Journal of Inclusion Phenomena and Molecular Recognition in Chemistry **9**: 153–160, 1990.

Crystal Structure of the 1 : 4 Complex between 18-crown-6 and a Polyfunctional Guest: 2-Hydroxymethyl-4-(1,1,3,3-tetramethylbutyl)phenol

M. PERRIN,* E. MAHDAR, S. LECOCQ and C. BAVOUX
Laboratoire de Minéralogie-Cristallographie, Associé au CNRS (UA805), Université Claude Bernard, LYON I, 43 boulevard du 11 Novembre 1918, 69622 Villeurbanne Cedex, France.

(Received 29 March 1989; in final form: 20 June 1989)

Abstract. Single crystal X-ray analysis of the 1 : 4 complex between 18-crown-6 and 2-hydroxymethyl-4-(1,1,3,3-tetramethylbutyl)phenol is reported. Crystals of the complex are triclinic $P\bar{1}$ with $a = 11.929$ (2), $b = 18.655$ (2), $c = 8.313$ (1) Å, $\alpha = 93.14$ (1), $\beta = 94.02$ (1), $\gamma = 100.89$ (1)°, and $D_c = 1.111$ g cm^{-3} for $Z = 1$. The R index is 0.057 for 5935 reflections measured at 293 K. The complex lies on a center of symmetry, the macroring has the C_i symmetry ($g^+g^+aag^-aag^-ag^-g^-aag^+aag^+a$). The CH_2—OH group at the *ortho* position to the phenolic OH lies near the benzene ring plane; this conformation differs from the one found for uncomplexed phenol-alcohol molecules. The packing is characterized by layers of hydrogen bonded entities parallel to *ac*; along *b* the layers are stabilized by van der Waals interactions.

Key words. Crown ether, phenol–alcohol, crystal structure, hydrogen bonding, host–guest complex.

Supplementary Data relating to this article are deposited with the British Library as supplementary Publication No. 82088 (48 pages).

1. Introduction

The crystal structures of several complexes between 18-crown-6 and neutral molecules have been reported during the last few years. Often the guest molecules are complexed via water molecules to increase the possibility of hydrogen bonds [1–4]. In our laboratory, the guest molecules used are substituted phenols [5–8]. We present here a complex with a polyfunctional guest having two OH groups (phenol and alcohol functions) at neighbouring positions: 2-hydroxymethyl-4-(1,1,3,3-tetramethylbutyl)phenol. The structure of the 1 : 4 complex is solved and we describe the conformation of both molecules (host and guest) as well as the different hydrogen bonds found in the crystal.

2. Experimental

Crystals were obtained by dissolution of 18-crown-6 and the named substituted phenol in toluene followed by crystallisation at 8°C. The crystals are needle shaped; one of them about 0.5 × 0.4 × 1.4 mm was sealed in a glass capillary; preliminary unit cell parameters were obtained by Weissenberg photographs; accurate values

---

* Author for correspondence.

Table I. Crystal data

Formula: $C_{12}O_6H_{24}, 4C_{15}O_2H_{24}$.
Lattice type : Triclinic
Space group : $P\bar{1}$
T : 293 K

Cell parameters:	
$a = 11.929(2)$ Å	$\alpha = 93.14(1)°$
$b = 18.655(2)$ Å	$\beta = 94.02(1)°$
$c = 8.313(1)$ Å	$\gamma = 100.89(1)°$

Volume: 1807.8(4) $Å^3$
Density: 1.111 g. cm^{-3}
Z:1
Absorption: $\mu = 5.66\ cm^{-1}$

were determined by least squares refinement of the setting angles of 25 reflections ($17° < \theta < 47°$) using an Enraf-Nonius CAD-4 diffractometer with CuK_α radiation ($\lambda = 1.54178$ Å). Table I gives the crystal data. The ω-2θ scan technique was used to collect intensities of reflections with $\theta < 73°$. One standard reflection (473) was measured every 60 min to monitor the intensity variations and the same at 100 reflection intervals to monitor the orientation of the crystal. Of 7221 unique data, 5998 having $I > 3\sigma(I)$ were used in the structure determination. The intensities were corrected for Lorentz and polarisation effects but not for absorption.

The structure was solved using direct methods (MULTAN 80) [9]. An E-map showed all non-H atoms for both molecules, crown ether and substituted phenol. After isotropic refinement by SHELX 76 [10] anisotropic thermal parameters were calculated and refined; difference maps revealed some hydrogen atoms; the positions of the others were calculated theoretically; during the last cycles, the isotropic U values of hydrogen atoms were refined. The final R with unit weights is 0.057 for 5935 reflections. All atomic scattering factors used were those incorporated in SHELX 76. Goodness of fit = 0.65. The residual electron density in the final Fourier difference map is 0.14 e $Å^{-3}$.

3. Discussion

The final atomic coordinates are tabulated in Table II as well as the equivalent thermal vibrations with the numbering scheme given in Figure 1. Bond distances and angles are given in Tables III and IV for half the crown molecule and the independent phenol alcohol molecules. Characteristic torsion angles are reported in Table V.

The crown molecule lies on the inversion center; the macroring has the $(g^+ g^+ a a g^- a a g^- a g^- g^- a a g^+ a a g^+ a)$ conformation with C_i symmetry. Average C—O distances are 1.425(8) Å; average C—C distances are 1.491(8) Å; average C—O—C angles are 112.5(1)° and average O—C—C angles are 110.4(2)°, all being similar to those found in other 18-crown-6 complexes [1–8].

For phenol, the C—C average distances in the benzene ring are respectively 1.389(12) and 1.390(10) Å for the two independent molecules. The C—O distances are 1.359(3) and 1.358(2) Å for hydroxyl groups and 1.425(3) and 1.428(3) Å for

Table II. Fractional positional parameters and equivalent isotropic thermal parameters with esd's in parentheses for nonhydrogen atoms

Atom	x/a	y/b	z/c	$B(\text{Å}^2)$	Atom	x/a	y/b	z/c	$B(\text{Å}^2)$
O(1)	0.5727(2)	0.42287(9)	0.5331(2)	4.22(4)	C(22)	0.3720(2)	0.4096(1)	0.6818(3)	3.87(5)
O(2)	0.2658(1)	0.39042(9)	0.7522(2)	4.00(4)	C(101)	1.0225(2)	0.3481(1)	0.4860(3)	3.52(5)
O(101)	1.0750(1)	0.40699(9)	0.5847(2)	5.04(4)	C(102)	0.9149(2)	0.3514(1)	0.4095(2)	3.35(5)
O(102)	0.7569(1)	0.41737(9)	0.3641(2)	3.88(4)	C(103)	0.8567(2)	0.2915(1)	0.3134(3)	3.53(5)
O(201)	0.7478(1)	0.45382(9)	1.0431(2)	4.35(4)	C(104)	0.8997(2)	0.2275(1)	0.2871(3)	3.65(5)
O(202)	0.5064(1)	0.38698(9)	1.0696(2)	4.87(4)	C(105)	1.0074(2)	0.2267(1)	0.3625(3)	4.17(6)
O(203)	0.2770(1)	0.41101(9)	1.0871(2)	4.61(4)	C(106)	1.0680(2)	0.2862(1)	0.4597(3)	4.15(6)
C(1)	0.5267(2)	0.3538(1)	0.5686(2)	3.36(5)	C(107)	0.8291(2)	0.1628(1)	0.1806(3)	4.23(5)
C(2)	0.4233(2)	0.3433(1)	0.6424(2)	3.18(4)	C(108)	0.8851(4)	0.0945(2)	0.1830(3)	7.2(1)
C(3)	0.3745(2)	0.2738(1)	0.6783(3)	3.41(5)	C(109)	0.7111(3)	0.1409(2)	0.2524(4)	7.01(9)
C(4)	0.4239(2)	0.2128(1)	0.6468(3)	3.65(5)	C(110)	0.8026(2)	0.1828(1)	0.0079(3)	4.39(6)
C(5)	0.5283(2)	0.2256(1)	0.5777(3)	4.03(5)	C(111)	0.8943(2)	0.2134(2)	−0.1070(3)	4.96(6)
C(6)	0.5788(2)	0.2942(1)	0.5387(3)	3.84(5)	C(112)	0.9757(5)	0.1645(3)	−0.1435(8)	11.9(2)
C(7)	0.3713(2)	0.1357(1)	0.6903(3)	4.40(6)	C(113)	0.8270(4)	0.2190(3)	−0.2661(4)	7.9(1)
C(8)	0.4498(3)	0.1156(2)	0.8284(4)	6.7(1)	C(114)	0.9607(4)	0.2879(2)	−0.0504(5)	9.4(1)
C(9)	0.2518(3)	0.1325(2)	0.7536(5)	6.48(9)	C(122)	0.8686(2)	0.4201(1)	0.4411(4)	4.50(6)
C(10)	0.3670(2)	0.0778(1)	0.5476(4)	5.07(7)	C(201)	0.6739(2)	0.4042(2)	0.9273(3)	4.82(6)
C(11)	0.2914(2)	0.0748(1)	0.3885(4)	5.58(7)	C(202)	0.5926(2)	0.3511(1)	1.0096(3)	4.84(6)
C(12)	0.1650(3)	0.0419(2)	0.4045(6)	8.4(1)	C(203)	0.4190(2)	0.3377(2)	1.1337(3)	5.16(7)
C(13)	0.3346(4)	0.0209(3)	0.2715(6)	9.1(1)	C(204)	0.3395(3)	0.3801(2)	1.2098(3)	5.39(7)
C(14)	0.2981(4)	0.1476(2)	0.3138(5)	7.1(1)	C(205)	0.2020(2)	0.4538(2)	1.1535(3)	5.14(7)
					C(206)	0.1628(2)	0.4994(2)	1.0268(3)	4.86(6)

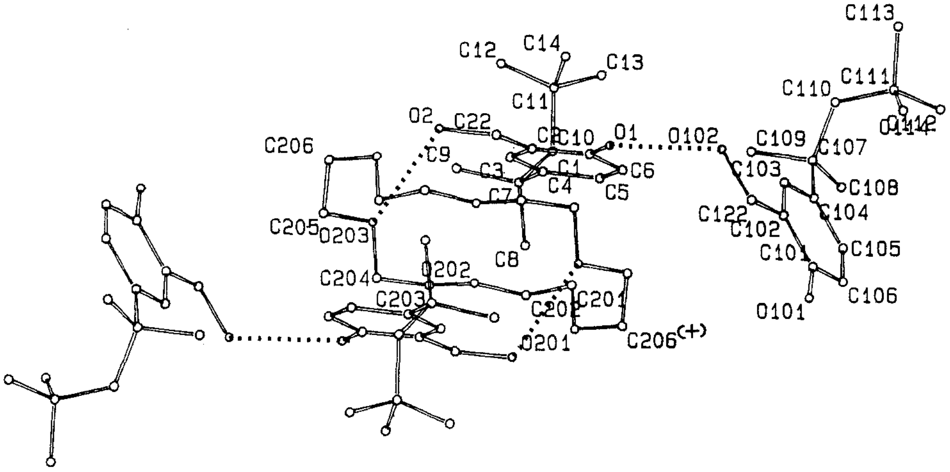

Fig. 1. View of the molecules with the numbering scheme.

alcohol groups. In the 1,1,3,3-tetramethylbutyl chains, the C—C distances lie between 1.487(7) and 1.559(4) Å. The angles in the ring are less than 120° (116.4(2) and 116.5(2)°) at positions *para* to the phenol OH group because of the electron releasing character of the substituent. In the chains we note the large values of C(7)—C(10)—C(11) : 123.8(2)° and C(107)—C(110)—C(111) : 124.7(2)°. Large values are also found for the same substituent in *p*-(1,1,3,3-tetramethylbutyl)phenol

Table III. Bond Distances (Å) with esd's in parentheses

O(1)—C(1)	1.359(3)	O(101)—C(101)	1.358(2)
C(1)—C(2)	1.401(3)	C(101)—C(102)	1.405(3)
C(2)—C(3)	1.375(3)	C(102)—C(103)	1.379(3)
C(3)—C(4)	1.399(3)	C(103)—C(104)	1.397(3)
C(4)—C(5)	1.394(3)	C(104)—C(105)	1.393(3)
C(5)—C(6)	1.374(3)	C(105)—C(106)	1.387(3)
C(1)—C(6)	1.392(3)	C(101)—C(106)	1.379(3)
C(2)—C(22)	1.511(3)	C(102)—C(122)	1.505(4)
C(22)—O(2)	1.425(3)	C(122)—O(102)	1.428(3)
C(4)—C(7)	1.530(3)	C(104)—C(107)	1.532(3)
C(7)—C(8)	1.537(5)	C(107)—C(108)	1.547(5)
C(7)—C(9)	1.545(4)	C(107)—C(109)	1.559(4)
C(7)—C(10)	1.551(4)	C(107)—C(110)	1.529(3)
C(10)—C(11)	1.540(4)	C(110)—C(111)	1.551(4)
C(11)—C(12)	1.535(5)	C(111)—C(112)	1.487(7)
C(11)—C(13)	1.543(6)	C(111)—C(113)	1.517(5)
C(11)—C(14)	1.515(5)	C(111)—C(114)	1.497(5)
O(203)—C(205)	1.426(4)	C(201)—C(202)	1.482(4)
O(203)—C(204)	1.432(3)	O(201)—C(201)	1.427(3)
C(203)—C(204)	1.496(5)	O(201)—C(206)(+)	1.424(3)
O(202)—C(203)	1.410(3)	C(205)—C(206)	1.494(4)
O(202)—C(202)	1.431(3)		

With the symmetry code
(+): $-x+1, -y+1, -z+2$

Table IV. Bond Angles (deg) with esd's in parentheses

O(1)—C(1)—C(2)	117.7(2)	O(101)—C(101)—C(102)	116.8(2)
O(1)—C(1)—C(6)	123.1(2)	O(101)—C(101)—C(106)	123.8(2)
C(2)—C(1)—C(6)	119.2(2)	C(102)—C(101)—C(106)	119.4(2)
C(1)—C(2)—C(3)	118.8(2)	C(101)—C(102)—C(103)	118.6(2)
C(1)—C(2)—C(22)	118.0(2)	C(101)—C(102)—C(122)	117.6(2)
C(3)—C(2)—C(22)	123.1(2)	C(103)—C(102)—C(122)	123.7(2)
C(2)—C(3)—C(4)	123.2(2)	C(102)—C(103)—C(104)	123.2(2)
C(3)—C(4)—C(5)	116.4(2)	C(103)—C(104)—C(105)	116.5(2)
C(3)—C(4)—C(7)	123.3(2)	C(103)—C(104)—C(107)	120.2(2)
C(5)—C(4)—C(7)	120.3(2)	C(105)—C(104)—C(107)	123.3(2)
C(4)—C(5)—C(6)	122.0(2)	C(104)—C(105)—C(106)	121.5(2)
C(1)—C(6)—C(5)	120.4(2)	C(101)—C(106)—C(105)	120.7(2)
C(4)—C(7)—C(8)	107.3(2)	C(104)—C(107)—C(108)	112.0(2)
C(4)—C(7)—C(9)	111.8(2)	C(104)—C(107)—C(109)	107.8(2)
C(4)—C(7)—C(10)	112.1(2)	C(104)—C(107)—C(110)	112.4(2)
C(8)—C(7)—C(9)	107.3(3)	C(108)—C(107)—C(109)	106.3(2)
C(8)—C(7)—C(10)	106.8(2)	C(108)—C(107)—C(110)	111.6(2)
C(9)—C(7)—C(10)	111.2(2)	C(109)—C(107)—C(110)	106.2(2)
C(7)—C(10)—C(11)	123.8(2)	C(107)—C(110)—C(111)	124.7(2)
C(10)—C(11)—C(12)	113.0(3)	C(110)—C(111)—C(112)	114.7(3)
C(10)—C(11)—C(13)	105.2(3)	C(110)—C(111)—C(113)	104.9(2)
C(10)—C(11)—C(14)	114.5(2)	C(110)—C(111)—C(114)	113.2(3)
C(12)—C(11)—C(13)	105.9(3)	C(112)—C(111)—C(113)	106.0(3)
C(12)—C(11)—C(14)	108.5(3)	C(112)—C(111)—C(114)	109.2(3)
C(13)—C(11)—C(14)	109.2(3)	C(113)—C(111)—C(114)	108.4(3)
C(2)—C(22)—O(2)	111.8(2)	C(102)—C(122)—O(102)	113.8(2)
C(201)—O(201)—C(206)(+)	113.3(2)	O(202)—C(203)—C(204)	108.8(2)
C(202)—O(202)—C(203)	112.0(2)	O(203)—C(204)—C(203)	109.7(2)
C(204)—O(203)—C(205)	112.1(2)	O(203)—C(205)—C(206)	109.3(2)
O(201)—C(201)—C(202)	110.5(2)	O(201)(+)—C(206)—C(205)	115.0(2)
O(202)—C(202)—C(201)	108.9(2)		

With the symmetry code
(+): $-x+1,\ -y+1,\ -z+2$

Table V. Torsion angles (°)

O(201)—C(201)—C(202)—O(202)	74.9(3)
C(201)—C(202)—O(202)—C(203)	173.5(2)
C(202)—O(202)—C(203)—C(204)	173.9(2)
O(202)—C(203)—C(204)—O(203)	68.9(3)
C(203)—C(204)—O(203)—C(205)	−178.4(2)
C(204)—O(203)—C(205)—C(206)	165.5(2)
O(203)—C(205)—C(206)—O(201)(+)	−56.7(3)
C(205)—C(206)—O(201)(+)—C(201)(+)	−57.6(3)
C(206)—O(201)(+)—C(201)(+)—C(202)(+)	−172.7(2)
C(1)—C(2)—C(22)—O(2)	178.4(2)
C(3)—C(2)—C(22)—O(2)	−2.8(3)
C(4)—C(7)—C(10)—C(11)	−66.8(3)
C(101)—C(102)—C(122)—O(102)	−177.5(2)
C(103)—C(102)—C(122)—O(102)	1.0(4)
C(104)—C(107)—C(110)—C(111)	55.7(3)

With the symmetry code
(+): $-x+1,\ -y+1,\ -z+2$

[11] and the calix [4] arene formed from *p*-(1,1,3,3-tetramethylbutyl) phenol and its 1 : 1 complex with toluene [12]. The external ring angles around C—OH phenol bonds deviate significantly from 120°: 123.1(2) and 117.7(2)° around C(1), 123.8(2) and 116.8 (2)° around C(101); for both molecules the H atom is situated on the side of the C—O bond where the angle has its larger value. This effect has been often observed and explained [13]. The values of the torsion angles for C(103)—C(102)—C(122)—O(102) and C(3)—C(2)—C(22)—O(2) are respectively +1.0 (4)° and −2.8(3)° showing that the —CH_2—OH groups are near the ring plane. This conformation is different compared to the one found in some uncomplexed phenol alcohol molecules. Indeed the —CH_2—OH group at the *ortho* position is clearly out of the ring plane for monohydroxymethyl derivatives [14]; for

Table VI. Least square planes and atom deviations (Å × 10^3) from the planes

Plane I/Crown-ether mean plane:
$+0.1229X + 0.2579Y + 0.9583Z = +10.7095$

O(201)*	−0.374(2)	O(101)	2.880(2)
C(201)*	0.840(3)	O(102)(×)	2.703(2)
C(202)*	0.553(3)	O(2)	2.2847(2)
O(202)*	0.063(2)	O(1)	3.946(2)
C(203)*	−0.092(3)	with the symmetry code	
C(204)*	−0.744(3)	(×): $x, y, z+1$	
O(203)*	0.161(2)		
C(205)*	−0.425(3)		
C(206)*	0.413(3)		

Plane II/Phenol-alcohol molecule 1:
$0.4257X + 0.4342Y - 0.7939Z = 4.0147$

C(101)*	−0.009(2)	C(122)	−0.009(3)
C(102)*	0.006(2)	O(101)	−0.061(2)
C(103)*	0.002(2)	O(102)	−0.050(2)
C(104)*	−0.006(2)	C(107)	−0.030(3)
C(105)*	0.002(3)		
C(106)*	0.008(3)		

Plane III/Phenol-alcohol molecule 2:
$0.4105X + 0.1013Y + 0.9062Z = 6.8288$

C(1)*	0.012(2)	C(22)	−0.069(3)
C(2)*	−0.009(2)	O(1)	0.014(2)
C(3)*	0.000(2)	O(2)	−0.048(2)
C(4)*	0.010(2)	C(7)	−0.015(3)
C(5)*	−0.010(2)		
C(6)*	−0.004(2)		

Plane I–Plane II	53.38(7)°
Plane I–Plane III	19.09(6)°
Plane II–Plane III	59.95(7)°

* atoms defining the plane

dihydroxymethyl derivatives, one is in the plane, the other out of the plane [15, 16].

In the complex, the oxygen atoms O(1), O(2), O(101) and O(102) lie 3.946(2), 2.847(2), 2.880(2) and 2.703(2) Å from the mean plane of the crown. Mean planes equations were calculated (Table VI); from the results it is shown that the benzene ring plane of the phenol molecule C(1)—C(2)—C(3)—C(4)—C(5)—C(6) is almost parallel to the mean plane of 18-crown-6; it is different for the second molecule with the C(101)—C(102)—C(103)—C(104)—C(105)—C(106) benzene ring plane making an angle of 53.38(7)° with the crown ether mean plane. The two vectors O(1)—C(7) and O(101)—C(107) form angles of 90.31(5)° and 142.47(7)° with the normal of the crown mean plane.

18-Crown-6 molecules and phenol-alcohol molecules are linked by hydrogen bonds; their geometry is given in Table VII. O(1)···O(102) and O(101)···O(2) bonds link phenol alcohol molecules in a chain formed alternatively by the two independent phenol alcohol molecules; in those hydrogen bonds, hydroxyl groups are the donors and alcohol groups the acceptors. Oxygen atoms O(2) and O(102), are donors to oxygen atoms O(203) and O(201) of two crown ether molecules related by a *c* translation. So one macrocycle is connected via four H-bonds with four phenol alcohol molecules, two of them above the mean plane, the two other under the mean plane as seen in Figure 2. Their O···O distances vary from 2.662(2) to 2.789(2) Å. The centrosymmetric 18-crown-6 molecules connect two symmetry related sheets of phenolic molecules leading to layers perpendicular to the *b* axis. Consequently, in crystals there are layers of molecules bound in the *a* and *c* directions; these layers are repeated along the *b* axis with van der Waals interactions between the (1,1,3,3-tetramethylbutyl) groups. Figure 2 shows the crystal packing viewed along the [100] direction.

We observe that no water molecule is involved in the interaction of the guest with 18-crown-6 as in other similar phenolic complexes found in the literature. Herein, as in the DyCl$_3$(triethylene glycol) complex with 18-crown-6 and in similar isostructural complexes [17, 18], the alcohol OH group itself is connected to the 18-crown-6.

Table VII. Hydrogen bonds (Å and °)

O(1)—H(1)	O(1)···O(102)$^{\mathrm{I}}$	H(1)···O(102)	O(1)—H(1)···O(102)$^{\mathrm{I}}$
0.67(3)	2.704(3)	2.08(3)	155(4)
O(101)—H(11)	O(101)···O(2)$^{\mathrm{II}}$	H(11)···O(2)	O(101)—H(11)···O(2)$^{\mathrm{II}}$
0.70(3)	2.662(2)	1.99(3)	163(4)
O(2)—H(2)	O(2)···O(203)$^{\mathrm{I}}$	H(2)···O(203)	O(2)—H(2)···O(203)$^{\mathrm{I}}$
0.81(3)	2.780(2)	1.99(3)	166(3)
O(102)—H(22)	O(102)···O(201)$^{\mathrm{III}}$	H(22)···O(201)	O(102)—H(22)···O(201)$^{\mathrm{III}}$
0.82(2)	2.789(2)	1.98(2)	170(2)

with the following symmetry codes:
$^{\mathrm{I}}$ x, y, z
$^{\mathrm{II}}$ $x + 1, y, z$
$^{\mathrm{III}}$ $x, y, z - 1$

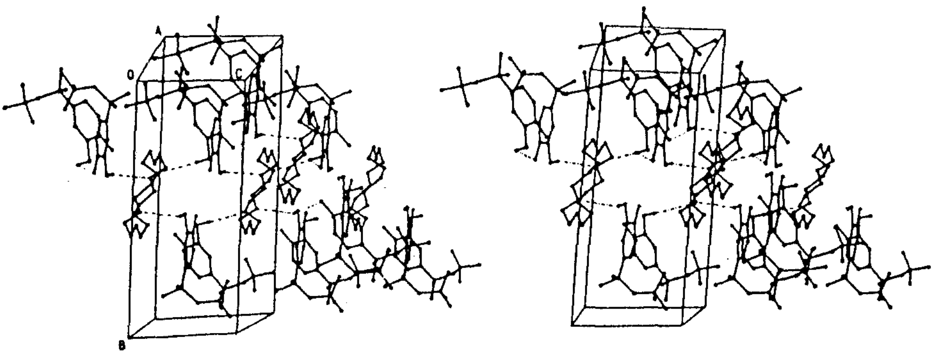

Fig. 2. Stereoscopic view of the crystal structure with the bonding scheme.

The resulting hydrogen bonds have average values (O···O distances 2.780(2) and 2.789(2) Å) in the title compound; they are very strong in the compounds MCl_3(triethylene glycol) (*ca* 2.66 Å for O···O distances), and very weak in most compounds with phenolic guest and participation of water molecules (distances vary between 2.80 and 3.00 Å for the oxygens of the water molecules and the oxygens of the 18-crown-6).

In conclusion, it is important to remark that this complex is the first between a 18-crown-6 molecule and a precursor of phenolic resins. It is interesting to see that the complex is built without water molecules. This may be due to the presence of alcohol OH groups that determine the interactions between host and guest molecules.

References

1. A. Elbasyouny, H. J. Brügge, K. Von Deuten, M. Dickel, A. Knöchel, K. U. Koch, J. Kopf, D. Melzer and G. Rudolph: *J. Am. Chem. Soc.* **105**, 65 (1983).
2. E. H. Nordlander and J. H. Burns: *Org. Chim. Acta.* **115**, 31 (1986).
3. W. H. Watson, J. Galloy, D. A. Grossie, F. Vögtle and W. M. Müller: *J. Org. Chem.* **49**, 347 (1984).
4. M. R. Caira, W. H. Watson, F. Vögtle and W. M. Müller: *Acta Crystallogr.* **C40**, 491 (1984).
5. B. Belamri, C. Bavoux and M. Perrin: *J. Cryst. Spectrosc. Res.* **17**, 177 (1987).
6. Belamri and C. Bavoux: *Acta Crystallogr.* **C44**, 2173 (1988).
7. C. Bavoux: Thesis, Lyon (1986).
8. B. Belamri, C. Bavoux and A. Thozet: *J. Incl. Phenom.* **8**, 383 (1990).
9. P. Main, S. J. Fiske, S. E. Hull, L. Lessinger, G. Germain, J. P. Declercq and M. M. Woolfson: *MULTAN 80.* A System of Computer Programs for the Automatic Solution of Crystal Structures from X-Ray Diffraction Data. Univ. of York, England and Univ. of Louvain, Belgium (1980).
10. G. Sheldrick: *SHELX 76*, Program for Crystal Structure Determination. Univ. of Cambridge (1976).
11. E. Mahdar, M. Perrin and S. Lecocq: *Acta Crystallogr.* (to be published).
12. G. R. Andreetti, A. Pochini and R. Ungaro: *J. Chem. Soc. Perkin Trans. 2*, 1773 (1989).
13. F. L. Hirshfeld: *Isr. J. Chem.* **2**, 87 (1964).
14. M. Halit, D. Oehler, M. Perrin, J. J. Grabowski and A. Stepien: *J. Cryst. Spectrosc. Res.* **17**, 597 (1987).
15. D. Oehler, A. Thozet and M. Perrin: *Acta Crystallogr.* **C41**, 1766 (1985).
16. M. Perrin and M. Cherared: *Acta Crystallogr.* **C42**, 1623 (1986).
17. R. D. Rogers and R. D. Etzenhouser: *Acta Crystallogr.* **C44**, 1533 (1988).
18. R. D. Rogers, E. J. Voss and R. D. Etzenhouser: *Inorg Chem.* **27**, 533 (1988).