				
C(1)	0·0501 (3)	0·3050 (2)	0·3642 (3)	0·051 (2)
C(2)	−0·0422 (3)	0·3537 (3)	0·2970 (4)	0·059 (2)
C(3)	−0·0389 (3)	0·4492 (3)	0·2956 (4)	0·061 (2)
C(4)	0·0562 (3)	0·4964 (2)	0·3630 (4)	0·055 (2)
C(4*a*)	0·1533 (3)	0·4475 (2)	0·4337 (3)	0·045 (2)
C(9)	0·2500	0·3001 (3)	0·5000	0·041 (2)
C(9*a*)	0·1513 (3)	0·3506 (2)	0·4348 (3)	0·042 (2)
N(10)	0·2500	0·4941 (3)	0·5000	0·047 (2)
C(11)	0·2500	0·5952 (4)	0·5000	0·072 (4)
C(1′)	0·2500	0·1985 (3)	0·5000	0·040 (2)
C(2′)	0·3248 (3)	0·1485 (2)	0·4420 (3)	0·044 (2)
C(3′)	0·3244 (3)	0·0545 (2)	0·4413 (3)	0·044 (2)
C(4′)	0·2500	0·0030 (3)	0·5000	0·040 (2)
N(7′)	0·2500	−0·0905 (3)	0·5000	0·053 (2)
C(8′)	0·3287 (4)	−0·1434 (3)	0·4434 (5)	0·078 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*
C(1)—C(2)	1·378 (5)	1·359 (5)
C(1)—C(9*a*)	1·424 (5)	1·425 (5)
C(2)—C(3)	1·391 (6)	1·393 (6)
C(3)—C(4)	1·371 (5)	1·373 (5)
C(4)—C(4*a*)	1·414 (5)	1·413 (5)
C(4*a*)—C(9a)	1·409 (5)	1·412 (5)
C(4*a*)—N(10)	1·382 (4)	1·379 (4)
C(9)—C(9*a*)	1·423 (4)	1·421 (4)
C(9)—C(1′)	1·471 (7)	1·481 (7)
N(10)—C(11)	1·490 (7)	1·473 (7)
C(1′)—C(2′)	1·405 (5)	1·400 (5)
C(2′)—C(3′)	1·364 (5)	1·370 (5)
C(3′)—C(4′)	1·415 (5)	1·413 (5)
C(4′)—N(7′)	1·354 (7)	1·362 (6)
N(7′)—C(8′)	1·466 (6)	1·449 (6)
C(2)—C(1)—C(9*a*)	120·8 (3)	120·7 (4)
C(1)—C(2)—C(3)	119·0 (4)	120·4 (3)
C(2)—C(3)—C(4)	122·7 (4)	121·1 (4)
C(3)—C(4)—C(4*a*)	118·7 (4)	119·7 (4)
C(4)—C(4*a*)—C(9*a*)	120·2 (3)	119·6 (3)
C(4)—C(4*a*)—N(10)	119·4 (4)	120·1 (3)
C(9*a*)—C(4*a*)—N(10)	120·3 (3)	120·2 (3)
C(9*a*)—C(9)—C(1′)	121·4 (2)	121·2 (2)
C(9*a*)—C(9)—C(9*a*)*	117·3 (4)	117·7 (4)
C(1)—C(9*a*)—C(4*a*)	118·5 (3)	118·5 (3)
C(1)—C(9*a*)—C(9)	120·7 (4)	121·0 (3)
C(4*a*)—C(9*a*)—C(9)	120·7 (3)	120·5 (3)
C(4*a*)—N(10)—C(11)	119·7 (2)	119·6 (2)
C(4*a*)—N(10)—C(4*a*)*	120·6 (4)	120·9 (4)
C(9)—C(1′)—C(2′)	121·7 (2)	121·3 (3)
C(2′)—C(1′)—C(2′)*	116·7 (4)	117·3 (4)
C(1′)—C(2′)—C(3′)	121·8 (4)	121·4 (3)
C(2′)—C(3′)—C(4′)	122·0 (4)	122·1 (4)
C(3′)—C(4′)—N(7′)	122·1 (2)	122·1 (2)
C(3′)—C(4′)—C(3′)*	115·8 (4)	115·8 (4)
C(4′)—N(7′)—C(8′)	122·0 (3)	122·1 (3)
C(8′)—N(7′)—C(8′)*	115·9 (4)	115·7 (4)

*These atoms are derived from those in Table 1 by the operations $1\frac{1}{2}-x$, y, $-z$ (molecule *A*) and $\frac{1}{2}-x$, y, $1-z$ (molecule *B*).

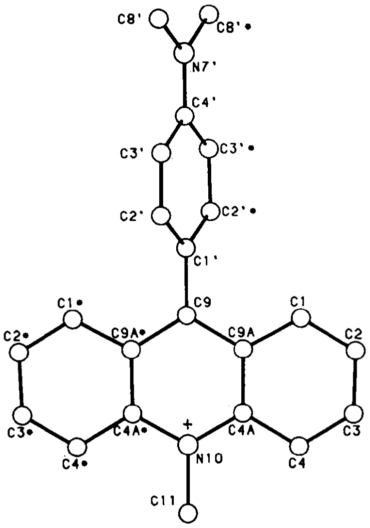


Fig. 1. *PLUTO* drawing of molecule *A*.

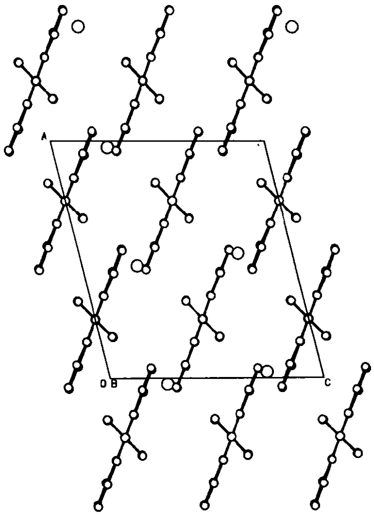


Fig. 2. Projection of the structure along [010].

1148 reflections were below the 2·5σ level and were treated as unobserved. Two standard reflections (141 and $\bar{1}13$) were measured hourly and showed no systematic change. Corrections for Lorentz and polarization effects were applied. Lattice parameters from least-squares adjustment to sinθ values of 23 reflections in the range $60 < 2\theta < 70°$.

The structure was solved from the Patterson synthesis. The asymmetric unit was found to comprise two independent half molecules straddling the crystallographic diad axes at $x = \frac{3}{4}$, $z = 0$ and $x = \frac{1}{4}$, $z = \frac{1}{2}$.

Refinement on F proceeded by means of block-diagonal least-squares calculations, anisotropic for the non-H and isotropic for H atoms. The H atoms were very well indicated in a ΔF synthesis, except those at C(11) which have been omitted. An empirical absorption correction (*DIFABS*; Walker & Stuart, 1983) was applied, with correction in the range 0·50–1·51. A weighting scheme $w = 1/(5{\cdot}2 + F_o + 0{\cdot}0043F_o^2)$ was employed and the anomalous dispersion of Cl was taken into account. The final R value was 0·063 ($wR = 0{\cdot}085$), $(\Delta/\sigma)_{\max} = 0{\cdot}47$. The residual density in a final difference synthesis was between $-0{\cdot}2$ and $0{\cdot}3$ e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968). All calculations were carried out with *XRAY76* (Stewart *et al.*, 1976).

Discussion. Final parameters for the non-H atoms are listed in Table 1.* Bond lengths and angles are given in Table 2. The atomic numbering is indicated in the *PLUTO* drawing of Fig. 1. The two independent molecules are very similar. There is a slight difference in the angle between the planes of the acridinium moiety and the phenyl group (*A* 65·1, *B* 63·8°). The Cl ion is surrounded by six C atoms at distances of 3·4–3·8 Å. The packing is indicated in Fig. 2.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52625 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Goubitz, K., Reiss, C. A. & Heijdenrijk, D. (1989*a*). *Acta Cryst.* **C45**, 1356–1358.

Goubitz, K., Reiss, C. A. & Heijdenrijk, D. (1989*b*). *Acta Cryst.* **C45**, 1364–1366.

Goubitz, K., Reiss, C. A., Heijdenrijk, D., Jonker, S. A. & Verhoeven, J. W. (1989). *Acta Cryst.* **C45**, 1348–1350.

Häming, L. P., Reiss, C. A., Goubitz, K. & Heijdenrijk, D. (1990). *Acta Cryst.* **C46**, 462–465.

Jonker, S. A., Ariese, F. & Verhoeven, J. W. (1989). *Recl Trav. Chim. Pays-Bas*, **108**, 109–115.

Kronenburg, M. J., Goubitz, K., Reiss, C. A. & Heijdenrijk, D. (1989*a*). *Acta Cryst.* **C45**, 1352–1353.

Kronenburg, M. J., Goubitz, K., Reiss, C. A. & Heijdenrijk, D. (1989*b*). *Acta Cryst.* **C45**, 1361–1363.

Reiss, C. A., Goubitz, K. & Heijdenrijk, D. (1989*a*). *Acta Cryst.* **C45**, 1350–1352.

Reiss, C. A., Goubitz, K. & Heijdenrijk, D. (1989*b*). *Acta Cryst.* **C45**, 1354–1356.

Reiss, C. A., Goubitz, K., Zoutberg, M. C. & Heijdenrijk, D. (1989). *Acta Cryst.* **C45**, 1366–1369.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Zoutberg, M. C., Reiss, C. A., Goubitz, K. & Heijdenrijk, D. (1989). *Acta Cryst.* **C45**, 1359–1361.

Acta Cryst. (1990). **C46**, 1081–1084

Crystal Studies of Acridinium Dyes. XIII.* The Structures of 10-Methyl-9-(4-methylphenyl)acridinium Perchlorate (*A*) and 10-Methyl-9-(4-methoxyphenyl)-acridinium Perchlorate (*B*)

By Kees Goubitz, Celeste A. Reiss and Dick Heijdenrijk

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

(*Received* 3 *July* 1989; *accepted* 6 *September* 1989)

Abstract. (*A*): $C_{21}H_{18}N^+.ClO_4^-$, $M_r = 383{\cdot}8$, monoclinic, $P2_1/n$, $a = 8{\cdot}3480\ (8)$, $b = 16{\cdot}9618\ (14)$, $c = 13{\cdot}2145\ (8)$ Å, $\beta = 96{\cdot}106\ (6)°$, $V = 1860{\cdot}5\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 23{\cdot}7$ cm^{-1}, $F(000) = 800$, room temperature. Final $R = 0{\cdot}067$ for 2242 observed reflections. (*B*):

---

* Part XII, preceding paper.