of gravity of the hetero atoms in the crown ether is 1·76 Å, the angle N(1″)–centre of gravity–Ba is 88°.

The O atoms of the crown ether deviate alternately about 0·08 Å above and below their mean plane; the N atom lies 1·063 Å below this plane.

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 distances from the Ba ion to the N atom in the crown ether and to the O(7) atom of the unidentate perchlorate are each about 3·3 Å. Thus, in analogy with the sixth structure in this series (Zoutberg et al., 1989), it can be argued that the Ba ion is 11- rather than 9-coordinated.

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Crystal Studies of Acridinium Dyes. XI. 10-Methyl-9-[2-methyl-4-(1,4,7,10-tetraoxa-13-aza-13-cyclopentadecyl)phenyl]acridinium Lithium Perchlorate

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Abstract. $C_{31}H_{37}N_2O_4^+.Li^+.2ClO_4^-$, $M_r = 707.5$, orthorhombic, $Pbca$, $a = 13.064$ (1), $b = 15.940$ (2), $c = 31.575$ (4) Å, $V = 6575$ (1) Å^3, $Z = 8$, $D_x = 1.43$ g cm^{-3}, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 23.5$ cm^{-1}, $F(000) = 2960$, room temperature. Final $R = 0.082$ for 2259 observed reflections. The angle between the planes of the acridinium and the phenyl groups is 66°. The Li atom is coordinated by the four O atoms of the crown ether [1·99 (2)–2·20 (2) Å] and by one O atom of one of the perchlorate ions [1·99 (2) Å].

Introduction. This paper is the eleventh in our series of structural studies of acridinium dyes (I: Goubitz, Reiss, Heijdenrijk, Jonker & Verhoeven, 1989; II, IV: Reiss, Goubitz & Heijdenrijk, 1989a,b; III, VII: Kronenburg, Goubitz, Reiss & Heijdenrijk, 1989a,b; V, VIII: Goubitz, Reiss & Heijdenrijk, 1989a,b; VI: Zoutberg, Reiss, Goubitz & Heijdenrijk, 1989; IX: Reiss, Goubitz, Zoutberg & Heijdenrijk, 1989; X: Häming, Reiss, Goubitz & Heijdenrijk, 1989). In this case the phenyl ring is substituted with both a methyl group and an aza[15]crown-5 group which has formed a complex with lithium perchlorate.

Experimental. A dark-brown plate-shaped crystal (dimensions 0·03 × 0·35 × 0·35 mm approximately) was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation and ω–2θ scans. A total of 4089 unique reflections was measured within the range $0 \le h \le 13$, $0 \le k \le 16$, $0 \le l \le 33$. Of these, 2259 were above the significance level of $2.5 \sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0·53 Å^{-1}. Two standard reflections (213, 004) were measured hourly, no significant decrease was observed during the 46 h collection time. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $49 < 2\theta < 54°$. Corrections for Lorentz and polarization effects were applied. The structure was solved by direct methods using the program

$$C_{31}H_{37}N_2O_4^+ \cdot Li^+ \cdot 2ClO_4^-$$
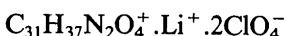
Table 1. *Fractional coordinates of the non-hydrogen atoms and equivalent isotropic thermal parameters*

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

	x	y	z	$U_{eq}(\text{Å}^2)$
Cl(1)	0·2369 (2)	1·2155 (1)	0·38552 (7)	0·067 (1)
Cl(2)	0·1891 (2)	0·3945 (2)	0·16712 (8)	0·082 (2)
Li	0·331 (1)	1·0755 (9)	0·3260 (4)	0·067 (9)
C(1)	0·1252 (6)	1·1246 (5)	0·4960 (2)	0·054 (5)
C(2)	0·0598 (7)	1·1899 (5)	0·4991 (3)	0·066 (6)
C(3)	−0·0096 (7)	1·1911 (6)	0·5337 (3)	0·068 (6)
C(4)	−0·0127 (6)	1·1275 (5)	0·5618 (3)	0·059 (5)
C(5)	0·1001 (7)	0·8524 (5)	0·6088 (3)	0·071 (6)
C(6)	0·1681 (8)	0·7892 (6)	0·6065 (3)	0·082 (7)
C(7)	0·2457 (7)	0·7880 (5)	0·5760 (3)	0·068 (6)
C(8)	0·2548 (6)	0·8531 (5)	0·5484 (3)	0·056 (5)
C(9)	0·1962 (6)	0·9898 (4)	0·5226 (2)	0·042 (4)
C(11)	−0·0401 (8)	0·9899 (7)	0·6163 (3)	0·088 (7)
N(10)	0·0438 (4)	0·9898 (4)	0·5840 (2)	0·052 (4)
O(1)	0·2689 (5)	1·1322 (3)	0·3760 (2)	0·067 (4)
O(2)	0·2995 (7)	1·2756 (4)	0·3681 (3)	0·118 (6)
O(3)	0·254 (1)	1·2291 (5)	0·4301 (3)	0·154 (8)
O(4)	0·1408 (7)	1·2258 (6)	0·3772 (6)	0·26 (1)
O(7)	0·2602 (8)	0·3482 (6)	0·1898 (2)	0·140 (7)
C(4a)	0·0506 (6)	1·0573 (5)	0·5579 (2)	0·050 (4)
C(8a)	0·1872 (6)	0·9216 (5)	0·5499 (2)	0·048 (4)
C(9a)	0·1263 (6)	1·0564 (5)	0·5246 (2)	0·046 (4)
C(10a)	0·1072 (6)	0·9236 (5)	0·5819 (2)	0·053 (5)
O(5a)	0·157 (2)	0·473 (1)	0·1791 (7)	0·146 (7)†
O(6a)	0·2303 (10)	0·4037 (8)	0·1207 (4)	0·075 (3)†
O(8a)	0·109 (1)	0·346 (1)	0·1530 (6)	0·119 (5)†
O(5b)	0·222 (1)	0·474 (1)	0·1703 (5)	0·111 (5)†
O(6b)	0·167 (2)	0·363 (2)	0·1289 (8)	0·172 (9)†
O(8b)	0·097 (2)	0·400 (1)	0·1930 (7)	0·149 (7)†
C(2″)	0·5603 (6)	1·0554 (6)	0·3802 (2)	0·060 (5)
C(3″)	0·5248 (7)	1·1395 (5)	0·3642 (3)	0·067 (6)
C(5″)	0·4696 (7)	1·2060 (6)	0·2999 (3)	0·078 (6)
C(6″)	0·4053 (8)	1·1894 (7)	0·2622 (3)	0·090 (7)
C(8″)	0·237 (1)	1·1310 (8)	0·2468 (4)	0·113 (9)
C(9″)	0·152 (1)	1·0891 (9)	0·2708 (5)	0·13 (1)
C(11″)	0·1983 (9)	0·9491 (8)	0·2845 (4)	0·108 (9)
C(12″)	0·295 (1)	0·9137 (7)	0·2922 (5)	0·13 (1)
C(14″)	0·4684 (8)	0·9265 (6)	0·3134 (3)	0·078 (6)
C(15″)	0·4943 (7)	0·9178 (5)	0·3595 (3)	0·061 (5)
N(1″)	0·4798 (5)	0·9940 (4)	0·3840 (2)	0·055 (4)
O(4″)	0·4785 (4)	1·1284 (3)	0·3232 (2)	0·062 (4)
O(7″)	0·3100 (5)	1·1572 (5)	0·2775 (2)	0·089 (5)
O(10″)	0·1852 (5)	1·0304 (5)	0·2993 (2)	0·089 (5)
O(13″)	0·3633 (6)	0·9576 (5)	0·3105 (3)	0·110 (6)
C(1′)	0·2775 (6)	0·9909 (4)	0·4887 (2)	0·044 (4)
C(2′)	0·3560 (5)	1·0504 (4)	0·4874 (2)	0·041 (4)
C(3′)	0·4234 (6)	1·0490 (4)	0·4526 (2)	0·046 (4)
C(4′)	0·4141 (5)	0·9920 (4)	0·4194 (2)	0·045 (4)
C(5′)	0·3358 (6)	0·9336 (5)	0·4219 (2)	0·053 (5)
C(6′)	0·2709 (6)	0·9316 (5)	0·4565 (2)	0·053 (5)
C(7′)	0·3731 (6)	1·1128 (5)	0·5219 (3)	0·060 (5)

† Refined isotropically.

SIMPEL (Schenk & Kiers, 1983). After isotropic refinement of the non-H atoms, a ΔF synthesis revealed some peaks in the vicinity of one of the perchlorate ions [Cl(2)]. These peaks were interpreted as due to disorder, representing alternative sites for the atoms O(5), O(6) and O(8). We therefore introduced two half atoms for each of these O atoms. In the other perchlorate ion, one of the O atoms [O(4)] is extremely anisotropic. The positions of the H atoms were initially calculated and subsequently refined. Block-diagonal least-squares refinement on F, anisotropic for the non-hydrogen atoms and isotropic for the H atoms and the disordered O atoms, converged to $R = 0.082$, $wR = 0.117$, $(\Delta/\sigma)_{max} = 0.72$, 844 refined parameters, $w = (7.3 + F_{obs} + 0.011F_{obs}^2)^{-1}$. An empirical absorption correction was applied, with corrections in the range 0·78–1·79

(*DIFABS*; Walker & Stuart, 1983). A final difference synthesis revealed residual electron density between −0·3 and 0·5 e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968); *International Tables for X-ray Crystallography* (1974). Anomalous dispersion for Cl was corrected for. All calculations were performed with *XRAY76* (Stewart *et al.*, 1976), unless stated otherwise.

Discussion. Final positional parameters for the non-H atoms are listed in Table 1,* bond lengths and

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52353 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
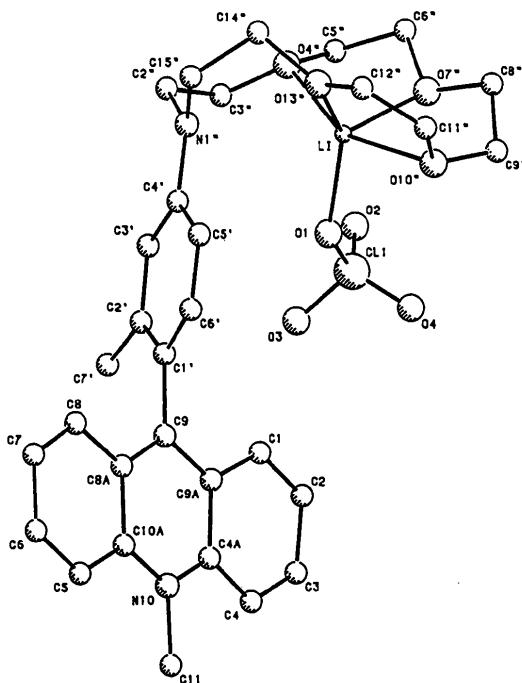

Fig. 1. Structure of $[C_{31}N_2O_4LiClO_4]^+$ showing the numbering scheme.
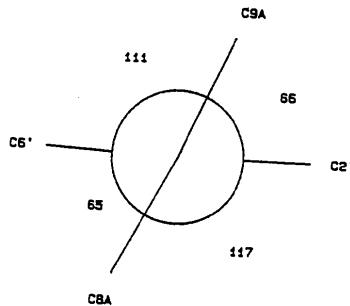
Fig. 2. The Newman projection along the C(9)—C(1′) bond.

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

Bond	Length	Bond	Length
Cl(1)—O(1)	1·424 (6)	N(10)—C(10a)	1·343 (10)
Cl(1)—O(2)	1·374 (8)	C(4a)—C(9a)	1·44 (1)
Cl(1)—O(3)	1·442 (9)	C(8a)—C(10a)	1·45 (1)
Cl(1)—O(4)	1·293 (10)	O(5a)—O(5b)	0·89 (3)
Cl(2)—O(7)	1·39 (1)	O(5a)—O(8b)	1·47 (3)
Cl(2)—O(5a)	1·37 (2)	O(6a)—O(6b)	1·08 (3)
Cl(2)—O(6a)	1·57 (1)	O(8a)—O(6b)	1·11 (3)
Cl(2)—O(8a)	1·38 (2)	O(8a)—O(8b)	1·54 (3)
Cl(2)—O(5b)	1·34 (2)	C(2")—C(3")	1·51 (1)
Cl(2)—O(6b)	1·34 (3)	C(2")—N(1")	1·44 (1)
Cl(2)—O(8b)	1·46 (2)	C(3")—O(4")	1·44 (1)
Li—O(1)	1·99 (2)	C(5")—C(6")	1·48 (1)
Li—O(4")	2·11 (2)	C(5")—O(4")	1·44 (1)
Li—O(7")	2·03 (2)	C(6")—O(7")	1·43 (1)
Li—O(10")	2·20 (2)	C(8")—C(9")	1·50 (2)
Li—O(13")	1·99 (2)	C(8")—O(7")	1·42 (1)
C(1)—C(2)	1·35 (1)	C(9")—O(10")	1·37 (2)
C(1)—C(9a)	1·41 (1)	C(11")—C(12")	1·40 (2)
C(2)—C(3)	1·42 (1)	C(11")—O(10")	1·39 (1)
C(3)—C(4)	1·35 (1)	C(12")—O(13")	1·27 (2)
C(4)—C(4a)	1·40 (1)	C(14")—C(15")	1·50 (1)
C(5)—C(6)	1·35 (1)	C(14")—O(13")	1·46 (1)
C(5)—C(10a)	1·42 (1)	C(15")—N(1")	1·45 (1)
C(6)—C(7)	1·40 (1)	N(1")—C(4')	1·410 (9)
C(7)—C(8)	1·36 (1)	C(1')—C(2')	1·397 (10)
C(8)—C(8a)	1·41 (1)	C(1')—C(6')	1·39 (1)
C(9)—C(8a)	1·392 (10)	C(2')—C(3')	1·408 (10)
C(9)—C(9a)	1·40 (1)	C(2')—C(7')	1·49 (1)
C(9)—C(1')	1·508 (10)	C(3')—C(4')	1·39 (1)
C(11)—N(10)	1·50 (1)	C(4')—C(5')	1·39 (1)
N(10)—C(4a)	1·358 (10)	C(5')—C(6')	1·38 (1)

Angle	Value	Angle	Value
O(1)—Cl(1)—O(2)	113·0 (4)	C(8)—C(8a)—C(10a)	119·5 (7)
O(1)—Cl(1)—O(3)	107·5 (4)	C(9)—C(8a)—C(10a)	118·3 (7)
O(1)—Cl(1)—O(4)	111·1 (5)	C(1)—C(9a)—C(9)	124·1 (7)
O(2)—Cl(1)—O(3)	101·2 (6)	C(1)—C(9a)—C(4a)	116·8 (7)
O(2)—Cl(1)—O(4)	114·1 (7)	C(9)—C(9a)—C(4a)	119·1 (6)
O(3)—Cl(1)—O(4)	109 (1)	C(5)—C(10a)—N(10)	123·9 (7)
O(7)—Cl(2)—O(5a)	123·2 (10)	C(5)—C(10a)—C(8a)	116·4 (7)
O(7)—Cl(2)—O(6a)	107·6 (6)	N(10)—C(10a)—C(8a)	119·7 (7)
O(7)—Cl(2)—O(8a)	112·2 (8)	Cl(2)—O(5a)—O(5b)	69 (2)
O(7)—Cl(2)—O(5b)	104·5 (8)	Cl(2)—O(5a)—O(8b)	62 (1)
O(7)—Cl(2)—O(6b)	114 (1)	O(5b)—O(5a)—O(8b)	128 (2)
O(7)—Cl(2)—O(8b)	107·2 (9)	Cl(2)—O(6a)—O(6b)	57 (1)
O(5a)—Cl(2)—O(6a)	106·1 (10)	Cl(2)—O(8a)—O(6b)	64 (2)
O(5a)—Cl(2)—O(8a)	112 (1)	Cl(2)—O(8a)—O(8b)	60 (1)
O(5a)—Cl(2)—O(5b)	38 (1)	O(6b)—O(8a)—O(8b)	120 (2)
O(5a)—Cl(2)—O(6b)	122 (1)	Cl(2)—O(5b)—O(5a)	73 (2)
O(5a)—Cl(2)—O(8b)	63 (1)	Cl(2)—O(6b)—O(6a)	80 (2)
O(6a)—Cl(2)—O(8a)	90·6 (9)	Cl(2)—O(6b)—O(8a)	68 (2)
O(6a)—Cl(2)—O(5b)	82·6 (8)	O(6a)—O(6b)—O(8a)	146 (3)
O(6a)—Cl(2)—O(6b)	43 (1)	Cl(2)—O(8b)—O(5a)	56 (1)
O(6a)—Cl(2)—O(8b)	143·3 (10)	Cl(2)—O(8b)—O(8a)	55 (1)
O(8a)—Cl(2)—O(5b)	143 (1)	O(5a)—O(8b)—O(8a)	98 (2)
O(8a)—Cl(2)—O(6b)	48 (1)	C(3")—C(2")—N(1")	114·1 (7)
O(8a)—Cl(2)—O(8b)	66 (1)	C(2")—C(3")—O(4")	108·8 (7)
O(5b)—Cl(2)—O(6b)	119 (1)	C(6")—C(5")—O(4")	107·6 (8)
O(5b)—Cl(2)—O(8b)	100 (1)	C(5")—C(6")—O(7")	106·6 (8)
O(6b)—Cl(2)—O(8b)	110 (1)	C(9")—C(8")—O(10")	106·4 (10)
O(1)—Li—O(4")	103·0 (7)	C(8")—C(9")—O(10")	114 (1)
O(1)—Li—O(7")	104·6 (7)	C(12")—C(11")—O(10")	115 (1)
O(1)—Li—O(10")	95·7 (7)	C(11")—C(12")—O(13")	119 (1)
O(1)—Li—O(13")	135·3 (8)	C(15")—C(14")—O(13")	107·7 (8)
O(4")—Li—O(7")	80·5 (6)	C(14")—C(15")—N(1")	114·2 (7)
O(4")—Li—O(10")	154·9 (8)	C(2")—N(1")—C(15")	115·4 (6)
O(4")—Li—O(13")	100·0 (7)	C(2")—N(1")—C(4')	121·7 (6)
O(7")—Li—O(10")	78·7 (6)	C(15")—N(1")—C(4')	118·9 (6)
O(7")—Li—O(13")	116·7 (8)	Li—O(4")—C(3")	113·3 (6)
O(10")—Li—O(13")	77·3 (6)	Li—O(4")—C(5")	106·9 (6)
C(2)—C(1)—C(9a)	123·6 (7)	C(3")—O(4")—C(5")	112·7 (6)
C(1)—C(2)—C(3)	118·0 (8)	Li—O(7")—C(6")	111·6 (7)
C(2)—C(3)—C(4)	121·0 (8)	Li—O(7")—C(8")	114·6 (8)
C(3)—C(4)—C(4a)	121·8 (8)	C(6")—O(7")—C(8")	117·3 (8)
C(6)—C(5)—C(10a)	121·5 (8)	Li—O(10")—C(9")	107·6 (8)
C(5)—C(6)—C(7)	121·7 (9)	Li—O(10")—C(11")	109·1 (7)
C(6)—C(7)—C(8)	119·6 (8)	C(9")—O(10")—C(11")	117·1 (9)
C(7)—C(8)—C(8a)	121·1 (7)	Li—O(13")—C(12")	118·9 (9)
C(8a)—C(9)—C(9a)	120·6 (7)	Li—O(13")—C(14")	120·3 (7)
C(8a)—C(9)—C(1')	120·5 (6)	C(12")—O(13")—C(14")	120·0 (9)
C(9a)—C(9)—C(1')	118·8 (6)	C(9)—C(1')—C(2')	123·1 (6)
C(11)—N(10)—C(4a)	117·4 (7)	C(9)—C(1')—C(6')	117·9 (6)
C(11)—N(10)—C(10a)	119·1 (7)	C(2')—C(1')—C(6')	119·0 (6)
C(4a)—N(10)—C(10a)	123·5 (6)	C(1')—C(2')—C(3')	118·1 (6)
Cl(1)—O(1)—Li	135·2 (6)	C(1')—C(2')—C(7')	122·7 (6)
C(4)—C(4a)—N(10)	122·8 (7)	C(3')—C(2')—C(7')	119·1 (6)
C(4)—C(4a)—C(9a)	118·5 (7)	C(2')—C(3')—C(4')	122·9 (6)
N(10)—C(4a)—C(9a)	118·6 (7)	N(1")—C(4')—C(3')	121·9 (6)

Table 2 (cont.)

Angle	Value	Angle	Value
C(8)—C(8a)—C(9)	122·2 (7)	N(1")—C(4')—C(5')	120·7 (7)
		C(3')—C(4')—C(5')	117·4 (7)
		C(4')—C(5')—C(6')	120·9 (7)
		C(1')—C(6')—C(5')	121·6 (7)

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 atoms of the acridinium group are coplanar to within 0·089 Å. The phenyl ring is planar to within 0·016 Å. The angle between the two planes is 66°.

The O atoms of the crown ether are alternately about 0·3 Å above and below their mean plane; the N atom lies 1·58 Å below this plane. For the mean cavity radius, as defined by Mathieu *et al.* (1978), a value of 0·84 Å was obtained. The distance from the Li ion to the centre of gravity of the hetero atoms in the crown ether is 0·59 Å, the angle N(1")–centre of gravity–Li is 109°. The Li ion is coordinated by the four O atoms of the crown ether and by one O atom [O(1)] of one of the perchlorate ions.

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