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[2-(1-Aza-4,7,10-trioxacyclododecyl)ethyl]dimethylammonium Iodide. An Intramolecular Trifurcated Hydrogen Bond

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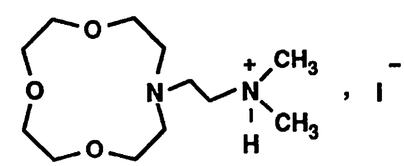
Abstract. $C_{12}H_{27}N_2O_3^+.I^-$, $M_r = 374{\cdot}3$, monoclinic, $P2_1/c$, $a = 23{\cdot}925$ (3), $b = 7{\cdot}7933$ (8), $c = 19{\cdot}182$ (3) Å, $\beta = 113{\cdot}113$ (10)°, $V = 3289{\cdot}5$ (8) Å^3, $Z = 8$, $D_x = 1{\cdot}511$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 19{\cdot}3$ cm^{-1}, $F(000) = 1520$, $T = 296$ K, $R = 0{\cdot}045$ for 3684 observations having $I > 1\sigma(I)$ (of 5785 unique data). There are two independent formula units in the asymmetric unit. The two macrocyclic cations have nearly identical conformations, with the 12-membered ring in the crown conformation, having all four donor atoms on the same side of the ring. The side arm, which contains the quaternary ammonium, is folded over the ring, with the H atom pointing towards the ring. The two independent cations exhibit a mean difference in 15 torsion angles describing their conformations of only 1·5°, with a maximum individual difference of 4·7 (11)°. The N—H hydrogen atom is involved in a trifurcated intramolecular hydrogen bond with the ring N and two O atoms of the ring. The N···acceptor distance range is 2·828 (8)–3·158 (8) Å, and the H···acceptor distance range is 2·25 (10)–2·40 (10) Å.

Experimental. A light-yellow irregular crystal fragment of (1), m.p. 475–476 K, grown by slow evaporation from ethanol, having approximate dimensions 0·38 × 0·35 × 0·32 mm, mounted in a glass capillary in random orientation, was used for data collection on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite-crystal incident-beam monochromator and Mo $K\alpha$ radiation. Cell dimensions were obtained from setting angles of 25 reflections having $25 < \theta < 30°$ using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54184$ Å). Space group determined to be $P2_1/c$ from systematic absences $h0l$ with l odd, $0k0$ with k odd.



(1)

One quadrant of data was collected ($0 \le h \le 28$, $0 \le k \le 9$, $-22 \le l \le 20$) using ω–2θ scans, within $1 < \theta < 25°$. Scan rates varied 1·0–4·0° min^{-1}. Three standard reflections (800, 040, 008), measured every 10 000 s, decreased in intensity by 9·7%; thus a linear decay correction was applied. Lorentz and polarization corrections were applied. An empirical absorption correction, based on a series of ψ scans, yielded relative transmission coefficients ranging from 0·798 to 0·997.

The transformation (001, $\bar{2}0\bar{1}$, $0\bar{1}0$) yields a C-centered cell with near-orthorhombic metric, cell dimensions $a = 19{\cdot}182$ (3), $b = 44{\cdot}010$ (6), $c = 7{\cdot}7933$ (8) Å, $\alpha = \beta = 90$, $\gamma = 90{\cdot}52$ (1)°. Diffraction patterns resemble symmetry mmm. In order to ascertain that the symmetry is $2/m$, a full sphere of low-angle data having $1 < \theta < 15°$, $-17 < h < 17$, $-5 < k < 5$, $-13 < l < 13$ was collected in the same fashion as the original quadrant. The value of R_{int} for the low-angle data averaged under mmm symmetry was 0·113, while R_{int} for all 10 739 data averaged

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)
I1	0·12935 (2)	0·73438 (6)	0·51798 (2)	4·587 (9)
I2	0·63045 (2)	0·22080 (6)	0·10940 (2)	4·686 (9)
O1*A*	0·5739 (2)	0·7954 (7)	0·7677 (2)	5·3 (1)
O2*A*	0·6652 (2)	0·9560 (7)	0·8935 (3)	5·4 (1)
O3*A*	0·7077 (2)	0·6542 (7)	0·9869 (3)	5·5 (1)
N1*A*	0·6115 (2)	0·4977 (7)	0·8641 (3)	4·5 (1)
N2*A*	0·5739 (2)	0·7477 (7)	0·9435 (2)	3·9 (1)
C1*A*	0·5818 (4)	0·496 (1)	0·7810 (4)	5·7 (2)
C2*A*	0·5976 (4)	0·647 (1)	0·7458 (4)	6·3 (2)
C3*A*	0·6044 (4)	0·949 (1)	0·7629 (4)	6·2 (2)
C4*A*	0·6668 (4)	0·9641 (9)	0·8214 (4)	5·7 (2)
C5*A*	0·7249 (4)	0·930 (1)	0·9540 (4)	6·7 (2)
C6*A*	0·7446 (3)	0·746 (1)	0·9592 (4)	6·7 (2)
C7*A*	0·7081 (4)	0·470 (1)	0·9736 (5)	6·6 (2)
C8*A*	0·6708 (4)	0·418 (1)	0·8948 (4)	6·0 (2)
C9*A*	0·5703 (2)	0·452 (1)	0·8996 (4)	5·0 (2)
C10*A*	0·5332 (3)	0·605 (1)	0·9026 (4)	5·2 (2)
C11*A*	0·5973 (4)	0·727 (1)	1·0272 (4)	6·3 (2)
C12*A*	0·5447 (3)	0·918 (1)	0·9214 (4)	5·3 (2)
O1*B*	0·0732 (2)	0·2104 (7)	0·3043 (2)	5·1 (1)
O2*B*	0·1618 (2)	0·0280 (6)	0·2712 (2)	4·5 (1)
O3*B*	0·2117 (2)	0·3170 (6)	0·2205 (2)	5·3 (1)
N1**B**	0·1171 (3)	0·4959 (7)	0·2454 (3)	4·6 (1)
N2*B*	0·0757 (2)	0·2432 (7)	0·1305 (2)	4·1 (1)
C1*B*	0·0886 (3)	0·509 (1)	0·3002 (4)	5·4 (2)
C2*B*	0·1004 (4)	0·353 (1)	0·3505 (4)	5·8 (2)
C3*B*	0·0978 (3)	0·052 (1)	0·3381 (4)	5·5 (2)
C4*B*	0·1603 (3)	0·018 (1)	0·3448 (4)	5·4 (2)
C5*B*	0·2224 (3)	0·042 (1)	0·2743 (4)	5·5 (2)
C6*B*	0·2445 (3)	0·226 (1)	0·2863 (4)	5·7 (2)
C7*B*	0·2148 (3)	0·501 (1)	0·2293 (5)	6·7 (2)
C8*B*	0·1779 (4)	0·568 (1)	0·2717 (5)	6·0 (2)
C9*B*	0·0762 (3)	0·541 (1)	0·1697 (4)	5·4 (2)
C10*B*	0·0369 (3)	0·392 (1)	0·1313 (4)	4·9 (2)
C11*B*	0·0986 (3)	0·256 (1)	0·0694 (3)	5·8 (2)
C12*B*	0·0435 (3)	0·076 (1)	0·1238 (4)	5·6 (2)
H2N*A*	0·610 (4)	0·763 (13)	0·927 (4)	9 (3)
H2N*B*	0·110 (3)	0·235 (12)	0·179 (4)	8 (2)

Table 2. *Bond distances* (Å) *and angles* (°)

O1*A*—C2*A*	1·42 (1)	O1*B*—C2*B*	1·413 (9)
O1*A*—C3*A*	1·42 (1)	O1*B*—C3*B*	1·413 (9)
O2*A*—C4*A*	1·40 (1)	O2*B*—C4*B*	1·428 (9)
O2*A*—C5*A*	1·457 (8)	O2*B*—C5*B*	1·433 (9)
O3*A*—C6*A*	1·40 (1)	O3*B*—C6*B*	1·392 (8)
O3*A*—C7*A*	1·46 (1)	O3*B*—C7*B*	1·44 (1)
N1*A*—C1*A*	1·468 (8)	N1*B*—C1*B*	1·46 (1)
N1*A*—C8*A*	1·444 (9)	N1*B*—C8*B*	1·45 (1)
N1*A*—C9*A*	1·44 (1)	N1*B*—C9*B*	1·440 (8)
N2*A*—C10*A*	1·481 (8)	N2*B*—C10*B*	1·488 (9)
N2*A*—C11*A*	1·487 (8)	N2*B*—C11*B*	1·48 (1)
N2*A*—C12*A*	1·480 (9)	N2*B*—C12*B*	1·49 (1)
N2*A*—H2N*A*	1·0 (1)	N2*B*—H2N*B*	0·96 (6)
C1*A*—C2*A*	1·48 (1)	C1*B*—C2*B*	1·51 (1)
C3*A*—C4*A*	1·48 (1)	C3*B*—C4*B*	1·47 (1)
C5*A*—C6*A*	1·50 (1)	C5*B*—C6*B*	1·51 (1)
C7*A*—C8*A*	1·48 (1)	C7*B*—C8*B*	1·51 (1)
C9*A*—C10*A*	1·50 (1)	C9*B*—C10*B*	1·49 (1)

C2*A*—O1*A*—C3*A*	113·2 (7)	C2*B*—O1*B*—C3*B*	113·3 (4)
C4*A*—O2*A*—C5*A*	113·3 (6)	C4*B*—O2*B*—C5*B*	112·4 (5)
C6*A*—O3*A*—C7*A*	112·9 (7)	C6*B*—O3*B*—C7*B*	114·7 (6)
C1*A*—N1*A*—C8*A*	114·7 (6)	C1*B*—N1*B*—C8*B*	114·2 (6)
C1*A*—N1*A*—C9*A*	112·3 (5)	C1*B*—N1*B*—C9*B*	112·6 (6)
C8*A*—N1*A*—C9*A*	116·4 (6)	C8*B*—N1*B*—C9*B*	116·7 (6)
C10*A*—N2*A*—C11*A*	112·4 (6)	C10*B*—N2*B*—C11*B*	112·5 (5)
C10*A*—N2*A*—C12*A*	112·3 (4)	C10*B*—N2*B*—C12*B*	112·2 (5)
C10*A*—N2*A*—H2N*A*	112 (5)	C10*B*—N2*B*—H2N*B*	109 (5)
C11*A*—N2*A*—C12*A*	109·9 (5)	C11*B*—N2*B*—C12*B*	109·2 (6)
C11*A*—N2*A*—H2N*A*	110 (4)	C11*B*—N2*B*—H2N*B*	110 (6)
C12*A*—N2*A*—H2N*A*	100 (5)	C12*B*—N2*B*—H2N*B*	104 (5)
N1*A*—C1*A*—C2*A*	112·8 (6)	N1*B*—C1*B*—C2*B*	112·3 (7)
O1*A*—C2*A*—C1*A*	108·0 (7)	O1*B*—C2*B*—C1*B*	108·0 (5)
O1*A*—C3*A*—C4*A*	114·0 (6)	O1*B*—C3*B*—C4*B*	114·8 (7)
O2*A*—C4*A*—C3*A*	109·6 (7)	O2*B*—C4*B*—C3*B*	108·9 (5)
O2*A*—C5*A*—C6*A*	111·5 (6)	O2*B*—C5*B*—C6*B*	111·7 (6)
O3*A*—C6*A*—C5*A*	106·6 (7)	O3*B*—C6*B*—C5*B*	107·8 (5)
O3*A*—C7*A*—C8*A*	113·9 (6)	O3*B*—C7*B*—C8*B*	112·9 (7)
N1*A*—C8*A*—C7*A*	113·8 (7)	N1*B*—C8*B*—C7*B*	113·6 (6)
N1*A*—C9*A*—C10*A*	110·1 (6)	N1*B*—C9*B*—C10*B*	110·9 (6)
N2*A*—C10*A*—C9*A*	109·9 (5)	N2*B*—C10*B*—C9*B*	109·6 (5)

under $2/m$ symmetry was 0·024. The number of unique data was 5785, of which 3684 had $I > 1\sigma(I)$ and were used in the refinement.

The structure was solved using heavy-atom methods, and refined by weighted full-matrix least squares. Anisotropic thermal parameters were varied for non-H atoms. H atoms were located by ΔF and

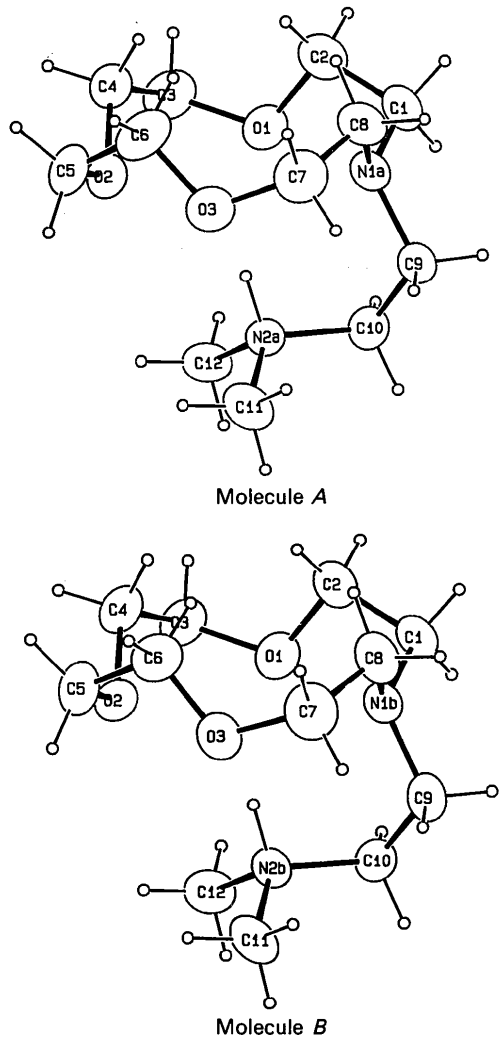

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles of arbitrary radii.

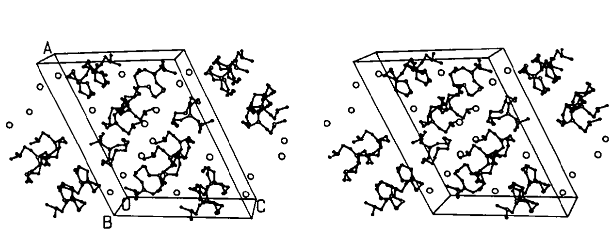

Fig. 2. Stereoview of the unit cell.

included as fixed contributions with C—H 0·95 Å and $B = 1{\cdot}3B_{eq}$ of the bonded C atom. The N—H hydrogen atoms were refined isotropically.

The function minimized was $\sum w(|F_o| - |F_c|)^2$, and weights were assigned as $w = 4F_o^2\text{Lp}[S^2(C + R^2B) + (0{\cdot}02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, Lp = Lorentz–polarization factor, using the Enraf–Nonius *SDP* system (Frenz & Okaya, 1980); scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1) and anomalous coefficients from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). The final cycle included 334 variables and converged (largest $\Delta/\sigma = 0{\cdot}01$) to $R = 0{\cdot}045$, $wR = 0{\cdot}039$, $S = 1{\cdot}109$. An extinction coefficient refined to $g = 1{\cdot}4$ (4) $\times 10^{-8}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Maximum and minimum residual electron densities were 0·58 and −0·39 e Å^{-3}. Table 1 shows the final positions and equivalent isotropic thermal parameters of these two molecules. Table 2 shows bond lengths and bond angles.* Fig. 1 shows the two molecules and numbering scheme, and Fig. 2 shows the unit cell. Table 3 reports the requisite parameters for evaluation of hydrogen bonding to the four possible acceptor atoms in the macrocycle.

Related literature. Complexation of alkali-metal cations by lariat ethers can be found in Gandour, Fronczek, Gatto, Minganti, Schultz, White, Arnold, Mazzocchi, Miller & Gokel (1986). An example of intramolecular hydrogen bonding between two tertiary nitrogens is in Shkol'nikova, Polyanchuk, Dyatlova & Polyakova (1984). The crystal structures of 10,10′-ethylenebis(1,4,7-trioxa-10-azacyclododecane) and its lithium complex are presented in Groth (1984*a*,*b*), respectively, and the potassium complex of *N*-(3,7,10-trioxaundecyl)-1,4,7-trioxa-10-azacyclododecane is presented in White, Arnold, Fronczek, Gandour & Gokel (1985).

Table 3. *Selected parameters for evaluation of hydrogen bonding*

X	N2Y*···X (Å)	N2Y*—H···X (°)	N2Y*—H···X (Å)
O1A	3·393 (7)	114 (5)	2·83 (8)
O1B	3·367 (7)	113 (6)	2·86 (9)
O2A	3·158 (8)	144 (8)	2·25 (10)
O2B	3·158 (6)	140 (7)	2·36 (8)
O3A	3·061 (7)	127 (6)	2·32 (8)
O3B	3·079 (6)	133 (7)	2·34 (8)
N1A	2·828 (8)	104 (6)	2·40 (10)
N1B	2·830 (7)	109 (6)	2·37 (9)

* If X is an atom in molecule A, then $Y = A$. If X is in molecule B, then $Y = B$.

* Tables of H-atom coordinates, anisotropic thermal parameters, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53554 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Polymorphs of 3,5-Dinitrobenzoic Acid

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Abstract. $C_7H_4N_2O_6$, $M_r = 212{\cdot}1$, monoclinic, $P2_1/c$, $a = 10{\cdot}0290$ (6), $b = 8{\cdot}8711$ (7), $c = 9{\cdot}514$ (2) Å, $\beta = 95{\cdot}639$ (12)°, $V = 842{\cdot}4$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}672$ g cm^{-3}; λ(Cu $K\alpha$) = 1·54184 Å, μ = 12·74 cm^{-1}, $F(000) = 432$, $T = 297$ K, $R = 0{\cdot}042$ for 1610 observations (of 1721 unique data); monoclinic, $C2/c$, $a = 21{\cdot}036$ (2), $b = 8{\cdot}7331$ (6), $c = 9{\cdot}7659$ (8) Å, $\beta = 111{\cdot}051$ (7)°, $V = 1674{\cdot}3$ (5) Å^3, $Z = 8$, $D_x = 1{\cdot}683$ g cm^{-3}; λ(Cu $K\alpha$) = 1·54184 Å, μ = 12·82 cm^{-1}, $F(000) = 864$, $T = 296$ K, $R = 0{\cdot}043$ for

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