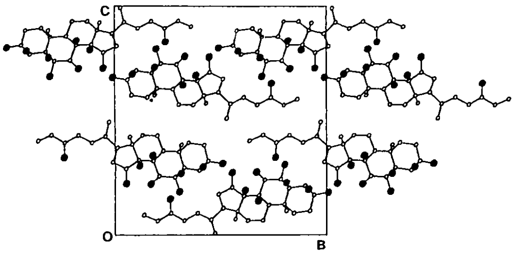

Fig. 2. Crystal packing along the **a** direction; O atoms are represented as filled circles.

Table 3. *Geometry of shortest distances involving hydroxyl groups*

D–H···*A*	*D*···*A* (Å)	H···*A* (Å)	*D*–H···*A* (°)
O(3)–H···O(15^{i})	3·117 (6)	2·32	135
O(4)–H···O(15^{ii})	2·945 (4)	2·01	155
O(5)–H···O(4^{iii})	3·662 (5)	2·72	159
O(6)–H···O(24^{ii})	2·862 (5)	2·02	140
O(7)–H···O(24^{ii})	2·967 (5)	1·98	171
O(8)–H···O(14^{iv})	2·963 (4)	2·10	144
O(14)–H···O(3^{v})	3·533 (5)	2·58	160
O(15)–H···O(7)	2·688 (4)	1·88	135
O(15)–H···O(3^{vi})	3·215 (6)	2·77	108
O(24)–H···O(5^{v})	2·800 (4)	1·91	146
O(24)–H···O(6^{v})	3·386 (5)	2·58	138

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $-1+x, y, z$; (iv) $1+x, y, z$; (v) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (vi) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

packing shown in Fig. 2 is a consequence of these amphiphilic properties. The four axial hydroxyl groups connect molecules translated along the *a* axis by means of an H bond between O(8) and O(14) and a much weaker interaction between O(4) and O(5) (see Table 3): the remaining hydroxyl groups hold together molecules related by the screw axis parallel to **b**. Altogether the hydroxyl groups form an intricate extensive network of hydrogen bonds which link molecules in double layers parallel to the *ab* plane. The double layers are related by the screw axis parallel to **a** and interact through their hydrophobic surfaces.

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Structure of a 1:2:2 Complex Between 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6), 2-Isopropyl-3-methylphenol and Water

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Abstract. $C_{12}H_{24}O_6.2C_{10}H_{14}O.2H_2O$, $M_r = 600{\cdot}8$, monoclinic, $P2_1/c$, $a = 14{\cdot}977$ (2), $b = 8{\cdot}351$ (1), $c = 13{\cdot}946$ (2) Å, $\beta = 91{\cdot}55$ (1)°, $V = 1743{\cdot}6$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}145$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\,K\alpha) = 6{\cdot}06$ cm^{-1}, $F(000) = 656$, $T = 293$ K, $R = 0{\cdot}067$ for 3053 unique observed reflections. A crown ether molecule on an inversion centre accepts a hydrogen bond from each of two inversion-related water molecules that accept a hydrogen bond from two inversion-related phenolic molecules. Binding between host and guest is thus effected by bridging water molecules.

Introduction. This paper is part of our investigation on the ability of the phenols to complex with 18-crown-6. X-ray analyses of complexes with 2,3-

dichlorophenol (Belamri, Bavoux & Perrin, 1987) and 3,5-dichlorophenol (Bavoux, 1986) have shown the role played by a water molecule in stabilizing the compounds: both structures consist of one phenol molecule linked to one crown ether molecule *via* a water molecule. The subject of the present study is a complex including a phenol with alkyl substituents.

Experimental. A mixture of crown ether and phenol was dissolved in toluene. The solution was allowed to stand at room temperature for some hours; the resulting crystals are elongated, pseudohexagonal prisms. Suitable crystal for the X-ray experiment 0·3 × 0·2 × 0·2 mm cut from a long crystal and sealed in a capillary tube. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$, lattice parameters from least-squares fit with 25 reflections $16 < \theta < 45°$, $\omega/2\theta$ scan. Two standard reflections were measured: one every 60 min to control the intensity and the other every 100 reflections to control the alignment of the crystal. 7400 reflections measured: $1 \leq \theta \leq 73°$, *hkl* range: h −27→27, k −12→12, l 0→7.

After averaging ($R_{\rm int} = 0{\cdot}0366$): 3401 unique reflections, 3053 observed reflections with $I > 3\sigma(I)$. Structure solved by direct methods using *MULTAN*80 (Main *et al.*, 1980), refinement on F by full-matrix least squares using *SHELX*76 (Sheldrick, 1976). H atoms located in difference Fourier map. 302 independent parameters. Non-H atoms refined anisotropically. H atoms with isotropic thermal parameters. Residual electron density in difference map within −0·08 and +0·11 e Å^{-3}; $(\Delta/\sigma)_{\rm average} = 0{\cdot}08$; $(\Delta/\sigma) = 1{\cdot}26$ (y coordinate of the water oxygen atom) and $(\Delta/\sigma)_{\rm max} = 1{\cdot}32$ (U_{33} of the same atom), that may be due to the high thermal parameter of this atom. Unit weights used, $R = 0{\cdot}067$. Goodness of fit = 0·63. Atomic scattering factors from *SHELX*76.

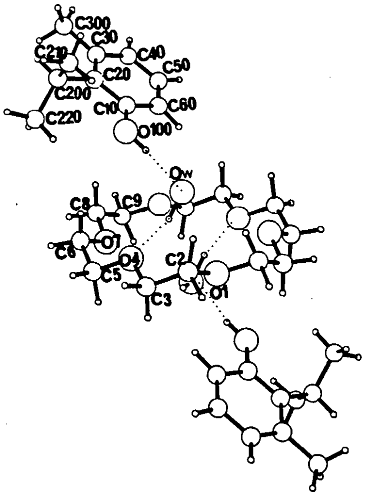

Fig. 1. Structure and numbering scheme of a cluster.

Table 1. *Positional parameters* (×10^4) *and equivalent isotropic temperature factors for non*-H *atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
O(100)	2728 (1)	2452 (2)	746 (1)	5·50 (5)
C(10)	2067 (1)	1460 (3)	1064 (2)	4·34 (6)
C(20)	1183 (1)	1928 (3)	915 (1)	3·99 (5)
C(30)	513 (1)	922 (3)	1253 (1)	4·32 (6)
C(40)	745 (2)	−506 (3)	1710 (2)	5·14 (7)
C(50)	1618 (2)	−945 (3)	1839 (2)	5·58 (8)
C(60)	2289 (2)	27 (3)	1517 (2)	5·10 (6)
C(200)	951 (2)	3506 (3)	419 (2)	4·65 (6)
C(210)	1282 (2)	3597 (3)	−610 (2)	5·88 (9)
C(220)	1261 (2)	4943 (4)	1009 (3)	6·23 (9)
C(300)	−469 (2)	1310 (4)	1123 (2)	5·51 (8)
O(1)	3822 (1)	394 (2)	−1556 (1)	6·11 (6)
C(2)	3715 (3)	2030 (4)	−1797 (2)	7·28 (11)
C(3)	4585 (3)	2778 (5)	−1906 (2)	7·69 (11)
O(4)	5011 (1)	2953 (2)	−981 (1)	6·61 (6)
C(5)	5842 (2)	3716 (4)	−1010 (3)	7·40 (10)
C(6)	6247 (2)	3759 (4)	−47 (3)	7·25 (11)
O(7)	6439 (1)	2167 (2)	247 (1)	6·62 (6)
C(8)	6855 (2)	2069 (5)	1165 (2)	7·43 (11)
C(9)	7003 (2)	356 (5)	1405 (2)	6·90 (10)
O(water)	4405 (2)	1251 (4)	747 (2)	8·71 (8)

Table 2. *Interatomic distances* (Å), *angles* (°) *and torsion angles* (°) *involving non*-H *atoms with e.s.d.'s in parentheses*

C(10)–C(20)	1·392 (3)	O(1)–C(2)	1·414 (4)
C(20)–C(30)	1·400 (3)	C(2)–C(3)	1·456 (5)
C(30)–C(40)	1·391 (3)	C(3)–O(4)	1·431 (4)
C(40)–C(50)	1·366 (4)	O(4)–C(5)	1·400 (4)
C(50)–C(60)	1·376 (4)	C(5)–C(6)	1·459 (5)
C(10)–O(100)	1·374 (3)	C(6)–O(7)	1·418 (4)
C(60)–C(10)	1·389 (3)	O(7)–C(8)	1·412 (4)
C(20)–C(200)	1·524 (3)	C(8)–C(9)	1·485 (5)
C(200)–C(210)	1·533 (4)	C(9)–O(1$^{\rm i}$)	1·406 (4)
C(200)–C(220)	1·520 (4)		
C(30)–C(300)	1·512 (3)		

C(20)–C(10)–O(100)	118·3 (2)	O(1)–C(2)–C(3)	110·1 (3)
C(60)–C(10)–O(100)	120·1 (2)	C(2)–C(3)–O(4)	109·1 (3)
C(60)–C(10)–C(20)	121·6 (2)	C(3)–O(4)–C(5)	113·3 (3)
C(10)–C(20)–C(30)	117·9 (2)	O(4)–C(5)–C(6)	109·4 (3)
C(10)–C(20)–C(200)	121·0 (2)	C(5)–C(6)–O(7)	108·6 (3)
C(30)–C(20)–C(200)	121·0 (2)	C(6)–O(7)–C(8)	113·4 (3)
C(20)–C(30)–C(40)	119·8 (2)	O(7)–C(8)–C(9)	108·6 (3)
C(20)–C(30)–C(300)	122·2 (2)	C(8)–C(9)–O(1$^{\rm i}$)	109·6 (3)
C(40)–C(30)–C(300)	118·0 (2)	C(9)–O(1$^{\rm i}$)–C(2$^{\rm i}$)	111·8 (3)
C(30)–C(40)–C(50)	121·1 (2)		
C(40)–C(50)–C(60)	120·2 (2)		
C(50)–C(60)–C(10)	119·3 (2)		
C(20)–C(200)–C(210)	113·1 (2)		
C(20)–C(200)–C(220)	112·0 (2)		
C(210)–C(200)–C(220)	111·4 (2)		

O(1)–C(2)–C(3)–O(4)	−72·1 (4)	C(6)–O(7)–C(8)–C(9)	178·7 (3)
C(2)–C(3)–O(4)–C(5)	−177·8 (3)	O(7)–C(8)–C(9)–O(1$^{\rm i}$)	−69·3 (3)
C(3)–O(4)–C(5)–C(6)	−176·7 (3)	C(8)–C(9)–O(1$^{\rm i}$)–C(2$^{\rm i}$)	−179·3 (3)
O(4)–C(5)–C(6)–O(7)	65·6 (3)	C(9)–O(1$^{\rm i}$)–C(2$^{\rm i}$)–C(3$^{\rm i}$)	−177·5 (3)
C(5)–C(6)–O(7)–C(8)	178·5 (3)		

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

Discussion. The structure and the numbering scheme used are shown in Fig. 1. Positional parameters and isotropic temperature factors ($B_{\rm eq}$) are given in Table 1, bond distances and angles in Table 2.* The conformation of the 2-isopropyl-3-methylphenol molecule exhibits the same features in this molecular compound as when uncomplexed (Thozet, 1981). Substitution on the phenol molecule produces a deformation of the benzene ring: C(40)–C(50) = 1·366 (4); C(20)–C(30) = 1·400 (3) Å; C(60)–C(10)–C(20) = 121·6 (2); C(10)–C(20)–C(30) = 117·9 (2)°. These values can be compared with those obtained for the uncomplexed molecule: C(40)–C(50) = 1·368 (4); C(20)–C(30) = 1·416 (3) Å; C(60)–C(10)–C(20) = 123·3 (2); C(10)–C(20)–C(30) = 116·8 (5)°. They are connected with the nature and the position of the substituents on the benzene ring (Domenicano, Vaciago & Coulson, 1975). The phenol ring is planar ($\chi^2 = 15$). The angle between the mean plane of the ring and the plane defined by C(200)–C(210)–C(220) is 88·8 (2)°. C(210) and C(220) carbon atoms are nearly symmetrically situated with respect to the plane of the phenol ring [+1·215 (3) and −1·305 (3) Å respectively] and are oriented in the hydroxyl direction. A similar disposition was found in the uncomplexed substance. The C(10)–O(100) distance [1·374 (3) Å] differs notably from the one found in the uncomplexed molecule [1·403 (3) Å]. It can be interpreted as due to the environment of the H atom and the strength of the hydrogen bond in the two compounds. The values of distances and angles in the crown ether molecule are similar to those commonly observed: average values for C–C = 1·47 (2); C–O = 1·41 (2) Å; O–C–C = 109 (1); C–O–C = 113 (1)°. Examination of torsion angles (Table 2) shows that the molecule exhibits the approximate D_{3d} symmetry often found in complexes instead of the elongated shape it has when it is uncomplexed.

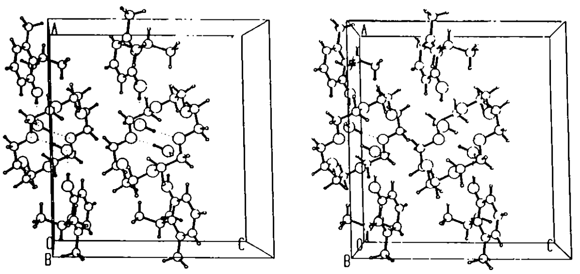

Fig. 2. Stereoscopic view of the structure drawn by *PLUTO* (Motherwell & Clegg, 1978).

The structure consists of clusters: the crown ether molecule is situated on an inversion centre; two inversion-related water molecules are linked with the crown ether molecule *via* H bonds between oxygen of the host molecule and a hydrogen of H_2O. These H bonds are rather long and bent [O–O: 2·954 (3) Å; O–H···O = 147 (4)°]. Each water molecule accepts a hydrogen bond from the hydroxyl group of a phenolic molecule. The distance O···O = 2·704 (3) Å and angle O–H···O = 173 (2)° describe the geometry of these H bonds between the guest and H_2O. Binding between host and guest is thus effected by a bridging water molecule. This arrangement was found in other complexes with phenol: 2,3-dichlorophenol, 3,5-dichlorophenol, 2,4-dinitrophenol (Caira, Watson, Vögtle & Müller, 1984); 3-nitrophenol (Watson, Galloy, Grossie, Vögtle & Müller, 1984). The $-CH_3$ and $-CH_2$ groups are not involved in the bond system as they are in many other complexes between crown ether and small neutral molecules: *N,N'*-dimethylthiourea (Weber, 1983), adiponitrile (Elbassyouny *et al.*, 1983), dimethyl acetylenedicarboxylate (Goldberg, 1975), bis(dimethyl sulfone) (Bandy & Truter, 1981), nitromethane (de Boer, Reinhouldt, Harkema, van Hummel & de Jong, 1982). The packing of the molecules is shown in Fig. 2. Contact distances between two clusters correspond to van der Waals interactions.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51271 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.