



Fig. 1. An ORTEP (Johnson, 1965) projection of the molecule along [001].

this class of compounds. In fact, in (UO₂saloden).CHCl₃ (Bombieri, Forsellini, Benetollo & Fenton, 1979) the O(1)–U–O(2) angle is 177.3 (6)° and the shortest

contact is 2.88 Å: the bending diminishes as the contacts lengthen.

In the overall geometry of the molecule, there are no substantial differences from the previous determination.

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Structure of 1,20-Bis(8-quinolyloxy)-3,6,9,12,15,18-hexaoxaicosane–Rubidium Iodide*

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Abstract. C₃₂H₄₀N₂O₈.RbI, monoclinic, C2/c, $a = 42.169$ (13), $b = 10.804$ (7), $c = 15.946$ (8) Å, $\beta = 100.42$ (8)°, $M_r = 793.06$, $Z = 8$, $d_c = 1.474$, $d_o = 1.465$ Mg m⁻³ (floatation in bromobenzene/ethyl acetate), $\mu = 2.388$ mm⁻¹, $R_w = 0.074$ for 4668 unique data. The ligand displays a distorted helical conformation with one and a half turns which provides the cation with tenfold coordination. Eight coordination distances are slightly longer and two are considerably longer than expected for Rb⁺...O,N. There is no interaction between the shielded cation and the twofold-disordered anion.

Introduction. The title compound was recrystallized from a mixture of methanol and ethyl acetate (Weber, Saenger, Vögtle & Sieger, 1979). Data were collected from a crystal 0.5 × 0.5 × 0.5 mm with a four-circle diffractometer, Mo K α radiation, a graphite monochromator and the θ –2 θ step-scan mode up to $\sin \theta = 0.3817$. They were corrected for polarization effects.

The structure was solved by direct methods and subsequent Fourier syntheses (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined by full-matrix least squares (Sheldrick, 1976), omitting four reflexions because of obvious strong secondary extinction. The weighting scheme was based on counting statistics (Stout & Jensen, 1968). H-atom positions were located from difference Fourier syntheses and recalculated according to stereochemical criteria. H atoms were

* Structures of Polyether Complexes. IX. Part VIII: Weber & Saenger (1979).

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Table 1. Fractional atomic coordinates ($\times 10^4$) of the non-hydrogen atoms and isotropic *B* values (mean *e.s.d.* ca 0.3–0.4 Å²) calculated from the deposited anisotropic *U* values

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å ²)
Rb	3771 (0)	2862 (1)	10927 (1)	4.2
I(1)	5000 (0)	2360 (1)	7500 (0)	6.2
I(2)	2543 (1)	2805 (2)	4692 (2)	13.2
N(1)	3923 (2)	2697 (7)	12935 (5)	6.0
C(2)	4209 (2)	2177 (8)	13360 (5)	4.2
C(3)	4374 (2)	1358 (8)	12891 (6)	4.2
O(4)	4222 (2)	1172 (6)	12069 (4)	6.4
C(5)	4349 (2)	346 (10)	11546 (6)	6.3
C(6)	4101 (3)	-531 (9)	11184 (8)	7.8
O(7)	3840 (2)	119 (6)	10660 (4)	6.2
C(8)	3558 (3)	-672 (9)	10490 (8)	7.9
C(9)	3274 (3)	71 (9)	10101 (7)	7.0
O(10)	3222 (1)	1005 (6)	10689 (4)	5.7
C(11)	2924 (2)	1589 (9)	10448 (7)	6.4
C(12)	2887 (2)	2605 (10)	11056 (7)	6.3
O(13)	3097 (1)	3571 (6)	10940 (4)	5.5
C(14)	3037 (2)	4648 (10)	11387 (7)	6.6
C(15)	3275 (3)	5636 (9)	11269 (7)	6.7
O(16)	3589 (2)	5223 (6)	11676 (4)	5.7
C(17)	3820 (3)	6202 (9)	11761 (7)	6.5
C(18)	4139 (3)	5684 (10)	12101 (7)	7.0
O(19)	4236 (1)	4942 (6)	11469 (4)	5.7
C(20)	4541 (3)	4388 (9)	11711 (7)	7.2
C(21)	4666 (2)	4012 (10)	10941 (7)	7.2
O(22)	4458 (1)	3117 (6)	10469 (4)	5.6
C(23)	4501 (2)	3068 (11)	9623 (7)	6.9
C(24)	4264 (3)	2082 (10)	9141 (6)	6.8
O(25)	3944 (1)	2544 (6)	9145 (4)	5.6
C(26)	3699 (2)	1951 (9)	8644 (6)	5.3
C(27)	3398 (2)	2571 (9)	8554 (5)	4.8
N(28)	3381 (2)	3644 (7)	8972 (5)	4.9
C(29)	3100 (2)	4225 (10)	8851 (7)	5.9
C(30)	2831 (2)	3770 (11)	8298 (7)	7.3
C(31)	2844 (3)	2704 (10)	7895 (7)	7.0
C(32)	3131 (3)	2055 (10)	8008 (6)	6.1
C(33)	3173 (3)	922 (13)	7608 (7)	8.9
C(34)	3467 (4)	304 (12)	7709 (8)	10.3
C(35)	3725 (3)	832 (9)	8253 (6)	6.9
C(36)	3779 (3)	3448 (9)	13388 (6)	6.5
C(37)	3901 (3)	3785 (9)	14226 (7)	7.0
C(38)	4180 (3)	3258 (9)	14650 (6)	5.8
C(39)	4344 (2)	2432 (8)	14219 (6)	4.2
C(40)	4626 (2)	1868 (9)	14596 (6)	5.6
C(41)	4768 (2)	1098 (10)	14138 (6)	6.5
C(42)	4651 (2)	808 (8)	13280 (6)	5.1

assigned the isotropic thermal parameters of the attached C atoms. Results are given in Tables 1, 2 and 3* and in Fig. 1. A stereoview of the complex is in Fig. 2.

Discussion. The structures of RbI–polyether complexes with similar but shorter ligands than the present one

* Lists of structure factors, temperature factors and H-atom positions have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34736 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å) involving non-hydrogen atoms

N(1)–C(2)	1.394 (11)	C(21)–O(22)	1.426 (11)
N(1)–C(36)	1.305 (13)	O(22)–C(23)	1.394 (13)
C(2)–C(3)	1.418 (13)	C(23)–C(24)	1.562 (15)
C(2)–C(39)	1.412 (11)	C(24)–O(25)	1.442 (11)
C(3)–O(4)	1.365 (11)	O(25)–C(26)	1.348 (11)
O(3)–C(42)	1.356 (11)	C(26)–C(27)	1.418 (13)
O(4)–C(5)	1.393 (13)	C(26)–C(35)	1.374 (13)
C(5)–C(6)	1.450 (13)	C(27)–N(28)	1.345 (11)
C(6)–O(7)	1.440 (11)	C(27)–C(32)	1.408 (13)
O(7)–C(8)	1.449 (13)	N(28)–C(29)	1.324 (11)
C(8)–C(9)	1.482 (15)	C(29)–C(30)	1.392 (13)
C(9)–O(10)	1.420 (13)	C(30)–C(31)	1.325 (16)
O(10)–C(11)	1.398 (11)	C(31)–C(32)	1.384 (15)
C(11)–C(12)	1.490 (15)	C(32)–C(33)	1.406 (16)
C(12)–O(13)	1.401 (11)	C(33)–C(34)	1.392 (20)
O(13)–C(14)	1.412 (13)	C(34)–C(35)	1.386 (16)
C(14)–C(15)	1.502 (15)	C(36)–C(37)	1.390 (13)
C(15)–O(16)	1.435 (11)	C(37)–C(38)	1.372 (15)
O(16)–C(17)	1.427 (11)	C(38)–C(39)	1.383 (13)
C(17)–C(18)	1.468 (15)	C(39)–C(40)	1.374 (11)
C(18)–O(19)	1.405 (13)	C(40)–C(41)	1.321 (15)
O(19)–C(20)	1.409 (11)	C(41)–C(42)	1.404 (13)
C(20)–C(21)	1.476 (16)		

Table 3. Bond angles (°) involving non-hydrogen atoms

C(36)–N(1)–C(2)	115.5 (8)	O(25)–C(24)–C(23)	106.1 (8)
C(3)–C(2)–N(1)	117.3 (7)	C(26)–O(25)–C(24)	116.8 (8)
C(39)–C(2)–N(1)	123.7 (8)	C(27)–C(26)–O(25)	114.2 (8)
C(39)–C(2)–C(3)	119.0 (8)	C(35)–C(26)–O(25)	124.8 (9)
O(4)–C(3)–C(2)	114.2 (7)	C(35)–C(26)–C(27)	120.9 (9)
C(42)–C(3)–C(2)	119.7 (8)	N(28)–C(27)–C(26)	119.1 (8)
C(11)–C(3)–O(4)	126.0 (9)	C(32)–C(27)–C(26)	118.4 (9)
C(5)–O(4)–C(3)	120.4 (7)	C(32)–C(27)–N(28)	122.5 (9)
C(6)–C(5)–O(4)	109.3 (9)	C(29)–N(28)–C(27)	117.7 (8)
O(7)–C(6)–C(5)	109.5 (8)	C(30)–C(29)–N(28)	121.8 (10)
C(8)–O(7)–C(6)	109.7 (7)	C(31)–C(30)–C(29)	121.1 (10)
C(9)–C(8)–O(7)	109.5 (8)	C(32)–C(31)–C(30)	119.2 (9)
O(10)–C(9)–C(8)	108.5 (8)	C(31)–C(32)–C(27)	117.6 (10)
C(11)–O(10)–C(9)	112.1 (7)	C(33)–C(32)–C(27)	118.3 (10)
C(12)–C(11)–O(10)	110.4 (7)	C(33)–C(32)–C(31)	124.1 (10)
O(13)–C(12)–C(11)	108.7 (9)	C(34)–C(33)–C(32)	123.2 (11)
C(14)–O(13)–C(12)	111.9 (8)	C(35)–C(34)–C(33)	117.2 (11)
C(15)–C(14)–O(13)	109.7 (9)	C(34)–C(35)–C(26)	121.8 (11)
O(16)–C(15)–C(14)	107.8 (8)	C(37)–C(36)–N(1)	124.6 (10)
C(17)–O(16)–C(15)	111.7 (7)	C(38)–C(37)–C(36)	120.0 (10)
C(18)–C(17)–O(16)	108.5 (8)	C(39)–C(38)–C(37)	119.0 (9)
O(19)–C(18)–C(17)	108.6 (8)	C(38)–C(39)–C(2)	117.3 (8)
C(20)–O(19)–C(18)	114.5 (7)	C(40)–C(39)–C(2)	120.1 (9)
C(21)–C(20)–O(19)	109.4 (8)	C(40)–C(39)–C(38)	122.6 (8)
O(22)–C(21)–C(20)	110.4 (8)	C(41)–C(40)–C(39)	118.8 (9)
C(23)–O(22)–C(21)	111.2 (8)	C(42)–C(41)–C(40)	124.3 (9)
C(24)–C(23)–O(22)	108.5 (9)	C(41)–C(42)–C(3)	118.1 (9)

have been investigated before: The ligand with three O atoms wraps around the cation circularly (Saenger & Reddy, 1979), and the ligand with five O atoms wraps around the cation in a helical manner with nearly one turn (Saenger, Brand, Vögtle & Weber, 1977; Saenger & Brand, 1979).

The present achiral linear polyether encloses the cation to form a chiral, helical complex with one and a

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Structure of (1,3,6,8,10,13,16,19-Octaazabicyclo[6.6.6]icosane)platinum(IV) Dithionate 2½ Hydrate

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Abstract. [Pt(C₁₂H₃₀N₈)](S₂O₆)₂·2½H₂O, C₁₂H₃₀N₈·Pt⁴⁺·2S₂O₆²⁻·2½H₂O, FW = 846.78, monoclinic, *P*2₁/*c*, *a* = 18.250 (3), *b* = 9.264 (1), *c* = 17.461 (3) Å, β = 121.50 (1)°, *U* = 2614.8 (5) Å³, *Z* = 4, μ = 6.04 mm⁻¹, *D*_x = 2.151 Mg m⁻³. The cage-shaped ligand in which the metal ion is encapsulated is sexidentate in this complex. The geometry of the cage structure depends on the nature of the central metal atom. The three non-equivalent N–C bonds differ significantly from each other [1.498 (7), 1.421 (8) and 1.532 (8) Å]. The tris(ethylenediamine)Pt^{IV} moiety of the complex has a *lel*₃ conformation. The complex is hydrogen bonded to dithionate ions and water molecules to form a network in the *c* and *b* directions.

Introduction. The macrocyclic title complex offers interesting prospects for the study of intramolecular rearrangements, electron transfer and spectroscopic properties. With this ligand, a metal ion could be in different oxidation states without seriously changing its coordination number or the structure. Moreover, cage complexes are usually mononuclear (Creaser, Harrowfield, Herlt, Sargeson, Springborg, Geue & Snow, 1977). In this connection, the crystal structure of the title complex, trivially represented as [Pt(sepulchrate)](S₂O₆)₂·2½H₂O, was determined. The [Pt(sepulchrate)]⁴⁺ was prepared by condensation of a

formaldehyde and an ammonia with an ethylenediamine complex of Pt. The prismatic transparent crystals were kindly supplied by A. Sargeson of the Australian National University. The crystal used for the intensity measurements was bounded by ±(110), (1̄10), (001) and (111), with interplanar spacings of 0.10, 0.16, 0.15 and 0.25 mm respectively. X-ray diffraction data were collected within the range 2θ < 65° on an automated four-circle diffractometer, using graphite-monochromated Mo *K*α radiation (λ = 0.7107 Å). Four standard reflections counted at 50-reflection intervals indicated considerable decay (5–15%) during the two weeks of data collection. The intensity measurements were completed by employing six different sets of setting parameters based on the same 18 reflections in order to obtain the average structure.

Structure determination was carried out on 6305 independent reflections with |*F*_o| ≥ 3σ(|*F*|). Corrections were applied to the net intensities to account for Lorentz and polarization effects and for absorption. The lattice constants showed 0.08 Å elongations in *a* and *c*, while that of *b* showed a shrinkage of 0.06 Å during the period of data collection. The coordinates of the Pt and S atoms, derived from the sharpened Patterson maps, were used for a structure factor calculation which gave an *R* value of 0.382. Successive Fourier and difference syntheses revealed the remaining