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# Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics

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### Crystal Structure of the Inclusion Complex of β-Cyclodextrin with 3,3-Dimethylbutylamine

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## CRYSTAL STRUCTURE OF THE INCLUSION COMPLEX OF β-CYCLODEXTRIN WITH 3,3-DIMETHYLBUTYLAMINE

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Abstract Single crystal X-ray analysis of the 1:1 3,3-dimethylbutylamine inclusion complex of -cyclodextrin ( $\beta$ -CD) is reported. The inclusion complexes form dimers. The guest molecule occupies the host cavity with its polar group towards the site of the secondary hydroxyl groups of  $\beta$ -CD and is hydrogen-bonded to a water molecule located inside the dimer.

### INTRODUCTION

The structure is part of a systematic study of inclusion complexes of  $\beta$ -cyclodextrin ( $\beta$ -CD) with guests of the same family that differ from one another only in terms of polarity, hydrogen bond ability, length etc. The purpose of the study is to determine what characteristics of the guest influence the geometry of the inclusion complex, the dimer formation, the packing of the macrocycle molecules, as well as the formation of the hydrogen bonds that holds them together. The structure is isomorphous with those of the inclusion complexes of  $\beta$ -CD with biphenyl or benzophenone (G.LeBas, PhD thesis, Univ. Pierre et Marie Curie 1985).

### **EXPERIMENTAL**

Prismatic crystals of dimensions 0.25x0.5x0.5mm were grown by slow cooling of an aqueous solution of the complex. During data collection the crystal was enclosed in a glass capillary. A summary of the experimental conditions are listed in Table I.

TABLE I Summary of Data Collection and Data Refinement.

Space group	C2
a	19.187(9)A
b	24.56(1)A
c	15.893(7)A
β	108.77(4)°
v	7091 A <sup>3</sup>
Z	4
λ	Moka
$\theta_{ extbf{max}}$	44°
Scaning Mode	$\theta - 2\theta$
No of measured reflections	4485
No of observed reflections ( $F_{obs} > 2\sigma F_{obs}$ )	3132
R	0.109
No of H <sub>2</sub> O mols	11
No of H <sub>2</sub> O positions	14

As initial coordinates for  $\beta$ -CD those of the  $\beta$ -CD benzophenone complex were used. The guest molecule was located inside the cyclodextrin cavity after an initial refinement of the  $\beta$ -CD and water coordinates had been performed.

### STRUCTURE DESCRIPTION

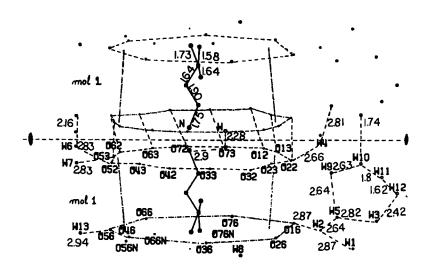


FIGURE 1 Schematic representation of the dimer and its surroundings.

The guest molecule is ordered and it has full occupancy. It is aligned within the cavity with its amino end towards the secondary hydroxyl groups and it is hydrogen-bonded to a water molecule (Fig.1).

The  $\beta$ -CD molecules pack in head to head dimers (Fig. 2) as in most  $\beta$ -CD inclu-

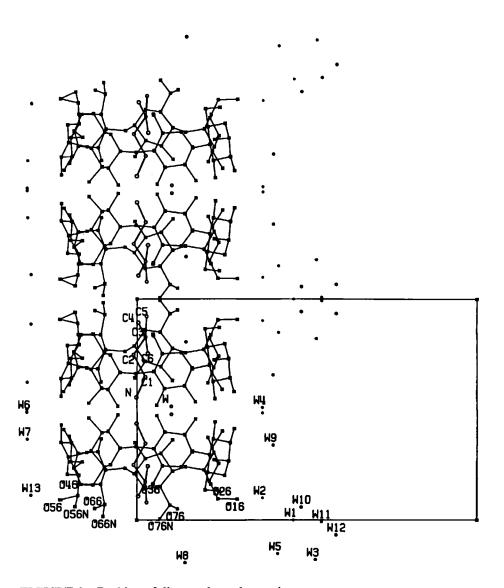


FIGURE 2 Packing of dimers along the c axis.

sion complexes. The dimers seem to constitute a stable entity. The lengths of the

hydrogen bonds between the secondary hydroxyl groups are very close to the other inclusion complexes of  $\beta$ -CD (Table II). Even the water molecules that surround the dimers have similar positions despite the different modes of association of the complexes (G. Le Bas, PhD thesis, Univ. Pierre et Marie Curie, 1985)

TABLE II Intra and Intermolecular Hydrogen Bonds Between the Secondary Hydroxyl Groups.

mol 1 mol 1	mol 1 mol 2
O12O13 = 2.85A	O22 O22 = 3.11A
O13O22 = 2.77A	O13 O23 = 2.79A
O22O23 = 2.88A	O12O32 = 3.01A
O23 O32 = 2.81A	O73 O33 = 2.86A
O32O33 = 2.90A	O72O42 = 2.90A
O33 O42 = 2.82A	O63 O43 = 2.75A
O42O43 = 2.87A	O53 O53 = 2.82A
O43 O52 = 2.85A	O62 O52 = 2.99A
O52O53 = 2.85A	
O53 O62 = 2.79A	
O62O63 = 2.85A	
O63 O72 = 2.82A	
O72O73 = 2.85A	
O73 O12 = 2.79A	

The eleven molecules of water distribute themselves in forteen positions according to the occupancies of Table III. Three primary hydroxyl groups exhibit two con-

TABLE III Occupancies of Water Molecules.

W1 = 0.5	W8 = 0.5
W2 = 1.16	W9 = 0.86
W3 = 1.01	W10 = 0.52
W4 = 0.96	W11 = 0.77
W5 = 1.22	W12 = 0.63
W6 = 0.74	W13 = 0.81
W7 = 0.78	W = 0.66

formations, one with the OH group away from the center of the macrocycle (gauche-gauche conformation), the other towards the center (gauche-trans conformation).