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X-ray structures of hexakis(2,6-di-*O*-methyl)-*α*-cyclodextrin in two crystal forms

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Hexakis(2,6-di-*O*-methyl)-*α*-cyclodextrin (DMαCD) crystallizes in the space group $P2_1$ from 10% NaCl aqueous solution at room temperature and in the space group $P2_12_12_1$ by the slow evaporation of water at ca. 90 °C. The $P2_1$ crystal shows a typical cage-type packing structure and a water molecule is included within its macrocyclic cavity. In the $P2_12_12_1$ crystal, DMαCD molecules are arranged in a helically extended polymeric chain formed by the inclusion of an O-6CH_3 methoxyl group of the adjacent molecule related by the twofold screw axis. In spite of the difference in the packing structure, the macrocyclic conformation of DMαCD molecules in the two crystals is nearly identical. The round structure of the macrocycle is maintained by the intramolecular O-3H---O-2 hydrogen bonds formed between adjacent residues. The root-mean-square distance between equivalent two atoms calculated after the least-squares superposition of the two molecules is 0.36 Å for non-hydrogen atoms except O-6 and methyl carbon atoms.

INTRODUCTION

Hexakis(2,6-di-*O*-methyl)-α–cyclodextrin (DMαCD) is a chemically modified αCD, in which all O-2H and O-6H hydroxyl groups are *O*-methylated. The methylation extends the cavity of macrocycle and affects the geometry of guest inclusion.[1] In the crystals of the DMαCD complexes with iodine and 1-propanol,[2] the guest molecules are included in the orientation similar to that observed in the corresponding αCD complexes,[3,4] but they are shifted to the O-2, O-3 side in the cavity. On the other hand, 3-iodopropionic acid is found to be included upside down in the DMαCD complex.[5] The macrocyclic structure of αCD, which is distorted in the uncomplexed state, becomes more symmetrical by the accommodation of the guest molecule. The DMαCD molecule in the complexed state is also in a round structure which is maintained by the intramolecular O-3H---O-2 hydrogen bonds.[2,5] To elucidate the property of molecular recognition and the conformational change upon complex formation, the knowledge of the structural details of DMαCD in the uncomplexed state is essential. We have obtained crystals of uncomplexed DMαCD in two forms. In this paper, we discuss the structure of DMαCD in these two crystals in comparison with that of the complexed state.

RESULTS

DMαCD was crystallized in two forms. The crystals with a space group $P2_1$ were obtained at room temperature by the slow evaporation of an aqueous solution containing 10% NaCl. The slow evaporation of an aqueous DMαCD solution at ca. 90 °C provided the crystals of another form with the space group $P2_12_12_1$.

Atomic numbering of DMαCD in two crystal forms are shown in Figure 1. Geometrical data describing the macrocyclic conformation is given in Table 1.

Structure of the low temperature form

The DMαCD molecule has a round structure and includes a water molecule which is disordered and occu-

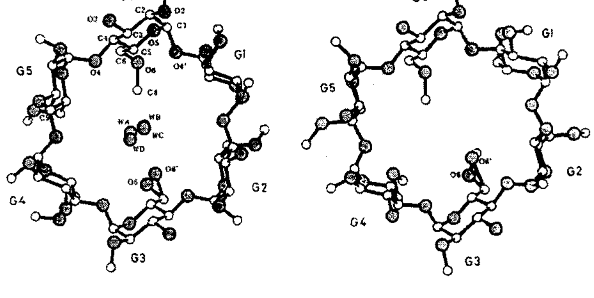

Figure 1 Structure and atomic numbering of DMαCD of the low temperature form (left) and high temperature form (right). Oxygen atoms are shaded. Water sites in the low temperature form are denoted by WA, WB, WC, and WD with the respective occupancies of 0.18, 0.20, 0.27, and 0.35.

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Table 1 Geometrical Data for the Macrocyclic Conformation

1. Radius of the O-4 hexagon (Å)

Residue	*Low temperature form*	*High temperature form*
G1	4.32	4.10
G2	4.27	4.36
G3	4.18	4.17
G4	4.32	4.18
G5	4.31	4.33
G6	4.15	4.27
Average	4.26	4.24

2. O-4 – O-4′ distance between adjacent residues (Å)

Residue	*Low temperature form*	*High temperature form*
G1 – G2	4.26	4.19
G2 – G3	4.22	4.16
G3 – G4	4.36	4.30
G4 – G5	4.18	4.27
G5 – G6	4.36	4.14
G6 – G1	4.18	4.38
Average	4.26	4.22

3. O-2 – O-3′ distance between adjacent residues (Å)

Residue	*Low temperature form*	*High temperature form*
G1 – G2	2.93	2.96
G2 – G3	2.86	2.84
G3 – G4	3.05	3.03
G4 – G5	3.19	3.14
G5 – G6	2.88	2.99
G6 – G1	2.98	2.78
Average	2.98	2.96

4. Tilt-angle and O-4 angle (°)

Residue	*Low temperature form*		*High temperature form*	
	Tilt-angle	*O-4 angle*	*Tilt-angle*	*O-4 angle*
G1	7.7	119	14.4	117
G2	11.6	119	3.1	121
G3	21.6	119	27.2	119
G4	11.1	120	14.2	120
G5	14.3	119	5.9	123
G6	20.9	118	18.0	122
Average	14.5	119	13.8	120

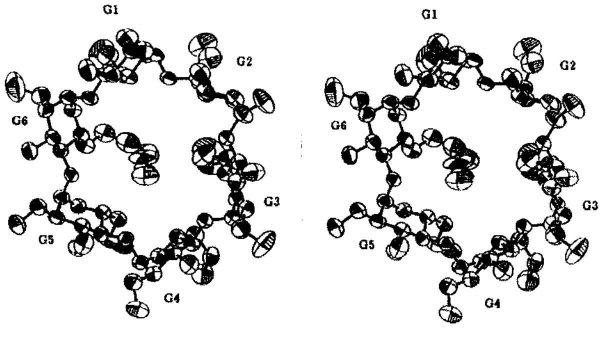

Figure 2 Stereo-drawing of DMαCD of the low temperature form.

pies four sites as shown in Figure 2. The crystal contains 3-*O* monomethylated DMαCD which is estimated to be 48% from the occupancy factor of the methyl group. Methyl groups attached to O-2 point away from the center of the macrocycle. The C-6-O-6 bonds show two types of conformation, (+)-gauche in the G3 and G6 residues and (-)-gauche in the other residues. In the G4 and G6 residues, the O-6CH_3 group is gauche to the C-5-C-6 bond while the trans conformation is observed in the other residues.

The radius of the hexagon composed of glycosidic O-4 atoms which are coplanar within 0.15 Å deviation is in the range from 4.15 to 4.32 Å and the side lengths are 4.18-4.36 Å. The tilt-angle indicates that the 2,6-di-*O*-methylglucopyranose residues incline with their O-6 side towards the inside of the macrocycle. The distance between O-2 and O-3 of the adjacent residue, which are in the range of 2.88-3.05 Å, indicates the formation of intramolecular O-3H---O-2 hydrogen bonds.

Crystal structure is shown in Figure 3. The DMαCD molecules are stacked along the b axis to form a typical cage-type packing structure. Both ends of the DMαCD cavity are blocked by adjacent molecules and a water molecule is enclosed within the isolated "cage". Intermolecular distances are listed in Table 2. The DMαCD molecules are in van der Waals contact with adjacent molecules. The O-3(G3)---O-W(B) distance indicates that the water molecule at the B site form a weak hydrogen bond with an O-3 of the adjacent DMαCD molecule.

Structure of the high temperature form

The stereo-drawing of the structure is shown in Figure 4. As indicated by the tilt-angle, the molecule has a pseudo twofold symmetry. The G3 and G6 residues are tilted more than other residues. The O-6CH_3 group of these residues points to the inside of the macrocycle and caps the O-6 side of the cavity. The C-6-O-6 bonds in the G3,

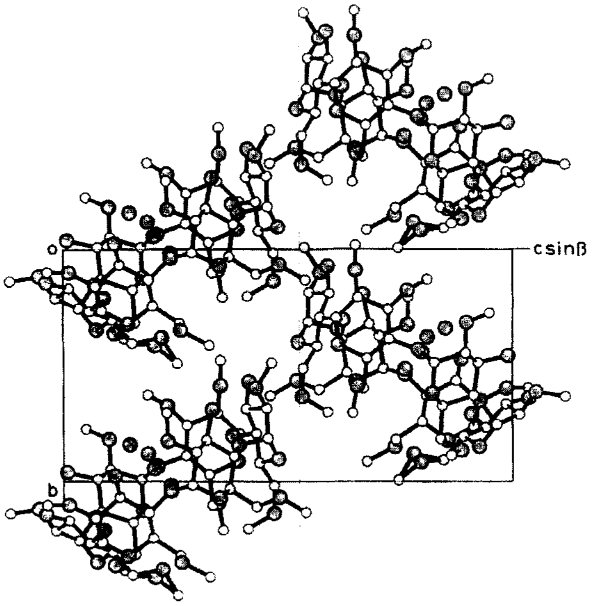

Figure 3 Crystal structure of the low temperature form viewed along the a axis. Oxygen atoms are shaded.

Table 2 Intermolecular Distances (Å) less than 3.6 Å

Atom-Atom	*Distance*	*Atom-Atom*	*Distance*
1. Low temperature form			
C-1(G1) - C-8(G5)	3.57 (a)	O-2(G2) - O-5(G3)	3.30 (f)
C-1(G1) - O-6(G5)	3.44 (a)	O-5(G2) - C-7(G3)	3.40 (d)
C-7(G1) - O-3(G5)	3.25 (a)	O-6(G2) - C-8(G5)	3.55 (a)
C-8(G1) - O-3(G1)	3.59 (g)	C-6(G3) - O-3(G3)	3.51 (d)
O-3(G1) - C-8(G2)	3.30 (c)	C-8(G3) - O-3(G6)	3.41 (b)
O-5(G1) - C-8(G5)	3.30 (a)	O-3(G3) - O-W(B)	3.26 (d)
O-5(G1) - O-6(G5)	3.51 (a)	C-6(G4) - C-9(G5)	3.39 (b)
C-7(G2) - C-9(G5)	3.36 (d)	C-1(G5) - O-6(G6)	3.35 (h)
C-7(G2) - O-5(G3)	3.24 (f)	C-6(G6) - O-3(G6)	3.33 (e)
C-8(G2) - C-8(G5)	3.18 (a)		
2. High temperature form			
C-8(G1) - C-7(G3)	3.46 (i)	C-8(G5) - O-3(G5)	3.46 (j)
C-8(G1) - O-2(G3)	3.25 (i)	C-3(G6) - O-5(G2)	3.52 (k)
C-8(G1) - O-3(G3)	3.29 (i)	C-6(G6) - O-5(G3)	3.54 (o)
O-6(G1) - C-6(G4)	3.51 (o)	C-7(G6) - O-5(G5)	3.23 (n)
C-2(G2) - O-2(G5)	3.53 (i)	C-7(G6) - C-7(G4)	3.41 (p)
C-7(G2) - O-3(G4)	3.54 (m)	O-3(G6) - O-5(G2)	3.35 (k)
O-3(G2) - C-8(G3)	3.46 (k)	O-3(G6) - O-7(G1)	3.55 (l)
O-3(G3) - C-7(G4)	3.55 (m)	O-6(G6) - C-1(G3)	3.45 (o)

Symmetry operator

a	1 + x,	y,	z	i	1/2 + x,	1/2 - y,	1 - z
b	x,	1 + y,	z	j	1/2 + x,	3/2 - y,	1 - z
c	x,	-1 + y,	z	k	-1/2 + x,	1/2 - y,	1 - z
d	- x,	1/2 + y,	-z	l	- x,	1/2 + y,	3/2 - z
e	- x,	1/2 + y,	1 - z	m	- x,	-1/2 + y,	1/2 - z
f	- x,	-1/2 + y,	- z	n	- x,	-1/2 + y,	3/2 - z
g	1 - x,	1/2 + y,	1 - z	o	1/2 - x,	1 - y,	1/2 + z
h	1 - x,	-1/2 + y,	1 - z	p	-1/2 - x,	1 - y	1/2 + z

G4, and G6 residues are in the (+)-gauche conformation and the (-)-gauche conformation is observed in the other residues.

The six O-4 atoms are coplanar with the maximum deviation of 0.13 Å. The radius of the O-4 hexagon is in the range of 4.10-4.36 Å and the side lengths are distributed in the range from 4.14 to 4.38 Å. The average values of these parameters are slightly smaller than those of the low temperature form. The distance between O-2 and O-3 of the adjacent residue indicates the formation of O-3H---O-2 hydrogen bonds which maintain the round macrocyclic structure.

Crystal packing is shown in Figure 5. The DMαCD molecules are arranged along the twofold screw axis parallel to the a axis. The O-6CH$_3$ methoxyl group of the G2 residue is inserted into the cavity of the adjacent molecule and the repetition of the intermolecular inclusion produces the helically extended polymeric chain.

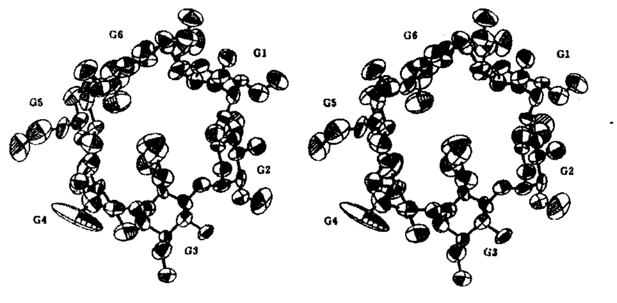

Figure 4 Stereo-drawing of DMαCD of the high temperature form.

DISCUSSION

DMαCD is highly soluble in water and did not crystallize at room temperature. The crystallization was successfully done at high temperature or in the presence of sodium chloride. DMαCD has many methyl groups and the hydrophobic aggregation, which may lead to the crystallization, may be enhanced by increasing temperature or ionic strength. It is well known that methylated CDs are less soluble at higher temperature.[6] The crystallization of the low temperature form of DMαCD indicates that the solubility decreases in the presence of inorganic salts. Such a phenomenon has been frequently observed in the crystallization of proteins.[7]

The present X-ray study demonstrated that DMαCD is in the round structure in the uncomplexed state. The macrocyclic conformation does not significantly differ between the two forms in spite of the different packing structures. The macrocycle of native αCD is distorted and changes more symmetrical in the complexed state. The round structure of DMαCD is maintained by the O-3H---O-2 hydrogen bonds between adjacent residues. The importance of such intramolecular hydrogen bonds to maintain the round structure is indicated from the comparison of the structure with permethylated αCD,[8] in which the macrocyclic ring having no intramolecular hydrogen bond is markedly distorted. In the crystal of αCD, the secondary hydroxyl groups are also hydrogen-bonded to water or adjacent αCD molecules. In the crystal of DMαCD, however, the O-3H hydroxyl groups only form a hydrogen bond with the adjacent residue and the hydrogen bonds may be stronger in the lower dielectric environment. To estimate the conformational difference of DMαCD between the two crystal forms, the structure of the high temperature form was superim-

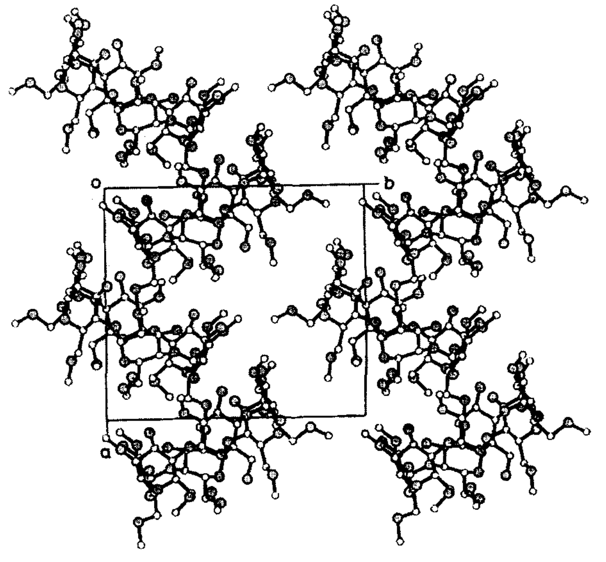

Figure 5 Crystal structure of the high temperature form viewed along the c axis. Oxygen atoms are shaded. Molecules related by the twofold screw axis along the c axis are not drawn because of the clarity.

posed on the structure of the low temperature form by the least-squares method. The methyl groups and O-6 atoms were not included in the calculation because of the conformational flexibility. The superimposed two structures are shown in Figure 6. The r.m.s.d. value of 0.36 Å indicates the close resemblance of the two structures. The conformational difference is observed only in some O-6CH_3 methoxyl groups.

The crystal of the low temperature form is isomorphous with those of the complexes with 1-propanol or iodine.[2] The macrocyclic structure does not significantly change upon exchange of water for 1-propanol or iodine. On the other hand, the conformational change has been observed in the complex with 3-iodopropionic acid.[5] Because of the inclusion of a bulkier molecule, the O-6 side of the DMαCD becomes wider as indicated by the smaller values of tilt-angles in the 3-iodopropionic acid complex; the average value is 11.5°. The DMαCD in the high temperature form includes no guest molecule, instead a methoxyl group of the adjacent molecule is inserted into the cavity from the O-2, O-3 side. A similar packing structure has been observed in the DMβCD complex with p-nitrophenol,[9] in which the guest molecule is accommodated not in the host cavity but in the intermolecular space. The intermolecular inclusion of the substituent group has been also observed in the crystal of monosubstituted CDs.[10-13] In the crystal, the DMαCD molecules of the high temperature form are more closely packed than those of the low temperature form as indicated by the comparison of the volume per molecule: 1476.0 Å^3 for the high temperature form and 1560.5 Å^3 for the low temperature form.

EXPERIMENTAL

DMαCD was purchased from Toshin Chemical Co. and recrystallized from ethanol.

Crystal of the low temperature form

Crystals were obtained from 10% NaCl solution by the slow evaporation. 200mg DMαCD was dissolved in 5ml 10% NaCl solution at 5 °C. After the filtration through 0.45 μm filter, the solution in a loosely capped 20ml vial was allowed to stand at room temperature and ca. half the solution was slowly evaporated in 3 months. Crystals were transparent needles. X-ray diffraction data were measured on a Nicolet P3/F diffractometer with graphite-monochromated CuKα radiation. A set of 4730 unique reflections was obtained in the 2θ range to 118° using a θ-2θ scan mode. Crystal data were as follows: $C_{48}H_{84}O_{30}\cdot H_2O$, F.W. = 1159.2, space group $P2_1$, a = 14.144(3), b = 10.687(2), c = 21.488(3) Å, β = 106.03(1)°, Z = 2, D_x = 1.233 g.cm^{-3}.

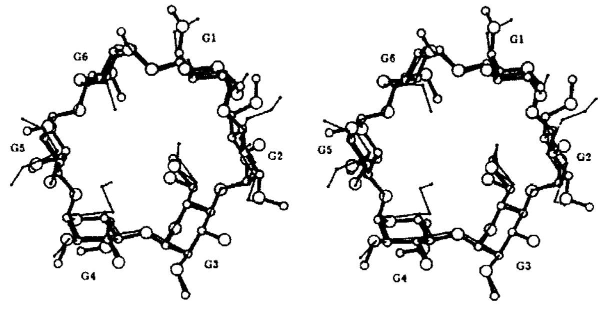

Figure 6 Stereo-drawing of the superimposed structures of DMαCD for two crystal forms. The DMαCD molecule in the high temperature form is shown with thin lines.

The structure was determined by the molecular replacement method using a set of DMαCD coordinates of the 1-propanol complex.[2] The refinement of the structure was carried out by the block-diagonal least-squares method. The quantity minimized was $\Sigma w(|F_o|-|F_c|)^2$ with w = 1.0 for all the reflections. Positions of the hydrogen atoms attached to methylene and methine groups were calculated and others were estimated on the difference-Fourier map. The final R-value was 0.061 for 4331 reflections with $|F_o|>3\sigma(F)$. The maximum values of positive and negative residual electron-density were 0.33 and -0.18 eÅ^{-3}, respectively.

Crystal of the high temperature form

100mg DMαCD was dissolved in 1ml distilled water. After the filtration through 0.45 μm filter, the solution in a loosely capped 10ml vial was slowly evaporated to dryness at ca. 90°C in an air bath. Small plate-like crystals obtained were stable at room temperature. X-ray measurements were carried out on an Enraf-Nonius CAD4 diffractometer equipped with an Elliott GX21 generator (40kV, 60mA, focal spot size 0.3 × 3mm, CuKα radiation). By using a θ-2θ scan mode, 4158 unique reflections were collected in the 2θ range to 110°. Crystal data were as follows: $C_{48}H_{84}O_{30}$, F.W. = 1141.2, space group $P2_12_12_1$, a = 15.195(2), b = 17.012(1), c = 22.481(2), Z = 4, D_x = 1.284 g.cm^{-3}.

The structure was solved by the molecular replacement method using a computer-generated symmetrical model of DMαCD, and refined by the block-diagonal least-squares method. The quantity minimized was $\Sigma w(|F_o|-|F_c|)^2$ with unit weight for all the reflections. Positions of the hydrogen atoms attached to the methine and methylene groups were calculated and included in the final least-squares refinement. The refinement converged at the R-value of 0.095 for 2692 reflections with $|F_o|>3\sigma(F)$. The maximum values of positive and negative residual electron density were 0.28 and -0.26 eÅ^{-3}, respectively.

Atomic coordinates of non-hydrogen atoms are listed in Table 3. Parameters of hydrogen atoms, anisotropic temperature factors of non-hydrogen atoms, bond distances, angles, torsion angles, and observed and calculated structure factors are deposited as supplementary material.

Table 3 Atomic coordinates and B_{eq} values

Atom	*Occupancy*	*x*	*y*	*z*	$B_{eq}(Å^2)$
(1) Low temperature form					
C-1(G1)		4193(6)	153(9)	2883(4)	5.9
C-2(G1)		4322(6)	-1256(9)	2968(4)	5.7
C-3(G1)		3467(6)	-1772(9)	3189(4)	5.9
C-4(G1)		3409(5)	-1087(9)	3801(4)	5.4
C-5(G1)		3346(6)	316(9)	3699(4)	5.6
C-6(G1)		3379(7)	1072(11)	4301(4)	6.9
C-7(G1)		4759(7)	-2935(10)	2316(5)	7.4
C-8(G1)		4198(14)	1323(21)	5391(6)	16.9
O-2(G1)		4344(4)	-1723(7)	2337(3)	6.7
O-3(G1)		3613(5)	-3083(7)	3327(4)	8.0
O-4(G1)		2503(4)	-1560(6)	3904(2)	5.4
O-5(G1)		4184(4)	694(6)	3488(3)	6.0
O-6(G1)		4157(5)	666(9)	4810(3)	9.7
C-1(G2)		2003(6)	2642(8)	861(4)	5.3
C-2(G2)		2660(6)	1590(9)	726(4)	5.5
C-3(G2)		2891(6)	661(9)	1266(4)	5.5
C-4(G2)		3322(5)	1318(8)	1908(4)	5.0
C-5(G2)		2688(6)	2439(9)	2002(4)	5.5
C-6(G2)		3103(6)	3204(10)	2598(5)	6.7
C-7(G2)		2633(8)	991(12)	-341(5)	8.7
C-8(G2)		4609(11)	4146(14)	3247(8)	13.8
O-2(G2)		2133(4)	998(7)	138(3)	6.6
O-3(G2)		3584(4)	-243(6)	1159(3)	6.6
O-4(G2)		3319(3)	388(5)	2403(3)	5.1
O-5(G2)		2501(4)	3237(6)	1448(3)	5.8
O-6(G2)		4093(6)	3511(8)	2623(4)	10.6
C-1(G3)		-1900(6)	2564(9)	348(4)	5.3
C-2(G3)		-1429(6)	2137(9)	-165(4)	5.9
C-3(G3)		-392(6)	1684(10)	139(4)	5.7
C-4(G3)		210(6)	2682(9)	597(4)	5.3
C-5(G3)		-319(5)	3059(8)	1092(4)	4.8
C-6(G3)		160(7)	4077(10)	1534(4)	6.7
C-7(G3)		-2424(10)	1372(12)	-1169(5)	10.0
C-8(G3)		215(14)	4933(22)	2542(7)	18.5
O-2(G3)		-2007(5)	1168(7)	-523(3)	7.2
O-3(G3)		67(4)	1428(8)	-365(3)	7.6
O-4(G3)		1120(4)	2101(5)	911(2)	5.0
O-5(G3)		-1298(4)	3500(5)	749(3)	5.3
O-6(G3)	0.7	-268(7)	4170(15)	2039(5)	10.7
O-6'(G3)	0.3	183(21)	3818(28)	2205(13)	10.8
C-1(G4)		-3699(5)	-101(8)	1750(4)	5.1
C-2(G4)		-3921(6)	-512(9)	1053(4)	5.2
C-3(G4)		-3080(5)	-96(8)	778(4)	4.9
C-4(G4)		-2934(5)	1304(8)	864(4)	4.9
C-5(G4)		-2850(5)	1753(8)	1556(4)	4.8
C-6(G4)		-2937(6)	3178(9)	1602(4)	6.1
C-7(G4)		-4852(7)	-2240(11)	520(5)	7.4
C-8(G4)		-4588(8)	3871(14)	1365(6)	10.3
O-2(G4)		-4039(4)	-1826(6)	1030(3)	6.2
O-3(G4)		-3307(4)	-393(6)	98(3)	6.4
O-4(G4)		-2001(4)	1524(5)	717(2)	5.0
O-5(G4)		-3636(4)	1222(5)	1784(3)	5.2
O-6(G4)		-3758(5)	3660(6)	1141(3)	6.9
C-1(G5)		-1406(6)	-2045(9)	3927(4)	5.4
C-2(G5)		-2053(5)	-2935(9)	3442(4)	5.5
C-3(G5)		-2280(5)	-2412(8)	2765(4)	4.9
C-4(G5)		-2729(5)	-1114(8)	2741(3)	4.7
C-5(G5)		-2047(6)	-292(8)	3249(4)	4.8
C-6(G5)		-2447(6)	1005(9)	3320(4)	6.0
C-7(G5)		-2134(8)	-5190(10)	3493(7)	9.7
C-8(G5)		-3709(7)	2078(12)	3553(7)	9.9
C-9(G5)	0.48	-2626(15)	-3709(20)	1899(9)	7.0
O-2(G5)		-1550(4)	-4110(6)	3497(3)	6.7
O-3(G5)		-2947(4)	-3239(6)	2336(3)	6.0
O-4(G5)		-2807(3)	-650(5)	2105(2)	4.6
O-5(G5)		-1861(4)	-874(6)	3884(3)	5.2
O-6(G5)		-3354(4)	896(6)	3454(3)	7.1
C-1(G6)		2481(6)	-1842(10)	4552(4)	6.0
C-2(G6)		2024(6)	-3139(10)	4531(4)	6.2
C-3(G6)		990(6)	-3104(9)	4080(4)	5.4
C-4(G6)		408(5)	-2053(9)	4294(4)	4.9
C-5(G6)		978(6)	-804(9)	4359(4)	5.2
C-6(G6)		479(7)	169(10)	4675(5)	6.7
C-7(G6)		2938(14)	-5088(16)	4626(11)	18.0
C-8(G6)		570(9)	2034(13)	4102(7)	10.8
O-2(G6)		2603(5)	-4011(7)	4283(3)	8.0
O-3(G6)		525(4)	-4282(6)	4123(3)	6.4
O-4(G6)		-486(4)	-1952(5)	3770(2)	4.7
O-5(G6)		1935(4)	-970(6)	4783(3)	5.8
O-6(G6)		930(5)	1359(7)	4680(3)	8.1
O-W(A)	0.18	457(24)	-1198(95)	1892(33)	23.5
O-W(B)	0.20	-177(24)	-1577(35)	1491(18)	8.8
O-W(C)	0.27	-111(17)	-556(42)	2028(17)	13.0
O-W(D)	0.35	424(19)	172(39)	2397(11)	16.1
(2) High temperature form					
C-1(G1)		2519(12)	1292(9)	6016(7)	8.4
C-2(G1)		1778(11)	1084(9)	6404(5)	6.8
C-3(G1)		1208(12)	1811(10)	6527(6)	8.1
C-4(G1)		1778(12)	2475(10)	6752(6)	8.6
C-5(G1)		2595(11)	2628(10)	6365(7)	8.2
C-6(G1)		3219(12)	3214(11)	6611(8)	9.5
C-7(G1)		747(16)	-60(13)	6422(8)	13.0
C-8(G1)		4040(14)	2588(13)	7363(12)	14.3
O-2(G1)		1251(10)	519(8)	6104(5)	10.8
O-3(G1)		555(8)	1628(7)	6984(5)	9.0
O-4(G1)		1192(7)	3161(6)	6744(4)	7.4
O-5(G1)		3010(8)	1917(6)	6297(4)	8.1
O-6(G1)		3379(9)	3149(9)	7248(6)	12.0
C-1(G2)		2530(11)	1678(10)	3721(6)	7.4
C-2(G2)		1840(12)	1059(7)	3950(7)	7.4
C-3(G2)		1619(10)	1227(9)	4599(6)	6.7
C-4(G2)		2476(11)	1297(9)	4952(7)	7.3
C-5(G2)		3146(11)	1810(9)	4684(5)	6.7
C-6(G2)		4144(19)	1875(13)	4950(10)	15.5
C-7(G2)		509(14)	485(14)	3532(11)	14.2
C-8(G2)		5417(16)	1235(21)	5115(14)	20.3
O-2(G2)		1075(8)	1134(6)	3571(4)	8.7
O-3(G2)		1100(10)	605(7)	4815(5)	10.6
O-4(G2)		2198(7)	1599(6)	5492(4)	7.1
O-5(G2)		3288(7)	1623(7)	4081(4)	8.4
O-6(G2)		4403(11)	1161(9)	5014(7)	13.7
C-1(G3)		1636(11)	4448(9)	2876(6)	7.4
C-2(G3)		1319(11)	3672(8)	2554(5)	5.9
C-3(G3)		1365(10)	3014(9)	2995(6)	6.7
C-4(G3)		2240(10)	2974(8)	3312(6)	6.4
C-5(G3)		2516(10)	3762(7)	3564(6)	6.0
C-6(G3)		3440(12)	3761(13)	3811(9)	11.1
C-7(G3)		370(13)	3552(10)	1751(7)	8.8
C-8(G3)		3961(23)	4220(21)	4682(11)	21.3
O-2(G3)		467(7)	3773(7)	2339(5)	8.6
O-3(G3)		1198(8)	2303(6)	2689(4)	7.6
O-4(G3)		2136(6)	2410(6)	3775(4)	6.7
O-5(G3)		2493(7)	4311(6)	3122(4)	7.1
O-6(G3)	0.7	3637(13)	4344(12)	4167(10)	13.4
O-6'(G3)	0.3	3157(25)	4011(31)	4504(21)	13.1
C-1(G4)		-223(11)	6451(10)	4087(6)	7.4
C-2(G4)		-738(10)	6063(9)	3597(5)	7.0
C-3(G4)		-322(10)	5328(10)	3460(6)	7.0
C-4(G4)		664(11)	5399(9)	3337(6)	7.2
C-5(G4)		1151(11)	5890(7)	3806(6)	6.5
C-6(G4)		2113(10)	6081(10)	3637(7)	8.3
C-7(G4)		-2265(14)	6342(26)	3492(12)	27.0
C-8(G4)		3345(14)	6456(15)	4135(12)	15.5
O-2(G4)		-1619(7)	5953(7)	3757(5)	8.2
O-3(G4)		-735(8)	4929(8)	2953(5)	10.4
O-4(G4)		988(6)	4631(6)	3295(4)	6.3
O-5(G4)		671(7)	6594(6)	3898(4)	7.7
O-6(G4)		2456(8)	6365(9)	4204(7)	12.6

Table 3 *Continued*

Atom	*Occupancy*	*x*	*y*	*z*	$B_{eq}(Å^2)$
C-1(G5)		-488(11)	6122(9)	6346(7)	7.3
C-2(G5)		-1356(10)	6130(8)	6044(7)	6.6
C-3(G5)		-1198(9)	5868(8)	5401(6)	5.5
C-4(G5)		-523(10)	6399(8)	5137(6)	5.8
C-5(G5)		323(10)	6506(9)	5477(7)	7.2
C-6(G5)		984(12)	7142(9)	5279(8)	8.9
C-7(G5)		-2534(14)	6057(12)	6732(8)	11.2
C-8(G5)		800(17)	8522(11)	4816(11)	15.0
O-2(G5)		-1962(7)	5676(7)	6334(4)	8.1
O-3(G5)		-2008(7)	5937(7)	5105(5)	8.2
O-4(G5)		-302(6)	6043(5)	4580(4)	6.0
O-5(G5)		138(7)	6690(7)	6080(5)	8.1
O-6(G5)		423(11)	7838(9)	5217(7)	14.5
C-1(G6)		1152(12)	3626(9)	7259(6)	7.7
C-2(G6)		146(13)	3701(11)	7421(7)	8.5
C-3(G6)		-264(10)	4261(10)	6924(6)	7.2
C-4(G6)		246(8)	4988(9)	6835(5)	5.5
C-5(G6)		1215(12)	4838(10)	6699(6)	7.9
C-6(G6)		1783(12)	5536(13)	6652(8)	10.7
C-7(G6)		-872(14)	2852(14)	7912(8)	11.6
C-8(G6)		2707(18)	5393(18)	5837(11)	17.3
O-2(G6)		-236(10)	2962(8)	7449(5)	11.2
O-3(G6)		-1156(7)	4438(6)	7096(4)	7.8
O-4(G6)		-124(6)	5379(6)	6321(4)	6.1
O-5(G6)		1540(7)	4373(7)	7177(4)	8.2
O-6(G6)		2627(7)	5382(9)	6455(6)	12.2

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