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Crown Ether Complexes of Alkaline-Earth Metal Ions. III. Structures of 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6) Complexed with Magnesium and Barium Thiocyanates

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Abstract. (1): 1,4,7,10,13,16-Hexaoxacyclooctadecane–magnesium thiocyanate tetrahydrate, $C_{12}H_{24}O_6.Mg(SCN)_2.4H_2O$, $M_r = 476{\cdot}85$, orthorhombic, $Pna2_1$, $a = 9{\cdot}225$ (2), $b = 19{\cdot}256$ (7), $c = 13{\cdot}267$ (4) Å, $V = 2357$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}34$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}99$ cm^{-1}, $F(000) = 1016$, $T = 291$ K, $R = 0{\cdot}041$ for 1279 observed reflections. (2): 1,4,7,10,13,16-Hexaoxacyclooctadecane–barium thiocyanate monohydrate, $C_{12}H_{24}O_6.Ba(SCN)_2.H_2O$, $M_r = 535{\cdot}84$, monoclinic, $P2_1$, $a = 8{\cdot}030$ (3), $b = 17{\cdot}304$ (11), $c = 8{\cdot}551$ (7) Å, $\beta = 110{\cdot}78$ (5)°, $V = 1111$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}60$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 20{\cdot}55$ cm^{-1}, $F(000) = 536$, $T = 291$ K, $R = 0{\cdot}021$ for 1582 observed reflections. The Mg^{2+} ion is not bonded directly to the ether O atoms, but complex (1) involves $Mg(SCN)_2.4H_2O$ units that are hydrogen-bonded through the water molecules to the O atoms of the crown ether. The Ba^{2+} ion is coordinated to the six O atoms of the crown ether, to the two N atoms of the anions on one side and to the O of the water molecule on the opposite side. The cation is displaced 0·59 Å from the mean plane of the hexaether. In both molecular (1) and ionic (2) complexes the 18-crown-6 possesses approximate D_{3d} symmetry.

Introduction. As part of a systematic analysis of the structures of crown ethers complexed with alkaline-earth thiocyanates (Wei, Tinant, Declercq, Van Meerssche & Dale, 1988), we report here the structures of two new 18-crown-6 compounds: the tetrahydrated $C_{12}H_{24}O_6.Mg(SCN)_2$ (1) and the monohydrated $C_{12}H_{24}O_6.Ba(SCN)_2$ complexes.

Experimental. Both crystals obtained by slow evaporation from water. Crystal sizes: (1) 0·30 × 0·15 × 0·20, (2) 0·288 × 0·160 × 0·144 mm. Lattice parameters refined using 15 reflections in the range $5 \leq 2\theta \leq 25°$. Syntex $P2_1$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω scan width 1·3°, scan rate 1·3–30° min^{-1}, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}561$ Å^{-1}. For (1) 1648 independent *hkl* reflections measured ($0 \leq h \leq 10$, $0 \leq k \leq 21$, $0 \leq l \leq 14$), 1279 observed [criterion $I \geq 2{\cdot}5\sigma(I)$]. For (2) 1700 $hk\pm l$ measured ($0 \leq h \leq 9$, $0 \leq k \leq 19$, $-9 \leq l \leq 8$), 1582 observed. Standard reflections 062 (1) and $24\bar{3}$ (2) checked every 50 reflections: no significant deviation. Absorption corrections: none for (1), numerical corrections for crystal defined by its faces for (2) using *SHELX76* (Sheldrick, 1976); max./min. transmission 0·817, 0·704. Structure solution: position of Mg and S atoms (1), Ba (2) from an $E \times F$ Patterson map (*SHELXS86*, Sheldrick, 1985); the remaining non-H atoms from *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, Van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981). Anisotropic least-squares refinement (*SHELX76*, Sheldrick, 1976) using *F*. H atoms of three water molecules in (1) and of the crown ether except

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those on C(9) and C(12) in (2) located from a difference Fourier synthesis and refined with common isotropic temperature factors. All other H atoms of the crown ethers included in the refinement in idealized positions (C—H = 1·08 Å, H—C—H = 109·5°). 280 (1) and 235 (2) LS parameters. Weighting scheme $w = [\sigma^2(F) + gF^2]^{-1}$; $g = 0·0158$ (1) and 0·0005 (2). (1) $R = 0·041$, $wR = 0·048$, $S = 0·52$ for 1279 observed reflections. (2) $R = 0·021$, $wR = 0·022$, $S = 0·96$ for 1582 observed reflections. $(\Delta/\sigma)_{\max}$ in final refinement cycle: (1) 0·31 [U_{12} of atom O(10)], (2) 0·10 [U_{11} of atom N(1)]. Max. and min. heights in final Fourier synthesis 0·27, −0·34 (1), 0·69, −0·38 e Å^{-3} (2). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2) *for* (1)

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Mg	6179 (1)	219 (1)	0	2·50 (2)
S(1)	9694 (2)	2139 (1)	−22 (5)	6·56 (3)
C(13)	8584 (5)	1474 (2)	22 (8)	3·57 (4)
N(1)	7842 (4)	996 (2)	20 (7)	4·61 (4)
S(2)	9138 (2)	−1881 (1)	−13 (4)	5·24 (2)
C(14)	8255 (4)	−1146 (2)	−18 (8)	3·13 (4)
N(2)	7655 (4)	−620 (2)	−14 (7)	3·97 (4)
O(1)	3408 (6)	−1180 (3)	−1860 (5)	3·53 (4)
O(2)	1963 (7)	103 (4)	−2101 (5)	3·94 (4)
O(3)	3895 (6)	1290 (4)	−2140 (5)	3·99 (4)
O(4)	6627 (6)	1213 (3)	−3214 (5)	3·67 (4)
O(5)	8071 (5)	−90 (4)	−2966 (5)	3·51 (4)
O(6)	6190 (6)	−1291 (3)	−2855 (5)	3·55 (4)
O(7)*w*	5914 (6)	183 (3)	−1544 (5)	2·95 (4)
O(8)*w*	4426 (3)	−493 (2)	−2 (6)	3·68 (3)
O(9)*w*	4672 (3)	1058 (1)	17 (7)	3·90 (4)
O(10)*w*	5907 (6)	208 (3)	1530 (5)	3·42 (4)
C(1)	1899 (9)	−1131 (5)	−2011 (8)	5·23 (5)
C(2)	1355 (7)	−493 (5)	−1522 (7)	4·21 (4)
C(3)	1736 (8)	750 (6)	−1572 (6)	4·32 (4)
C(4)	2269 (7)	1301 (4)	−2160 (6)	3·49 (4)
C(5)	4384 (7)	1831 (4)	−2753 (6)	3·54 (4)
C(6)	6069 (8)	1845 (4)	−2790 (6)	4·20 (5)
C(7)	8162 (7)	1152 (4)	−3019 (7)	3·62 (4)
C(8)	8699 (9)	483 (5)	−3446 (6)	4·28 (4)
C(9)	8330 (8)	−709 (5)	−3492 (7)	4·32 (4)
C(10)	7683 (9)	−1336 (6)	−2883 (8)	5·63 (5)
C(11)	5497 (10)	−1802 (4)	−2161 (8)	6·12 (5)
C(12)	3992 (9)	−1799 (3)	−2343 (7)	5·06 (4)

Table 2. *Atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2) *for* (2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ba	193 (1)	0	3517 (1)	2·99 (1)
S(1)	5416 (5)	−1587 (3)	8544 (4)	5·31 (8)
C(13)	3826 (16)	−1140 (7)	7066 (16)	4·3 (2)
N(1)	2706 (20)	−831 (7)	6048 (17)	8·1 (2)
S(2)	5433 (5)	1541 (3)	8517 (4)	5·15 (7)
C(14)	3766 (14)	1239 (6)	6958 (14)	3·6 (2)
N(2)	2574 (17)	1023 (6)	5849 (15)	6·0 (1)
O(1)	−2043 (6)	1109 (3)	4245 (6)	3·8 (1)
O(2)	−316 (6)	1515 (3)	2041 (6)	4·2 (1)
O(3)	2002 (7)	461 (4)	1369 (6)	5·9 (1)
O(4)	1210 (8)	−1120 (4)	1640 (7)	6·0 (1)
O(5)	−806 (7)	−1572 (3)	3510 (7)	5·0 (1)
O(6)	−2658 (7)	−432 (3)	4506 (7)	4·8 (1)
O(7)*w*	−2508 (6)	−6 (6)	462 (6)	6·9 (1)
C(1)	−2502 (18)	1789 (6)	3227 (14)	4·7 (1)
C(2)	−927 (16)	2072 (6)	2923 (14)	5·7 (1)
C(3)	1235 (18)	1747 (8)	1740 (19)	6·6 (3)
C(4)	1568 (14)	1186 (7)	551 (12)	6·9 (2)
C(5)	2302 (13)	−128 (11)	292 (12)	8·0 (2)
C(6)	2710 (15)	−881 (8)	1237 (15)	7·4 (2)
C(7)	1360 (21)	−1879 (8)	2291 (18)	7·5 (3)
C(8)	−400 (16)	−2081 (6)	2418 (15)	6·2 (2)
C(9)	−2470 (16)	−1710 (7)	3733 (15)	5·2 (2)
C(10)	−2653 (12)	−1228 (5)	5037 (11)	5·3 (2)
C(11)	−3024 (11)	123 (7)	5581 (10)	5·6 (2)
C(12)	−3497 (11)	849 (5)	4638 (12)	5·6 (2)

Table 3. *Bond lengths and coordination distances* (Å)

	(1) $M = Mg^{2+}$	(2) $M = Ba^{2+}$
C(1)—O(1)	1·41 (1)	1·43 (1)
C(12)—O(1)	1·46 (1)	1·40 (1)
C(2)—O(2)	1·49 (1)	1·41 (1)
C(3)—O(2)	1·45 (1)	1·42 (2)
C(4)—O(3)	1·50 (1)	1·42 (1)
C(5)—O(3)	1·40 (1)	1·45 (2)
C(6)—O(4)	1·44 (1)	1·43 (1)
C(7)—O(4)	1·44 (1)	1·41 (2)
C(8)—O(5)	1·40 (1)	1·40 (1)
C(9)—O(5)	1·40 (1)	1·43 (1)
C(10)—O(6)	1·38 (1)	1·45 (1)
C(11)—O(6)	1·49 (1)	1·43 (1)
C(2)—C(1)	1·48 (1)	1·46 (2)
C(4)—C(3)	1·41 (1)	1·50 (2)
C(6)—C(5)	1·55 (1)	1·51 (2)
C(8)—C(7)	1·49 (1)	1·50 (2)
C(10)—C(9)	1·57 (1)	1·44 (2)
C(12)—C(11)	1·41 (1)	1·47 (1)
C(13)—S(1)	1·64 (1)	1·64 (1)
N(1)—C(13)	1·15 (1)	1·14 (2)
C(14)—S(2)	1·63 (1)	1·60 (1)
N(2)—C(14)	1·15 (1)	1·14 (1)
M···O(1)	—	2·843 (5)
M···O(2)	—	2·875 (5)
M···O(3)	—	2·830 (7)
M···O(4)	—	2·815 (7)
M···O(5)	—	2·836 (5)
M···O(6)	—	2·808 (6)
M···N(1)	2·144 (4)	2·779 (12)
M···N(2)	2·113 (4)	2·839 (11)
M···O(7)*w*	2·064 (6)	2·740 (4)
M···O(8)*w*	2·119 (3)	—
M···O(9)*w*	2·132 (3)	—
M···O(10)*w*	2·046 (6)	—

Discussion. Final atomic parameters for both structures are listed in Tables 1 and 2.* Bond lengths and coordination distances are compared in Table 3 and valence and torsion angles in the two hexaether rings in Table 4. Top views depicting the atom-numbering scheme (Figs. 1 and 2) and side views (Fig. 3) drawn by the program *PLUTO* (Motherwell & Clegg, 1978) show the coordination and the conformation of the crown ether for the two complexes.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44396 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Bond angles* (°) *and torsion angles* (°) ($\sigma = 3°$) *in* 18-*crown*-6

	(1)	(2)
C(12)–O(1)–C(1)	111·0 (7)	110·7 (8)
C(3)–O(2)–C(2)	111·1 (6)	113·4 (9)
C(5)–O(3)–C(4)	107·6 (6)	112·1 (8)
C(7)–O(4)–C(6)	110·4 (6)	113·7 (10)
C(9)–O(5)–C(8)	111·9 (6)	115·6 (8)
C(11)–O(6)–C(10)	113·7 (7)	114·5 (7)
C(2)–C(1)–O(1)	109·2 (7)	109·3 (10)
C(1)–C(2)–O(2)	106·6 (6)	110·6 (9)
C(4)–C(3)–O(2)	109·3 (6)	108·7 (10)
C(3)–C(4)–O(3)	109·2 (6)	108·1 (9)
C(6)–C(5)–O(3)	110·7 (6)	108·7 (9)
C(5)–C(6)–O(4)	110·8 (6)	109·8 (9)
C(8)–C(7)–O(4)	109·1 (6)	107·2 (11)
C(7)–C(8)–O(5)	111·7 (7)	110·5 (9)
C(10)–C(9)–O(5)	109·5 (7)	111·2 (8)
C(9)–C(10)–O(6)	110·1 (8)	107·3 (8)
C(12)–C(11)–O(6)	108·3 (6)	107·0 (7)
C(11)–C(12)–O(1)	107·0 (7)	109·4 (7)
C(12)–O(1)–C(1)–C(2)	180	177
O(1)–C(1)–C(2)–O(2)	68	62
C(1)–C(2)–O(2)–C(3)	−169	−179
C(2)–O(2)–C(3)–C(4)	−178	−170
O(2)–C(3)–C(4)–O(3)	−73	−66
C(3)–C(4)–O(3)–C(5)	178	179
C(4)–O(3)–C(5)–C(6)	−179	−179
O(3)–C(5)–C(6)–O(4)	62	63
C(5)–C(6)–O(4)–C(7)	−166	170
C(6)–O(4)–C(7)–C(8)	179	−172
O(4)–C(7)–C(8)–O(5)	−64	−61
C(7)–C(8)–O(5)–C(9)	168	180
C(8)–O(5)–C(9)–C(10)	177	172
O(5)–C(9)–C(10)–O(6)	67	63
C(9)–C(10)–O(6)–C(11)	−171	174
C(10)–O(6)–C(11)–C(12)	−169	−164
O(6)–C(11)–C(12)–O(1)	−77	−63
C(11)–C(12)–O(1)–C(1)	180	176

The Mg^{2+} ion chelates 15-crown-5 (Wei *et al.*, 1988) but not 12-crown-4 (Neuman, Steiner, van Remoortere & Boer, 1975). The reason is that its size [effective ionic radius 0·72 Å (Shannon, 1976)] matches the size of the ligand cavity in the former complex sufficiently well to overcome its affinity for water, but not for the second one. Thus it is not surprising that the $Mg(SCN)_2$.-18-crown-6 compound (1) described here turned out not to be a cation complex, but a molecular complex with four cation-coordinated water molecules in addition to the two thiocynate anions. The $Mg(SCN)_2$.-$4H_2O$ entities have an octahedral arrangement with the two N atoms of the anions on one side of a square. As can be seen in Table 3, the Mg···O distances fall into two classes: O(7) and O(10) above and below at 2·050 (6) Å, O(8) and O(9) at 2·138 (4) Å. Each of these $Mg(SCN)_2.4H_2O$ units is hydrogen-bonded through the four water molecules to the 18-crown-6 rings (Fig. 4). The equatorial O atoms [O(8), O(9)] are hydrogen-bonded to two symmetry-related crown molecules (Table 5) so that the structure consists of columns aligned along the *c* axis. The 18-crown-6 molecules adopt the regular D_{3d} conformation with exclusively $ag^{\pm}a$ units of alternating sign. The ether O atoms lie alternatively about 0·3 Å above and below the plane defined by the six O atoms (Table 5) and are 2·826 (7)–2·899 (8) Å distant from each other. This conformation, also observed for (2) as discussed hereafter, is the most common one for 18-crown-6 when complexed with molecules and ions (Dunitz, Dobler, Seiler & Phizackerley, 1974; Dale, 1980).

The structure of complex (2) is very similar to those observed for the complexes of 18-crown-6 with alkali thiocyanates. The latter have been discussed in some detail (Dunitz *et al.*, 1974). It has been shown that even

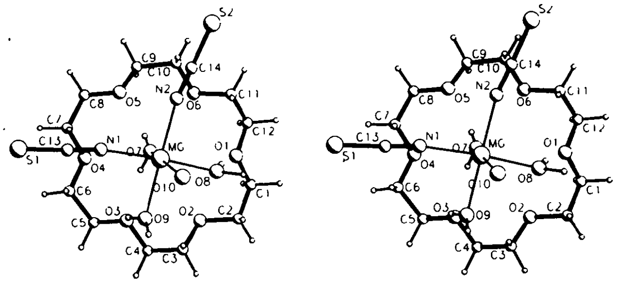


Fig. 1. Top view of (1) and atom numbering.

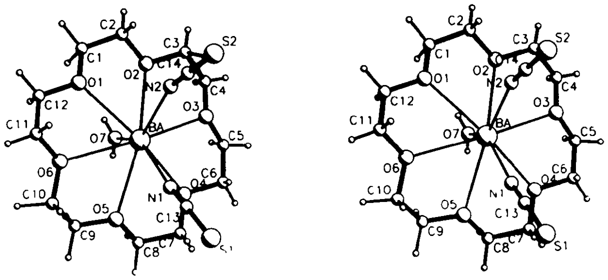
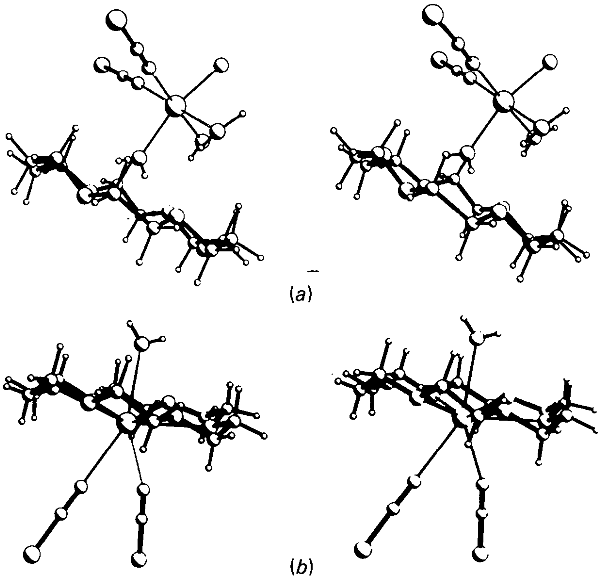


Fig. 2. Top view of (2) and atom numbering.


(*a*)

(*b*)

Fig. 3. Side views of (*a*) (1) and (*b*) (2).

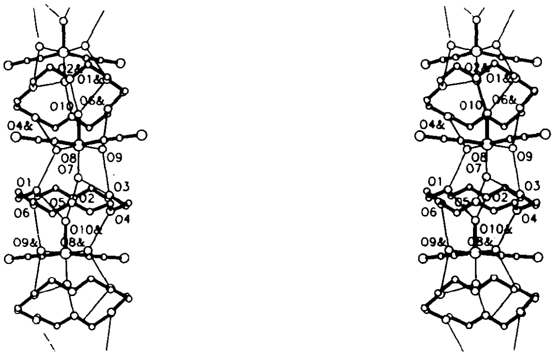


Fig. 4. Hydrogen bonding in (1).

when the size of the cation is too large to fit into the ligand cavity, the hexaether preserves the D_{3d} symmetry practically unchanged, while the cation adjusts its position out on one side of the ring. Thus the cations Rb^+ and Cs^+ are displaced respectively by 1·19 and 1·44 Å from the mean plane of the hexagon. This is also what we observe here for the $Ba(SCN)_2$ complex (2). The crown ether approximates D_{3d} symmetry with all torsion angles about C—C bonds close to 65° and those about C—O close to 180°. The ligating ether O atoms are above and below their mean plane by about 0·1–0·2 Å and the Ba^{2+} lies 0·59 Å out of this plane (Roberts & Sheldrick, 1975). The cation, also coordinated by the two N atoms of the anions on one side and the O of the water molecule on the opposite side of the ring, is displaced from the equatorial plane towards the thiocyanate anions. The Ba···O distances range from 2·808 (6) to 2·875 (5) Å with an average value of 2·834 (6) Å which corresponds to the sum of the ionic and van der Waals radii (1·47 + 1·40 = 2·87 Å) (Shannon, 1976; Pauling, 1960). The O···O distances ranging from 2·734 (9) to 2·835 (9) Å, average 2·786 (9) Å, are slightly shorter than those observed for (1).

There are no obvious linkages between the $Ba(SCN)_2.18\text{-crown-}6.H_2O$ complex units.

Table 5. *Hydrogen bonds* (Å) *in* (1)

	O···O	H···O
O(7)–H(7)···O(3^{i})	2·939 (8)	2·09 (2)
O(7)–H(7')···O(5^{i})	2·792 (8)	2·10 (2)
O(9)–H(9)···O(3^{i})	2·984 (8)	2·23 (2)
O(9)–H(9')···O(6^{ii})	2·967 (8)	2·04 (2)
O(8)–H(8)···O(4^{ii})	2·914 (8)	1·86 (2)
O(8)–H(8')···O(1^{i})	2·952 (8)	2·22 (2)
O(10)···O(2^{ii})	2·742 (8)	
O(10)···O(1^{ii})	2·910 (8)	

Symmetry code: (i) x, y, z; (ii) $1-x, -y, 0·5+z$.

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Structure of [*N*,*N*-Bis(diphenylphosphino)ethylamine]tricarbonyliron

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Abstract. $[Fe(C_{26}H_{25}NP_2)(CO)_3]$, $M_r = 553·32$, monoclinic, $C2/c$, $a = 35·741\ (8)$, $b = 10·008\ (2)$, $c = 15·049\ (2)$ Å, $\beta = 95·72\ (1)°$, $V = 5356·2$ Å^3, $Z = 8$, $D_m = 1·368$, $D_x = 1·372$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 0·706$ mm^{-1}, $F(000) = 2288$, $R = 0·083$ ($wR = 0·045$) for 4696 unique reflections (none

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