

Cromer & Mann (1968); *International Tables for X-ray Crystallography* (1974). Anomalous dispersion for Cl was corrected for. All calculations were performed with XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), unless stated otherwise.*

Discussion. Final positional parameters for the non-H atoms are listed in Table 1, bond lengths and bond angles in Table 2. A PLUTO (Motherwell & Clegg, 1978) drawing of the molecule is given in Fig. 1 and the Newman projection along the C(9)—C(1') bond in Fig. 2. A least-squares plane through the acridinium atoms shows that the maximum distance from the plane is 0.070 Å [C(9)]. N(10) lies 0.055 Å from the plane, all other distances are less than 0.048 Å. The phenyl ring is planar {max. distance

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51845 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

from the best plane is 0.014 Å [C(4')]]. The angle between the least-squares planes through these moieties is 68°.

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Crystal Studies of Acridinium Dyes. II. 9-(4-Dimethylamino-2-methylphenyl)-10-methylacridinium Chloride Dihydrate

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Abstract. C₂₃H₂₃N₂⁺.Cl⁻.2H₂O, *M*_r = 398.9, triclinic, *P* $\bar{1}$, *a* = 9.3079 (6), *b* = 12.9187 (9), *c* = 9.1687 (6) Å, α = 107.577 (10), β = 91.752 (7), γ = 86.121 (7)°, *V* = 1048.6 (2) Å³, *Z* = 2, *D*_x = 1.26 g cm⁻³, λ (Cu *K* α) = 1.5418 Å, μ = 16.6 cm⁻¹, *F*(000) = 424, room temperature. Final *R* = 0.063 for 2698 observed reflections. The acridinium group and the phenyl ring are each nearly planar. The angle between the acridinium plane and the phenyl ring is 72°. There are no unusual bond lengths or angles.

Introduction. The title compound is the second structure in a series of acridinium dyes [I: Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven (1989)]. In the structure here presented the phenyl ring is substituted with a methyl and a dimethylamino group.

Experimental. A blue-purple plate-shaped crystal (dimensions 0.08 × 0.38 × 0.38 mm approximately)

was used for data collection on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Cu *K* α radiation and θ - 2θ scan. A total of 3963 unique reflections was measured within the range $-11 \leq h \leq 11$, $-15 \leq k \leq 15$, $0 \leq l \leq 11$. Of these, 2698 were above the significance level of $2.5\sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0.61 Å⁻¹. Two standard reflections (221, 002) were measured hourly, the intensity decrease was 5.1% during the 45 h collecting time. Unit-cell parameters were refined by a least-squares-fitting procedure using 23 reflections with $70 < 2\theta < 80^\circ$. Corrections for Lorentz and polarization effects were applied. The structure was determined by direct methods using the program SIMPEL (Schenk & Kiers, 1985). From a ΔF synthesis the water molecules and the H atoms were derived. Block-diagonal least-squares refinement on *F*, anisotropic for the non-H and isotropic for the H atoms, converged to *R* = 0.063,

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Table 1. Fractional coordinates of the non-H atoms and equivalent isotropic thermal parameters

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Cl	0.9992 (1)	0.14324 (10)	0.2657 (1)	0.0658 (6)
C(1)	0.3671 (4)	0.1659 (3)	0.3973 (4)	0.045 (2)
C(2)	0.4040 (4)	0.1061 (3)	0.2537 (4)	0.057 (2)
C(3)	0.5415 (5)	0.0512 (4)	0.2274 (4)	0.062 (2)
C(4)	0.6361 (4)	0.0530 (3)	0.3429 (4)	0.052 (2)
C(5)	0.7455 (4)	0.1617 (3)	0.8871 (5)	0.057 (2)
C(6)	0.7086 (5)	0.2159 (4)	1.0320 (5)	0.069 (3)
C(7)	0.5792 (5)	0.2798 (4)	1.0648 (5)	0.070 (3)
C(8)	0.4882 (4)	0.2864 (3)	0.9487 (4)	0.054 (2)
C(9)	0.4264 (3)	0.2341 (4)	0.6730 (4)	0.038 (2)
C(11)	0.8404 (4)	0.0638 (4)	0.5816 (5)	0.063 (3)
N(10)	0.6926 (3)	0.1153 (2)	0.6144 (3)	0.042 (2)
C(4a)	0.5997 (3)	0.1135 (3)	0.4941 (4)	0.040 (2)
C(8a)	0.5202 (4)	0.2293 (3)	0.7946 (4)	0.041 (2)
C(9a)	0.4624 (3)	0.1723 (3)	0.5242 (4)	0.037 (2)
C(10a)	0.6553 (4)	0.1676 (3)	0.7618 (4)	0.042 (2)
O(1s)	0.7691 (4)	0.3163 (3)	0.4694 (5)	0.092 (3)
O(2s)	0.1324 (5)	0.0766 (3)	0.9313 (5)	0.099 (3)
C(1')	0.2881 (3)	0.3028 (3)	0.7034 (4)	0.039 (2)
C(2')	0.2705 (3)	0.3981 (3)	0.6628 (4)	0.042 (2)
C(3')	0.1400 (4)	0.4600 (3)	0.6909 (4)	0.045 (2)
C(4')	0.0239 (4)	0.4274 (3)	0.7553 (4)	0.044 (2)
C(5')	0.0444 (4)	0.3320 (3)	0.7973 (4)	0.047 (2)
C(6')	0.1745 (4)	0.2711 (3)	0.7722 (4)	0.046 (2)
C(7')	0.3910 (5)	0.4384 (4)	0.5908 (6)	0.065 (3)
C(9')	-0.1168 (5)	0.5960 (4)	0.7616 (6)	0.071 (3)
C(10')	-0.2197 (4)	0.4607 (4)	0.8605 (5)	0.064 (2)
N(8')	-0.1087 (3)	0.4863 (3)	0.7729 (4)	0.061 (2)

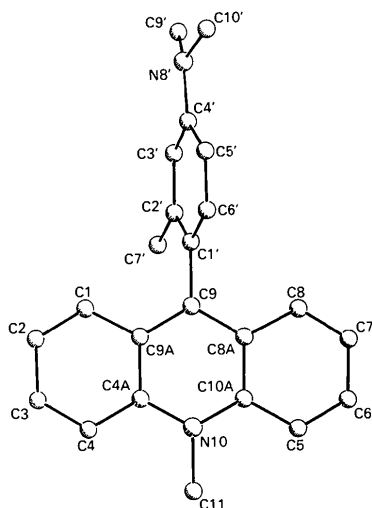


Fig. 1. Structure of $[\text{C}_{23}\text{H}_{23}\text{N}_2]^+$ showing the numbering scheme.

$wR = 0.082$, $(\Delta/\sigma)_{\text{max}} = 0.45$. A weighting scheme $w = (5.04 + F_{\text{iso}} + 0.081F_{\text{obs}}^2)^{-1}$ was used. The isotropic secondary-extinction coefficient refined to $2.6 (6) \times 10^{-4}$ (Zachariassen, 1968). An empirical absorption correction was applied, with corrections in the range 0.44–1.63 (DIFABS; Walker & Stuart, 1983). A final difference Fourier map revealed a residual electron density between -0.2 and 0.5 e \AA^{-3} . Scattering factors were taken from Cromer & Mann

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

C(1)—C(2)	1.351 (5)	N(10)—C(4a)	1.376 (4)
C(1)—C(9a)	1.427 (5)	N(10)—C(10a)	1.360 (4)
C(2)—C(3)	1.411 (6)	C(4a)—C(9a)	1.432 (4)
C(3)—C(4)	1.352 (6)	C(8a)—C(10a)	1.432 (5)
C(4)—C(4a)	1.409 (5)	C(1')—C(2')	1.388 (5)
C(5)—C(6)	1.344 (6)	C(1')—C(6')	1.392 (5)
C(5)—C(10a)	1.420 (6)	C(2')—C(3')	1.394 (5)
C(6)—C(7)	1.400 (7)	C(2')—C(7')	1.515 (6)
C(7)—C(8)	1.359 (6)	C(3')—C(4')	1.394 (5)
C(8)—C(8a)	1.413 (5)	C(4')—C(5')	1.398 (6)
C(9)—C(8a)	1.409 (5)	C(4')—N(8')	1.393 (5)
C(9)—C(9a)	1.395 (4)	C(5')—C(6')	1.385 (5)
C(9)—C(1')	1.497 (4)	C(9')—N(8')	1.449 (7)
C(11)—N(10)	1.485 (5)	C(10')—N(8')	1.442 (6)
C(2)—C(1)—C(9a)	121.5 (3)	C(1)—C(9a)—C(9)	122.3 (3)
C(1)—C(2)—C(3)	119.4 (4)	C(1)—C(9a)—C(4a)	117.6 (3)
C(2)—C(3)—C(4)	121.9 (3)	C(9)—C(9a)—C(4a)	120.1 (3)
C(3)—C(4)—C(4a)	119.8 (3)	C(5)—C(10a)—N(10)	122.0 (3)
C(6)—C(5)—C(10a)	121.3 (4)	C(5)—C(10a)—C(8a)	117.9 (3)
C(5)—C(6)—C(7)	121.1 (4)	N(10)—C(10a)—C(8a)	120.0 (3)
C(6)—C(7)—C(8)	119.7 (4)	C(9)—C(1')—C(2')	120.9 (3)
C(7)—C(8)—C(8a)	121.6 (4)	C(9)—C(1')—C(6')	120.1 (3)
C(8a)—C(9)—C(9a)	119.2 (3)	C(2')—C(1')—C(6')	119.1 (3)
C(8a)—C(9)—C(1')	120.4 (3)	C(1')—C(2')—C(3')	119.7 (3)
C(9a)—C(9)—C(1')	120.3 (3)	C(1')—C(2')—C(7')	121.7 (3)
C(11)—N(10)—C(4a)	119.0 (3)	C(3')—C(2')—C(7')	118.6 (4)
C(11)—N(10)—C(10a)	119.1 (3)	C(2')—C(3')—C(4')	121.8 (4)
C(4a)—N(10)—C(10a)	121.8 (3)	C(3')—C(4')—C(5')	117.6 (3)
C(4)—C(4a)—N(10)	121.2 (3)	C(3')—C(4')—N(8')	121.0 (4)
C(4)—C(4a)—C(9a)	119.7 (3)	C(5')—C(4')—N(8')	121.4 (4)
N(10)—C(4a)—C(9a)	119.1 (3)	C(4')—C(5')—C(6')	120.9 (4)
C(8)—C(8a)—C(9)	122.4 (3)	C(1')—C(1')—C(5')	120.9 (4)
C(8)—C(8a)—C(10a)	118.3 (3)	C(4')—N(8')—C(9')	119.2 (4)
C(9)—C(8a)—C(10a)	119.3 (3)	C(9')—N(8')—C(10')	119.7 (4)
C(1)—C(2)—C(3)—C(4)	121.5 (3)	C(1)—C(9a)—C(9a)—C(9)	122.3 (3)
C(2)—C(3)—C(4)—C(4a)	119.8 (3)	C(1)—C(9a)—C(4a)—C(9a)	117.6 (3)
C(3)—C(4)—C(4a)—C(9a)	119.8 (3)	C(9)—C(9a)—C(4a)—C(9a)	120.1 (3)
C(4)—C(4a)—C(9a)—C(9)	119.8 (3)	C(5)—C(10a)—N(10)—C(10a)	122.0 (3)
C(5)—C(6)—C(7)—C(8)	121.3 (4)	C(5)—C(10a)—C(8a)—C(10a)	117.9 (3)
C(6)—C(7)—C(8)—C(8a)	121.1 (4)	N(10)—C(10a)—C(8a)—C(10a)	120.0 (3)
C(7)—C(8)—C(8a)—C(9)	119.7 (4)	C(9)—C(1')—C(2')—C(1')	120.9 (3)
C(8)—C(8a)—C(9)—C(9a)	121.6 (4)	C(9)—C(1')—C(6')—C(1')	120.1 (3)
C(8a)—C(9)—C(9a)—C(9)	119.2 (3)	C(2')—C(1')—C(6')—C(1')	119.1 (3)
C(8a)—C(9)—C(1')—C(1')	120.4 (3)	C(1')—C(2')—C(3')—C(2')	119.7 (3)
C(9a)—C(9)—C(1')—C(1')	120.3 (3)	C(1')—C(2')—C(7')—C(2')	121.7 (3)
C(11)—N(10)—C(4a)—C(4a)	119.0 (3)	C(3')—C(2')—C(7')—C(2')	118.6 (4)
C(11)—N(10)—C(10a)—C(10a)	119.1 (3)	C(2')—C(3')—C(4')—C(3')	121.8 (4)
C(4a)—N(10)—C(10a)—C(10a)	121.8 (3)	C(3')—C(4')—C(5')—C(4')	117.6 (3)
C(4)—C(4a)—N(10)—C(10a)	121.2 (3)	C(3')—C(4')—N(8')—C(4')	121.0 (4)
C(4)—C(4a)—C(9a)—C(9a)	119.7 (3)	C(5')—C(4')—N(8')—C(4')	121.4 (4)
N(10)—C(4a)—C(9a)—C(9a)	119.1 (3)	C(4')—C(5')—C(6')—C(4')	120.9 (4)
C(8)—C(8a)—C(9)—C(9a)	122.4 (3)	C(1')—C(1')—C(5')—C(1')	120.9 (4)
C(8)—C(8a)—C(10a)—C(10a)	118.3 (3)	C(4')—N(8')—C(9')—C(4')	119.2 (4)
C(9)—C(8a)—C(10a)—C(10a)	119.3 (3)	C(9')—N(8')—C(10')—C(9')	119.7 (4)

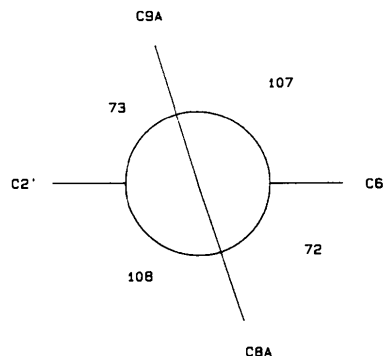


Fig. 2. The Newman projection along the C(9)—C(1') bond.

(1968); *International Tables for X-ray Crystallography* (1974). Anomalous dispersion for Cl was corrected for. All calculations were performed with XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), unless stated otherwise.*

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51846 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Final positional parameters for the non-H atoms are listed in Table 1, bond lengths and bond angles in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule is given in Fig. 1 and the Newman projection along the C(9)—C(1') bond in Fig. 2. A least-squares plane through the acridinium atoms shows that the maximum distance from the plane is 0.095 Å [C(2)]. N(10) lies 0.085, C(6) 0.085 and C(9) 0.078 Å from the plane, all other distances are less than 0.061 Å. The phenyl ring is planar {max. distance from the best plane is 0.014 Å [C(3')]}]. The angle between the two planes is 72°.

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Crystal Studies of Acridinium Dyes. III. 10-Methyl-9-(2-methylphenyl)acridinium Perchlorate

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(Received 13 October 1988; accepted 28 April 1989)

Abstract. C₂₁H₁₈N⁺.ClO₄⁻, *M_r* = 383.8, monoclinic, *P*2₁/*a*, *a* = 14.738 (2), *b* = 11.866 (2), *c* = 11.041 (2) Å, β = 105.75 (2)°, *V* = 1858.4 (6) Å³, *Z* = 4, *D_x* = 1.37 g cm⁻³, λ(Mo *Kα*) = 0.71069 Å, μ = 2.28 cm⁻¹, *F*(000) = 800, room temperature. Final *R* = 0.066 for 1830 observed reflections. The acridinium group and phenyl ring are each nearly planar. The angle between the acridinium plane and the phenyl ring is 76°. There are no unusual bond lengths or angles.

Introduction. The title compound is the third structure in a series of acridinium dyes [I: Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven (1989); II: Reiss, Goubitz & Heijdenrijk (1989)]. In the structure here presented the phenyl ring is substituted with a methyl group.

Experimental. A bright-yellow cubic-shaped crystal (dimensions 0.30 × 0.30 × 0.30 mm approximately) was used for data collection on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Mo *Kα* radiation and θ-2θ scan. A total of 3862 reflections was measured within the range -18 ≤ *h* ≤ 18, 0 ≤ *k* ≤ 14, 0 ≤ *l* ≤ 13. Of these, 1830 were above the significance level of 2.5σ(*I*). The maximum value of (sinθ)/λ was 0.61 Å⁻¹. Two standard reflections (021, 201) were measured hourly, the

intensity decrease was 4.6% during 43 h collection time. Unit-cell parameters were refined by a least-

Table 1. Fractional coordinates of the non-H atoms and equivalent isotropic thermal parameters

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i> (Å ²)
Cl	0.3611 (1)	0.7971 (1)	0.3894 (1)	0.0694 (9)
C(1)	0.3063 (4)	1.2380 (5)	0.2828 (5)	0.058 (3)
C(2)	0.3859 (4)	1.2725 (5)	0.3670 (5)	0.067 (4)
C(3)	0.3850 (4)	1.3793 (6)	0.4258 (5)	0.075 (4)
C(4)	0.3084 (4)	1.4443 (5)	0.4021 (5)	0.066 (4)
C(5)	-0.0238 (4)	1.5021 (5)	0.1840 (6)	0.069 (4)
C(6)	-0.1019 (4)	1.4693 (5)	0.0957 (6)	0.076 (4)
C(7)	-0.1028 (4)	1.3710 (6)	0.0247 (6)	0.070 (4)
C(8)	-0.0242 (4)	1.3064 (5)	0.0464 (5)	0.056 (3)
C(9)	-0.1404 (3)	1.2693 (4)	0.1629 (4)	0.045 (3)
C(11)	0.1404 (5)	1.5751 (6)	0.3677 (7)	0.086 (5)
N(10)	0.1421 (3)	1.4700 (3)	0.2954 (4)	0.054 (3)
O(1)	0.2812 (5)	0.7775 (7)	0.2859 (6)	0.156 (6)
O(2)	0.4387 (4)	0.7579 (5)	0.3481 (5)	0.113 (4)
O(3)	0.3764 (8)	0.9076 (6)	0.4172 (10)	0.214 (9)
O(4)	0.3512 (6)	0.7408 (6)	0.4935 (6)	0.146 (6)
C(4a)	0.2235 (4)	1.4080 (4)	0.3175 (4)	0.051 (3)
C(8a)	0.0594 (3)	1.3358 (4)	0.1389 (4)	0.047 (3)
C(9a)	0.2225 (3)	1.3025 (4)	0.2535 (4)	0.048 (3)
C(10a)	0.0605 (4)	1.4382 (4)	0.2086 (5)	0.050 (3)
C(1')	0.1395 (3)	1.1614 (4)	0.0911 (4)	0.045 (3)
C(2')	0.1335 (4)	1.0567 (4)	0.1447 (5)	0.052 (3)
C(3')	0.1358 (4)	0.9610 (5)	0.0716 (5)	0.063 (3)
C(4')	0.1390 (4)	0.9692 (5)	-0.0508 (5)	0.065 (4)
C(5')	0.1422 (4)	1.0713 (5)	-0.1052 (5)	0.065 (4)
C(6')	0.1429 (4)	1.1696 (5)	-0.0349 (5)	0.055 (3)
C(7')	0.1245 (5)	1.0448 (6)	0.2755 (6)	0.079 (4)

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