

A Complex of 2,11-Diaza-5,8,14,17-tetraoxa[8^{2,11}][12](4,4'')*o*-terphenylophane, Sodium Thiocyanate and Methanol (1 : 1 : 1)

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Abstract

$C_{32}H_{40}N_2O_4 \cdot NaSCN \cdot CH_3OH$, $M_r = 629.80$, is triclinic, space group $P1$; $a = 11.965$ (4), $b = 12.148$ (5), $c = 14.116$ (6) Å, $\alpha = 90.33$ (2), $\beta = 113.16$ (3), $\gamma = 114.57$ (3)°, $Z = 2$, $D_c = 1.245$ Mg m^{-3} , $\mu = 0.146$ mm⁻¹, $R = 0.058$, $R_w = 0.056$ for 3262 independent reflexions. The 'boat' configuration of the crown-analogous section of the ligand enables all six hetero atoms to coordinate to the cation in a bipical square-pyramidal arrangement, whilst the terphenyl residue folds away. There is no direct contact between Na^+ and SCN^- ; the anion is hydrogen bonded to a methanol molecule.

Introduction

According to the size of their inner cavities, macrobicyclic ligands display considerable selectivity in ligating spherical electron acceptors (Lehn & Sauvage, 1975). In complexes thus formed, 'fitting' cations are well shielded from their environments (Mathieu, Metz, Moras & Weiss, 1978; Moras, Metz & Weiss, 1973). Substitution of one coordinating chain by a large and rigid organic residue reduces the complexing ability of the ligand to that of the respective crown analogue. On the other hand it might provide an intramolecular cavity sufficiently large to accommodate a cation-anion pair (Rossa & Vögtle, 1981).

Experimental

Colourless irregular blocks of the title complex were grown from a mixture of methanol and ethyl acetate (Rossa & Vögtle, 1981). A crystal of approximate size 0.3 × 0.2 × 0.2 mm was used for collecting 4697 diffractometer data ($6^\circ < 2\theta < 46^\circ$) in a profile-fitting procedure (Clegg, 1981) with monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). Corrections for Lp and crystal delay (ca 7%) were applied. Averaging equivalents gave 3270 unique reflexions with $F > 4\sigma(F)$.

Cell dimensions were obtained from a least-squares analysis of angle measurements of 24 strong reflexions between $2\theta = 20$ and 25° .

The structure was solved by direct methods. Anisotropic refinement of all non-H atoms, followed by difference syntheses, showed the positions of all H atoms (except that of the hydroxyl group), which were idealized (distances 0.96 Å, angles 109.5° , aromatic H on external bisector). H atoms were assigned fixed isotropic thermal parameters 1.2 times the U values of attached C atoms; in further refinement a riding model was employed. Omission of eight poorly agreeing low-angle reflexions (probably caused by machine error) and application of an empirical extinction correction [$F_c^* = F_c(1 - xF_c^2/\sin \theta)$, $x = 3.9$ (5) × 10^{-7}] led to a final $R = 0.058$ and $R_w = 0.056$, where $R = \sum \Delta / \sum F_o$, $R_w = \sum w^{1/2} \Delta / \sum w^{1/2} F_o$, $\Delta = |F_o| - |F_c|$ and $w^{-1} = \sigma^2(F_o) + 0.00025 F_o^2$.

Coordinates and thermal parameters of non-H atoms are given in Table 1, with derived bond lengths and angles in Tables 2 and 3.† Views of the complex are shown in Fig. 1; the numbering scheme is arbitrary.

Results and discussion

The title ligand is reported to form stable crystalline complexes with $Ba(SCN)_2$ and $NaSCN$ (Rossa & Vögtle, 1981). In general, the diameter of the inner cavity of an 18-membered annular oligoether is such that Ba^{2+} and similar sized cations fit well (Dunitz, Dobler, Seiler & Phizackerley, 1974; Hughes, Mortimer & Truter, 1978; Moras, Metz, Herceg & Weiss, 1972). With smaller cations like Na^+ , however, such a ligand has to adapt its original conformation to the ionic radius of the guest (Dobler, Dunitz & Seiler, 1974). This also holds for the present structure.

† Lists of structure factors, anisotropic thermal parameters, H atom parameters and a complete list of torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36102 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
Na	-1743 (1)	1870 (1)	2113 (1)	51 (1)
S	6667 (1)	6454 (1)	76 (1)	79 (1)
C	8237 (4)	7307 (3)	891 (3)	57 (2)
N	9366 (4)	7930 (3)	1475 (3)	82 (3)
N(1)	-386 (3)	1841 (2)	992 (2)	45 (2)
N(2)	-2513 (3)	2021 (3)	3647 (2)	47 (2)
O(1)	-3212 (2)	250 (2)	514 (2)	54 (2)
O(2)	-2594 (2)	-90 (2)	2624 (2)	53 (1)
O(3)	-3322 (3)	2807 (2)	1692 (2)	63 (2)
O(4)	-740 (3)	3877 (2)	1722 (2)	71 (2)
C(1)	517 (4)	1230 (3)	1257 (3)	52 (2)
C(2)	-1496 (4)	1229 (3)	-76 (3)	55 (2)
C(3)	-2580 (4)	11 (4)	-78 (3)	56 (2)
C(4)	-4046 (4)	-849 (3)	761 (3)	61 (2)
C(5)	-3206 (4)	-1098 (3)	1758 (3)	58 (2)
C(6)	-3400 (4)	-260 (4)	3176 (3)	61 (3)
C(7)	-2708 (4)	865 (3)	4031 (3)	60 (3)
C(8)	-1613 (4)	3078 (4)	4558 (3)	60 (3)
C(9)	-3832 (4)	2047 (4)	3077 (3)	60 (2)
C(10)	-3753 (4)	3030 (4)	2440 (3)	65 (3)
C(11)	-2913 (4)	3816 (4)	1175 (4)	94 (3)
C(12)	-1483 (5)	4565 (4)	1668 (5)	103 (4)
C(13)	-307 (4)	3935 (4)	914 (3)	65 (3)
C(14)	422 (4)	3166 (3)	1037 (3)	55 (2)
C(15)	1546 (4)	1658 (3)	2397 (3)	46 (2)
C(16)	2874 (4)	2563 (3)	2708 (3)	52 (3)
C(17)	3775 (4)	3022 (3)	3759 (3)	50 (2)
C(18)	3384 (4)	2591 (3)	4536 (3)	44 (2)
C(19)	2071 (4)	1629 (3)	4224 (3)	47 (2)
C(20)	1174 (4)	1168 (3)	3174 (3)	48 (2)
C(21)	4317 (3)	3166 (3)	5671 (3)	46 (2)
C(22)	5612 (4)	3240 (4)	6098 (3)	58 (3)
C(23)	6489 (4)	3735 (4)	7153 (3)	69 (3)
C(24)	6092 (4)	4170 (4)	7789 (3)	64 (3)
C(25)	4808 (4)	4125 (3)	7376 (3)	53 (2)
C(26)	3904 (4)	3634 (3)	6312 (3)	42 (2)
C(27)	2535 (4)	3596 (3)	5899 (3)	44 (2)
C(28)	1705 (4)	3103 (4)	6405 (3)	54 (2)
C(29)	394 (4)	2958 (3)	5995 (3)	57 (2)
C(30)	-162 (4)	3288 (3)	5043 (3)	50 (2)
C(31)	688 (4)	3830 (3)	4554 (3)	50 (2)
C(32)	2010 (4)	3986 (3)	4971 (3)	47 (2)
O(5)	11836 (5)	7977 (5)	2755 (4)	170 (4)
C(33)	12932 (5)	9128 (5)	3060 (4)	121 (5)

* $U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$

Geometry of the ligand

The 'non-fitting' cation and additional steric requirements of the rigid non-coordinating terphenyl bridge force the coordinating section of the ligand into a strained 'boat' configuration. This differs considerably from that found in the analogous sodium complex with 1,4,7,10,13,16-hexaoxacyclooctadecane ('18-crown-6') (Dobler, Dunitz & Seiler, 1974) and is somewhat similar to those observed in Na^+ cryptates with 4,7,13,16,21-pentaoxa-1,10-diazabicyclo[8.8.5]-tricosane ('2.2.1') (Mathieu *et al.*, 1978) and 4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo[8.8.8]hex-

Table 2. Bond lengths (\AA)

C(1)-N(1)	1.487 (7)	C(1)-C(15)	1.509 (5)
C(2)-N(1)	1.476 (5)	C(2)-C(3)	1.508 (6)
C(3)-O(1)	1.426 (7)	C(4)-O(1)	1.439 (6)
C(4)-C(5)	1.492 (7)	C(5)-O(2)	1.436 (5)
C(6)-O(2)	1.418 (7)	C(6)-C(7)	1.504 (6)
C(7)-N(2)	1.468 (6)	C(8)-N(2)	1.485 (5)
C(9)-N(2)	1.478 (7)	C(10)-O(3)	1.413 (8)
C(10)-C(9)	1.493 (7)	C(11)-O(3)	1.438 (7)
C(11)-C(12)	1.415 (7)	C(12)-O(4)	1.436 (9)
C(13)-O(4)	1.417 (8)	C(13)-C(14)	1.492 (9)
C(14)-N(1)	1.478 (5)	C(15)-C(16)	1.386 (6)
C(15)-C(20)	1.388 (7)	C(16)-C(17)	1.381 (6)
C(17)-C(18)	1.380 (7)	C(18)-C(19)	1.397 (6)
C(18)-C(21)	1.494 (5)	C(19)-C(20)	1.380 (6)
C(21)-C(22)	1.386 (7)	C(21)-C(26)	1.403 (8)
C(22)-C(23)	1.385 (6)	C(23)-C(24)	1.360 (9)
C(24)-C(25)	1.389 (8)	C(25)-C(26)	1.398 (6)
C(26)-C(27)	1.487 (7)	C(27)-C(28)	1.387 (8)
C(27)-C(32)	1.393 (6)	C(28)-C(29)	1.372 (8)
C(29)-C(30)	1.390 (6)	C(30)-C(8)	1.498 (7)
C(30)-C(31)	1.394 (8)	C(31)-C(32)	1.379 (7)
C(33)-O(5)	1.373 (8)		
C-S	1.610 (4)	C-N	1.159 (5)

Table 3. Bond angles ($^\circ$)

C(1)-N(1)-C(2)	108.4 (4)	C(1)-N(1)-C(14)	109.2 (4)
C(2)-N(1)-C(14)	110.6 (4)	C(7)-N(2)-C(8)	109.0 (4)
C(7)-N(2)-C(9)	110.9 (4)	C(8)-N(2)-C(9)	109.1 (5)
C(3)-O(1)-C(4)	112.8 (4)	C(5)-O(2)-C(6)	112.6 (4)
C(10)-O(3)-C(11)	114.4 (5)	C(12)-O(4)-C(13)	114.6 (5)
N(1)-C(1)-C(15)	111.8 (5)	N(1)-C(2)-C(3)	111.7 (4)
O(1)-C(3)-C(2)	108.2 (5)	O(1)-C(4)-C(5)	110.4 (4)
O(2)-C(5)-C(4)	111.1 (5)	O(2)-C(6)-C(7)	108.5 (4)
N(2)-C(7)-C(6)	114.1 (4)	N(2)-C(8)-C(30)	112.7 (5)
N(2)-C(9)-C(10)	113.2 (4)	O(3)-C(10)-C(9)	108.2 (5)
O(3)-C(11)-C(12)	111.4 (5)	O(4)-C(12)-C(11)	112.6 (5)
O(4)-C(13)-C(14)	109.7 (5)	N(1)-C(14)-C(13)	115.5 (5)
C(1)-C(15)-C(16)	121.7 (5)	C(1)-C(15)-C(20)	120.5 (4)
C(16)-C(15)-C(20)	117.8 (4)	C(15)-C(16)-C(17)	121.2 (5)
C(16)-C(17)-C(18)	121.1 (4)	C(17)-C(18)-C(19)	117.7 (4)
C(17)-C(18)-C(21)	121.0 (4)	C(19)-C(18)-C(21)	121.2 (5)
C(18)-C(19)-C(20)	121.0 (5)	C(15)-C(20)-C(19)	120.9 (4)
C(18)-C(21)-C(22)	119.6 (5)	C(18)-C(21)-C(26)	121.0 (5)
C(22)-C(21)-C(26)	119.4 (4)	C(21)-C(22)-C(23)	121.1 (6)
C(22)-C(23)-C(24)	120.1 (6)	C(23)-C(24)-C(25)	119.8 (5)
C(24)-C(25)-C(26)	121.2 (6)	C(21)-C(26)-C(25)	118.3 (5)
C(21)-C(26)-C(27)	122.0 (4)	C(25)-C(26)-C(27)	119.7 (5)
C(26)-C(27)-C(28)	120.3 (5)	C(26)-C(27)-C(32)	121.9 (5)
C(28)-C(27)-C(32)	117.7 (5)	C(27)-C(28)-C(29)	121.7 (5)
C(28)-C(29)-C(30)	121.0 (5)	C(8)-C(30)-C(29)	121.7 (5)
C(8)-C(30)-C(31)	120.9 (5)	C(29)-C(30)-C(31)	117.4 (5)
C(30)-C(31)-C(32)	121.6 (5)	C(27)-C(32)-C(31)	120.5 (5)
S-C-N	179.5 (5)		

acosane ('2.2.2') (Moras & Weiss, 1973). Four C-O-C torsion angles of about 90° (see Table 4) instead of the expected 180° (Truter, 1973), and two N-C-C torsion angles of about -100° instead of 60° , reflect the resulting ring strain. Moreover, most X-C-C bond angles ($X = \text{O}, \text{N}$) (range $108.2-115.5$, mean 111.4°) appear significantly widened, the greatest disparities being found next to the branching N atoms (see Table 3). C-N-C angles (range $108.4-110.9$, mean 109.5°), however, reveal almost ideal hybridization tetrahedra for both N atoms of the ligand.

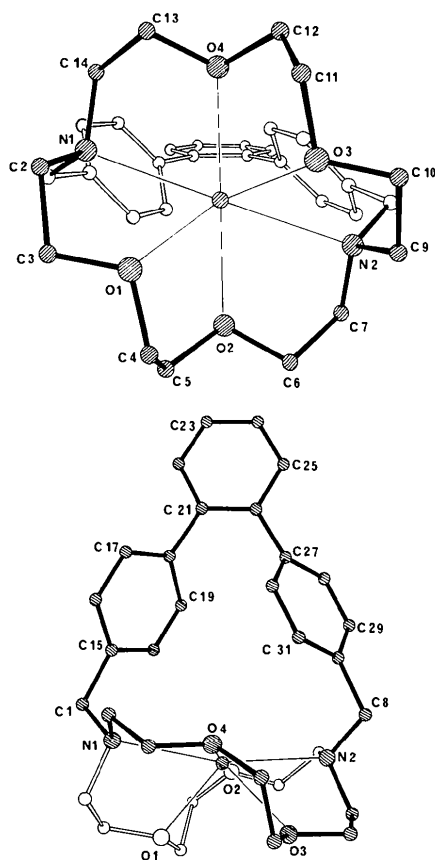


Fig. 1. Two perspective views of the complex including the numbering scheme adopted. Radii of atoms are arbitrary.

Table 4. Selected torsion angles (°)

C(2)—N(1)—C(1)—C(15)	-170.8 (4)	C(14)—N(1)—C(1)—C(15)	68.7 (5)
C(1)—N(1)—C(2)—C(3)	77.8 (6)	C(14)—N(1)—C(2)—C(3)	-162.5 (5)
C(1)—N(1)—C(14)—C(13)	-173.1 (4)	C(2)—N(1)—C(14)—C(13)	67.8 (6)
C(8)—N(2)—C(7)—C(6)	-170.1 (5)	C(9)—N(2)—C(7)—C(6)	69.7 (6)
C(7)—N(2)—C(8)—C(30)	71.1 (6)	C(9)—N(2)—C(8)—C(30)	-167.6 (4)
C(7)—N(2)—C(9)—C(10)	-165.3 (5)	C(8)—N(2)—C(9)—C(10)	74.6 (6)
C(4)—O(1)—C(3)—C(2)	-167.1 (4)	C(3)—O(1)—C(4)—C(5)	87.3 (5)
C(6)—O(2)—C(5)—C(4)	92.9 (5)	C(5)—O(2)—C(6)—C(7)	-177.4 (4)
C(11)—O(3)—C(10)—C(9)	-167.1 (4)	C(10)—O(3)—C(11)—C(12)	95.9 (7)
C(13)—O(4)—C(12)—C(11)	97.6 (6)	C(12)—O(4)—C(13)—C(14)	-178.7 (4)
N(1)—C(1)—C(15)—C(16)	-98.5 (6)	N(1)—C(1)—C(15)—C(20)	80.1 (6)
N(1)—C(2)—C(3)—O(1)	64.6 (6)	O(1)—C(4)—C(5)—O(2)	60.1 (6)
O(2)—C(6)—C(7)—N(2)	59.7 (6)	N(2)—C(8)—C(30)—C(29)	-105.1 (6)
N(2)—C(8)—C(30)—C(31)	75.2 (6)	N(2)—C(9)—C(10)—O(3)	60.1 (5)
O(3)—C(11)—C(12)—O(4)	56.3 (8)	O(4)—C(13)—C(14)—N(1)	59.6 (5)

A complete list of torsion angles has been deposited. See deposition footnote.

Whereas C—O bond distances (range 1.413–1.439, mean 1.428 Å) and C—N bond distances (range 1.468–1.487, mean 1.479 Å) are more or less normal, C—C bond lengths seem somewhat shortened [range 1.492–1.508, mean 1.498 Å, neglecting C(11)—C(12)] as is often observed in oligoethers (Dunitz *et al.*, 1974). The abnormally short C(11)—C(12) distance, 1.415 (7) Å, can be explained by the large and anisotropic vibration ellipsoids of these atoms perpendicular to the bond axis.

Table 5. Coordination distances (Å) and angles (°)

Na—O(1)	2.451 (4)	Na—O(2)	2.410 (4)
Na—O(3)	2.476 (5)	Na—O(4)	2.407 (4)
Na—N(1)	2.685 (5)	Na—N(2)	2.698 (5)
N(1)—Na—N(2)	165.8 (2)	N(1)—Na—O(1)	68.5 (2)
N(2)—Na—O(1)	122.4 (2)	N(1)—Na—O(2)	110.2 (2)
N(2)—Na—O(2)	68.5 (2)	O(1)—Na—O(2)	71.8 (2)
N(1)—Na—O(3)	124.3 (2)	N(2)—Na—O(3)	66.9 (2)
O(1)—Na—O(3)	93.5 (2)	O(2)—Na—O(3)	112.9 (2)
N(1)—Na—O(4)	69.8 (2)	N(2)—Na—O(4)	110.4 (2)
O(1)—Na—O(4)	111.8 (2)	O(2)—Na—O(4)	175.8 (2)
O(3)—Na—O(4)	69.7 (2)		

The terphenyl bridge displays no unusual geometrical features except that the three aromatic rings are not coplanar. The central benzene ring (II) is arranged perpendicular [90 (2)°] to plane (IV) formed by hetero atoms N(1), N(2), O(2) and O(4); the remaining dihedral angles are: (I)—(II) = 55 (2), (I)—(III) = 40 (2), (II)—(III) = 49 (2), (IV)—(I) = 69 (2), (IV)—(III) = 71 (2)° [plane (I) = C(15) to C(20), plane (III) = C(27) to C(32)]. These twists are probably due to steric repulsion; shortest interatomic non-bonded distances H(31)···O(4) = 2.804 (8) and H(20)···O(2) = 3.030 (7) Å correspond approximately to the sum of the van der Waals radii of H and O (2.6 Å).

Environment of the cation

The cavity thus provided by the ligand precludes a cation–anion contact but allows all hetero atoms to coordinate to Na⁺. Since N(1), N(2), O(2) and O(4) are coplanar to within ±0.123 Å, and since O(1) and O(3) are located on the same side of this plane at distances of 1.95 (1) and 1.85 (1) Å, the polyhedron of ligating atoms may best be described as a square pyramid with two apices. The cation lies almost in the basal plane [deviation 0.211 (8) Å] and therefore lacks strong interactions in one hemisphere of its environment. The base of the pyramid is considerably distorted into a parallelogram, due to the long Na⁺···N, as compared to Na⁺···O, distances (see Table 5). The latter, even those to apical atoms, exceed only marginally the sum of their respective ionic and van der Waals radii, whilst Na⁺···N contacts are about 0.2 Å longer than calculated values.

Comparing complexes of the analogous unbridged ligand 1,7,10,16-tetraoxa-4,13-diazacyclooctadecane with K⁺ (Moras *et al.*, 1972), Pb²⁺ (Metz & Weiss, 1973) and Cu²⁺ (Herceg & Weiss, 1973) shows that N···N distances depend on the radius of the coordinated cation in an almost linear manner [Cu²⁺: *r* = 0.72, N···N *ca* 4.0 Å; Pb²⁺: *r* = 1.20, N···N *ca* 5.5 Å; K⁺: *r* = 1.33, N···N *ca* 5.7 Å; *cf.* N···N 5.8 Å in the free ligand (Herceg & Weiss, 1972)]. The value of 5.341 (6) Å in the present structure does not fit the function assumed above, possibly due to the

terphenyl bridge. On the other hand, non-bonding distances of those equatorially coordinating O atoms opposite each other are not much influenced by the size of the guest (*ca* 5.4 Å for the Cu²⁺ complex, 5.7 Å for the Pb²⁺ complex, 5.7 Å for the K⁺ complex, 5.6 Å for the free ligand). The rather short O(2)···O(4) distance of 4.814 (8) Å in the title structure also seems to be an effect of the terphenyl group.

Intermolecular interactions

The title compound was originally reported to contain two water molecules per formula unit (Rossa & Vögtle, 1981). *A priori*, therefore, an H—O—H···S—C—N···H—O—H linkage similar to those in crystalline complexes of Cs⁺ and Rb⁺ with the cryptand '2.2.2' (Moras, Metz & Weiss, 1973) seemed possible. The current structure analysis, however, reveals methanol to be the only solvent molecule present, to which the anion is probably hydrogen bonded [N···O(5) = 2.768 (9) Å]. Since solvents used for crystallization had not been specially dried, replacement of water by the less polar methanol (even if present in large excess) is rather unusual. Normally the uptake of water is preferred with complexes of oligoethers because of the possibilities of forming more extended hydrogen-bond systems (*e.g.* Bush & Truter, 1971; Weber & Saenger, 1980).

There are no other strong intermolecular interactions.

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Structure of 4-Homosulfanilamide Hydrochloride

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Abstract

4-Homosulfanilamide hydrochloride, C₇H₁₀N₂O₂S·HCl, *M_r* = 222.8, is a 'sulfa' drug, which crystallizes in the tetragonal space group *I4₁cd* with *a* = *b* = 23.719 (8), *c* = 7.697 (14) Å, *Z* = 16, *D_m* = 1.44,

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D_c = 1.43 Mg m⁻³ (flotation in C₆H₆/CHBr₃), *V* = 4330 Å³, λ(Cu *Kα*) = 1.5418 Å, μ(Cu *Kα*) = 4.9 mm⁻¹. The structure was solved by direct methods and refined to a final *R* value of 0.065 for 835 counter reflections. Structural features in the compound are compared with those of other 'sulfa' drugs. The

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