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Molecular Structure of 21-Crown-7-(1,2)Benzeno-2,6,18-dione-KSCN 1:1 Complex*

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Abstract

$C_{18}H_{24}O_9 \cdot KSCN$, $M_r = 481.57$, is monoclinic, $P2_1/n$, with $a = 8.631$ (1), $b = 11.338$ (1), $c = 24.479$ (3) Å, $\beta = 109.58$ (2)°, $V = 2257.0$ (8) Å³, $D_c = 1.417$ Mg m⁻³, $Z = 4$, $F(000) = 1008$. The structure, after inclusion of a conformational disorder for the C(9) and C(10) atoms, was refined to $R = 0.050$ for 3122 diffractometer (CAD-4) data. The 21-crown-7 ligand is fairly puckered, wrapping round the guest cation; K^+ is located near the least-squares plane of the seven ligating ether O atoms (their mean deviation is 0.56 Å). All $K \cdots O$ distances (2.77–3.07 Å) are longer than the sum of the corresponding ionic and van der Waals radii (2.73 Å). The eighth atom coordinated to K^+ is N of SCN^- at a distance of 2.80 Å. The keto O atoms, pointing outward from the macro ring, do not coordinate to K^+ . The established conformational disorder of atoms C(9) and C(10) suggests that these large rings may be subject to a low-energy-barrier pseudorotation.

Introduction

The alkali-metal salts are known to form various types of complexes with crown ethers. These polyether complexes may be classified into four groups according

to the positioning of the cation relative to the ligand atoms (Dalley, 1978). Examination of the cation and ligand size parameters indicates that K^+ is just able to fit the cavity of an 18-crown-6 ligand (Seiler, Dobler & Dunitz, 1974). Accordingly, the title complex, KSCN with 21-crown-7, should fall into the third group, *i.e.* the cation is smaller than the cavity. To our knowledge, based on a search of the Cambridge Crystallographic Data File containing 27 988 entries to October 1980, no crystal structure of a K^+ (or any other metal) complex with a 21-crown-7 ligand has been reported hitherto. Only the structure of a substituted uncomplexed 21-crown-7 is known (Owen & Nowell, 1978). The structures of K^+ complexes with dibenzo-24-crown-8 and dibenzo-30-crown-10 ligands have been described by Mercer & Truter (1973*b*) and Bush & Truter (1972). In these cases the cavities are even larger than in the title complex. Owing to the actual host-guest relationship in the title compound, complexation of the keto O atoms was expected (Vögtle, 1980).

Experimental

Crystals of the compound were kindly supplied by Professor F. Vögtle (University of Bonn). Intensities were collected on a CAD-4 diffractometer with graphite-monochromated $Cu K\alpha$ ($\lambda = 1.5418$ Å) radiation. Cell constants were determined by least squares from the setting angles of 25 reflexions; 3122

* Nomenclature is as suggested by Weber & Vögtle (1980). Alternative name: 2,5,8,11,14,17,20-Heptaaxabicyclo[19.4.0]pentacosane-1(25),21,23-triene-4,18-dione-KSCN complex.

of the 4570 independent reflexions with $I > 2.5\sigma(I)$ were taken as observed. No absorption correction was applied. Phases for 193 reflexions having $E \geq 1.70$ were obtained by *MULTAN* (Germain, Main & Woolfson, 1971). The E map computed from a phase set with the best consistency revealed 28 of the 31 non-hydrogen atoms ($R = 0.39$ for 1664 observations). The remaining three non-hydrogen atoms were located in a Fourier map ($R = 0.34$). The structural model was

Table 1. Fractional coordinates ($\times 10^5$ for K and $\times 10^4$ for the other atoms) and mean temperature factors for non-hydrogen atoms

E.s.d.'s are given in parentheses, $B_{eq} = 4[B_{11}B_{22}B_{33}/(a^*b^*c^*)]^{1/3}$.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (Å ²)
K	83129 (9)	43348 (5)	32933 (3)	4.02 (2)
S	4938 (1)	4319 (1)	3773 (1)	5.76 (4)
C(1)	-525 (4)	-2619 (3)	510 (1)	3.57 (11)
O(2)	1116 (2)	-2311 (2)	741 (1)	3.90 (8)
C(3)	2267 (4)	-3249 (3)	856 (1)	4.41 (13)
C(4)	2394 (4)	-3938 (3)	1394 (1)	4.19 (13)
O(5)	2148 (3)	-3251 (2)	1799 (1)	4.61 (9)
C(6)	2373 (5)	-3810 (3)	2361 (2)	5.51 (16)
C(7)	1737 (5)	-2997 (4)	2697 (2)	7.57 (19)
O(8)	2561 (4)	-1965 (2)	2810 (1)	6.30 (12)
C(9A)	1718 (8)	-1328 (6)	3159 (3)	6.74 (14)
C(10A)	2887 (8)	-169 (6)	3335 (3)	6.54 (14)
C(9B)	2865 (10)	-1236 (8)	3332 (4)	7.27 (19)
C(10B)	1746 (10)	-201 (7)	3206 (3)	7.01 (18)
O(11)	2147 (4)	519 (2)	2770 (1)	5.95 (12)
C(12)	3245 (5)	1462 (3)	2904 (1)	5.66 (17)
C(13)	2967 (5)	2215 (3)	2383 (1)	5.08 (15)
O(14)	3207 (3)	1518 (2)	1935 (1)	4.28 (9)
C(15)	3165 (4)	2168 (3)	1436 (1)	4.61 (14)
C(16)	1454 (4)	2507 (3)	1068 (1)	4.38 (13)
O(17)	552 (3)	1410 (2)	865 (1)	4.10 (9)
C(18)	-1035 (4)	1539 (3)	561 (1)	4.00 (12)
C(19)	-1933 (4)	421 (3)	342 (1)	4.10 (13)
O(20)	-866 (2)	-573 (2)	482 (1)	3.91 (8)
C(21)	-1603 (4)	-1658 (3)	359 (1)	3.82 (12)
C(22)	-3264 (4)	-1858 (3)	103 (1)	4.87 (15)
C(23)	-3862 (4)	-3020 (3)	16 (2)	5.44 (16)
C(24)	-2801 (5)	-3954 (3)	177 (2)	5.38 (15)
C(25)	-1117 (4)	-3768 (3)	423 (1)	4.49 (13)
O(26)	2748 (4)	-4963 (2)	1456 (1)	5.84 (13)
O(27)	-1725 (3)	2483 (2)	473 (1)	5.44 (11)
C(28)	2945 (4)	4220 (3)	3485 (1)	4.37 (12)
N(29)	1537 (4)	4147 (3)	3288 (1)	6.07 (14)

Table 2. Interatomic distances (Å) and bond angles (°) with their e.s.d.'s in parentheses

S—C(28)	1.630 (3)	O(11)—C(10B)	1.474 (6)
C(1)—O(2)	1.382 (4)	C(12)—C(13)	1.486 (3)
C(1)—C(21)	1.400 (5)	C(13)—O(14)	1.422 (3)
C(1)—C(25)	1.389 (5)	O(14)—C(15)	1.417 (3)
O(2)—C(3)	1.418 (4)	C(15)—C(16)	1.499 (4)
C(3)—C(4)	1.503 (3)	C(16)—O(17)	1.463 (4)
C(4)—O(5)	1.333 (3)	O(17)—C(18)	1.330 (4)
C(4)—O(26)	1.198 (4)	C(18)—C(19)	1.489 (5)
O(5)—C(6)	1.467 (4)	C(18)—O(27)	1.209 (4)
C(6)—C(7)	1.459 (5)	C(19)—O(20)	1.423 (4)
C(7)—O(8)	1.349 (5)	O(20)—C(21)	1.371 (4)
C(9A)—C(10A)	1.625 (9)	C(21)—C(22)	1.377 (5)
C(9B)—C(10B)	1.485 (12)	C(22)—C(23)	1.405 (5)
O(8)—C(9A)	1.482 (6)	C(23)—C(24)	1.369 (5)
O(8)—C(9B)	1.470 (8)	C(24)—C(25)	1.390 (5)
O(11)—C(12)	1.393 (5)	C(28)—N(29)	1.150 (5)
O(11)—C(10A)	1.529 (6)		
O(2)—C(1)—C(21)	114.2 (4)	C(15)—C(16)—O(17)	106.9 (4)
O(2)—C(1)—C(25)	125.0 (4)	C(16)—O(17)—C(18)	115.3 (4)
C(21)—C(1)—C(25)	120.8 (4)	O(17)—C(18)—C(19)	115.0 (4)
C(1)—O(2)—C(3)	116.5 (4)	O(17)—C(18)—O(27)	123.4 (5)
O(2)—C(3)—C(4)	113.8 (4)	C(19)—C(18)—O(27)	121.6 (4)
C(3)—C(4)—O(5)	111.4 (4)	C(18)—C(19)—O(20)	111.8 (4)
C(3)—C(4)—O(26)	123.8 (5)	C(19)—O(20)—C(21)	116.3 (4)
O(5)—C(4)—O(26)	124.8 (5)	C(1)—C(21)—O(20)	115.0 (4)
C(4)—O(5)—C(6)	116.2 (4)	C(1)—C(21)—C(22)	119.4 (4)
O(5)—C(6)—C(7)	107.4 (5)	O(20)—C(21)—C(22)	125.7 (4)
C(6)—C(7)—O(8)	113.0 (6)	C(21)—C(22)—C(23)	119.8 (5)
C(7)—O(8)—C(9A)	102.4 (6)	C(22)—C(23)—C(24)	120.4 (5)
C(7)—O(8)—C(9B)	126.5 (7)	C(23)—C(24)—C(25)	120.6 (6)
C(12)—O(11)—C(10A)	97.7 (5)	C(1)—C(25)—C(24)	119.1 (5)
C(12)—O(11)—C(10B)	124.1 (6)	S—C(28)—N(29)	179.2 (5)
O(11)—C(12)—C(13)	109.7 (4)	O(8)—C(9A)—C(10A)	99.5 (7)
C(12)—C(13)—O(14)	108.6 (4)	O(11)—C(10A)—C(9A)	98.0 (7)
C(13)—O(14)—C(15)	114.1 (4)	O(8)—C(9B)—C(10B)	110.5 (10)
O(14)—C(15)—C(16)	112.8 (4)	O(11)—C(10B)—C(9B)	107.1 (9)

refined anisotropically by the full-matrix least-squares technique to an R value of 0.046 for 2917 reflexions above the $3.0\sigma(I)$ level. The contributions of H positions generated from assumed geometries were also included. The H atoms were given the isotropic temperature factors of the heavy atoms to which they are bound. At this stage the distance C(9)—C(10) appeared unreasonably short (1.29 Å). Hence positional disorder of these atoms was assumed. A subsequent difference electron density synthesis revealed a spurious peak in the vicinity of C(9). A slant-plane difference map in the plane defined by this peak and the original C(9) and C(10) positions enabled us to resolve four atomic sites, the corresponding bonds of which intersect on this plane. Isotropic vibrational parameters were assigned to these atoms and their occupan-

Table 3. Torsion angles with their e.s.d.'s in parentheses (°)

C(4)—C(3)—O(2)—C(1)	-76.6 (4)	O(20)—C(19)—C(18)—O(17)	-2.9 (4)	C(25)—C(1)—C(21)—C(22)	-2.2 (5)
O(5)—C(4)—C(3)—O(2)	-33.0 (4)	O(20)—C(21)—C(1)—O(2)	-2.1 (4)	C(25)—C(24)—C(23)—C(22)	-0.7 (5)
C(6)—O(5)—C(4)—C(3)	-175.3 (5)	C(21)—C(1)—O(2)—C(3)	-174.9 (5)	O(26)—C(4)—C(3)—O(2)	150.2 (6)
C(7)—C(6)—O(5)—C(4)	-168.9 (6)	C(21)—O(20)—C(19)—C(18)	172.7 (4)	O(26)—C(4)—O(5)—C(6)	1.4 (5)
O(8)—C(7)—C(6)—O(5)	-62.6 (5)	C(22)—C(21)—C(1)—O(2)	177.4 (5)	O(27)—C(18)—O(17)—C(16)	-2.9 (5)
O(11)—C(10A)—C(9A)—O(8)	80.6 (6)	C(22)—C(21)—O(20)—C(19)	1.5 (5)	O(27)—C(18)—C(19)—O(20)	178.3 (6)
O(11)—C(10B)—C(9B)—O(8)	-63.2 (8)	C(23)—C(22)—C(21)—C(1)	2.3 (5)	C(9A)—O(8)—C(7)—C(6)	-178.7 (6)
O(14)—C(13)—C(12)—O(11)	-59.5 (4)	C(23)—C(22)—C(21)—O(20)	-178.2 (6)	C(9A)—C(10A)—O(11)—C(12)	-175.1 (8)
C(15)—O(14)—C(13)—C(12)	-173.0 (5)	C(23)—C(24)—C(25)—C(1)	0.8 (5)	C(10A)—O(11)—C(12)—C(13)	171.6 (6)
C(16)—C(15)—O(14)—C(13)	-75.9 (4)	C(24)—C(23)—C(22)—C(21)	-0.9 (5)	C(10A)—C(9A)—O(8)—C(7)	174.3 (8)
O(17)—C(16)—C(15)—O(14)	-62.7 (4)	C(24)—C(25)—C(1)—O(2)	-178.9 (6)	C(9B)—O(8)—C(7)—C(6)	-147.1 (9)
C(18)—O(17)—C(16)—C(15)	175.7 (5)	C(24)—C(25)—C(1)—C(21)	0.7 (5)	C(9B)—C(10B)—O(11)—C(12)	93.6 (9)
C(19)—C(18)—O(17)—C(16)	178.4 (5)	C(25)—C(1)—O(2)—C(3)	4.7 (5)	C(10B)—O(11)—C(12)—C(13)	-161.9 (8)
C(19)—O(20)—C(21)—C(1)	-179.0 (4)	C(25)—C(1)—C(21)—O(20)	178.3 (5)	C(10B)—C(9B)—O(8)—C(7)	-103.3 (11)

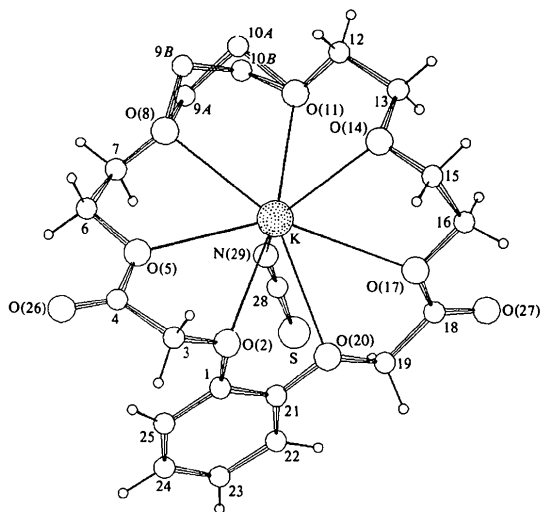


Fig. 1. A view of the complex projected onto the plane of O(5), O(14) and O(20), showing atomic numbering and the conformational disorder of C(9) and C(10). Atoms are carbon unless indicated otherwise, and H atoms are numbered according to the carbon to which they are linked. No H atoms were assigned to the disordered C(9) and C(10) atomic pairs (*A* and *B*).

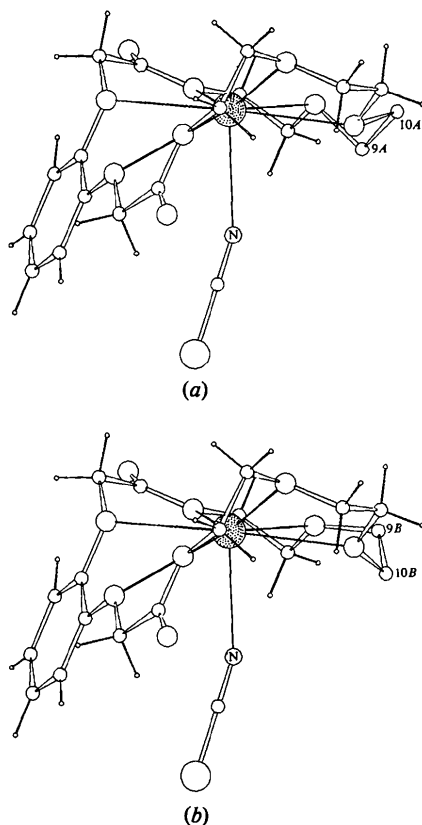


Fig. 2. A side view of the molecule showing the puckered polyether ring. For better visualization the disordered $-\text{CH}_2-\text{CH}_2-$ moiety is presented in two separate diagrams: (a) for C(9A) and C(10A) and (b) for C(9B) and C(10B).

cies were allowed to vary. Though no constraints were imposed on these atomic pairs [C(9A)—C(10A) and C(9B)—C(10B)] and their bonded H atoms were omitted, the refinement for 283 variables with the 3122 observed reflexions led to reasonable values for both sums of occupancies [$0.56 + 0.44 = 1.0$ for C(9) and $0.54 + 0.46 = 1.0$ for C(10)], for isotropic *R* values and for C_A-C_B separation distances [C(9A)—C(9B) = $0.94(1)$, C(10A)—C(10B) = $0.93(1)$ Å]. The final *R* index is 0.050 ($R_w = 0.062$). A final difference map showed no features greater than $0.2 \text{ e } \text{Å}^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). All calculations were performed on a PDP-11/34 minicomputer with the Enraf-Nonius SDP-34 system. The final coordinates for the non-hydrogen atoms are given in Table 1. Interatomic distances and bond angles are presented in Table 2 and torsion angles in Table 3.*

Discussion

The general conformational features of the 21-crown-7 ligand complexed to KSCN are depicted in Fig. 1. The projection of the macroring exhibits a quasi-regular $\text{K}^+ \cdots \text{O}_7$ heptagon characterized by an average $\text{O} \cdots \text{K}^+ \cdots \text{O}$ angle of $55.1 \pm 3.6^\circ$. The distances of the seven ligating O atoms from their least-squares plane ($0.9975x - 0.0038y - 0.0699z = -0.2563$) vary, however, in a wide range, -0.96 – 1.12 Å (mean deviation 0.56 Å). As shown by Fig. 2, K^+ lies in the centre of this plane, embraced by the heptaether. The keto O atoms do not coordinate, but point outward from the macroring. The $\text{K} \cdots \text{O}$ distances are longer than the sum of the corresponding ionic and van der Waals radii (2.73 Å). They range from 2.769 to 3.071 Å (Table 4). The shortest $\text{K} \cdots \text{O}$ distance is formed by O(14) which is farthest from the least-squares plane of the seven ether O atoms. In comparison with the other types of K^+ cyclic polyether complexes, the title compound exhibits the widest range of $\text{K} \cdots \text{O}$ distances, as shown in Table 4. It seems, therefore, that the title complex is maintained by weak interactions. The cavity is large but not large enough to permit an additional guest ion, as in case of the 24-crown-8 ligand, or to fold completely around K^+ as in the 30-crown-10 ring. The seemingly weak host-guest interaction in the title compound is reflected in conformational disorder of one of the $-\text{CH}_2-\text{CH}_2-$ moieties. We are inclined to believe that these large rings are subject to a low-energy-barrier pseudorotation which may be present in 'empty' as well as

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36437 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

weakly complexed molecules even at relatively low temperatures (Goldberg, 1978). The ratios between the corresponding ionic radii and cavity diameters for Na⁺ embedded in an 18-crown-6 ligand [$2 \times 1.94/(2.6 + 3.2) = 0.67$] and for K⁺ sitting in a 21-crown-7 polyether [$2 \times 2.66/(3.4 + 4.3) = 0.69$] agree within experimental error. Nevertheless, the hexaether rings in NaBr-dibenzo-18-crown-6 (Bush & Truter, 1971) and NaBr-dicyclohexyl-18-crown-6 (Mercer & Truter, 1973a) are rather planar, while the ring in a hydrated NaSCN complex of 1,4,7,10,13,16-hexaoxacyclooctadecane (Dobler, Dunitz & Seiler, 1974) is more puckered. The significant differences in the ligand conformation between such weakly complexed NaX-18-crown-6 (X = Br, SCN) compounds and the title species may be attributed to small differences in the intramolecular energy balances. These energy balances depend on a complicated interplay of various factors which determine the host-guest relationship: e.g. ratios of the radii of complexed cations to the potential diameters of ligand cavities, coordination numbers of guest ions, the character of the anions, the effect of additional guest molecules (water, etc.). The substituents on the polyether rings may also affect the actual macroring conformation. The mean (0.36 Å) and the maximum (0.67 Å) deviations from the least-squares plane of the ether oxygens of the uncomplexed dimethyl-dibenzo-21-crown-7 (Owen & Nowell, 1978) are significantly smaller than those of the title complex (see above). The more pronounced puckering of the latter enables it to wrap around the K⁺ located in the centre of the polyether crown. The K⁺, as follows from the shortest K...O(14) distance, is slightly off the axis of this ring. The sequence of torsion angles is characterized by three synperiplanar (Klyne & Prelog, 1960) angles about the C(3)-C(4), C(18)-C(19) and C(1)-C(21) bonds. The angle about C(3)-C(4) is just on the margin -sp/-sc. The angles are determined by the presence of the phenyl substituent and two coplanar acetoxy groups formed around C(4) and C(18). The sequence between O(5) and O(14), even with the conformational disorder between C(9) and C(10), exhibits the usual ap,±sc,ap order. Between O(14) and O(17) the sequence is -sc,-sc,ap.

Table 4. *The characteristics of oxygen coordination around the K⁺ cation*

Ligand type	K...O (Å)	$4_{K...O}$ (Å)	Co-ordination number	References
15-Crown-5	2.777-2.955 (8)	0.178	10	Mallison & Truter (1972)
18-Crown-6	2.770-2.833	0.063	8	Seiler <i>et al.</i> (1974)
21-Crown-7	2.769-3.071 (2)	0.302	8	Present work
24-Crown-8	2.732-2.931 (6)	0.199	7 + 1	Mercer & Truter (1973a,b)
30-Crown-10	2.850-2.931 (6)	0.081	10	Bush & Truter (1972)

The eighth atom coordinated to K⁺ is N of SCN⁻ at a distance of 2.795 (3) Å. N is located near the axis of the polyether belt: O(17)...I⁺...N = 88.13 (7)° [$\Delta_{O(17)} = 0.12$ Å]. The K⁺...S distance is 3.486 (1) Å.

The mean bond length for the planar and regular phenyl ring is 1.388 (5) Å. The C-O bond lengths can be classified in four groups: C=O = 1.203 (4), C(sp²)-O = 1.332 (4), C_o-O = 1.382 (4) Å and the C(sp³)-O distances, the latter ranging from 1.393 (5) to 1.527 (6) Å. The shortening of the bonds maintained by C(7) indicates also an unresolved disorder of this atomic position. The mean C(sp²)-C(sp³) distance is 1.496 (4) Å. The reliable C(sp³)-C(sp³) distances in ordered parts of the crown [1.490 (5) Å] exhibit the same shortening discussed by Dunitz, Dobler, Seiler & Phizackerley (1974) and Maverick, Seiler, Schweizer & Dunitz (1980).

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