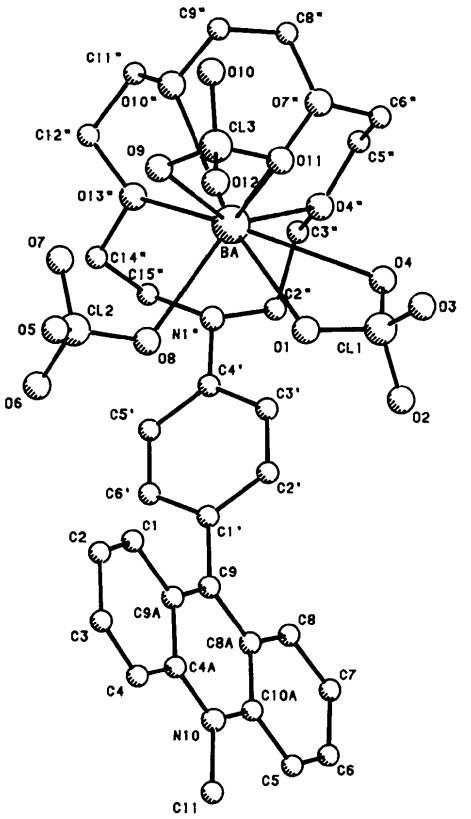

Fig. 1. The molecular structure of the title compound showing the atom numbering.

gravity—Ba is 105°. The Ba ion is coordinated by the four O atoms of the crown ether and by the three perchlorate ions, one unidentate the others bidentate (distances about 3·0 Å). The distance from the Ba ion to the N atom in the crown ether and to the O(7) atom of the unidentate perchlorate is about 3·3 Å. From this it can be argued that the Ba ion is 11 rather than 9 coordinated.

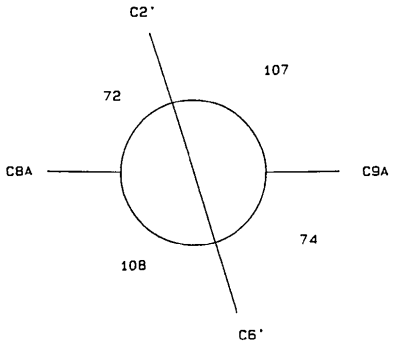

Fig. 2. The Newman projection along the C(9)—C(1′) bond.

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Crystal Studies of Acridinium Dyes. VII. 10-Methyl-9-[4-(1,4,7,10-tetraoxa-13-aza-13-cyclopentadecyl)phenyl]acridinium Silver Diperchlorate Dichloromethane Solvate

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(*Received* 11 *November* 1988; *accepted* 28 *April* 1989)

Abstract. $C_{30}H_{35}N_2O_4^+.Ag^+.2ClO_4^-.2CH_2Cl_2$, M_r = 964·3, monoclinic, $P2_1$, a = 14·861 (8), b = 14·810 (8), c = 9·157 (4) Å, β = 101·86 (5)°, V = 1972 (2) Å^3, Z = 2, D_x = 1.624 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 9·75 cm^{-1}, $F(000)$ = 980, room temperature. Final R = 0·055 for 2917 observed reflections. The angle between the acridinium and phenyl groups is 74°. The mean cavity radius of the crown ether is 1·13 Å.

0108-2701/89/091361-03$03.00

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

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

	x	y	z	U_{eq}(Å^2)
Ag	0·65473 (6)	0·5021 (1)	0·53015 (9)	0·0591 (4)
Cl(1)	0·8285 (2)	0·4159 (3)	0·3437 (4)	0·072 (2)
Cl(2)	0·3105 (3)	0·3883 (3)	0·9767 (4)	0·074 (2)
C(1)	0·8908 (9)	0·150 (1)	1·122 (1)	0·071 (8)
C(2)	0·938 (1)	0·124 (2)	1·255 (2)	0·10 (1)
C(3)	0·986 (1)	0·045 (2)	1·271 (2)	0·11 (1)
C(4)	0·981 (1)	−0·012 (1)	1·152 (2)	0·09 (1)
C(5)	0·880 (1)	−0·068 (1)	0·617 (2)	0·09 (1)
C(6)	0·841 (1)	−0·042 (1)	0·484 (2)	0·09 (1)
C(7)	0·789 (1)	0·043 (1)	0·463 (2)	0·085 (10)
C(8)	0·789 (1)	0·0947 (10)	0·579 (1)	0·069 (8)
C(9)	0·8347 (7)	0·1239 (8)	0·850 (1)	0·053 (6)
C(11)	0·969 (1)	−0·1338 (10)	0·902 (2)	0·09 (1)
N(10)	0·9266 (7)	−0·0419 (7)	0·884 (2)	0·072 (7)
O(1)	0·894 (2)	0·468 (2)	0·296 (4)	0·27 (3)
O(2)	0·855 (2)	0·344 (2)	0·405 (5)	0·34 (4)
O(3)	0·814 (3)	0·409 (4)	0·203 (3)	0·36 (5)
O(4)	0·752 (2)	0·451 (2)	0·357 (5)	0·32 (4)
O(5)	0·265 (2)	0·348 (2)	0·851 (3)	0·21 (2)
O(6)	0·396 (1)	0·350 (1)	1·010 (2)	0·16 (2)
O(7)	0·269 (2)	0·379 (1)	1·091 (3)	0·19 (2)
O(8)	0·322 (1)	0·482 (1)	0·944 (2)	0·15 (1)
C(4a)	0·9297 (7)	0·013 (1)	1·005 (1)	0·066 (7)
C(8a)	0·8342 (7)	0·0693 (8)	0·723 (1)	0·055 (6)
C(9a)	0·8816 (9)	0·0961 (10)	0·994 (1)	0·063 (7)
C(10a)	0·8820 (8)	−0·0149 (8)	0·743 (2)	0·062 (7)
Cl(1s)	0·0798 (3)	0·1564 (4)	0·9071 (6)	0·099 (3)
Cl(2s)	0·0447 (4)	0·2442 (4)	0·6202 (5)	0·108 (3)
Cl(3s)	0·6034 (4)	0·2840 (4)	0·4635 (5)	0·112 (3)
Cl(4s)	0·5895 (5)	0·1774 (4)	0·1942 (7)	0·129 (4)
C(1s)	0·125 (1)	0·182 (1)	0·749 (2)	0·09 (1)
C(2s)	0·586 (1)	0·287 (1)	0·263 (2)	0·09 (1)
C(2″)	0·711 (1)	0·537 (1)	0·898 (2)	0·085 (9)
C(3″)	0·704 (1)	0·630 (1)	0·822 (2)	0·085 (10)
C(5″)	0·758 (2)	0·703 (2)	0·626 (3)	0·16 (2)
C(6″)	0·693 (2)	0·726 (1)	0·504 (3)	0·13 (2)
C(8″)	0·585 (1)	0·675 (1)	0·290 (2)	0·10 (1)
C(9″)	0·551 (1)	0·588 (2)	0·205 (2)	0·12 (1)
C(11″)	0·439 (1)	0·531 (1)	0·330 (2)	0·09 (1)
C(12″)	0·4283 (9)	0·464 (1)	0·443 (2)	0·085 (9)
C(14″)	0·4913 (9)	0·439 (1)	0·703 (2)	0·081 (10)
C(15″)	0·5619 (9)	0·4727 (9)	0·834 (2)	0·068 (8)
N(1″)	0·6556 (8)	0·4678 (7)	0·807 (1)	0·062 (6)
O(4″)	0·742 (1)	0·6228 (9)	0·698 (1)	0·112 (9)
O(7″)	0·6651 (8)	0·6561 (8)	0·400 (1)	0·096 (8)
O(10″)	0·5293 (7)	0·5222 (8)	0·298 (1)	0·083 (6)
O(13″)	0·4933 (6)	0·4923 (9)	0·575 (1)	0·079 (6)
C(1′)	0·7865 (8)	0·2104 (8)	0·837 (1)	0·052 (6)
C(2′)	0·8210 (7)	0·2861 (9)	0·772 (1)	0·060 (7)
C(3′)	0·7775 (8)	0·3682 (7)	0·764 (1)	0·055 (6)
C(4′)	0·6943 (8)	0·3799 (7)	0·820 (1)	0·050 (6)
C(5′)	0·6602 (9)	0·3051 (9)	0·879 (1)	0·062 (7)
C(6′)	0·7040 (8)	0·2209 (9)	0·888 (1)	0·057 (6)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Ag—O(4)	2·47 (4)	N(10)—C(10a)	1·38 (2)
Ag—N(1″)	2·58 (1)	C(4a)—C(9a)	1·42 (2)
Ag—O(4″)	2·53 (1)	C(8a)—C(10a)	1·43 (2)
Ag—O(7″)	2·59 (1)	Cl(1s)—C(1s)	1·76 (2)
Ag—O(10″)	2·540 (9)	Cl(2s)—C(1s)	1·76 (2)
Ag—O(13″)	2·520 (10)	Cl(3s)—C(2s)	1·80 (2)
Cl(1)—O(1)	1·38 (3)	Cl(4s)—C(2s)	1·75 (2)
Cl(1)—O(2)	1·23 (4)	C(2″)—C(3″)	1·54 (2)
Cl(1)—O(3)	1·27 (3)	C(2″)—N(1″)	1·46 (2)
Cl(1)—O(4)	1·28 (3)	C(3″)—O(4″)	1·37 (2)
Cl(2)—O(5)	1·35 (2)	C(5″)—O(6″)	1·36 (4)
Cl(2)—O(6)	1·37 (2)	C(5″)—O(4″)	1·40 (3)
Cl(2)—O(7)	1·33 (3)	C(6″)—O(7″)	1·41 (3)
Cl(2)—O(8)	1·44 (2)	C(8″)—C(9″)	1·54 (3)
C(1)—C(2)	1·33 (2)	C(8″)—O(7″)	1·42 (2)
C(1)—C(9a)	1·40 (2)	C(9″)—O(10″)	1·38 (3)
C(2)—C(3)	1·36 (3)	C(11″)—C(12″)	1·47 (3)
C(3)—C(4)	1·37 (3)	C(11″)—C(10″)	1·44 (2)
C(4)—C(4a)	1·45 (2)	C(12″)—O(13″)	1·45 (2)
C(5)—C(6)	1·30 (2)	C(14″)—C(15″)	1·51 (2)
C(5)—C(10a)	1·39 (2)	C(14″)—O(13″)	1·42 (2)
C(6)—C(7)	1·47 (3)	C(15″)—N(1″)	1·47 (2)
C(7)—C(8)	1·31 (2)	N(1″)—C(4′)	1·42 (2)
C(8)—C(8a)	1·40 (2)	C(1′)—C(2′)	1·41 (2)
C(9)—C(8a)	1·42 (2)	C(1′)—C(6′)	1·41 (2)
C(9)—C(9a)	1·42 (2)	C(2′)—C(3′)	1·37 (2)
C(9)—C(1′)	1·46 (2)	C(3′)—C(4′)	1·44 (2)
C(11)—N(10)	1·49 (2)	C(4′)—C(5′)	1·37 (2)
N(10)—C(4a)	1·37 (2)	C(5′)—C(6′)	1·40 (2)

O(4)—Ag—N(1″)	133·1 (9)	Ag—N(1″)—C(2″)	108·1 (9)
O(4)—Ag—O(4″)	108·1 (8)	Ag—N(1″)—C(15″)	110·0 (8)
O(4)—Ag—O(7″)	82·9 (9)	Ag—N(1″)—C(4″)	100·5 (7)
O(4)—Ag—O(10″)	85·7 (8)	C(2″)—N(1″)—C(15″)	108 (1)
O(4)—Ag—O(13″)	142·0 (8)	C(2″)—N(1″)—C(4′)	115 (1)
N(1″)—Ag—O(4″)	69·2 (4)	C(15″)—N(1″)—C(4′)	114 (1)
N(1″)—Ag—O(7″)	129·5 (4)	Ag—O(4″)—C(3″)	108 (1)
N(1″)—Ag—O(10″)	134·4 (4)	Ag—O(4″)—C(5″)	115 (1)
N(1″)—Ag—O(13″)	69·0 (3)	C(3″)—O(4″)—C(5″)	117 (2)
O(4″)—Ag—O(7″)	65·6 (4)	Ag—O(7″)—C(6″)	112 (1)
O(4″)—Ag—O(10″)	128·4 (4)	Ag—O(7″)—C(8″)	112(1)
O(4″)—Ag—O(13″)	109·1 (4)	C(6″)—O(7″)—C(8″)	115 (2)
O(7″)—Ag—O(10″)	67·4 (4)	Ag—O(10″)—C(9″)	112·1 (9)
O(7″)—Ag—O(13″)	105·9 (4)	Ag—O(10″)—C(11″)	113·3 (8)
O(10″)—Ag—O(13″)	65·4 (3)	C(9″)—O(10″)—C(11″)	114 (1)
O(1)—Cl(1)—O(2)	117 (2)	Ag—O(13″)—C(12″)	112·0 (9)
O(1)—Cl(1)—O(3)	73 (3)	Ag—O(13″)—C(14″)	110·4 (7)
O(1)—Cl(1)—O(4)	120 (2)	C(12″)—O(13″)—C(14″)	114 (1)
O(2)—Cl(1)—O(3)	111 (3)	C(9)—C(1′)—C(2′)	121 (1)
O(2)—Cl(1)—O(4)	121 (3)	C(9)—C(1′)—C(6′)	121 (1)
O(3)—Cl(1)—O(4)	99 (3)	C(2′)—C(1′)—C(6′)	118 (1)
O(5)—Cl(2)—O(6)	107 (1)	C(1′)—C(2′)—C(3′)	121 (1)
O(5)—Cl(2)—O(7)	113 (2)	C(2′)—C(3′)—C(4′)	121 (1)
O(5)—Cl(2)—O(8)	108 (1)	N(1″)—C(4′)—C(3′)	116 (1)
O(6)—Cl(2)—O(7)	110 (1)	N(1″)—C(4′)—C(5′)	127 (1)
O(6)—Cl(2)—O(8)	108 (1)	C(3′)—C(4′)—C(5′)	117 (1)
O(7)—Cl(2)—O(8)	111 (1)	C(4′)—C(5′)—(6′)	122 (1)
		C(1)—C(6)—C(5′)	120 (1)
C(11)—N(10)—C(4a)	121 (1)	C(3″)—C(2″)—N(1″)	113 (1)
C(11)—N(10)—C(10a)	118 (1)	C(2″)—C(3″)—O(4″)	108 (1)
C(4a)—N(10)—C(10a)	121 (1)	C(6″)—C(5″)—O(4″)	116 (2)
Ag—O(4)—Cl(1)	146 (3)	C(5″)—C(6″)—O(7″)	116 (2)
C(4)—C(4a)—N(10)	122 (1)	C(9″)—C(8″)—O(7″)	110 (2)
C(4)—C(4a)—C(9a)	117 (1)	C(8″)—C(9″)—O(10″)	112 (1)
N(10)—C(4a)—C(9a)	122 (1)	C(12″)—C(11″)—O(10″)	109 (1)
C(8)—C(8a)—C(9)	123 (1)	C(11″)—C(12″)—O(13″)	104 (1)
C(8)—C(8a)—C(10a)	119 (1)	C(15″)—C(14″)—O(13″)	110 (1)
C(9)—C(8a)—C(10a)	118 (1)	C(14″)—C(15″)—N(1″)	112 (1)
C(1)—C(9a)—C(9)	124 (1)	C(2)—C(1)—C(9a)	123 (2)
C(1)—C(9a)—C(4a)	118 (1)	C(1)—C(2)—C(3)	121 (2)
C(9)—C(9a)—C(4a)	118 (1)	C(2)—C(3)—C(4)	120 (2)
C(5)—C(10a)—N(10)	122 (1)	C(3)—C(4)—C(4a)	121 (2)
C(5)—C(10a)—C(8a)	118 (1)	C(6)—C(5)—C(10a)	123 (2)
N(10)—C(10a)—C(8a)	120 (1)	C(5)—C(6)—C(7)	120 (2)
Cl(1s)—C(1s)—Cl(2s)	110 (1)	C(6)—C(7)—C(8)	119 (1)
Cl(3s)—C(2s)—Cl(4s)	109·6 (10)	C(7)—C(8)—C(8a)	122 (1)
		C(8a)—C(9)—C(9a)	121 (1)
		C(8a)—C(9)—C(1′)	121·0 (10)
		C(9a)—C(9)—C(1′)	118 (1)

The Ag atom is six-coordinated by five O atoms and one N atom at distances of about 2·5 Å.

Introduction. The title compound is one of a series of acridinium dyes [I: Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven (1989); II and IV: Reiss, Goubitz & Heijdenrijk (1989*a*,*b*); III: Kronenburg, Goubitz, Reiss & Heijdenrijk (1989); V: Goubitz, Reiss & Heijdenrijk (1989); VI: Zoutberg, Reiss, Goubitz & Heijdenrijk (1989)]. In this case the phenyl ring is substituted with an aza-15-crown-5 silver perchlorate complex.

Experimental. A red pillar-shaped crystal (dimensions 0·25 × 0·25 × 0·40 mm approximately) was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation and θ–2θ scan. A total of 5907 reflections was measured within the range $-19 \le h \le 19$, $0 \le k \le 19$, $0 \le l \le 12$. Of these, 2917 were above the significance level of $2{\cdot}5\sigma(I)$. The maximum

value of $(\sin\theta)/\lambda$ was 0·66 Å^{-1} ($\theta \leq 28°$). Two standard reflections (041, 301) were measured hourly, the intensity decrease was 5·3% during 67 h collection time. Unit-cell parameters were refined by a least-squares-fitting procedure using 23 reflections with $32 < 2\theta < 39°$. Corrections for Lorentz and polarization effects were applied. The structure was determined by the heavy-atom method, and appeared to contain two molecules of dichloromethane. The H atoms were derived from a ΔF synthesis. Block-diagonal least-squares refinement on F, anisotropic for the non-H atoms and isotropic with fixed temperature factors for the H atoms, converged to $R = 0{\cdot}055$, $wR = 0{\cdot}084$, $(\Delta/\sigma)_{max} = 0{\cdot}73$. Disorder in one of the perchlorate ions was found. A weighting scheme $w = (1{\cdot}08 + F_{obs} + 0{\cdot}0098F_{obs}^2)^{-1}$ was used. An empirical absorption correction was applied with corrections in the range 0·18–1·08 (*DIFABS;* Walker & Stuart, 1983). A final difference Fourier map revealed a residual electron density between −0·2 and 0·8 e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968); *International Tables for X-ray Crystallography* (1974). Anomalous dispersion for Ag and Cl was corrected for. All calculations were performed with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), unless stated otherwise.*

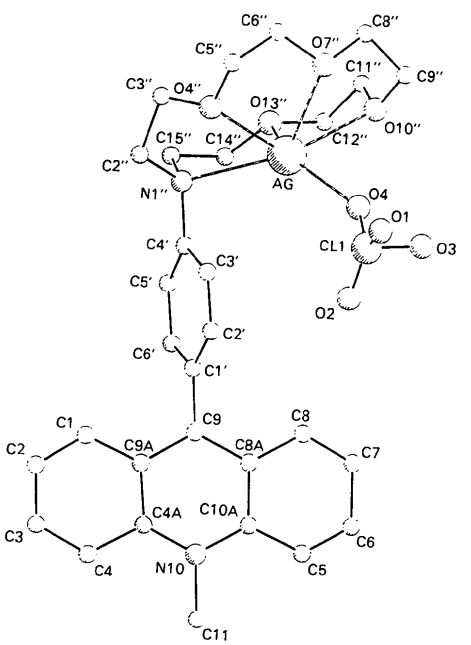

Fig. 1. Structure of $[C_{30}H_{35}N_2O_4Ag(ClO_4)]^+$ showing the numbering scheme.

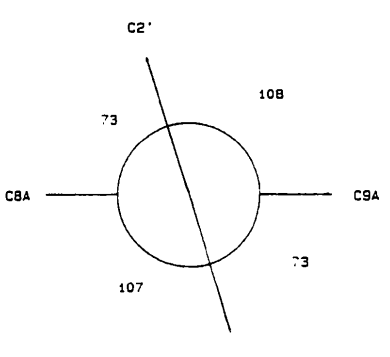

Fig. 2. The Newman projection along the C(9)—C(1′) bond.

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. The phenyl ring is planar within 0·013 Å and the maximum deviation from the acridinium plane is 0·078 Å. The angle between the two planes is 74°. The O atoms of the crown ether lie alternately about 0·3 Å above and below their mean plane; the N atom lies 1·08 Å below this plane. For the mean cavity radius, as defined by Mathieu, Metz, Moras & Weiss (1978), a value of 1·13 Å is obtained. The Ag ion is 6 coordinated by the hetero atoms of the crown ether and by the O(4) atom of the perchlorate ion (distances about 2·5 Å). The distance between the Ag ion and the centre of gravity of the hetero atoms is 0·99 Å, the angle N(1″)—centre of gravity—Ag is 79°.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Documenet Supply Centre as Supplementary publication No. SUP 51851 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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