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Structures of Polyether Complexes.
III.* Molecular and Crystal Structure of Bis[(*o*-methoxyphenoxy)ethoxy]ethane–Sodium Isothiocyanate

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$C_{20}H_{26}O_6$.NaSCN crystallizes from methanol/ethyl acetate in space group $P2_12_12_1$ with $a = 10{\cdot}146$ (3), $b = 15{\cdot}171$ (4), $c = 14{\cdot}855$ (4) Å, $Z = 4$. The structure was solved from 2054 X-ray diffractometer data by direct methods and refined to $R_w = 7{\cdot}0\%$. Starting with one oxyanisole residue the polyether ligand is wrapped around the Na^+ ion in a planar, annular structure with the C—O torsion angles *trans* and ethylenedioxy C—C angles *gauche*. To avoid a collision between the two oxyanisole residues, the two C—O bonds near the second oxyanisole residue are *gauche*, giving rise to a helical structure of the ligand with the normals to the phenyl rings forming an angle of 116°. The Na^+ ion is coordinated to all six O atoms of the ligand with $Na^+\cdots O$ distances ranging from 2·33 to 2·54 Å and is also connected to the N atom of the isothiocyanate group, $Na^+\cdots N = 2{\cdot}33$ Å.

Introduction

Cyclic crown ethers and cryptates have been of considerable interest as analogues of cyclic ionophore antibiotics (Ovchinnikov, Ivanov & Shkrob, 1974; Pedersen & Frensdorff, 1972; Lehn, 1973; Christensen, Eatough & Izatt, 1974) and have found application in preparative organic chemistry (Vögtle & Neumann, 1973). Recently, linear polyethers with good cation complexing properties have been synthesized (Vögtle & Weber, 1975; Vögtle & Sieger, 1977; Tümmler, Maass, Weber, Wehner & Vögtle, 1977). The structure analysis of bis(8-quinolyloxyethoxyethyl) ether–RbI (I.RbI; Saenger, Brand, Vögtle & Weber, 1977) revealed a helical complex structure. The present study

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* Part II: Saenger, Brand, Reddy, Suh & Weber (1978).

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of bis[(*o*-methoxyphenoxy)ethoxy]ethane–NaSCN (II.NaSCN) was carried out in order to furnish data for a ligand shorter than the previous one.

Experimental

Crystallographic data are summarized in Table 1. X-ray intensities were converted to normalized structure amplitudes $E_{\mathbf{h}}$. The strongest 300 $E_{\mathbf{h}}$ were used to solve the structure with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). An E map computed on the basis of the most probable phase set revealed all the non-hydrogen atoms except those of the isothiocyanate group and C(12) which were positioned from a subsequent Fourier synthesis. After full-matrix least-squares refinement (Busing, Martin & Levy, 1962) with weights from counting statistics (Stout & Jensen, 1968), H atoms could be located from difference syntheses. The refinement converged at R_w = 7·0% for all data.*

Results and discussion

Tables 2 and 3 list final atomic coordinates and intramolecular bond distances and angles. The molecular structure, stereochemistry and atom numbering are shown in Fig. 1. Fig. 1 also displays torsion angles of the polyether chain and $Na^+\cdots O$ and $Na^+\cdots N$ distances. In Figs. 2 and 3* thermal ellipsoid plots and a stereoscopic view along **a** of the crystal stucture are presented.

Table 1. *Crystallographic data*

II.NaSCN, $C_{20}H_{26}O_6$.NaSCN, M_r = 443·5
Chunky, prismatic crystals from hot, saturated methanol/ethyl acetate
Space group: orthorhombic, $P2_12_12_1$
a = 10·146 (3) Å Z = 4
b = 15·171 (4) d_o = 1·304 g cm^{-3} (flotation, bromobenzene/ethyl acetate)
c = 14·855 (4) d_c = 1·287
Data: 2054 intensities collected on a four-circle diffractometer, Ni-filtered Cu $K\alpha$ radiation;
$2\theta/\omega$ step scan (1° min^{-1}), 10 s stationary background counts on each side of scan. Data were corrected for Lorentz–polarization but not for absorption.
Size of crystal: 0·2 × 0·3 × 0·5 mm
μ(Cu $K\alpha$) = 13·25 cm^{-1}

Table 2. *Fractional atomic coordinates* (×10^4), *with e.s.d.'s in parentheses for heavy atoms*

Coordinates of the H atoms were located from difference syntheses. The H temperature factors used in the calculations were assigned from those of the attached C atoms.

	x	*y*	*z*
S	13478 (3)	4650 (2)	8960 (2)
Na	8870 (3)	3776 (2)	8143 (2)
C	12009 (10)	4264 (7)	8858 (7)
N	10957 (9)	3956 (6)	8775 (6)
O(1)	9962 (7)	3801 (4)	6702 (4)
O(2)	8669 (6)	2443 (4)	7156 (4)
O(3)	7430 (8)	2566 (4)	8730 (4)
O(4)	7868 (7)	4164 (4)	9516 (5)
O(5)	8708 (6)	5440 (3)	8261 (4)
O(6)	6881 (6)	4441 (3)	7433 (4)
C(1)	10778 (12)	4529 (6)	6525 (8)
C(2)	9628 (9)	3258 (6)	5969 (7)
C(3)	9943 (11)	3385 (6)	5101 (7)
C(4)	9459 (11)	2782 (8)	4457 (7)
C(5)	8718 (13)	2063 (7)	4673 (7)
C(6)	8426 (11)	1930 (6)	5581 (8)
C(7)	8884 (10)	2506 (6)	6221 (6)
C(8)	7937 (12)	1688 (5)	7475 (7)
C(9)	8017 (12)	1738 (6)	8440 (7)
C(10)	7331 (13)	2648 (7)	9683 (6)
C(11)	6854 (10)	3594 (6)	9873 (6)
C(12)	7506 (12)	5065 (7)	9624 (7)
C(13)	8618 (9)	5592 (5)	9159 (5)
C(14)	7767 (9)	5851 (5)	7722 (6)
C(15)	7763 (10)	6770 (6)	7576 (7)
C(16)	6806 (12)	7157 (6)	7076 (8)
C(17)	5826 (11)	6682 (7)	6700 (7)
C(18)	5801 (9)	5746 (6)	6841 (6)
C(19)	6794 (9)	5351 (5)	7290 (5)
C(20)	5884 (10)	3896 (5)	7083 (5)
H(11)	11800	4346	6355
H(12)	10406	4936	5940
H(13)	10824	5013	7140
H(3)	10555	3946	4896
H(4)	9683	2903	3749
H(5)	8333	1620	4158
H(6)	7880	1355	5795
H(81)	6920	1717	7275
H(82)	8391	1078	7257
H(91)	7487	1192	8739
H(92)	9050	1718	8644
H(101)	8271	2523	10000
H(102)	6593	2177	9933
H(111)	6766	3698	10597
H(112)	5929	3723	9539
H(121)	6571	5199	9283
H(122)	7444	5243	10330
H(131)	8475	6289	9272
H(132)	9563	5396	9465
H(15)	8529	7168	7885
H(16)	6844	7867	6966
H(17)	5073	6994	6287
H(18)	4974	5349	6614
H(201)	5045	3873	7589
H(202)	5448	4197	6476
H(203)	6151	3205	6933

(*a*) *Wrapping of the ligand around the cation*

Depending on its length, a linear ligand can wrap around the cation in at least three modes which could

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* Lists of structure factors and anisotropic thermal parameters, and Fig. 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33556 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

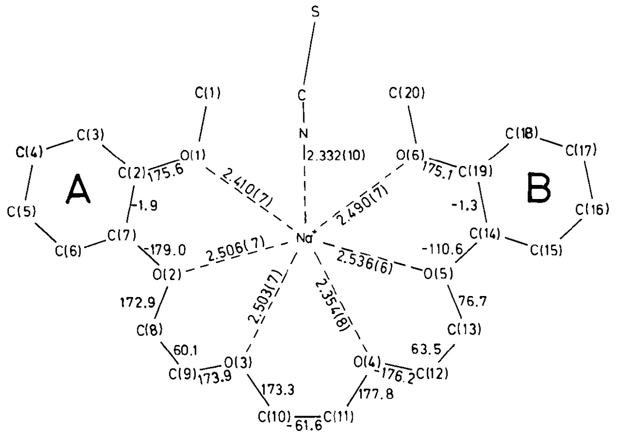
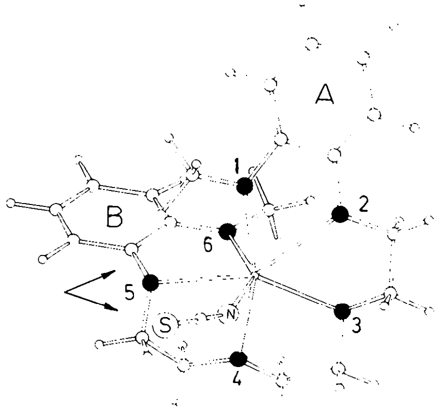


Fig. 1. A schematic drawing indicating the chemical structure and numbering scheme. Also shown are the torsion angles along the polyether chain and distances between Na$^+$ and hetero-atoms. E.s.d.'s for the latter are given in parentheses.



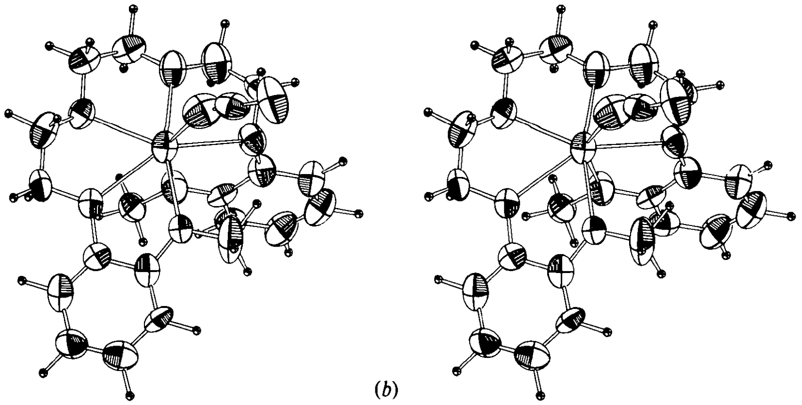
(a)

Fig. 2. (a) The structure of one complex molecule. ○ H, ◯ C, ● O. The *gauche* oriented C—O torsion angles are marked by arrows.

be demonstrated thus far by X-ray diffraction analyses: a short ligand forms an annular, disk-like structure, while ligands too long to wrap around the cation in only one turn form helical structures and very long ligands can wrap around two cations in an *S*-like configuration, one cation being accommodated in each turn of the *S* (Saenger, Brand, Reddy, Suh & Weber, 1978). In the present complex, all the six O atoms of the ligand coordinate with the Na$^+$ ion and the ligand adopts a helical structure. As in I.RbI, O(1), O(2), O(3) and O(4) are coplanar within 0·005 Å; the Na$^+$ ion is 0·101 Å away from this plane while O(5), O(6) and N deviate by −0·879, −2·385, and 2·144 Å. All the C—O torsion angles in this portion of the ligand are *trans* and the ethylenedioxy C—C torsion angles are *gauche*, conformations also observed in cyclic crown ether complexes (Truter, 1973). O(5) and O(6) deviate from this planar arrangement because a kink caused by *gauche* conformations about both C(13)–O(5) and O(5)–C(14) is introduced to avoid collision of the ω,ω'-substituted oxyanisole residues which would result if these torsion angles were in the common *trans* form. A similar kink was also found in I.RbI but here the radius of the cation, 1·47 Å, is considerably greater than the 0·97 Å of Na$^+$ (*Handbook of Chemistry and Physics*, 1974) and therefore the kink occurs one C—O bond later than in the present complex. The arrangement of the benzene ring planes *A* and *B* (Fig. 1) relative to each other is described by a dihedral angle of 116°; the angles between these planes and the plane through O(1), O(2), O(3) and O(4) are 23° for *A* and 96° for *B*.

(*b*) *Coordination of* Na$^+$

From the sum of the van der Waals radii [1·4 Å for O, 1·5 Å for N and 0·97 Å for Na$^+$ (*Handbook of Chemistry and Physics*, 1974)] the Na$^+\cdots$O distance should be 2·37 Å and we expect 2·47 Å for Na$^+\cdots$N. Fig. 1 shows that Na$^+\cdots$O is in the range 2·35 to 2·54 Å, the mean, 2·47 Å, being significantly greater than



(*b*)

Fig. 2 (*cont.*). (*b*) A stereoplot with thermal ellipsoids indicated.

the ideal, 2·37 Å. The largest deviation is observed for Na$^+\cdots$O(5), suggesting some irregularity around O(5) and indeed the kink occurs at this atom as described above.

Table 3. *Interatomic distances* (Å) *and angles* (°)

O(1)···O(2)	2·553 (9)	O(5)–C(13)–C(12)	113·6 (7)
O(2)···O(3)	2·661 (9)	O(5)–C(14)–C(15)	122·1 (8)
O(3)···O(4)	2·726 (8)	O(5)–C(14)–C(19)	120·3 (7)
O(4)···O(5)	2·820 (8)	C(15)–C(14)–C(19)	117·6 (8)
O(5)···O(6)	2·691 (8)	C(14)–C(15)–C(16)	120·9 (9)
S–C	1·609 (11)	C(15)–C(16)–C(17)	121·5 (10)
C–N	1·17 (1)	C(16)–C(17)–C(18)	118·8 (10)
O(1)–C(1)	1·41 (1)	C(17)–C(18)–C(19)	119·9 (9)
O(1)–C(2)	1·41 (1)	C(18)–C(19)–C(14)	120·8 (8)
O(2)–C(7)	1·41 (1)	C(18)–C(19)–O(6)	124·1 (8)
O(2)–C(8)	1·45 (1)	C(14)–C(19)–O(6)	114·8 (7)
O(3)–C(9)	1·46 (1)	O(1)–Na–O(2)	62·0 (2)
O(3)–C(10)	1·43 (1)	O(2)–Na–O(3)	64·2 (2)
O(4)–C(11)	1·44 (1)	O(3)–Na–O(4)	68·2 (2)
O(4)–C(12)	1·43 (1)	O(4)–Na–O(5)	70·3 (2)
O(5)–C(13)	1·36 (1)	O(5)–Na–O(6)	64·7 (2)
O(5)–C(14)	1·39 (1)	O(1)–Na–O(6)	89·5 (2)
O(6)–C(19)	1·40 (1)	N–Na–O(1)	86·5 (3)
O(6)–C(20)	1·41 (1)	N–Na–O(2)	113·8 (3)
C(2)–C(3)	1·34 (1)	N–Na–O(3)	118·4 (3)
C(2)–C(7)	1·42 (1)	N–Na–O(4)	90·8 (3)
C(3)–C(4)	1·41 (1)	N–Na–O(5)	85·1 (3)
C(4)–C(5)	1·36 (2)	N–Na–O(6)	149·2 (3)
C(5)–C(6)	1·40 (2)	C(1)–O(1)–C(2)	117·2 (8)
C(6)–C(7)	1·37 (1)	C(7)–O(2)–C(8)	117·1 (7)
C(8)–C(9)	1·44 (1)	C(9)–O(3)–C(10)	113·4 (7)
C(10)–C(11)	1·54 (1)	C(11)–O(4)–C(12)	110·5 (7)
C(12)–C(13)	1·55 (1)	C(13)–O(5)–C(14)	116·3 (6)
C(14)–C(15)	1·41 (1)	C(19)–C(6)–C(20)	118·5 (6)
C(14)–C(19)	1·40 (1)	O(1)–C(2)–C(3)	127·1 (9)
C(15)–C(16)	1·36 (2)	O(1)–C(2)–C(7)	113·2 (8)
C(16)–C(17)	1·35 (2)	C(3)–C(2)–C(7)	119·7 (9)
C(17)–C(18)	1·43 (1)	C(2)–C(3)–C(4)	118·3 (9)
C(18)–C(19)	1·35 (1)	C(3)–C(4)–C(5)	123·5 (10)
		C(4)–C(5)–C(6)	117·4 (11)
O(2)–C(8)–C(9)	104·8 (8)	C(5)–C(6)–C(7)	120·3 (10)
O(3)–C(9)–C(8)	108·5 (8)	C(6)–C(7)–C(2)	120·7 (9)
O(3)–C(10)–C(11)	106·6 (8)	O(2)–C(7)–C(2)	113·4 (8)
O(4)–C(11)–C(10)	105·5 (8)	O(2)–C(7)–C(6)	126·0 (9)
O(4)–C(12)–C(13)	105·0 (8)		

As already observed in the complex I.RbI and in the Na$^+$ complex of ferroverdin (Candeloro, Grdenić, Taylor, Thompson, Viswamitra & Hodgkin, 1969) cation$\cdots$ligand distances may exceed ideal values. This behaviour appears to reflect steric strain in the ligand when wrapping around the cation or, as discussed previously (Saenger, Brand, Vögtle & Weber, 1977), differences in the electronegativity of the hetero-atoms of the ligand.

The position of the cation relative to the ligand atoms can also be expressed by the O$\cdots$Na$^+\cdots$O angles ranging from 62·0 to 70·3°, the largest angle again involving the kink atom O(5). Compared with the O$\cdots$Rb$^+\cdots$O angles in I.RbI, the O$\cdots$Na$^+\cdots$O angles are greater by about 15°, a consequence of the smaller ionic radius of the Na$^+$ ion.

The isothiocyanate ion is coordinated to Na$^+$ *via* the N atom, the Na$^+\cdots$N distance, 2·33 Å, being significantly shorter than the theoretical value of 2·47 Å. This shortening may be attributed to the negative charge localized on the N atom.

(*c*) *Bond angles and distances*

As in crown ether complexes containing resorcin-like moieties (Bright & Truter, 1970), a tendency exists for C–C (phenyl) bonds between O atoms, *i.e.* C(2)–C(7) and C(14)–C(19), to be short. Further, the orientation of the aromatic C–O bonds relative to the phenyl rings is unsymmetrical, with angles O(1)–C(2)–C(7), O(2)–C(7)–C(2), O(5)–C(14)–C(19) and C(14)–C(19)–O(6) smaller than 120° while O(1)–C(2)–C(3), O(2)–C(7)–C(6), O(5)–C(14)–C(15) and C(18)–C(19)–O(6) are significantly greater than 120°, indicating attractive forces between the two phenolic O atoms. In agreement with previous data, the (aromatic)C–O–C angles, $\simeq$117°, are generally greater than the C–O–C angles involving ethylenedioxy C atoms, average 106·8°.

The isothiocyanate ion is almost linear with N–C–S = 177·7°. The short N–C bond, 1·17 Å, and the double C–S bond, 1·61 Å, reflect the double-bond character described for this molecule by the possible mesomeric states.

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Study of Photochromic Sydnones.
I. Structure of 3-Phenyl-4-(3-methyl-1-buten-2-yl)sydnone

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The structure of $C_{13}H_{14}N_2O_2$ has been determined by direct methods and refined to $R = 0{\cdot}049$ for 1641 counter reflexions. The cell is monoclinic, space group $P2_1/c$, with $a = 9{\cdot}732\,(6)$, $b = 11{\cdot}644\,(4)$, $c = 12{\cdot}098\,(6)$ Å, $\beta = 115{\cdot}83\,(4)^\circ$, $Z = 4$. An angle of 54·9° was found between the planes of the sydnone and phenyl rings; the sydnone ring structure is in agreement with earlier results.

Introduction

The structure determination of 3-phenyl-4-(3-methyl-1-buten-2-yl)sydnone is part of a study of the photochromic properties of 4-alkenylsydnones (Nešpůrek & Šorm, 1975, 1977; Šorm & Nešpůrek, 1975*a*,*b*).

The objective of this work is the evaluation of the effects of molecular conformation and intermolecular contacts on the photochromism of the compound.

Experimental

The title compound forms transparent white crystals. The sample was prepared by cooling a saturated solution in ethanol (Šorm & Nešpůrek, 1975*b*).

Cell parameters were obtained by least-squares calculations from a calibrated powder photograph and also from the positions of 22 reflexions recorded with a Syntex $P2_1$ diffractometer. Results of the two determinations were identical to within one standard deviation.

Crystal data

$C_{13}H_{14}N_2O_2$, FW 230·3, m.p. 105–106 °C, monoclinic, $P2_1/c$, $a = 9{\cdot}732\,(6)$, $b = 11{\cdot}644\,(4)$, $c = 12{\cdot}098\,(6)$ Å, $\beta = 115{\cdot}83\,(4)^\circ$, $V = 1234$ Å^3, $F(000) = 488$, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54178$ Å, $\mu(\text{Cu }K\alpha) = 6{\cdot}95$ cm^{-1}, $Z = 4$, $D_x = 1{\cdot}239$, $D_m = 1{\cdot}23\,(1)$ g cm^{-3}; the density was determined by flotation in aqueous KI solution; systematic absences (ψ scan check): $h0l$ for l odd; $0k0$ for k odd.

A crystal was ground to a sphere with $r = 0{\cdot}3$ mm. Intensities were measured with the diffractometer (graphite monochromator, θ–2θ scan, Cu $K\alpha$ radiation, scan rate varied according to the intensity of the reflexion from 0·5 to 29·3° min^{-1}). Up to $\sin\theta/\lambda = 0{\cdot}53$ Å^{-1}, 1691 independent reflexions were measured; 50 of these were classed as unobserved with $I < 1{\cdot}96\sigma(I)$. 24 reflexions with $I > 80\,000$ c.p.s. were measured with a lower anode current. Three standard reflexions measured after every 30 reflexions showed no intensity decrease (40 kV, 20 mA, 200 h). No corrections for absorption or extinction were made.

Determination and structure refinement

The positions of all non-hydrogen atoms were determined with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974). The correct solution was

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