

does not form any O...O hydrogen bonds, on account of steric reasons.

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## Structure of 4,11,17,24,29,32-Hexaoxa-1,14-diazatetracyclo[12.12.8.0<sup>5,10</sup>.0<sup>18,23</sup>]-tetratriaconta-5,7,9,18,20,22-hexaene

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**Abstract.** C<sub>26</sub>H<sub>36</sub>N<sub>2</sub>O<sub>6</sub>,  $M_r = 472.6$ , monoclinic,  $P2_1/c$ ,  $Z = 4$ ; at 138 K:  $a = 11.512$  (6),  $b = 8.493$  (4),  $c = 27.35$  (1) Å,  $\beta = 112.16$  (2)°,  $V = 2476.5$  Å<sup>3</sup>,  $D_c = 1.267$  Mg m<sup>-3</sup>; at 296 K:  $a = 11.565$  (7),  $b = 8.700$  (5),  $c = 27.33$  (2) Å,  $\beta = 111.62$  (3)°,  $V = 2556.4$  Å<sup>3</sup>,  $D_c = 1.228$  Mg m<sup>-3</sup>;  $F(000) = 1016$ . All intensity data were collected at  $138 \pm 2$  K. The structure was solved by direct methods and refined by blocked-full-matrix least-squares methods to a final  $R$  value of 0.066 for all 5087 independent reflections with  $2\theta(\text{Cu}) \leq 150^\circ$ .

**Introduction.** The complexation selectivity pattern of the title compound, cryptand 2<sub>B</sub>·2<sub>B</sub>·2, is markedly different from that of the parent compound 2.2.2 (Dietrich, Lehn & Sauvage, 1973; Lehn, 1973; Taylor, Springer & Ott, 1981). The structure determination of cryptand 2<sub>B</sub>·2<sub>B</sub>·2 was undertaken in order to determine the effects of the benzo rings on the cavity size and conformation of the ligand.

The compound was purchased from PCR Chemical Corporation and used without further purification.

Crystals were obtained from an aqueous solution in a thermal-gradient apparatus. A crystal 0.08 × 0.25 × 0.30 mm was used for data collection and unit-cell determination at  $138 \pm 2$  K using a Nonius CAD-4 automatic diffractometer controlled by a PDP8/e computer. The space group was determined uniquely as  $P2_1/c$  from systematic absences. The unit-cell dimensions were determined by a least-squares refinement of 48 reflections chosen from all octants of reciprocal space using Cu  $K\alpha_1$  radiation ( $\lambda = 1.54051$  Å). A total of 5087 independent reflections with  $2\theta(\text{Cu}) \leq 150^\circ$ , 3869 with  $I \geq 2\sigma(I)$ , were measured using  $\theta$ - $2\theta$  scans and Ni-filtered Cu  $K\alpha$  radiation ( $\lambda = 1.5418$  Å). The scan angle and receiving aperture were  $(0.90 + 0.14 \times \tan \theta)^\circ$  and  $(3.50 + 0.86 \tan \theta)$  mm, respectively. The receiving aperture, located 173 mm from the crystal, had a height of 4 mm. The maximum scan time per reflection was 90 s. For each reflection two thirds of the time was spent scanning the peak and the remaining one third of the time scanning the left and right backgrounds.

No significant changes were observed for the

measured intensities of three monitor reflections during the course of data collection. Three orientation-control reflections were checked after every 100 measurements. For any angular deviation  $>0.1^\circ$  a reorientation matrix was calculated using a set of 25 reflections. Reflections with  $I < 2\sigma(I)$  were assigned an intensity value equal to  $1.4\sigma(I)$ . The intensity data were corrected for Lorentz and polarization factors, but not for absorption ( $\mu = 6.5 \text{ cm}^{-1}$ ). Each structure amplitude was assigned an individual weight determined from  $\sigma(I)$  and based on counting statistics and intensity (Ealick & van der Helm, 1975).

The structure was solved with the aid of the direct-methods program *MULTAN* 80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). An *E* map calculated with 336 reflections ( $E \geq 1.83$ ), having a combined figure-of-merit equal to 3.0, gave the positions of all the non-H atoms. Following initial refinement with *SHELX* (Sheldrick, 1976), the H atoms were located from a difference Fourier map. Because of the size of the problem, further refinement was carried out with the matrix in three blocks, with anisotropic thermal parameters for the C, N and O atoms and isotropic thermal parameters for the H atoms. Refinement was discontinued when all parameter shifts were less than 25% of their corresponding e.s.d.'s. The final *R* factor for all reflections is 0.066 and the value of the weighted *R*  $\{[\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/2}\}$  is 0.054.

**Discussion.** A stereoview of a single molecule is shown in Fig. 1 and positional parameters for the C, N and O atoms are listed in Table 1.\* Bond distances and atom labeling for non-H atoms are shown in Fig. 2 while bond angles involving non-H atoms are listed in Table 2. Selected conformational angles are listed in Table 3.

The conformer of cryptand **2<sub>B</sub>.2<sub>B</sub>.2** present in the crystalline state has the *endo-endo* conformation (Simmons & Park, 1968; Lehn, 1973). The molecule possesses a more rounded shape than that of the

\* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and thermal and positional parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36775 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

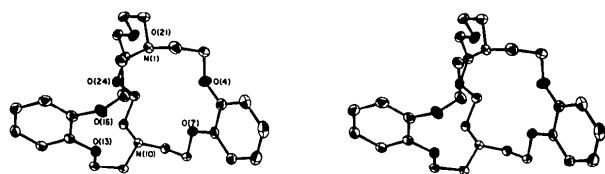


Fig. 1. Stereoview (*ORTEP*, Johnson, 1965) of cryptand **2<sub>B</sub>.2<sub>B</sub>.2**. Thermal ellipsoids are drawn at the 50% probability level.

Table 1. Positional parameters ( $\times 10^5$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ ) for the non-H atoms with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> *
N(1)	24845 (11)	80942 (17)	88183 (5)	212 (7)
C(2)	15747 (16)	89956 (22)	83823 (7)	270 (9)
C(3)	4846 (16)	79975 (25)	80459 (7)	291 (9)
O(4)	8955 (11)	68718 (15)	77563 (4)	294 (6)
C(5)	7711 (14)	71948 (21)	72474 (6)	219 (8)
C(6)	14547 (13)	61656 (20)	70525 (6)	183 (7)
O(7)	21896 (10)	51147 (14)	74153 (4)	219 (5)
C(8)	28405 (15)	39604 (21)	72375 (6)	220 (8)
C(9)	35144 (15)	29101 (20)	77040 (6)	213 (8)
C(27)	193 (15)	83400 (22)	69195 (7)	260 (8)
C(28)	-999 (16)	84213 (24)	63912 (7)	279 (9)
C(29)	5285 (16)	73731 (24)	61953 (7)	273 (9)
C(30)	13196 (15)	62415 (22)	65268 (6)	229 (8)
N(10)	45536 (11)	37134 (16)	81173 (5)	189 (6)
C(11)	56559 (14)	37063 (21)	79716 (6)	227 (8)
C(12)	67625 (14)	46163 (21)	83467 (6)	226 (8)
O(13)	63451 (10)	61751 (14)	83975 (4)	210 (5)
C(14)	70781 (14)	70434 (20)	88192 (6)	211 (7)
C(15)	65227 (14)	84239 (20)	89165 (6)	222 (8)
O(16)	53589 (10)	88938 (14)	85652 (5)	246 (6)
C(17)	42838 (15)	79827 (23)	85541 (7)	267 (9)
C(18)	37616 (16)	86588 (23)	89442 (7)	277 (9)
C(31)	83154 (14)	66919 (22)	91376 (6)	243 (8)
C(32)	89769 (15)	76878 (25)	95571 (6)	274 (9)
C(33)	84225 (16)	90202 (24)	96592 (7)	306 (9)
C(34)	71884 (16)	93920 (22)	93332 (7)	284 (8)
C(19)	20899 (16)	81033 (23)	92706 (7)	285 (9)
C(20)	27773 (16)	69423 (23)	97038 (6)	276 (9)
O(21)	26615 (11)	54089 (15)	94916 (4)	258 (6)
C(22)	32575 (16)	42487 (24)	98836 (6)	273 (9)
C(23)	33527 (16)	27395 (23)	96209 (7)	271 (8)
O(24)	42526 (10)	27841 (16)	93757 (4)	250 (6)
C(25)	37822 (16)	33532 (23)	88445 (6)	250 (8)
C(26)	48096 (15)	30217 (21)	86377 (6)	222 (8)

$$* U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}.$$

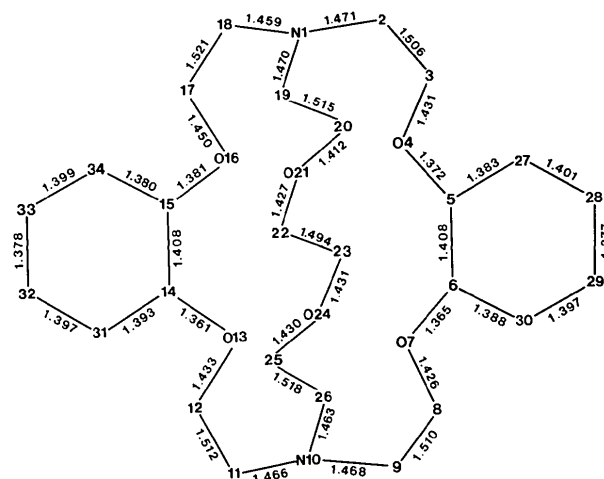


Fig. 2. Atom numbering and bond distances ( $\text{\AA}$ ) in cryptand **2<sub>B</sub>.2<sub>B</sub>.2**. The standard deviation for the bonds involving C, N and O atoms is 0.002–0.003  $\text{\AA}$ .

unsubstituted analog, cryptand 2.2.2, which has an ellipsoidal shape (Metz, Moras & Weiss, 1976). O atoms (13) and (16) are tangential to the central cavity, O atoms (4), (7), and (21) are directed toward the center of the ligand cavity while O atom (24) points away from the interior of the ligand cavity. If the metal-ion complexes of 2<sub>B</sub>.2<sub>B</sub>.2 are similar to those of 2.2.2 and 2.2.1, smaller conformational changes are required during the formation of the complexes of 2<sub>B</sub>.2<sub>B</sub>.2 than those for 2.2.2 and 2.2.1. The introduction of the two benzo rings forces O-atom pairs (4), (7), and (13), (16) into a *cis* configuration with O—C—C—O torsion angles of  $-4.8$  (2) and  $-6.1$  (2)°, respectively, while the corresponding torsion angle involving O(21) and O(24) is  $71.4$  (2)°. These values are in sharp contrast to the analogous torsion angles of cryptand 2.2.2, which have an average value of  $178^\circ$  (Metz *et*

*al.*, 1976). The torsion angle for O(21)—C—C—O(24) is similar to those found for the 2.2.2 cryptates of K, Rb, and Cs ions which have average values of 51, 65, and 70°, respectively (Moras, Metz & Weiss, 1973*a,b*). One consequence of the reduced O—C—C—O torsion angles in cryptand 2<sub>B</sub>.2<sub>B</sub>.2 is a decrease of the N(1)···N(10) distance to  $5.161$  (2) Å compared to the value of  $6.871$  (4) Å found for cryptand 2.2.2 (Metz *et al.*, 1976).

The N(1)···N(10) distance of cryptand 2<sub>B</sub>.2<sub>B</sub>.2 lies at the lower end of the range of values (4.97 to 6.07 Å) found for a variety of metal-cation complexes of cryptand 2.2.2 (Metz, Moras & Weiss, 1971; Metz & Weiss, 1974). The limited conformational freedom of the central O—C—C—O segment may hinder expansion of the ligand cavity to accommodate large cations. This is consistent with the observed trends in the complex-formation constants where the values of  $\log K_{\text{stab}}$  for the Na-ion complex increase from 6.95 to 7.3 while those for the K-ion complex decrease from 9.45 to 8.60 and those for the Ba-ion complex decrease from 11.5 to 8.5 (Lehn, 1973) going from 2.2.2 to 2<sub>B</sub>.2<sub>B</sub>.2. The TI<sup>I</sup> complexes are also weakened by the introduction of the benzo moieties (Taylor *et al.*, 1981). The C(sp<sup>3</sup>)—C(sp<sup>3</sup>) bond lengths vary from 1.494 (3) to 1.521 (2) Å with an average value of 1.511 (9) Å. These are shorter than the expected value of 1.537 (5) Å for aliphatic C—C bonds (Sutton, 1965) but are consistent with values found in a wide variety of macrocyclic polyethers which vary from 1.47–1.51 Å (Dunitz, Dobler, Seiler & Phizackerley, 1974; Hanson, Hughes & Truter, 1976; Hašek, Hlavatá & Huml, 1977; Goldberg, 1975*a,b*, 1978). This apparent shortening has been ascribed in part to inadequate treatment of curvilinear vibrational motion (Dunitz *et al.*, 1974); however, Goldberg (1975*a,b*, 1976, 1978) has found that the effect persists at low temperatures (178 to 113 K) and suggests that the shortening of the C—C bond is real and may be due to a specific interaction between the C and O atoms (Goldberg, 1975*a*, 1978). A detailed treatment of the structure of 18-crown-6 at 100 K results in thermal-motion correc-

Table 2. Bond angles (°)

C(2)—N(1)—C(18)	111.5	N(10)—C(26)—C(25)	112.9
C(2)—N(1)—C(19)	109.1	C(11)—C(12)—O(13)	108.1
C(18)—N(1)—C(19)	113.8	C(12)—O(13)—C(14)	117.4
N(1)—C(2)—C(3)	112.1	O(13)—C(14)—C(15)	115.5
C(2)—C(3)—O(4)	109.9	O(13)—C(14)—C(31)	125.1
C(3)—O(4)—C(5)	119.9	C(14)—C(15)—C(34)	120.1
O(4)—C(5)—C(6)	113.6	C(14)—C(31)—C(32)	119.7
O(4)—C(5)—C(27)	126.8	C(31)—C(32)—C(33)	120.8
C(5)—C(6)—C(30)	120.2	C(32)—C(33)—C(34)	119.5
C(5)—C(27)—C(28)	119.8	C(33)—C(34)—C(15)	120.4
C(27)—C(28)—C(29)	120.5	C(15)—C(14)—C(31)	119.4
C(28)—C(29)—C(30)	120.1	C(34)—C(15)—O(16)	119.6
C(29)—C(30)—C(6)	119.7	C(14)—C(15)—O(16)	120.0
C(6)—C(5)—C(27)	119.6	C(15)—O(16)—C(17)	117.2
C(30)—C(6)—O(7)	125.4	O(16)—C(17)—C(18)	110.1
C(5)—C(6)—O(7)	114.3	C(17)—C(18)—N(1)	109.9
C(6)—O(7)—C(8)	117.6	N(1)—C(19)—C(20)	114.8
O(7)—C(8)—C(9)	106.7	C(19)—C(20)—O(21)	109.8
C(8)—C(9)—N(10)	112.7	C(20)—O(21)—C(22)	112.6
C(9)—N(10)—C(11)	109.0	O(21)—C(22)—C(23)	109.4
C(9)—N(10)—C(26)	111.7	C(22)—C(23)—O(24)	113.6
C(11)—N(10)—C(26)	112.7	C(23)—O(24)—C(25)	114.9
N(10)—C(11)—C(12)	114.0	O(24)—C(25)—C(26)	105.4

E.s.d.'s range from 0.1–0.2°.

Table 3. Selected conformational angles (°) with e.s.d.'s for the last digit in parentheses

N(1)—C(2)—C(3)—O(4)	-69.1 (1)	C(14)—C(15)—O(16)—C(17)	72.5 (2)	C(2)—N(1)—C(19)—C(20)	167.6 (1)
C(2)—C(3)—O(4)—C(5)	-95.7 (2)	C(15)—O(16)—C(17)—C(18)	90.0 (2)	C(18)—N(1)—C(2)—C(3)	147.2 (1)
C(3)—O(4)—C(5)—C(6)	166.7 (1)	O(16)—C(17)—C(18)—N(1)	161.9 (1)	C(18)—N(1)—C(19)—C(20)	-67.2 (2)
O(4)—C(5)—C(6)—O(7)	-4.8 (2)	N(1)—C(19)—C(20)—O(21)	-55.7 (2)	C(19)—N(1)—C(2)—C(3)	-86.2 (2)
C(5)—C(6)—O(7)—C(8)	175.6 (1)	C(19)—C(20)—O(21)—C(22)	-178.2 (1)	C(19)—N(1)—C(18)—C(17)	155.7 (1)
C(6)—O(7)—C(8)—C(9)	-176.9 (1)	C(20)—O(21)—C(22)—C(23)	-167.2 (1)	C(9)—N(10)—C(11)—C(12)	175.4 (1)
O(7)—C(8)—C(9)—N(10)	-69.3 (2)	O(21)—C(22)—C(23)—O(24)	71.4 (2)	C(9)—N(10)—C(26)—C(25)	-69.9 (2)
N(10)—C(11)—C(12)—O(13)	-53.8 (2)	C(22)—C(23)—O(24)—C(25)	-87.4 (2)	C(11)—N(10)—C(9)—C(8)	-81.3 (2)
C(11)—C(12)—O(13)—C(14)	162.2 (1)	C(23)—O(24)—C(25)—C(26)	-170.1 (1)	C(11)—N(10)—C(26)—C(25)	166.9 (1)
C(12)—O(13)—C(14)—C(15)	-167.5 (1)	O(24)—C(25)—C(26)—N(10)	-172.6 (1)	C(26)—N(10)—C(9)—C(8)	153.6 (1)
O(13)—C(14)—C(15)—O(16)	-6.1 (2)	C(2)—N(1)—C(18)—C(17)	-80.3 (2)	C(26)—N(10)—C(11)—C(12)	-60.0 (2)

tion of 0.007 Å for the mean C—C bond length (Maverick, Seiler, Schweizer & Dunitz, 1980) which would give a corrected mean C—C bond length of 1.519 Å, close to the dioxane value of 1.523 Å (Davis & Hassel, 1963), but still much shorter than the paraffinic value of 1.537 (5) Å (Sutton, 1965). For the cryptands a small difference is observed between the average values of the C(sp<sup>3</sup>)—C(sp<sup>3</sup>) bonds in the O—C—C—O and N—C—C—O segments. Thus, for 2<sub>B</sub>.2<sub>B</sub>.2 the C—C bond length in O—C—C—O is 1.494 (3) Å while the average C—C bond length in N—C—C—O is 1.514 (6) Å. This pattern can also be observed for cryptand 2.2.2 where the respective average C—C bond lengths are 1.482 (10) and 1.508 (6) Å (Metz *et al.*, 1976) and for the macrocyclic analog, 4,7,13,16-tetraoxa-1,10-diazacyclooctadecane (2.2) which has average C—C bond lengths of 1.477 (9) and 1.513 (10) Å in the O—C—C—O and N—C—C—O segments, respectively (Herceg & Weiss, 1972).

The present results and those reported for similar structures seem to indicate that the C—C bond-length shortening is real and that the effect is larger when both neighbors of the C—C bond are O atoms compared to the case where N and O are the adjacent atoms. The average C(sp<sup>3</sup>)—O bond length of 2<sub>B</sub>.2<sub>B</sub>.2 is 1.430 (10) Å which is close to the expected value, 1.426 (5) Å, (Sutton, 1965) and those found in 2.2.2, 1.408 (19), and 2.2, 1.433 (26) Å. The average C(sp<sup>3</sup>)—N bond length in 2<sub>B</sub>.2<sub>B</sub>.2 is 1.466 (5) Å which is close to the expected value of 1.472 (5) Å (Sutton, 1965) and similar to those found in 2.2.2, 1.460 (12) Å, (Metz *et al.*, 1976), and 2.2, 1.456 (6) Å (Herceg & Weiss, 1972).

The aromatic C—C bond lengths range from 1.377 to 1.408 Å with an average value of 1.392 (12) Å which is very close to the expected bond length of 1.395 (3) Å (Sutton, 1965). The benzene rings show little deviation from planarity with an average difference of 0.009 (6) Å from the least-squares planes calculated for the individual rings. The largest deviations from the calculated planes are for C(5), 0.020; C(6), -0.016; C(29), 0.013; and C(14), -0.011 Å.

There is no possibility of hydrogen bonding, and the closest intermolecular contacts involving non-H atoms are: C(8)···O(13) 3.274 (2) Å and C(8)···C(14) 3.349 (2) Å, both O(13) and C(14) at  $1 - x, \frac{1}{2} + y, 1\frac{1}{2} - z$ ; and C(3)···O(7) 3.372 (2) Å, O(7) at  $-x, -\frac{1}{2} + y, \frac{1}{2} - z$ . There are no other non-H-atom intermolecular contacts less than 3.4 Å.

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