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Crystal Structure of Supramolecular Complexes Formed by 18-Crown-6 and *cis-anti-cis*-Dicyclohexano-18-crown-6 (Host) with 3,4-Diaminofurazane (Guest)

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Abstract. An X-ray–diffraction study is reported for two molecular complexes containing 3,4-diamino-1,2,5-oxadiazole as guest (G) with 18-crown-6 (18-C-6) and *cis-anti-cis*-dicyclohexano-18-crown-6 (DCH-6B) as host. Both complexes are of the polymeric-chain structure with the guest molecule bridging two crown neighbours. Complex **I**: [18-C-6 $*$ G $*$ H_2O], 1 : 1 : 1, monoclinic, $P2_1/n$, $a = 8.171(1)$, $b = 15.042(2)$, $c = 16.209(6)$ Å, $\beta = 101.15(2)°$, final R-factor 0.068. Complex **II**: [DCH-6B $*$ G], 1 : 1, monoclinic $C2/c$, $a = 21.212(4)$, $b = 9.380(2)$, $c = 13.049(3)$ Å, $\beta = 108.61(3)°$, final R 0.047.

Key words: 18-crown-6, diaminofurazane, crystal structure, host–guest complexes.

1. Introduction

Host–guest molecular complexes formed by 18-membered crown ethers (18-crown-6, *cis-syn-cis* and *cis-anti-cis* isomers of dicyclohexano-18-crown-6, DCH-6) are known [1,2] in which small organic molecules serve as the guests, and where their RH_2 (R = N, C) groups play the role of H-donor in hydrogen bonding. The RH_2-group geometry fits topologically to the 18-membered ring cavity with the possibility of complex formation, with a resultant stoichiometry of 1 : 2. Host–guest interactions occur with the macrocycle O-atoms divided by the —$(CH_2)_2$—O—$(CH_2)_2$— moiety. If the guest possesses several donor groups separated from each other, e.g., being in p-positions on an aromatic ring, both of them are able to interact independently and in a similar way according to the scheme described above. A number of reviews and original papers cover the data concerning host–guest complexes

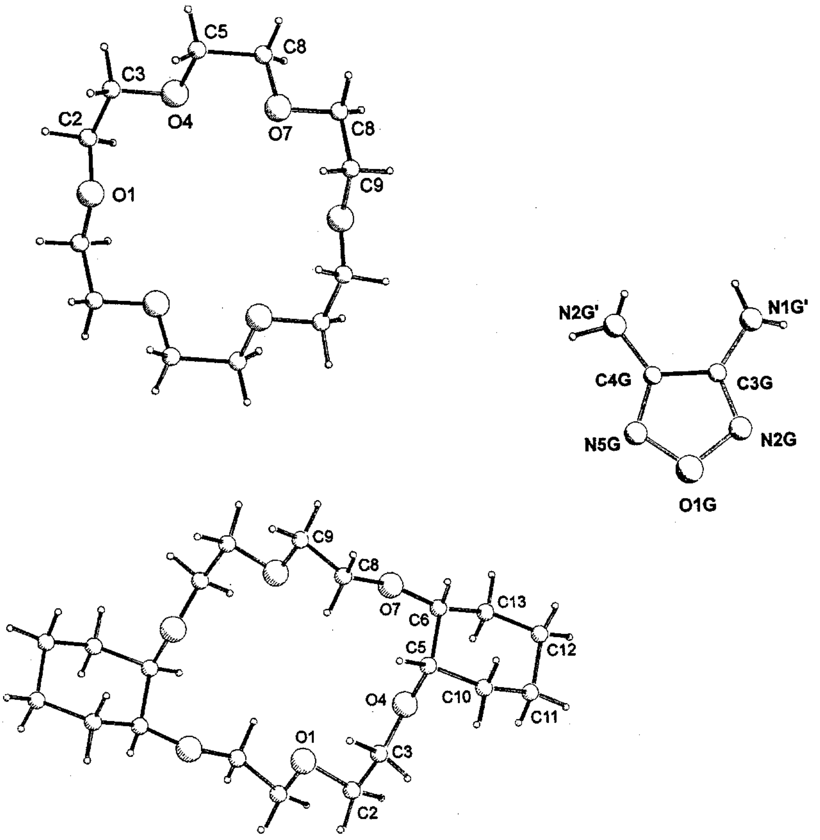

Figure 1. Numbering scheme of two host molecules (compounds **I** and **II**) and the guest molecule present in both complexes.

with guests bearing isolated NH_2 groups [3,4]. Besides, there are many papers in the literature devoted to the mode of interaction with tripod RNH_3^+ or H_3O^+ entities [5,6]. Crystal data concerning binding via other donor groups, e.g., >NH are virtually absent. There are, however, some interesting exceptions. For example, the complexes of 18-crown-6 and *cis-anti-cis*-DCH-6 with 6-chloro-7-sulphamido-3,4-dihydro-1,2,4-benzothiadiazine-1,1-dioxade [7,8] where two guest NH-groups form a rather rigid methylenediimino fragment —NH—CH_2—NH—, stipulate the unusual host-guest complementarity with the participation of both NH groups (18-crown-6) [7] or one only (DCH-6B) [8] in the contacts with the host. It seemed interesting to investigate the mode of complex organization with organic molecules containing H-donor groups in neighbouring positions. A suitable example of such a guest is 3,4-diamino-furazane. This paper presents experimental data on the synthesis and structure of its host : guest complexes with 18-crown-6 (complex **I**) and the *cis-anti-cis* isomer of DCH-6 (complex **II**).

Table I.

Compound	I	II
Formula	$C_{14}H_{30}N_4O_8$	$C_{22}H_{40}N_4O_7$
mol. wt.	382.42	472.58
Space group cell	$P2_1/n$	$C2/c$
a (Å)	8.170(1)	21.212(4)
b (Å)	15.042(2)	9.380(2)
c (Å)	16.209(6)	13.049(3)
β (°)	101.15(2)	108.61(3)
V (Å^3)	1954.4(8)	2460.6(9)
Z	4	4
$F(000)$	824	1024
d_{calc} (g/cm^3)	1.300	1.276
μ (mm^{-1})	0.106	0.095
max 2θ (°)	30.07	34.90
$h_{\min}$ $h_{\max}$	−11 11	−34 34
$k_{\min}$ $k_{\max}$	0 21	0 15
$l_{\min}$ $l_{\max}$	0 22	0 20
Scan mode	$\omega - -2\theta$	$\omega - -2\theta$
Number of refl. measured	5917	5550
Number of unique data	5741	5354
Data/parameters	5717/356	5337/230
R [a]	0.062	0.047
R_W [b]	0.160	0.119
Weighting scheme: a, b [c]	0.1140	0.066 0.690
S	1.027	1.038
$\Delta\rho$ (e Å^{-3})	+0.42 −0.38	+0.39 −0.36

[a] Conventional R based on F ($F_0 > 4\sigma(F_0)$).
[b] R_W based on F^2.
[c] $w = 1/[\sigma^2(F_0^2) + (aP)^2 + bP]$ where $P = (F_0^2 + 2F_c^2)/3$.

2. Synthesis

Both complexes have been obtained in a similar way. The appropriate crown ether (1 mmol) and 1 mmol of 3,4-diamino-furazane were dissolved simultaneously in 10 mL of methanol at 65 °C. The crystals precipitated after cooling to 20 °C were separated and air dried. Crystals suitable for X-ray crystallography were obtained by recrystallization from benzene : methanol (1 : 1) (**I**) or from methanol (**II**).

2.1. COMPLEX I

Colourless, transparent crystals, soluble in methanol, ethanol, acetone, m.p. 81–82 °C. NMR (Bruker AM 250) $(CD_3)_2CO$, d, 3.02 s (4H, H_2O, 3.64 (24H, CH_2O),

Table II. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **I**. $U_{(eq)}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{(eq)}$
O(1a)	4833(3)	1191(2)	6398(1)	31(1)
C(2a)	3177(4)	1251(2)	6539(2)	33(1)
C(3a)	2051(4)	1525(2)	5740(2)	32(1)
O(4a)	1973(3)	814(1)	5151(1)	30(1)
C(5a)	953(4)	1020(2)	4354(2)	34(1)
C(6a)	836(4)	214(2)	3812(2)	31(1)
O(7a)	2397(3)	51(1)	3574(1)	30(1)
C(8a)	2361(4)	−736(2)	3090(2)	33(1)
C(9a)	4030(4)	−886(2)	2873(2)	33(1)
O(1b)	1675(3)	1601(2)	966(1)	32(1)
C(2b)	1404(5)	1549(2)	1808(2)	35(1)
C(3b)	−404(5)	1392(2)	1776(2)	37(1)
O(4b)	−851(3)	503(1)	1512(1)	33(1)
C(5b)	−2599(4)	360(3)	1431(2)	39(1)
C(6b)	−3039(5)	−574(3)	1167(2)	37(1)
O(7b)	−2967(3)	−683(2)	300(1)	34(1)
C(8b)	−3559(4)	−1528(2)	−17(2)	35(1)
C(9b)	−3396(4)	−1592(3)	−924(2)	36(1)
O(1G)	−896(3)	−3156(2)	2255(1)	41(1)
N(2G)	−30(4)	−2496(2)	1902(2)	36(1)
C(3G)	−48(3)	−2752(2)	1132(2)	24(1)
C(4G)	−943(4)	−3576(2)	980(2)	25(1)
N(5G)	−1457(4)	−3822(2)	1657(2)	35(1)
N(1G′)	705(3)	−2305(2)	565(2)	30(1)
N(2G′)	−1304(4)	−4001(2)	224(2)	34(1)
O_W	1735(4)	−466(2)	862(2)	38(1)

5.31 br.s (4H, NH). *Anal. Calc.* (%) C = 41.52, H = 8.60, N = 14.01 for the composition $C_{14}H_{28}N_4O_7 \cdot 2H_2O$. *Found*: C = 41.78, H = 8.52, N = 13.92.

2.2. Complex **II**

Colourless, transparent crystals, soluble in methanol, ethanol, acetone, m.p. 153–154 °C. NMR (Bruker AM 250) $(CD_3)_2CO$, d, 1.29–1.88 m and 3.59–3.72 m (36H, CH, CH_2, CH_2O), 5.29 br.s (4H, NH). *Anal. Calc.* (%) C = 55.91, H = 8.53, N = 11.86 for the composition $C_{22}H_{40}N_4O_7$. *Found*: C = 56.12, H = 8.60, N = 11.95.

Table III. Bond lengths [Å] and angles [deg] for **I**.

O(1a)—C(2a)	1.418(4)	C(2a)—O(1a)—C(9a)#2	112.0(2)
O(1a)—C(9a)#2	1.430(4)	O(1a)—C(2a)—C(3a)	108.9(3)
C(2a)—C(3a)	1.494(5)	O(4a)—C(3a)—C(2a)	108.3(3)
C(3a)—O(4a)	1.428(4)	C(3a)—O(4a)—C(5a)	112.6(2)
O(4a)—C(5a)	1.430(4)	O(4a)—C(5a)—C(6a)	108.4(3)
C(5a)—C(6a)	1.490(5)	O(7a)—C(6a)—C(5a)	109.9(3)
C(6a)—O(7a)	1.423(4)	C(8a)—O(7a)—C(6a)	111.7(2)
O(7a)—C(8a)	1.417(4)	O(7a)—C(8a)—C(9a)	109.6(3)
C(8a)—C(9a)	1.491(5)	O(1a)#2—C(9a)—C(8a)	109.3(2)
C(9a)—O(1a)#2	1.430(4)	C(9b)#1—O(1b)—C(2b)	112.5(2)
O(1b)—C(9b)#1	1.421(4)	O(1b)—C(2b)—C(3b)	108.2(3)
O(1b)—C(2b)	1.427(4)	O(4b)—C(3b)—C(2b)	110.7(3)
C(2b)—C(3b)	1.487(5)	C(5b)—O(4b)-C(3b)	111.2(3)
C(3b)—O(4b)	1.430(4)	O(4b)—C(5b)—C(6b)	110.5(3)
O(4b)—C(5b)	1.425(4)	O(7b)—C(6b)—C(5b)	109.7(3)
C(5b)—C(6b)	1.492(5)	C(8b)—O(7b)—C(6b)	112.5(2)
C(6b)—O(7b)	1.427(4)	O(7b)—C(8b)—C(9b)	108.8(3)
O(7b)—C(8b)	1.421(4)	O(1b)#1—C(9b)—C(8b)	108.8(3)
C(8b)—C(9b)	1.505(5)	N(2G)—O(1G)—N(5G)	110.2(2)
C(9b)—O(1b)#1	1.421(4)	C(3G)—N(2G)—O(1G)	105.5(2)
O(1G)—N(2G)	1.404(4)	N(2G)—C(3G)—N(1G′)	124.8(3)
O(1G)—N(5G)	1.408(3)	N(2G)—C(3G)—C(4G)	109.3(3)
N(2G)—C(3G)	1.305(4)	N(1G′)—C(3G)—C(4G)	125.9(3)
C(3G)—N(1G′)	1.376(4)	N(5G)—C(4G)—N(2G′)	125.1(3)
C(3G)—C(4G)	1.435(4)	N(5G)—C(4G)—C(3G)	109.7(2)
C(4G)—N(5G)	1.303(4)	N(2G′)—C(4G)—C(3G)	125.0(3)
C(4G)—N(2G′)	1.363(4)	C(4G)—N(SG)—O(1G)	105.3(2)

Symmetry transformations used to generate equivalent atoms: #1 $-x, -y, -z$ #2 $-x+1, -y, -z+1$.

3. X-ray Diffraction Measurements

X-ray data for both complexes were collected on a KM4 (KUMA-Diffraction), four-circle diffractometer, graphite monochromated, $\lambda(\mathrm{Mo}K_\alpha) = 0.7108$ Å. Lorentz polarization corrections were made but absorption was ignored. The structures were solved by direct methods (SHELXS-86) and refined by a full-matrix least-squares procedure (SHELXL-93) on F^2. Table I contains full crystallographic data; atomic coordinates, bond lengths and angles are given in Tables II–V, respectively. H atoms for —NH_2 groups were found from ΔF maps, while the positions of those connected to C were put at calculated positions and refined without constraints with isotropic temperature factors.

Table IV. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for **II**. $U_{(eq)}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{(eq)}$
O(1)	4893(1)	−2075(1)	4123(1)	25(1)
C(2)	4292(1)	−2769(2)	3502(1)	28(1)
C(3)	3764(1)	−1668(2)	2998(1)	25(1)
O(4)	3550(1)	−1028(1)	3831(1)	21(1)
C(5)	3081(1)	106(2)	3428(1)	20(1)
C(6)	3050(1)	1001(2)	4400(1)	21(1)
O(7)	3682(1)	1508(1)	5079(1)	23(1)
C(8)	4029(1)	2369(2)	4535(1)	26(1)
C(9)	4606(1)	3100(2)	5359(1)	26(1)
C(10)	2380(1)	−452(2)	2803(1)	26(1)
C(11)	2070(1)	−1294(2)	3528(1)	34(1)
C(12)	2048(1)	−383(2)	4497(1)	34(1)
C(13)	2745(1)	164(2)	5134(1)	27(1)
OG1	5000	4228(2)	2500	36(1)
NG2	4642(1)	3370(2)	1610(1)	31(1)
CG3	4775(1)	2048(2)	1942(1)	22(1)
NG1′	4497(1)	865(2)	1355(1)	31(1)

4. Discussion

Complexes **I** and **II** have a host : guest ratio of 1 : 1. In the both cases guest G carries out the single bridge function and is bonded with the neighbouring macrocyclic moieties by H-bonds (crystallographically independent in **I** or with two equal faces of one crown ether in **II**). This fragment of the polymeric like structures for **I** and **II** are shown in Figures 2a and 3a. The infinite chains in **I** and **II** are rather different. This is due to the water molecule included in **I** where it might be considered as the second guest connected with 18-crown-6 by H-bonds. The numbering scheme of **I** and **II** is shown in Figure 1. The geometric parameters of H-bonds in **I** and **II** are given in Table VI.

4.1. CRYSTAL STRUCTURE OF I

Figure 2a shows the structure unit of the polymeric chain guest-crown in **I**. As mentioned above, G serves as a single bridge in the infinite chains. Its two —NH_2 groups interact in a different manner with two crystallographically independent host molecules. One of them (—N(2)G′) forms two single H-bonds N(2)G′ · · · O(1a)* = 3.109(4) Å and N(2)G′ · · · O(4a) = 3.060(4) Å with Ha(host a) O-atoms divided by the —$(CH_2)_2$—O—$(CH_2)_2$— moiety (Figure 2c). In this fragment the N(2)G′—

Table V. Bond lengths [Å] and angles [deg] for **II**.

O(1)—(2)	1.430(2)	C(2)—O(1)—C(9)#1	110.75(12)
O(1)—C(9)#1	1.432(2)	O(1)—C(2)—C(3)	109.78(13)
C(2)—C(3)	1.511(2)	O(4)—C(3)—C(2)	109.01(13)
C(3)—O(4)	1.437(2)	C(3)—O(4)—C(5)	112.42(11)
O(4)—C(5)	1.437(2)	O(4)—C(5)—C(6)	108.00(11)
C(5)—C(6)	1.539(2)	O(4)—C(5)—C(10)	112.34(12)
C(5)—C(10)	1.542(2)	C(6)—C(5)—C(10)	109.48(12)
C(6)—O(7)	1.432(2)	O(7)—C(6)—C(13)	106.31(11)
C(6)—C(13)	1.531(2)	O(7)—C(6)—C(5)	114.42(11)
O(7)—C(8)	1.427(2)	C(13)—C(6)—C(5)	111.95(12)
C(8)—C(9)	1.512(2)	C(8)—O(7)—C(6)	114.18(11)
C(9)—O(1)#1	1.432(2)	O(7)—C(8)—C(9)	109.38(13)
C(10)—C(11)	1.533(2)	O(1)#1—C(9)—C(8)	109.96(12)
C(11)—C(12)	1.539(2)	C(11)—C(10)—C(5)	112.28(13)
C(12)—C(13)	1.536(2)	C(10)—C(11)—C(12)	110.7(2)
OG1—NG2	1.418(2)	C(13)—C(12)—C(11)	110.67(14)
OG1—NG2#2	1.418(2)	C(6)—C(13)—C(12)	111.02(13)
NG2—CG3	1.315(2)	NG2—OG1—NG2#2	110.9(2)
CG3—NG1′	1.370(2)	CG3—NG2—OG1	105.20(13)
CG3—CG3#2	1.463(3)	NG2—CG3—NG1′	124.78(14)
		NG2—CG3—CG3#2	109.36(9)
		NG1′—CG3—CG3#2	125.71(9)

Symmetry transformations used to generate equivalent atoms: #1 $-x + 1$, $-y$, $-z + 1$, #2 $-x + 1, y, -z + 1/2$.

Table VI. H-bonds in **I** and **II**.

Complex **I**	D—H	D···A	H···A	D—H—A
N1G′—HN11···Ow	0.99(4)	2.905(4)	1.92(4)	175(3)
N1G′—HN12···O1b	0.89(5)	3.032(3)	2.19(5)	155(5)
Ow—H1w···O4b*	0.79(5)	2.924(4)	2.15(5)	166(4)
Ow—H2w···O7b	1.2(1)	2.878(4)	1.7(1)	163(8)
N2G′—HN21···O1a*	0.98(4)	3.109(4)	2.24(4)	148(3)
N2G′—HN22···O4a	0.83(4)	3.060(4)	2.23(4)	173(4)
Complex **II**				
N1G′—HN11···O1	0.89(2)	3.191(2)	2.38(2)	152(2)
N1G′—HN12···O4*	0.83(3)	3.269(2)	2.44(3)	171(2)

*Indicates symmetry equivalent atom from the same host molecule.

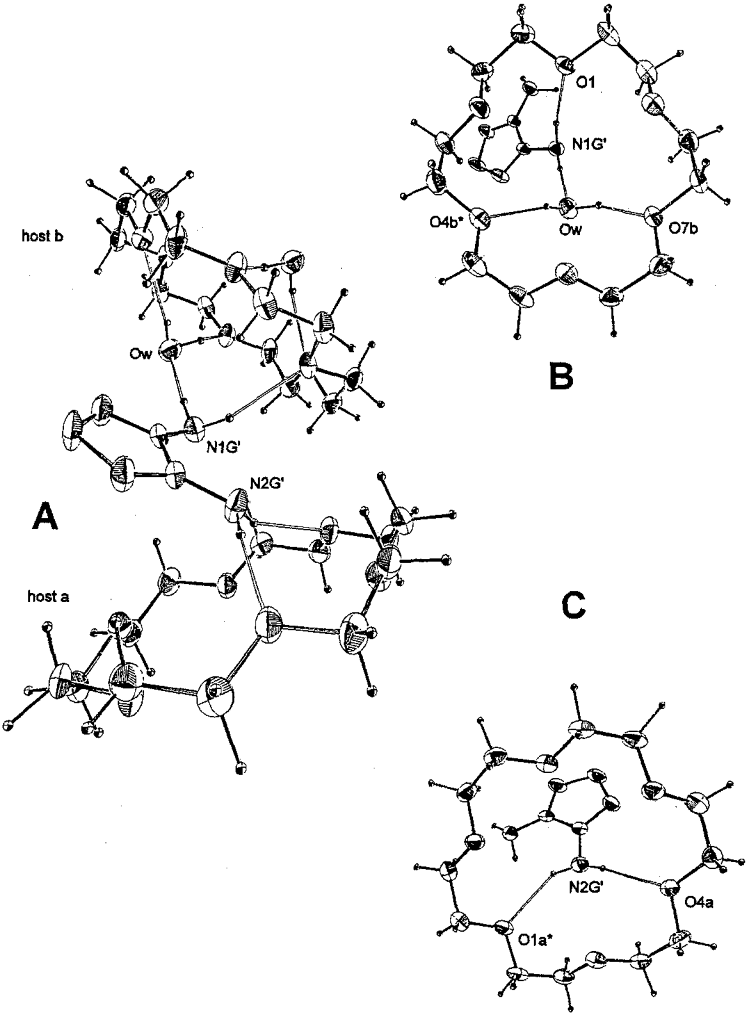

Figure 2. ORTEP drawing of complex **I**: fragment of infinite chain (A), hydrogen bonding scheme in G-Hb (B) and G-Ha (C).

C(4)G bond forms an angle of 135.8(2)° with the average plane of six O-atoms of Ha. N(2)G′ is at a distance of 1.848(3) Å above the same plane. The dihedral angle between the average planes through the guest heterocyclic moiety and host O-atoms is 129.57(8)°. The second NH_2 group uses its two H atoms in a different way (Figure 2b): one for direct contact with the crown ether N(1)G′ · · · O(1b) = 3.032(3) Å; the other as donor to the water molecule lone pair with the N(1)G′ · · · Ow distance equal to 2.905(4) Å. In its turn the water molecule interacts as an H donor with two other host O atoms also separated by one ethylene moiety: Ow· · ·O(4b) = 2.924(4) and Ow· · ·O(7b) = 2.878(4) Å. The water intermediate function is quite essential for the

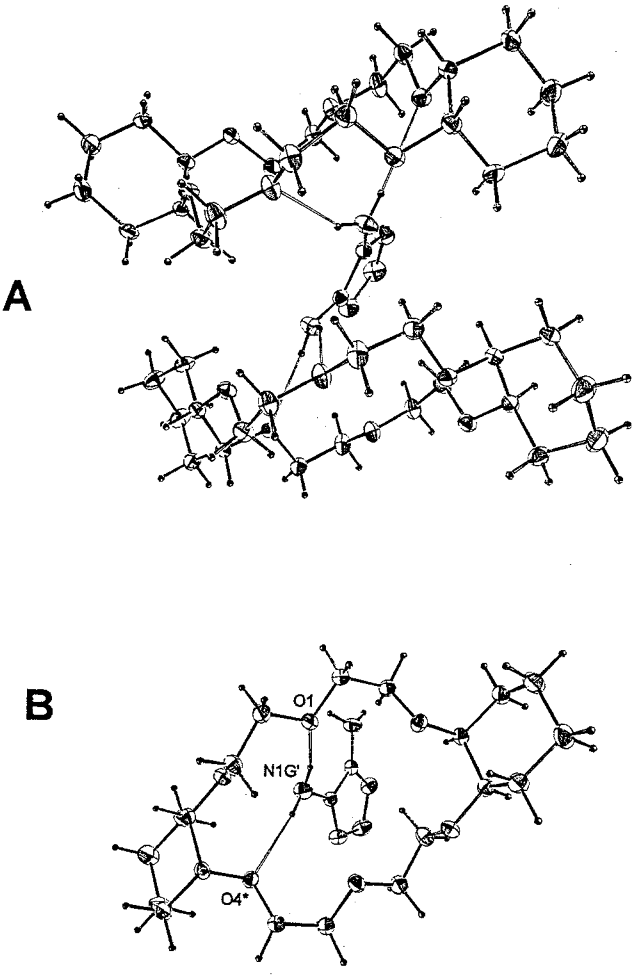

Figure 3. ORTEP drawing of complex **II**: fragment of infinite chain (a) and hydrogen bonding scheme (B).

realization of the optimum system of H-bonds in the supramolecular aggregate. The same water function was found in the complexes [18C6·*trans*-$SiF_4 \cdot H_2O$]·H_2O [9], and [18C6·$SnCl_4$·$2H_2O$]·$2H_2O$ [10]. Thus, the infinite chain in **I** might be drawn schematically as:

=Ha=G— Hb —G=Ha=
\ //\\ /
H_2O H_2O

Here the G=Ha=G entity is stabilized by four, while for

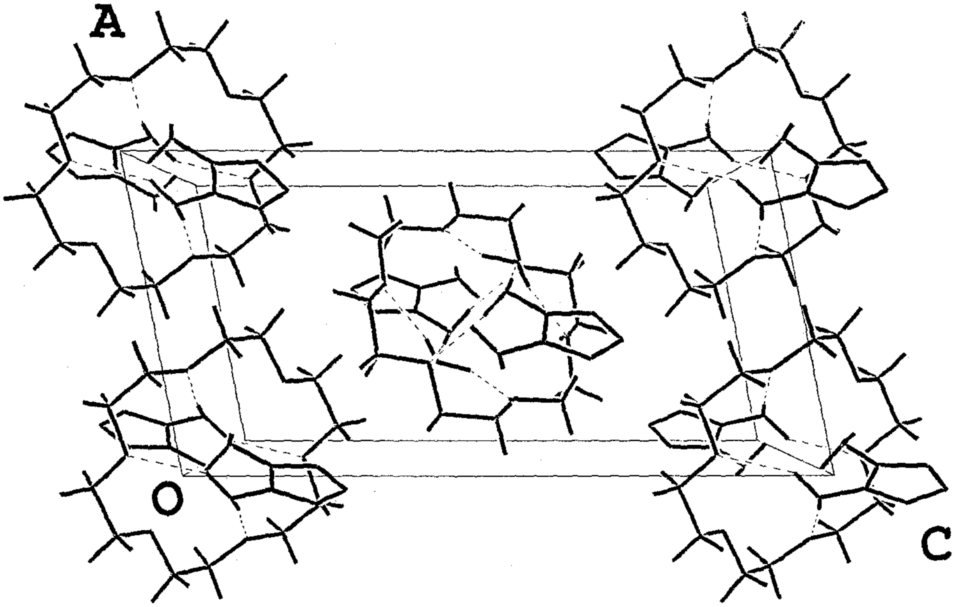

Figure 4. Perspective view of I along *b*.

G—H b—G
\ //\\ /
H_2O H_2O

there are six $R(=O,N)H_{guest}\cdots O_{crown}$ interactions. In this fragment the N(1)G′—C(3)G bond forms an angle of 114.2(1)° with the average plane of the six O atoms of Hb. N(1)G′ is at a distance of 3.026(3) É above the plane. The dihedral angle between the average planes through the guest heterocyclic moiety and host O atoms is 25.57(7)°. Thus, during chain structure formation two distinctive (in the binding manner) coordination 'knots' of the polymeric chain are formed, each of them possessing an inversion center. Describing these coordination centers, we would like to stress, that the structure consists of the 'standard elements' joining in one polymeric chain. Previously such a composition for the similar fragments has not been described. Despite the different way of host coordination, the conformations of the two independent host molecules are rather similar.

4.2. CRYSTAL STRUCTURE OF **II**

The C_2-symmetry unit fragment of complex **II** is shown in Figure 3a. The two fold axis passes through the center of the C=C bond and the O atom of the furazane guest moiety. One centrosymmetric coordination unit of the infinite chain in **II** is similar to that for the host molecule 'a' in **I**. H-bonds are formed with the host oxygen atoms divided by the —$(CH_2)_2$—O—$(CH_2)_2$— fragment: N(1)G′ $\cdots$ O(1)=3.191(2) and

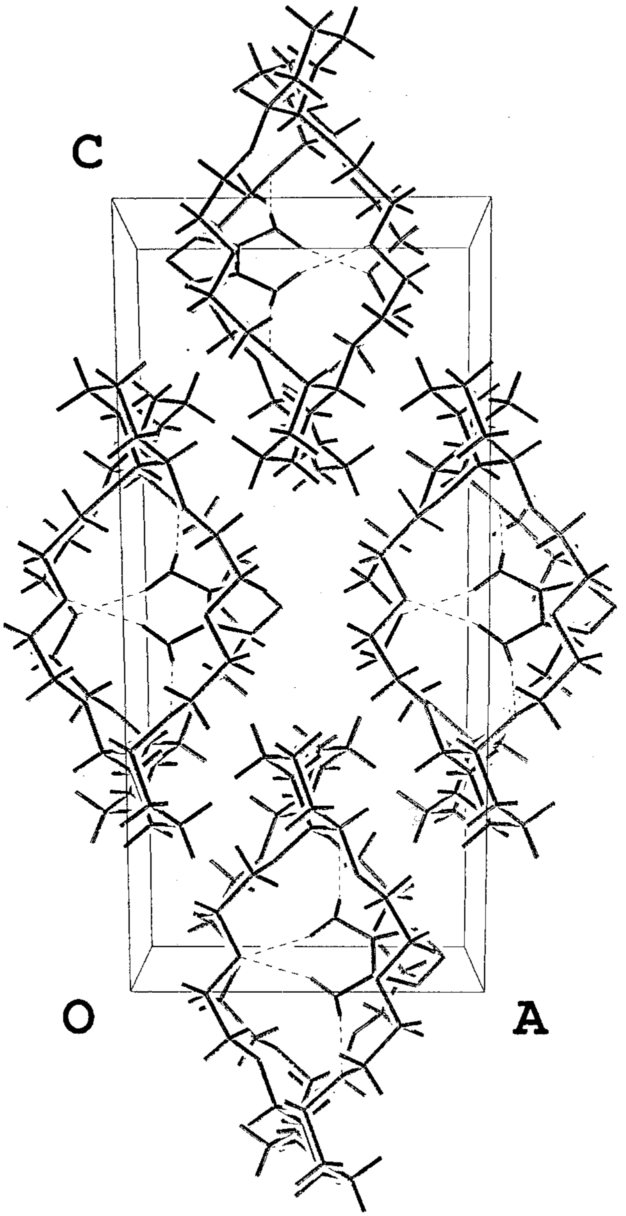

Figure 5. Perspective view of **II** along *b*.

N(1)G′ · · · O(4)*=3.269(2) Å. It is the most common way of coordination for 18-crown-6 and DCH-6B [11, 12]. In this fragment the N(1)G′—C(3)G bond forms an angle of 142.13(9)° with the average plane of six O atoms of the host. N(2)G′ is at a distance of 2.197(2) Å above the plane. The dihedral angle between the average planes through the guest heterocyclic moiety and host O atoms is equal to 120.50(4)°.

4.3. CROWN ETHER CONFORMATION AND GEOMETRY

4.3.1. *18-Crown-6*

Both independent molecules have D_{3d} symmetry. The *gauche* torsion angles are in the range −68 to 74 and 67 to 74°, while *anti* torsion angles fall within the range 173–178 and 169–179° (for Ha and Hb, respectively). The deviations of O atoms from coplanarity for Ha are 0.243(2)–0.256(2) Å and for HB 0.246(2)–0.275(2) Å. The rings are nearly regular hexagons with the distances between the neighbouring O atoms being 2.836(3)–2.929(3) Å for Ha and (2.841(3)–2.953(3) Å for Hb. Transannular O· · ·O distances are 5.522(3)–6.096(3) Å.

4.3.2. *Cis -anti-cis-DCH-6*

In the complexes with neutral organic molecules DCH-6B adopts the C_i symmetry with 8 *gauche* bonds (6 C—C and 2 C—O) and two $g \pm g\pm$ corner fragments along the ring. This arrangement could be different – they could be fixed at the bridge carbon atoms and the ring elongated in the direction of the cyclohexano substituents, with shortened O· · ·O distances between the central oxygen atoms. However, in the DCH-6B complex with 2-$CH_3PhSO_3NH_2$ the corner fragments are in the central part of the heterocyclic framework, the shortened O· · ·O distances being between one pair of cyclohexanediol atoms. In **II** the ring conformation is of the first type: the corner fragments are at the C(6) atom, the shortened transannular O· · ·O distances being between O(1) and O(1)*: 4,465(2) Å; the other O· · ·O distances are O(4)· · ·O(4)* : 6.236(2) Å; O(7)· · ·O(7)* : 6130(2) Å. The host oxygen atoms are coplanar within 0.052(1)–0.075(1) Å.

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