$wR = 0.039$
$S = 2.59$
899 reflections
90 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} < 0.0001$

Extinction correction: Zachariasen (1967) type II, Gaussian isotropic
Extinction coefficient: 84.58906
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N(1)	0.1947 (2)	0.5781 (3)	−0.05932 (10)	0.0521 (4)
N(2)	0.3630 (2)	0.2739 (3)	0.0370 (1)	0.0522 (4)
N(3)	0.0368 (2)	0.9761 (3)	0.1368 (1)	0.0664 (5)
N(4)	0.3822 (2)	0.4032 (3)	0.31350 (10)	0.0651 (5)
C(1)	0.2642 (2)	0.4808 (3)	0.03629 (10)	0.0402 (4)
C(2)	0.2355 (2)	0.5932 (3)	0.1342 (1)	0.0425 (4)
C(3)	0.1264 (2)	0.8045 (3)	0.1359 (1)	0.0466 (4)
C(4)	0.3151 (2)	0.4892 (3)	0.2333 (1)	0.0478 (4)

Table 2. *Selected geometric parameters (Å, °)*

C(1)—C(2)	1.413 (2)	N(4)—C(4)	1.148 (2)
N(1)—C(1)	1.331 (2)	N(1)—H(1)	0.90 (2)
N(2)—C(1)	1.330 (2)	N(1)—H(2)	0.92 (2)
C(2)—C(3)	1.407 (2)	N(2)—H(3)	0.89 (2)
C(2)—C(4)	1.404 (2)	N(2)—H(4)	0.96 (2)
N(3)—C(3)	1.148 (2)		
N(1)—C(1)—N(2)	117.8 (1)	C(1)—C(2)—C(4)	119.6 (1)
N(1)—C(1)—C(2)	121.5 (1)	C(3)—C(2)—C(4)	118.5 (1)
N(2)—C(1)—C(2)	120.7 (1)	N(3)—C(3)—C(2)	179.6 (1)
C(1)—C(2)—C(3)	121.9 (1)	N(4)—C(4)—C(2)	178.9 (1)
N(1)—C(1)—C(2)—C(3)	3.2 (2)	N(2)—C(1)—C(2)—C(3)	−177.2 (1)
N(1)—C(1)—C(2)—C(4)	−178.1 (1)	N(2)—C(1)—C(2)—C(4)	1.5 (2)

The scan width was $(1.26 + 0.35\tan\theta)^\circ$ with an ω scan rate of 8.0° min^{-1}. The weak reflections $[I < 2.5\sigma(I)]$ were rescanned a maximum of four times and the counts accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1153). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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β-Cyclodextrin (Z)-9-Dodecen-1-ol 2:1 Complex

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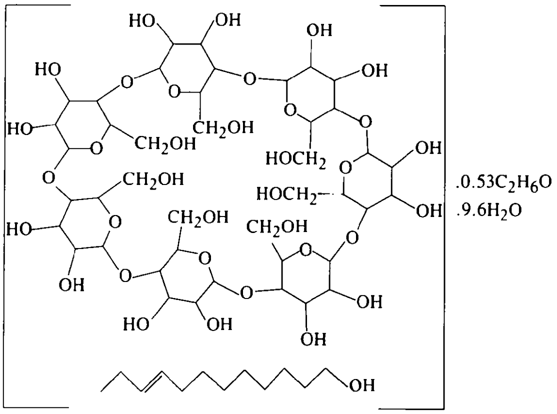
Abstract

The structure of the title complex, cycloheptaamylose–(*Z*)-9-dodecen-1-ol–ethanol–water (1/0.5/0.53/9.6), $C_{42}H_{70}O_{35}.0.5C_{12}H_{24}O.9.6H_2O.0.53C_2H_6O$, has been determined at 150 K. Two cycloheptaamylose (β-cyclodextrin, β-CD) monomers related by a twofold axis form dimers. The stoichiometry of host:guest is 2:1. Therefore, one guest molecule per dimer is enclosed, disordered over two sites related by the above-mentioned twofold axis. The hydrophobic end of the guest lies in the dimeric interface. The rest of the molecular chain transverses the hydrophobic cavity of either monomer and the extreme hydroxy group is found in the primary hydroxy region.

Comment

This work is part of a series of structure determinations of cyclomaltoheptose or cyclodextrin (CD) complexes, or their methylated derivatives, with linear molecules (Mentzafos, Mavridis & Schenk, 1994) that are components of insect pheromones. There is also an increasing interest in the inclusion of linear molecules inside CDs because of their ability to form composite structures such as molecular nanotube aggregates or polymer chains threaded with CD rings (Li & McGown, 1994; Harada, Li & Kamachi, 1993; Isnin & Kaifer, 1991; Wenz & Keller, 1993).



(I)

The main features of the β-CD host molecule are similar to those of other dimeric β-CD complexes. The host dimers are formed by O3···O3 hydrogen bonds between adjacent monomers related by a twofold axis and are packed in channels (see Fig. 2), as in all β-CD complexes that crystallize in the *C*2 space group (Mentzafos, Mavridis, Le Bas & Tsoucaris, 1991). Three primary hydroxy groups are disordered over two sites [O61 and O64, with site-occupation factor (s.o.f.) 0.76 for the major site; O65 with s.o.f. 0.77 for the major site] and one over three sites (O66, with s.o.f. set to 0.33 for all three sites). The non-disordered primary hydroxy groups and the disordered groups at the *A* and *C* sites have the *gauche–gauche* conformation, pointing outside the cavity, while those at the *B* site have the *gauche–trans* conformation, pointing inside the cavity (Table 2). This extensive disorder is interpreted as resulting from hydrogen bonding with the guest hydroxy group (see below).

The guest molecule was found to have some of its extreme atoms very close to the twofold axis, which causes a mutual overlap if both cavities of the dimer are occupied. However, the refinement of the occupancy of the guest gave a value very close to 0.5 and therefore it was restricted to that. The 2:1 host to guest ratio is in agreement with the low difference electron density observed inside the host cavity. The hydroxy O atom of the guest is found in the primary hydrophilic region of the CD cavity close to the *B* sites (inward) of two disordered primary hydroxy O atoms, O66*B* and O61*B* [O66*B*···O*G*(1−*x*, *y*, 1−*z*) = 3.01 Å, C66—O66*B*···O*G* = 113.8°; O61*B*···O*G*(*x*, *y*, *z*−1) = 3.49 Å, C61—O61*B*···O*G* = 96°]. In the last steps of the refinement we observed, consistently, two electron density peaks C*X*3 and C*X*2 (Fig. 1) close to atom C*G*4 which we have interpreted as the beginning of an alternative path of the hydroxy end of the guest molecule. C*X*2 is at a distance of 2.96 Å from two more disordered O atoms, O65*B* and O66*B*, which indicate the direction of the disordered chain, which in turn justifies their disorder. We have given an s.o.f. of 0.25 to the disordered guest atoms. The (*Z*)-9-dodecen-1-ol molecule, because of the *cis* double bond at position nine, has an L shape. Atoms C*G*8 to C*G*12 are accommodated in the dimeric interface (Fig. 2).

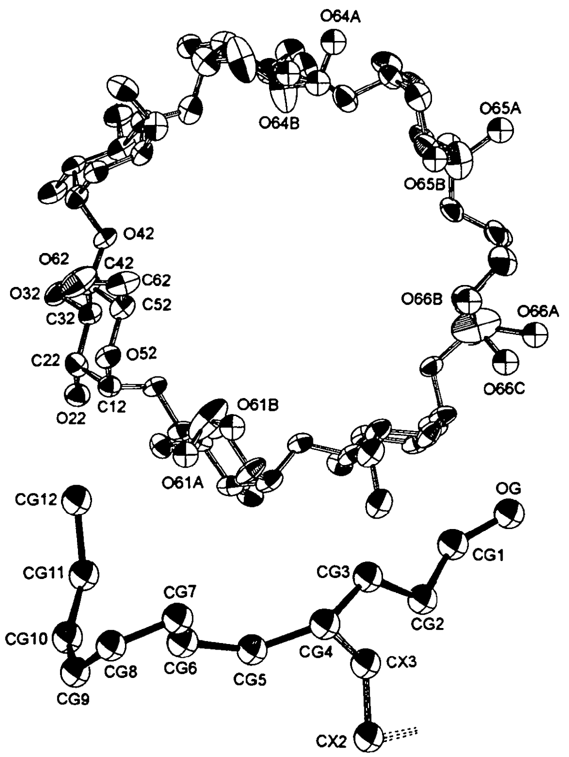


Fig. 1. The host and guest molecules with the atomic numbering scheme. C*mn* and O*mn* denote the *m*th atoms in the *n*th glucosidic residue. Probability ellipsoids are at the 50% level.

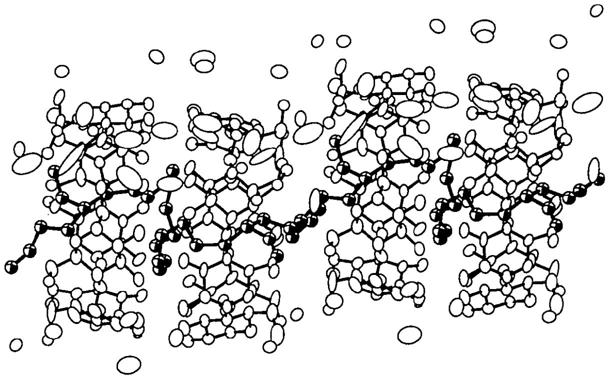


Fig. 2. A view of the β-CD channels with the guest molecules inside.

There are 9.6 water molecules per asymmetric unit distributed over 14 sites and one ethanol molecule with occupancy 0.53. All water molecules are within hydrogen bonding distance from primary or secondary hydroxy groups of the β-CD (O···*OW* = 2.50–3.06 Å, C—O···*OW* = 92–140°) and are labelled by the number of the closest β-CD O atom. The water network is very similar to that of other dimeric β-CD structures (Mentzafos, Mavridis, Le Bas & Tsoucaris, 1991). The hydroxy O atom of the ethanol forms strong hydrogen bonds with two water molecules (O*ET*···O*W*61*A* = 2.61, O*ET*···O*W*62*B* = 2.53 Å) and it is close to sites O64*A* and O64*B* (O*ET*···O64*A* = 2.08 and O*ET*···O64*B* = 2.655 Å).

Experimental

The complex was prepared from an aqueous solution of β-CD to which a tenfold excess of (*Z*)-9-dodecen-1-ol was added. It was then dissolved in a mixture of water and ethanol by heating to 75° and the solution was allowed to return to room temperature over a period of 7 d. The crystal was sealed in a glass capillary to prevent water loss.

Crystal data

$C_{42}H_{70}O_{35}.0.5C_{12}H_{24}O.$-
$9.6H_2O.0.53C_2H_6O$
$M_r = 1429.2$
Monoclinic
*C*2
$a = 19.238\,(5)$ Å
$b = 24.477\,(3)$ Å
$c = 15.79\,(2)$ Å
$\beta = 109.52\,(2)°$
$V = 7007.8\,(91)$ Å^3
$Z = 4$
$D_x = 1.337$ Mg m^{-3}
D_m not measured

Mo *Kα* radiation
$\lambda = 0.71069$ Å
Cell parameters from 250 reflections
$\theta = 5$–$20°$
$\mu = 0.120$ mm^{-1}
$T = 150\,(2)$ K
Diamond
0.70 × 0.50 × 0.25 mm
Colourless

Data collection

FAST TV area-detector diffractometer
ω steps of 0.2° (190° range at $\chi = 0$, 2 × 70° range at $\chi = 90°$)
Absorption correction: none
10959 measured reflections
9567 independent reflections

4589 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0286$
$\theta_{\rm max} = 24.78°$
$h = -22 \rightarrow 22$
$k = -28 \rightarrow 25$
$l = -17 \rightarrow 10$
no standard reflections

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0679$
$wR(F^2) = 0.1842$
$S = 0.732$
9550 reflections
869 parameters
H riding on C atoms [U(H) = $1.2U$(C)]
Unit weights applied

$(\Delta/\sigma)_{\rm max} = 0.927$
$\Delta\rho_{\rm max} = 0.433$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.272$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
C11	0.4407 (3)	0.7712 (3)	0.2450 (4)	0.045 (2)
C21	0.4025 (3)	0.7762 (3)	0.3131 (4)	0.042 (2)
O21	0.4548 (2)	0.7872 (3)	0.3988 (3)	0.046 (1)
C31	0.3593 (3)	0.7259 (3)	0.3145 (4)	0.036 (1)
O31	0.3183 (2)	0.7312 (3)	0.3764 (3)	0.044 (1)
C41	0.3054 (4)	0.7152 (3)	0.2202 (4)	0.043 (2)
C51	0.3460 (4)	0.7099 (4)	0.1540 (5)	0.050 (2)
C61	0.2997 (6)	0.7022 (5)	0.0579 (5)	0.098 (4)
O61*A*	0.2577 (5)	0.7410 (4)	0.0242 (6)	0.087 (2)†
O61*B*	0.3162 (8)	0.7107 (7)	−0.0108 (11)	0.033 (4)†
O51	0.3893 (3)	0.7595 (3)	0.1574 (3)	0.055 (1)
O41	0.2679 (2)	0.6646 (3)	0.2241 (3)	0.038 (1)
C12	0.1906 (3)	0.6638 (3)	0.1865 (4)	0.039 (1)
C22	0.1613 (4)	0.6396 (3)	0.2579 (5)	0.046 (2)
O22	0.1838 (3)	0.6735 (3)	0.3369 (3)	0.050 (1)
C32	0.1875 (4)	0.5818 (3)	0.2790 (4)	0.039 (1)
O32	0.1562 (3)	0.5586 (3)	0.3427 (3)	0.050 (1)
C42	0.1618 (3)	0.5486 (3)	0.1929 (4)	0.039 (1)
C52	0.1932 (4)	0.5742 (3)	0.1246 (4)	0.043 (2)
C62	0.1694 (5)	0.5472 (4)	0.0343 (5)	0.062 (2)
O62	0.0924 (4)	0.5454 (3)	−0.0041 (4)	0.086 (2)
O52	0.1674 (2)	0.6305 (3)	0.1082 (3)	0.046 (1)
O42	0.1929 (2)	0.4954 (3)	0.2145 (3)	0.040 (1)
C13	0.1438 (3)	0.4487 (3)	0.1899 (4)	0.041 (2)
C23	0.1626 (3)	0.4129 (3)	0.2737 (4)	0.040 (1)
O23	0.1507 (3)	0.4429 (3)	0.3465 (3)	0.050 (1)
C33	0.2409 (4)	0.3932 (3)	0.2996 (4)	0.043 (2)
O33	0.2563 (3)	0.3567 (3)	0.3739 (3)	0.048 (1)
C43	0.2506 (3)	0.3633 (3)	0.2192 (4)	0.040 (1)
C53	0.2253 (3)	0.3989 (3)	0.1340 (4)	0.040 (1)
C63	0.2227 (4)	0.3687 (4)	0.0509 (4)	0.047 (2)
O63	0.1696 (3)	0.3256 (3)	0.0317 (3)	0.056 (1)
O53	0.1530 (2)	0.4203 (3)	0.1178 (3)	0.040 (1)
O43	0.3259 (2)	0.3503 (3)	0.2417 (3)	0.049 (1)
C14	0.3453 (4)	0.2955 (4)	0.2287 (4)	0.055 (2)
C24	0.3987 (4)	0.2758 (4)	0.3169 (5)	0.054 (2)
O24	0.3641 (2)	0.2784 (3)	0.3840 (3)	0.047 (1)
C34	0.4693 (3)	0.3060 (4)	0.3423 (4)	0.046 (2)
O34	0.5220 (3)	0.2847 (3)	0.4231 (3)	0.053 (1)
C44	0.5009 (4)	0.3029 (4)	0.2673 (5)	0.058 (2)
C54	0.4456 (5)	0.3244 (6)	0.1815 (6)	0.103 (4)
C64	0.4725 (9)	0.3194 (7)	0.0960 (12)	0.149 (5)†
O64*A*	0.4942 (10)	0.2728 (8)	0.0892 (12)	0.186 (6)†
O64*B*	0.4074 (10)	0.3085 (8)	0.0163 (13)	0.047 (4)†
O54	0.3784 (3)	0.2933 (4)	0.1605 (3)	0.092 (2)
O44	0.5665 (3)	0.3374 (3)	0.2912 (3)	0.056 (1)
C15	0.6322 (4)	0.3136 (4)	0.2864 (5)	0.047 (2)
C25	0.6911 (4)	0.3219 (3)	0.3767 (5)	0.049 (2)
O25	0.6706 (3)	0.2982 (3)	0.4474 (3)	0.054 (1)
C35	0.7066 (4)	0.3822 (4)	0.3919 (4)	0.046 (2)
O35	0.7667 (3)	0.3922 (3)	0.4722 (3)	0.051 (1)
C45	0.7272 (4)	0.4063 (3)	0.3150 (5)	0.048 (2)
C55	0.6679 (4)	0.3934 (4)	0.2235 (5)	0.053 (2)
C65	0.6899 (5)	0.4079 (5)	0.1431 (6)	0.078 (3)
O65*A*	0.7524 (6)	0.3737 (5)	0.1491 (7)	0.112 (3)†
O65*B*	0.6326 (8)	0.4066 (7)	0.0551 (12)	0.032 (4)†
O55	0.6525 (3)	0.3357 (3)	0.2157 (3)	0.054 (1)
O45	0.7316 (3)	0.4643 (3)	0.3275 (3)	0.051 (1)
C16	0.7957 (4)	0.4916 (4)	0.3249 (5)	0.054 (2)
C26	0.8256 (4)	0.5255 (4)	0.4093 (5)	0.056 (2)
O26	0.8417 (3)	0.4932 (3)	0.4888 (4)	0.057 (1)
C36	0.7715 (3)	0.5713 (3)	0.4076 (4)	0.040 (1)
O36	0.8012 (2)	0.6060 (3)	0.4837 (3)	0.047 (1)
C46	0.7553 (4)	0.6040 (3)	0.3221 (5)	0.045 (2)
C56	0.7286 (5)	0.5666 (4)	0.2414 (5)	0.058 (2)
C66	0.7175 (8)	0.5965 (5)	0.1523 (6)	0.105 (4)
O66*A*	0.8043 (8)	0.6072 (7)	0.1621 (10)	0.057 (4)†
O66*B*	0.6869 (12)	0.5692 (9)	0.0777 (15)	0.097 (6)†
O66*C*	0.7583 (10)	0.6382 (8)	0.1483 (13)	0.075 (5)†
O56	0.7824 (3)	0.5232 (3)	0.2484 (4)	0.068 (2)
O46	0.6987 (2)	0.6426 (3)	0.3194 (3)	0.041 (1)

C17	0.7129 (4)	0.6984	0.3120 (4)	0.044 (2)
C27	0.6953 (4)	0.7286 (3)	0.3871 (4)	0.047 (2)
O27	0.7429 (3)	0.7099 (3)	0.4726 (3)	0.048 (1)
C37	0.6156 (3)	0.7209 (3)	0.3772 (4)	0.040 (1)
O37	0.5990 (2)	0.7517 (3)	0.4450 (3)	0.046 (1)
C47	0.5703 (3)	0.7419 (3)	0.2849 (4)	0.040 (2)
C57	0.5911 (3)	0.7147 (3)	0.2101 (4)	0.045 (2)
C67	0.5542 (4)	0.7396 (4)	0.1175 (5)	0.054 (2)
O67	0.5671 (2)	0.7968 (3)	0.1186 (3)	0.060 (1)
O57	0.6697 (2)	0.7194 (3)	0.2276 (3)	0.048 (1)
O47	0.4934 (2)	0.7283 (3)	0.2727 (3)	0.041 (1)
O*ET*	0.429 (3)	0.203 (2)	0.057 (4)	0.45 (3)†
C*ET*1O	0.370 (1)	0.183 (1)	0.075 (2)	0.115 (7)†
C*ET*2O	0.331 (2)	0.1931 (13)	0.139 (2)	0.15 (1)†
O*W*61*A*	0.3595 (13)	0.2758 (10)	−0.0627 (12)	0.100 (8)
O*W*62*A*	0	0.4599 (3)	0	0.050 (2)
O*W*62*B*	1/2	0.1332 (8)	0	0.208 (9)
O*W*62*C*	0.4235 (6)	0.0265 (4)	0.1831 (6)	0.140 (3)
O*W*63	0.0446 (2)	0.3723 (3)	0.9168 (3)	0.049 (1)
O*W*64*A*	0.471 (2)	0.1826 (17)	0.152 (3)	0.22 (3)
O*W*65*A*	0.1052 (3)	0.4188 (3)	0.7975 (4)	0.077 (2)
O*W*66*C*	0.4110 (17)	0.1374 (12)	0.213 (2)	0.118 (9)†
O*W*21	0.0887 (3)	0.3731 (3)	0.5262 (4)	0.080 (2)
O*W*22	0.4411 (9)	0.1745 (7)	0.6189 (13)	0.164 (7)
O*W*23	0.0243 (4)	0.3948 (4)	0.3403 (6)	0.082 (2)
O*W*24	0.4097 (4)	0.1793 (4)	0.4666 (8)	0.119 (4)
O*W*26	0	0.4765 (6)	1/2	0.139 (5)
O*W*32	0.4870 (5)	0.0415 (6)	0.6362 (9)	0.184 (5)
O*G*	0.4590	0.6150	1.0380	0.19 (2)†
C*G*1	0.4990	0.6030	0.9820	0.34 (4)†
C*G*2	0.5470	0.5590	0.9550	0.17 (2)†
C*G*3	0.5180	0.5430	0.8520	0.15 (1)†
C*G*4	0.5600	0.5040	0.8050	0.27 (2)†
C*G*5	0.6070	0.5120	0.7380	0.31 (3)†
C*G*6	0.6030	0.5050	0.6360	0.37 (3)†
C*G*7	0.5510	0.4630	0.5780	0.32 (3)†
C*G*8	0.5890	0.4550	0.5080	0.29 (3)†
C*G*9	0.6500	0.4960	0.5140	0.20 (1)†
C*G*10	0.6400	0.5510	0.5160	0.51 (6)†
C*G*11	0.5880	0.5980	0.5210	0.28 (2)†
C*G*12	0.5140	0.6280	0.4710	0.26 (2)†
C*X*2	0.6820	0.4840	0.9430	0.16 (2)†
C*X*3	0.6060	0.5090	0.8960	0.23 (4)†

† $U_{\rm iso}$.

Table 2. *Torsion angles of the primary hydroxy groups (°)*

	$n = 1$	$n = 2$	$n = 3$	$n = 4$	$n = 5$	$n = 6$	$n = 7$	Site
C4—C5—C6—O6	63	56	63	48	64	72	54	*A*
C4—C5—C6—O6	160			146	−169	−173		*B*
C4—C5—C6—O6						31		*C*
O5—C5—C6—O6	−58	−64	−60	−70	−58	−50	−67	*A*
O5—C5—C6—O6	38			26	70	66		*B*
O5—C5—C6—O6						−90		*C*

There was an absence of significant decay shown by comparison of equivalent reflections recorded near the beginning and end of data collection. Total measuring time was 8 h. The coordinates of the isomorphous 3,3-dimethylbutylamine β-CD complex (Mavridis, Hadjoudis & Tsoucaris, 1991) were used as initial coordinates for the skeleton atoms of β-CD. Subsequent $\Delta\rho$ maps revealed the primary C and O atoms as well as the atoms of the guest, water and ethanol molecules. The coordinates of the guest molecule have been optimized by fitting in the difference electron density map using the molecular graphics *O Molecular-Modelling Program* (Jones & Kjeldgaad, 1993), on a Crimson Silicon Graphics workstation; subsequently, they were kept constant during the final stages of the refinement. 12 H atoms belonging to hydroxy groups or water molecules were found from $\Delta\rho$ maps but their coordinates were not refined. The disordered O atoms were refined isotropically.

Data collection: *MADNES* (Pflugrath & Messerschmitt, 1991). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *O* (Jones & Kjeldgaad, 1993). Software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1191). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-8,10-Di-*n*-propyllobelidiol Hydrochloride Dihydrate

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Abstract

The definitive structure and the relative configuration of the title compound, perhydro-2(*R*)-[2(*S*)-hydroxypentyl]-6(*S*)-[2(*R*)-hydroxypentyl]-*N*-methylpyridinium chloride dihydrate, $C_{16}H_{34}NO_2^+.Cl^-.2H_2O$, a new alkaloid iso-