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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 (β–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 β-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 β-cyclodextrin (β-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 β-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 $A^3$ |
| Z | 4 |
| λ | Moka |
| $\theta_{max}$ | 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 $H_2O$ mols | 11 |
| No of $H_2O$ positions | 14 |

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

## STRUCTURE DESCRIPTION

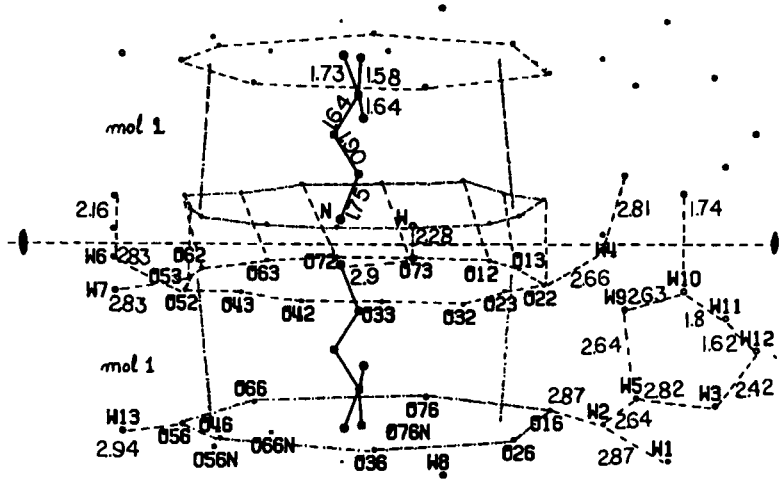


FIGURE 1 Schematic represantation of the dimer and its surroundings.

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 β-CD molecules pack in head to head dimers (Fig. 2) as in most β-CD inclu-

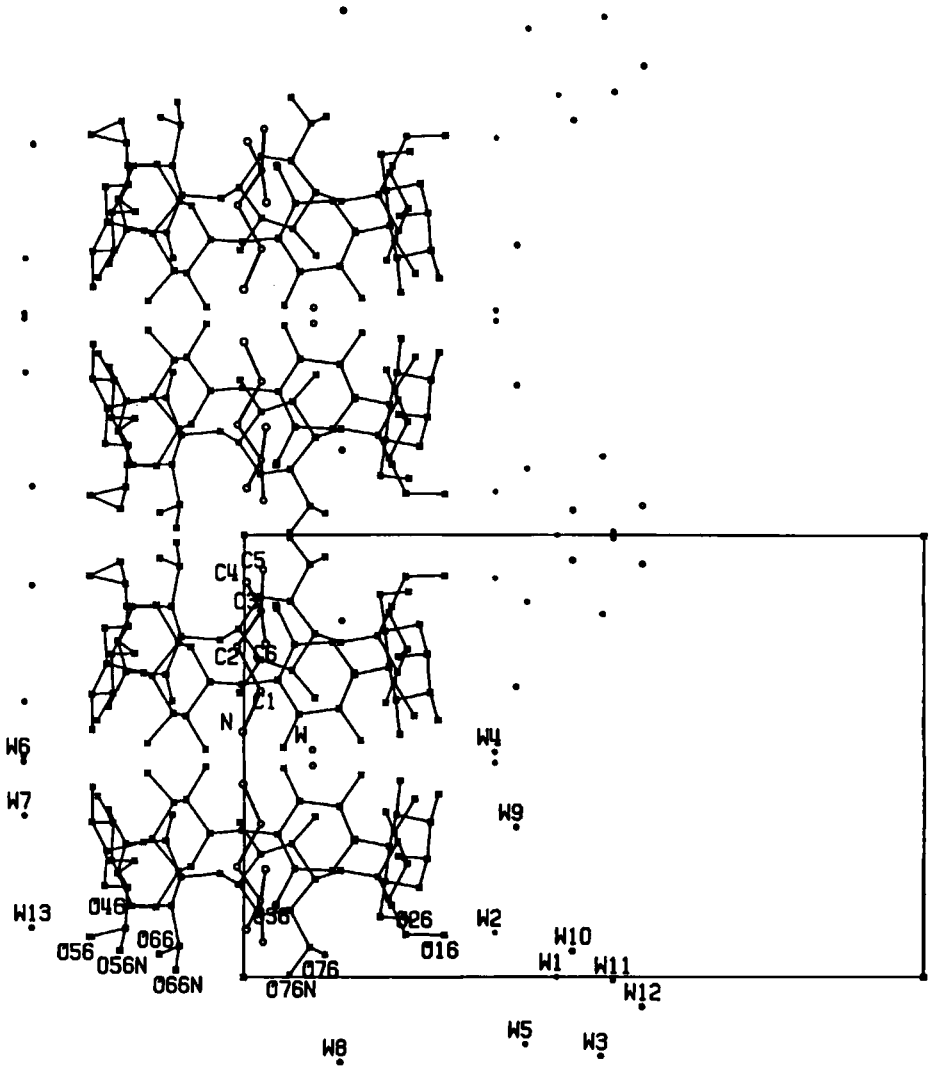


FIGURE 2 Packing of dimers along the c axis.

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 β-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 |
|---|---|
| O12 ...O13 = 2.85A | O22 ...O22 = 3.11A |
| O13 ...O22 = 2.77A | O13 ...O23 = 2.79A |
| O22 ...O23 = 2.88A | O12 ...O32 = 3.01A |
| O23 ...O32 = 2.81A | O73 ...O33 = 2.86A |
| O32 ...O33 = 2.90A | O72 ...O42 = 2.90A |
| O33 ...O42 = 2.82A | O63 ...O43 = 2.75A |
| O42 ...O43 = 2.87A | O53 ...O53 = 2.82A |
| O43 ...O52 = 2.85A | O62 ...O52 = 2.99A |
| O52 ...O53 = 2.85A | |
| O53 ...O62 = 2.79A | |
| O62 ...O63 = 2.85A | |
| O63 ...O72 = 2.82A | |
| O72 ...O73 = 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).