

1,4,7,10,13,16-Hexaoxacycloocta-
decane–ammonia (1/2)

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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.048
 wR factor = 0.123
Data-to-parameter ratio = 13.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, a 1:2 adduct of 18-crown-6 and ammonia, $C_{12}H_{24}O_6 \cdot 2NH_3$, has inversion symmetry. Two ammonia molecules are connected to the crown ether above and below the cyclic plane *via* $N-H \cdots O$ hydrogen bonds to form a disc-like adduct.

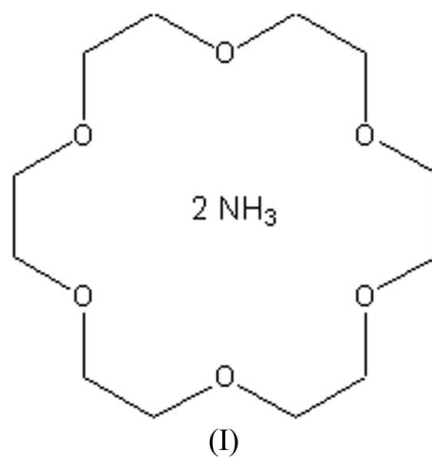
Received 19 August 2005

Accepted 31 August 2005

Online 7 September 2005

Comment

The group of cyclic polyethers, the so called crown ethers, is certainly one of the most useful substance classes in organic, metallorganic and inorganic chemistry. Crown ethers, which are tools to increase the solubility of inorganic salts in organic solvents (Yakshin, 2002), are able to coordinate to charged as well as neutral molecules (Elbasyouny *et al.*, 1983; Huang *et al.*, 1993; Doxsee *et al.*, 2000) and have been used to isolate such unusual compounds as alkalides and electrides (Dye, 1984). The fact that almost every week new crown ether compounds are published confirms this impression. We report here on the title compound, (I), the 1:2 adduct of 18-crown-6 and ammonia, which adds to the list of other solvent–crown ether adducts, starting with the water complexes of crown ethers (Newkome *et al.*, 1981; Mootz *et al.*, 1994) through acetonitrile compounds (Mosier-Boss & Popov, 1985; Mosier-Boss, 2005) to adducts containing formamide or nitromethane (Voegtle *et al.*, 1980; de Boer *et al.*, 1982).



Compound (I) has inversion symmetry (Fig. 1). The disc-like building blocks are stacked along the c axis with a tilt angle of $52.4(1)^\circ$ between the plane normal (line between the two N atoms) and the c axis (Fig. 2). The $N-H \cdots O$ hydrogen bonds (Table 1) connect the NH_3 molecules with the crown ether. The $H \cdots O$ distances [$2.30(3)$ – $2.43(3)$ Å] agree with values recently reported for the $\{[Li(NH_3)_4]_2[18\text{-crown-6}]\}^{2+}$ cation (Wiesler & Korber, 2005).

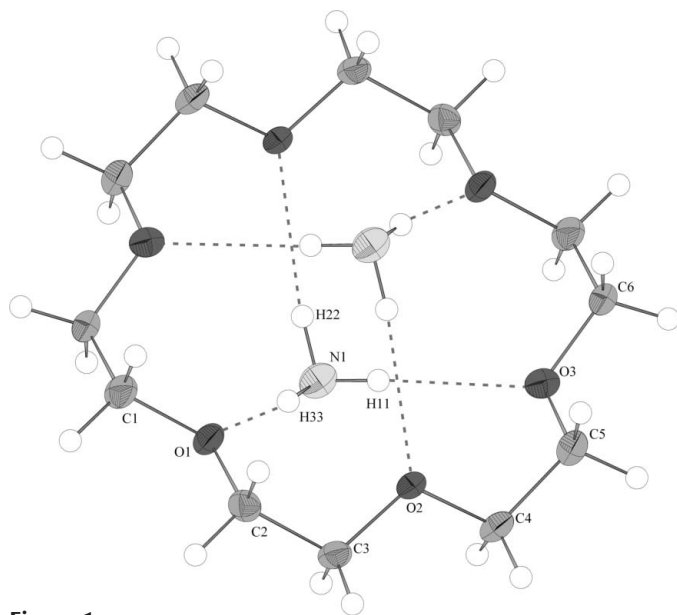


Figure 1

The molecular structure of (I) with the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by $(-x, -y, 1-z)$. Broken lines indicate hydrogen bonds.

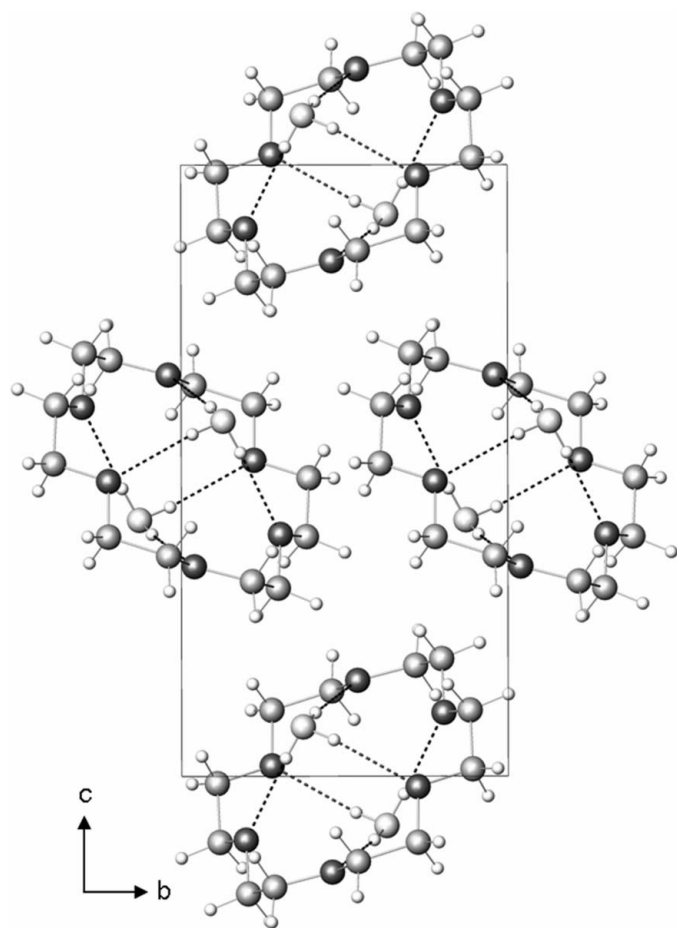


Figure 2

A packing scheme of (I), projected along the a axis. Broken lines indicate hydrogen bonds.

Experimental

Crystals of (I) were grown by slow evaporation of a saturated solution of 18-crown-6 in liquid ammonia at 195 K.

Crystal data

$C_{12}H_{24}O_6 \cdot 2H_3N$
 $M_r = 298.38$
 Monoclinic, $P2_1/c$
 $a = 7.4789$ (14) Å
 $b = 7.6632$ (14) Å
 $c = 14.374$ (3) Å
 $\beta = 91.754$ (4)°
 $V = 823.4$ (3) Å³
 $Z = 2$

$D_x = 1.203$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1983 reflections
 $\theta = 5.5\text{--}59.7^\circ$
 $\mu = 0.10$ mm⁻¹
 $T = 100$ (2) K
 Block, colourless
 $0.4 \times 0.2 \times 0.15$ mm

Data collection

Bruker SMART APEX diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.767$, $T_{\max} = 0.986$
 11143 measured reflections

1983 independent reflections
 1400 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$
 $\theta_{\max} = 28.0^\circ$
 $h = -9 \rightarrow 9$
 $k = -10 \rightarrow 10$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.123$
 $S = 1.05$
 1983 reflections
 151 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.1375P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H33 \cdots O1$	0.89 (3)	2.30 (3)	3.181 (2)	175 (2)
$N1-H22 \cdots O2^i$	0.90 (3)	2.42 (3)	3.320 (2)	174 (2)
$N1-H11 \cdots O3$	0.85 (3)	2.43 (3)	3.281 (2)	173 (2)

Symmetry code: (i) $-x, -y, -z + 1$.

All H atoms were located in difference maps and refined freely. The C—H and N—H distances are 0.93 (2)–1.01 (2) Å and 0.85 (3)–0.90 (3) Å, respectively.

Data collection: SMART32 (Bruker, 1997); cell refinement: SAINT32 (Bruker, 2003); data reduction: SAINT32; program(s) used to solve structure: SHELXTL (Sheldrick, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: ATOMS (Dowty, 2002); software used to prepare material for publication: SHELXTL.

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