

Table 2. *Hydrogen-bond distances* (Å)

A-B...C	AC	BC
N(1)–H(N1)···N(3) ⁱ	2.850 (6)	2.23 (8)
O(9)–H(O9)···O(10) ⁱⁱ	2.676 (6)	
N(11)–H(N11)···O(13) ⁱⁱⁱ	2.829 (6)	2.13 (8)

Symmetry code: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$; (iii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

As in L-histidine and 5-nitro-L-histidine, short intermolecular distances are present in the title compound. These are O(16)···O(10)ⁱ = 3.091 (6) and O(17)···N(15)ⁱⁱ = 3.143 (6) Å [(i) = $\frac{1}{2} - x, y, \frac{1}{2} + z$; (ii) = $\frac{1}{2} - x, \bar{y}, z - \frac{1}{2}$].

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A 2:1 Complex of 4-Nitro-1,2-benzenediamine and 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6)

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Abstract. $2C_6H_7N_3O_2 \cdot C_{12}H_{24}O_6$, $M_r = 570.60$, monoclinic, $P2_1/c$, $a = 9.740$ (2), $b = 10.178$ (3), $c = 14.615$ (3) Å, $\beta = 95.12$ (5)°, $Z = 2$, $d_c = 1.313$ Mg m⁻³, $\mu(\text{Mo } K\alpha) = 0.096$ mm⁻¹; $R = 0.047$, $R_w = 0.054$ for 2101 independent data. The two 4-nitro-1,2-benzenediamine molecules, related by a centre of inversion, are inclined with dihedral angles 120 (1)° 'above' and 'below' the plane formed by the six O atoms of the crown ether. They are linked to the latter *via* H bridges, none of which is bifurcated: six H atoms of the four NH₂ groups in the complex serve as e⁻ acceptors from only four O atoms, *i.e.* two O atoms receive two H bonds each, two receive one H bond each, and two O atoms remain without strong interactions. This pattern causes the macrocycle to adopt a conformation with a sequence of unique torsion angles $ag^+a ag^+a g^+g^+a$ which differs from the approximate D_{3d} symmetry normally found.

Introduction. A number of crystalline adducts of 18-crown-6 (or its derivatives) with H-bond donors have been prepared during the last decade (Pedersen, 1971; Gokel, Cram, Liotta, Harris & Cook, 1974; el Basyony, Klimes, Knöchel, Oehler & Rudolph, 1976; Vögtle & Müller, 1980, 1981). In these compounds, as in the well known complexes with 'fitting' metal ions (*e.g.* Dunitz, Dobler, Seiler & Phizackerley, 1974), the ligand usually adopts a conformation with approximate D_{3d} symmetry, *e.g.* with dimethyl acetylenedicarboxylate (Goldberg, 1975), malononitrile (Kaufmann, Knöchel, Kopf, Oehler & Rudolph, 1977), NH₄⁺ (Nagano, Kobayashi & Sasaki, 1978), benzylammonium (Bovill, Chadwick, Sutherland & Watkin, 1980), 2,4-dinitrophenylhydrazine (Hilgenfeldt & Saenger, 1981) and *p*-nitroaniline (Weber, 1981). Recently reported structures of adducts with urea (Harkema, van Hummel, Daasvatn & Reinhoudt,

1981) and 2,4-dinitroaniline (Weber & Sheldrick, 1981) have extended the known range of the less common 'biangular [99]' (Dale, 1973) conformation, previously observed in the benzenesulphonamide (Knöchel, Kopf, Oehler & Rudolph, 1978) and uranyl nitrate dihydrate complexes (Bombieri, de Paoli & Immirzi, 1978). The present study was undertaken to classify the conformation in the title compound.

Red-orange prisms of the adduct were grown from acetone (Vögtle & Müller, 1981). Cell dimensions were obtained from a least-squares analysis of 24 strong reflexions in the range $20^\circ \leq 2\theta \leq 25^\circ$. A crystal of approximate size $0.7 \times 0.5 \times 0.3$ mm was selected for the collection of 2538 unique data on an automated X-ray four-circle diffractometer in a profile-fitting procedure (Clegg, 1981) up to $2\theta = 50^\circ$ (monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å). Lp corrections were applied.

The structure was solved by direct methods; non-hydrogen atoms were refined anisotropically using 2104 reflexions with $F > 3\sigma(F)$. A difference map then revealed the positions of all H atoms. Those of the amine groups were refined without positional constraints; for other H atoms (in idealized positions, C—H = 0.96 Å) a riding model was employed. All H atoms were assigned fixed isotropic thermal parameters 1.2 times the U values of the attached atoms. Omission of three poorly agreeing low-angle reflexions and refinement of an empirical extinction coefficient x

Table 1. Atom coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}^*/U
O(1)	7058 (1)	-1223 (1)	8722 (1)	54 (1)
C(2)	8136 (2)	-317 (2)	8601 (1)	68 (1)
C(3)	8404 (2)	644 (2)	9381 (2)	68 (1)
O(4)	7236 (1)	1464 (1)	9416 (1)	57 (1)
C(5)	7343 (2)	2261 (2)	10219 (1)	67 (1)
C(6)	6112 (2)	3130 (2)	10215 (1)	66 (1)
O(7)	4906 (1)	2354 (1)	10260 (1)	57 (1)
C(8)	3767 (2)	3081 (2)	10524 (1)	62 (1)
C(9)	2588 (2)	2174 (2)	10586 (1)	58 (1)
C(10)	921 (2)	-100 (2)	6917 (1)	52 (1)
C(11)	1999 (2)	-985 (2)	6913 (1)	52 (1)
C(12)	3215 (2)	-763 (2)	7441 (1)	50 (1)
C(13)	3340 (2)	378 (2)	8001 (1)	48 (1)
C(14)	2246 (2)	1254 (2)	7974 (1)	54 (1)
C(15)	1031 (2)	1029 (2)	7437 (1)	56 (1)
N(16)	-347 (2)	-381 (2)	6352 (1)	69 (1)
O(17)	-406 (2)	-1366 (2)	5867 (1)	103 (1)
O(18)	-1324 (2)	369 (2)	6388 (1)	96 (1)
N(19)	4305 (2)	-1622 (2)	7420 (1)	83 (1)
N(20)	4543 (2)	621 (1)	8529 (1)	64 (1)
H(19a)	4182 (23)	-2341 (23)	7076 (16)	87
H(19b)	5005 (25)	-1562 (24)	7741 (16)	87
H(20a)	5115 (20)	-52 (17)	8697 (12)	69
H(20b)	4493 (19)	1211 (17)	8953 (13)	69

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

$[F^* = F(1 - xF^2/\sin \theta)$, $x = 8(1) \times 10^{-7}$] resulted in final discrepancy factors $R = 0.047$ and $R_w = 0.054$ with $w^{-1} = \sigma^2(F_o) + 0.0006F_o^2$.

Table 1 lists the coordinates of all refined atoms,

Table 2. Bond lengths (Å)

C(2)—O(1)	1.420 (3)	C(2)—C(3)	1.507 (4)
C(3)—O(4)	1.415 (3)	C(5)—O(4)	1.422 (3)
C(5)—C(6)	1.490 (4)	C(6)—O(7)	1.421 (3)
C(8)—O(7)	1.416 (3)	C(8)—C(9)	1.483 (4)
C(9)—O(1')	1.419 (3)	C(10)—C(11)	1.384 (4)
C(10)—C(15)	1.378 (4)	C(10)—N(16)	1.452 (3)
C(11)—C(12)	1.373 (3)	C(12)—C(13)	1.420 (3)
C(12)—N(19)	1.378 (4)	C(13)—C(14)	1.387 (4)
C(13)—N(20)	1.367 (3)	C(14)—C(15)	1.379 (4)
N(16)—O(17)	1.226 (4)	N(16)—O(18)	1.225 (3)
C(9)—O(1')	1.419 (3)	N(19)—H(19a)	0.889 (24)
N(19)—H(19b)	0.794 (23)	N(20)—H(20a)	0.903 (18)
N(20)—H(20b)	0.868 (18)		

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

C(2)—O(1)—C(9)'	113.3 (2)	O(1)—C(2)—C(3)	114.0 (3)
C(2)—C(3)—O(4)	109.0 (3)	C(3)—O(4)—C(5)	111.5 (2)
O(4)—C(5)—C(6)	109.6 (3)	C(5)—C(6)—O(7)	109.8 (3)
C(6)—O(7)—C(8)	113.1 (2)	O(7)—C(8)—C(9)	108.8 (2)
C(8)—C(9)—O(1)'	109.1 (2)	C(11)—C(10)—C(15)	121.6 (3)
C(11)—C(10)—N(16)	118.5 (3)	C(15)—C(10)—N(16)	119.9 (3)
C(10)—C(11)—C(12)	120.5 (3)	C(11)—C(12)—C(13)	118.9 (3)
C(11)—C(12)—N(19)	120.7 (3)	C(13)—C(12)—N(19)	120.3 (3)
C(12)—C(13)—C(14)	118.9 (3)	C(12)—C(13)—N(20)	119.9 (3)
C(14)—C(13)—N(20)	121.2 (3)	C(13)—C(14)—C(15)	121.8 (3)
C(10)—C(15)—C(14)	118.2 (3)	O(10)—N(16)—O(17)	118.9 (3)
C(10)—N(16)—O(18)	118.5 (3)	O(17)—N(16)—O(18)	122.6 (3)
C(12)—N(19)—H(19a)	118 (2)	C(12)—N(19)—H(19b)	124 (2)
H(19a)—N(19)—H(19b)	118 (2)	C(13)—N(20)—H(20a)	120 (1)
C(13)—N(20)—H(20b)	115 (1)	H(20a)—N(20)—H(20b)	114 (2)

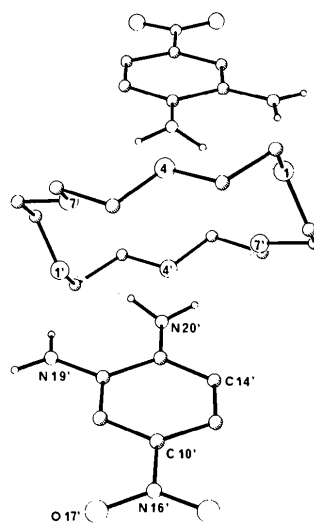


Fig. 1. A perspective view of the adduct. Primed atoms are related by inversion at the centre of 18-crown-6 ($1 - x, -y, 2 - z$). Radii are arbitrary.

with derived bond lengths and angles in Tables 2 and 3.* A view of the complex is given in Fig. 1.

Discussion. Recently reported molecular-mechanics calculations of ethers (Bovill, Chadwick, Sutherland & Watkin, 1980) showed the commonly observed D_{3d} conformation of 18-crown-6 to be $11.85 \text{ kJ mol}^{-1}$ less stable than the biangular conformation. The relatively high steric energy is mostly due to the arrangement of O atoms (mean distance *ca* 2.8 \AA) resulting in unfavourable dipole interactions. It is, however, more than compensated by favourable interactions of all the O atoms with an electrophilic centre. Hence a 'round', *i.e.* D_{3d} , conformation of the macrocycle would be indicated with spherical (*e.g.* alkaline cations) or pseudo-spherical (*e.g.* $R\text{-NH}_3^+$, NH_4^+ or $R\text{-CH}_3^{\delta+}$) e^- acceptors, whilst with more elongated guests (*e.g.* $R\text{-NH}_2$, $R\text{-CH}_2^{\delta+}$ or H_2O) the biangular conformation should be preferred. This simple rule (exceptions: Kaufmann, Knöchel, Kopf, Oehler & Rudolph, 1977; Weber, 1981) is supported by the present structure, as displayed in Fig. 2, with torsion angles close to theoretical values [except $\text{C}(5)\text{-C}(6)\text{-O}(7)\text{-C}(8) = 162.8(3)^\circ$, see Table 4].* Shortened C—C bonds (average 1.493 \AA), slightly widened C—O—C angles (average 112.6°), and almost ideal tetrahedral O—C—C angles [average 109.3° , neglecting $\text{O}(1)\text{-}$

* 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 36303 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

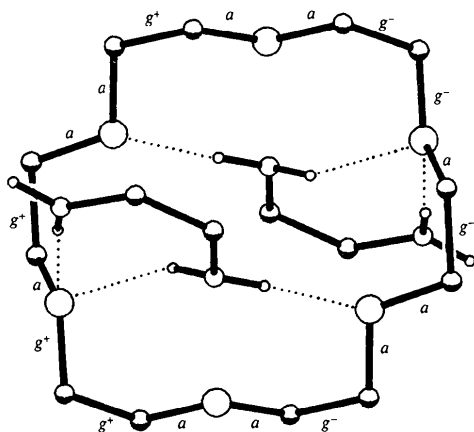


Fig. 2. The present conformation of the macrocycle ($a = \textit{anti}$, $g^\pm = \pm \textit{gauche}$) and the system of intermolecular H bridges illustrated in a projection normal to the best plane of the six O atoms.

For clarity, only the $\text{H}_2\text{N}-\overset{\textstyle |}{\text{C}}=\overset{\textstyle |}{\text{C}}-\text{NH}_2$ entities are included.

Table 4. Torsion angles ($^\circ$) in the crown ether

$\text{C}(8)\text{'-C}(9)\text{'-O}(1)\text{'-C}(2)$	$-179.1(3)$	$\text{C}(9)\text{'-O}(1)\text{'-C}(2)\text{'-C}(3)$	$-71.1(3)$
$\text{O}(1)\text{'-C}(2)\text{'-C}(3)\text{'-O}(4)$	$-65.4(3)$	$\text{C}(2)\text{'-C}(3)\text{'-O}(4)\text{'-C}(5)$	$171.3(3)$
$\text{C}(3)\text{'-O}(4)\text{'-C}(5)\text{'-C}(6)$	$178.5(3)$	$\text{O}(4)\text{'-C}(5)\text{'-C}(6)\text{'-O}(7)$	$63.7(3)$
$\text{C}(5)\text{'-C}(6)\text{'-O}(7)\text{'-C}(8)$	$162.8(3)$	$\text{C}(6)\text{'-O}(7)\text{'-C}(8)\text{'-C}(9)$	$-177.7(3)$
$\text{O}(7)\text{'-C}(8)\text{'-C}(9)\text{'-O}(1)\text{'}$	$62.4(3)$		

$\text{C}(2)\text{'-C}(3)\text{'}$ are well known characteristics of annular oligoethers (for references, see above and Maverick, Seiler, Schweizer & Dunitz, 1980); the unusual angle $\text{O}(1)\text{'-C}(2)\text{'-C}(3) = 114.0(3)^\circ$ may facilitate the twofold coordination of $\text{O}(1)$. The six O atoms of the ligand, located alternately about 0.2 \AA above and below their mean plane, form a distorted hexagon with $\text{O}(1)\text{'}\cdots\text{O}(4) = 2.916(3)$, $\text{O}(4)\text{'}\cdots\text{O}(7) = 2.827(3)$, $\text{O}(7)\text{'}\cdots\text{O}(1)\text{'} = 2.776(3) \text{ \AA}$, $\text{O}(7)\text{'}\cdots\text{O}(1)\text{'}\cdots\text{O}(4) = 103.0(2)$, $\text{O}(1)\text{'}\cdots\text{O}(4)\text{'}\cdots\text{O}(7) = 115.3(2)$ and $\text{O}(4)\text{'}\cdots\text{O}(7)\text{'}\cdots\text{O}(1)\text{'} = 136.0(2)^\circ$, and thus accommodate the elongated geometries of the

$\text{H}_2\text{N}-\overset{\textstyle |}{\text{C}}=\overset{\textstyle |}{\text{C}}-\text{NH}_2$ entities.

The two guest molecules, related by inversion at the centre of 18-crown-6, are inclined at $120(1)^\circ$ on either side of this hexagon, each thereby bringing three H atoms into positions suitable for hydrogen bonds. This is facilitated by a small rotation of the *m*-amino group against the phenyl plane: (I)/(II) = $1(3)$, (I)/(III) = $8(3)$, (I)/(IV) = $3(1)^\circ$ [(I) = C(10) to C(15); (II) = C(12), N(19), H(19a), H(19b); (III) = C(13), N(20), H(20a), H(20b); (IV) = C(10), N(16), O(17), O(18)]. The decreased acidity of the NH_2 groups, as compared to 2,4-dinitroaniline (Weber & Sheldrick, 1981), is reflected in C(12)—N(19) and C(13)—N(20) bonds of $1.378(4)$ and $1.367(3) \text{ \AA}$, respectively, comparable to C— NH_2 distances in non-complexing *o*-nitroaniline (Dhaneshwar, Tavale & Pant, 1978) and 3-aminopyridine (Chao, Schempp & Rosenstein, 1975) but slightly shorter than in 1,2-diaminobenzene and its hydrochloride (Stålhandske, 1976). Accordingly, a systematic distribution of bond lengths in the benzene ring is less distinct; but the lengthened C(12)—C(13) bond of $1.420(3) \text{ \AA}$ (between the two amine functions) may give some evidence for a contribution of quinonoid canonical forms to the overall resonance state.

In the molecular complexes of 18-crown-6 investigated so far, bifurcated H bridges (as defined by Donohue, 1968) are observed only when more than one electron-withdrawing group renders H atoms sufficiently acidic (Hilgenfeld & Saenger, 1981; Weber & Sheldrick, 1981). This also applies for the present structure, where the 4-nitro-1,2-benzenediamines are linked to the ligand only by 'normal' bonds (see Table 5). In view of the relatively short $\text{O}\cdots\text{N}$ distances (Table 5), H bridges to *all* O atoms seemed possible. However, $\text{O}(4)\text{'}\cdots\text{H}(20a) = 2.714(18)$, $\text{O}(4)\text{'}\cdots\text{H}(20b) = 2.710(18) \text{ \AA}$ and $\text{O}(4)\text{'}\cdots\text{H-N}$ angles of about 96° probably preclude strong inter-

Table 5. *Hydrogen-bond distances (Å) and angles (°)*

O(1)···N(19)	3.173 (4)	O(1)···H(19b)	2.380 (22)
O(1)···N(20)	3.079 (3)	O(1)···H(20a)	2.235 (19)
O(4)···N(20)	2.948 (3)	O(4)···H(20a)	2.714 (18)
		O(4)···H(20b)	2.710 (18)
O(7)···N(20)	3.078 (3)	O(7)···H(20b)	2.241 (19)
O(1)···H(19b)—N(19)	176 (2)	O(4)···H(20b)—N(20)	97 (1)
O(1)···H(20a)—N(20)	156 (1)	O(7)···H(20b)—N(20)	162 (1)
O(4)···H(20a)—N(20)	96 (1)		

actions, even when taking into account the short N—H bonds (≤ 0.9 Å).

A peculiarity of this structure is that O(1) accepts two H bonds of similar strength. A twofold co-ordination of one crown O has been observed before (Hilgenfeld & Saenger, 1981), but at substantially different distances.

Weak linkages between adducts might be indicated by $N(20) \cdots H(19a)^{ii} = 2.612(20)$ Å [(ii) = $1 - x, 0.5 + y, 1.5 - z$].

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Acta Cryst. (1982). **B38**, 632–635

Sulfate de *tert*-Butyl[(dihydroxy-3,5 phényl)-2 hydroxy-2 éthyl]ammonium (Sulfate de Terbutaline) Hydraté

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Abstract. $[NH_2(C_4H_9)(C_8H_9O_3)]^+ \cdot \frac{1}{2}SO_4^{2-} \cdot \frac{3}{2}H_2O$, $M_r = 301.35$, triclinic, $P1$, $a = 10.984(2)$, $b = 11.935(4)$, $c = 14.521(2)$ Å, $\alpha = 62.49(2)$, $\beta = 62.69(1)$, $\gamma = 78.80(2)^\circ$, $Z = 4$, $D_x = 1.340$ Mg m $^{-3}$. The structure was refined to $R = 0.076$ for

5742 observed reflections. The crystal structure is stabilized by a network of N—H···O, O—H···O hydrogen bonds. The two independent molecules are not very different and are compared with those of salbutamol.

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