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Effect of Cyclodextrin Complexation on Photo-Fries Rearrangement of Naphthyl Esters

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Abstract:: Photolysis of β-cyclodextrin inclusion complexes of 1- and 2-naphthyl esters (acetates and benzoates) in aqueous medium, results in rearrangement to give one isomer of acylnaphthol in excess, whereas the solid state irradiation of the cyclodextrin complexes yields selectively one isomer. In addition, formation of cleavage product is totally suppressed. This remarkable selectivity is attributed to specific modes of the complexation of the esters into the β-CD cavity.

INTRODUCTION

Considerable interest has been recently aroused in the photo-Fries rearrangement,[1,2] due to its general applicability for the manufacture of aromatic ketones. For example, the *para*-selective rearrangement of phenyl acetate is a key step in the Hoechst-Celanese process for the manufacture of 4-acetaminophenol.[3] The reaction also finds applications in the design of polymeric imaging systems. Our interest in utilising organised assemblies, for effecting selective chemical reactions, prompted us to undertake a detailed investigation on the effect of cyclodextrin (CD) complexation on the photo-Fries rearrangement of naphthyl esters.

Compared to homogeneous isotropic solvents, CDs are able to modify the reactivity of a large variety of molecules and to improve the efficiency and selectivity of product formation.[4-6] For example, in the photo-Fries rearrangement of phenyl esters, *para*-hydroxyacetophenone is formed with higher chemical yield and phenol is formed with lower yield, when phenyl acetate-β-CD (1 : 1) complexes are irradiated.[7] Preferential *para*-rearrangement and a decreased amount of escape products are also observed[8] in the analogous photoreactions of acetanilide, benzanilide and ethylphenyl carbonate and phenyl benzoate. The rearrangement of phenyl acetate in the CD environment has been studied extensively by de Rossi *et al*.[9] Remarkable *ortho*-selectivity has been achieved by Ramamurthy *et al*,[10] when a series of unsubstituted and *meta*-substituted phenyl esters and anilides are irradiated in the presence of a tenfold molar excess of β-CD. When the photolysis is carried out on the solid complexes, the *ortho*-selectivity is exclusive. The formation of *ortho*- products, which was not observed in homogeneous solution in solvents such as benzene and methanol, is induced by β-CD in the photorearrangement of benzenesulfonanilide, whereas in the solid state, the *ortho*-selectivity is > 99 %.[11] An impressive regioselectivity between the two *ortho*-isomers is observed in the photolysis of solid β-CD

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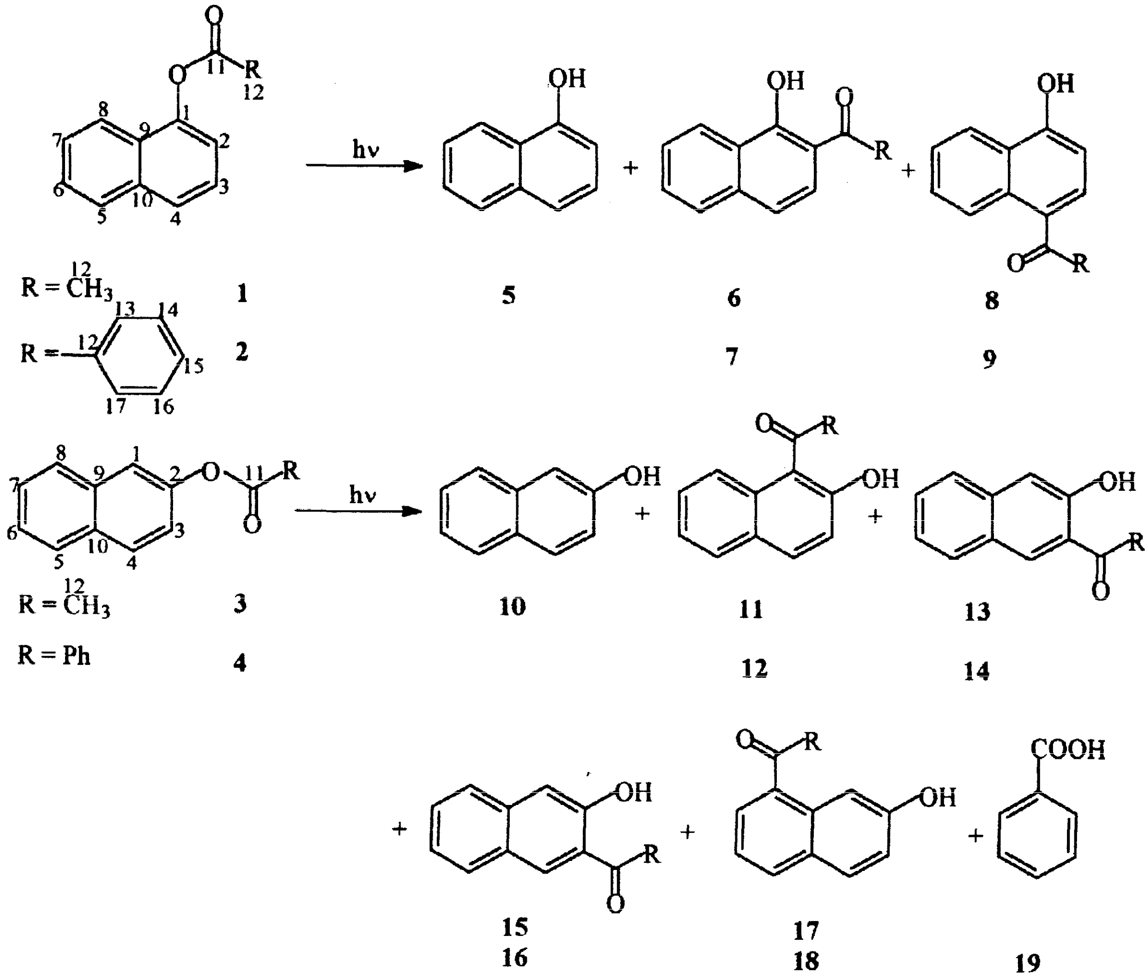
complexes of *meta*-substituted sulfonate esters.[12] The effect of CD complexation on photorearrangement of allyl phenyl ethers,[13a] benzyl phenyl ethers and alkyl phenoxyacetates has also been reported.[13b]

Though photo-Fries rearrangement of naphthyl esters have been studied[14-16] in solution, the influence of CD encapsulation on this title reaction has not been investigated, barring a brief report.[17] We present here, the salient features of our work on the effect of CD complexation of photo-Fries rearrangement of naphthyl esters.

RESULTS AND DISCUSSION

Characterization of β-CD-naphthyl ester complexes

The orientation of the guest molecules inside the CD cavity is governed by the shape of the guest molecule and also by the interaction between the guest and CD. This results in marked differences in spectral properties of the CD complexed and uncomplexed guest. IR, ^{1}H-, ^{13}C-NMR and X-ray diffraction studies have been used to characterize the CD complexes of 1- and 2-naphthyl acetates and benzoates (Scheme 1).



Scheme 1

^{1}H-NMR spectra. An insight into the extent to which the binding effects are operating can be obtained from an analysis of the changes in the chemical shifts and resonance line widths of β-CD and the substrates. They also provide effective methods for investigation of interaction sites since these parameters are sensitive to the molecular environment.

Table 1. 400 MHz ^{1}H-NMR spectral data of β-CD protons in complexes of 1- and 2-naphthyl esters with β-CD[a]

Compound	H_1	H_2	H_3	H_4	H_5	H_6
β-CD	2279.5	1358.5	1772.2	1339.5	1416.6	1456.4
1-β-CD	2278.3	1351.1	1799.3	1341.8	1430.6	1457.0
	(2280.8)[b]	(1345.4)	(1773.1)	(1334.6)	(1418.1)	(1458.2)
2-β-CD	2285.6	1355.5	1807.6	1337.5	1435.5	1459.9
	(2277.6)	(1359.5)	(1771.1)	(1338.4)	(1421.1)	(1459.5)
3-β-CD	2279.3	1352.5	1801.7	1343.3	1431.6	1458.0
	(2280.5)	(1357.1)	(1771.3)	(1336.7)	(1419.2)	(1458.7)
4-β-CD	2275.4	1355.5	1790.0	1333.9	1433.1	1457.5
	(2280.6)	(1359.0)	(1774.1)	(1334.1)	(1418.2)	(1458.1)

[a] Chemical shifts are expressed in ppm; Solvent DMSO-d_6; [b] Values in parentheses indicate the corresponding spectral data for a 1 : 1 physical mixture.

To confirm the complex formation between the esters and β-CD, ^{1}H-NMR spectra of CD protons in pure β-CD and its complexes with 1- and 2-naphthyl acetates and benzoates and their physical mixtures (a 1:1 mixture of β-CD and the guest) were recorded (Table 1). The results indicate that while H_1, H_2, H_4 and H_6 protons are unaffected, the H_3 and H_5 protons show considerable downfield shifts in all the cases. The stronger shifts arise due to the anisotropic effects of the naphthyl ring residing inside the cavity and are considered to be favourable evidence for inclusion complex formation.

^{13}C-NMR spectra. In many instances ^{13}C-NMR is advantageous over ^{1}H-NMR as it can provide direct and useful information of the position of all the carbon atoms of the guest molecules and hence on the degree of their penetration into the cavity. The ^{13}C-NMR chemical shifts for β-CD, **1**, **2** and their complexes with β-CD are presented in Table 2. As a result of complex formation, all the β-CD carbon atoms undergo shielding and it is significant in C_1, C_2, C_4 and C_6. With **1**, the C=O, C_6, C_7, C_8 and C_{12} carbons experience lesser shielding compared to other carbon atoms, indicating that in the penetration of the naphthyl ring into the CD cavity, these carbon atoms are the least affected.

The results point out that C_1 and C_4 of the naphthyl ring exhibit stronger shielding compared with the other carbons of **3** in the complex. Also, all the carbons of the phenyl group except C_{12} undergo strong shielding. This points out clearly the formation of a 2:1 (β-CD:Sub) complex in addition to (1:1) complex and this is also supported by the host:guest ratio measurements.

Table 2. 400 MHz ^{13}C-NMR chemical shifts (ppm) of β-CD, 1, 2 and their β-CD complexes[a,b]

β-CD		Δδ[c] in			carbons of 1		carbons of 2	
carbons	(δ)	1-β-CD	2-β-CD	carbon atoms[d]	(δ)	1-β-CD (Δδ)[c]	(δ)	2-β-CD (Δδ)[c]
1	102.018	-0.319	-0.319	C=O	169.443	-0.106	164.727	-0.156
2	72.419	-0.273	-0.273	C_1	146.402	-0.274	146.360	-0.247
3	72.905	-0.106	-0.091	C_2	118.472	-0.212	118.643	-0.186
4	81.648	-0.349	-0.364	C_3	126.602	-0.212	126.658	-0.156
5	71.933	-0.151	-0.136	C_4	121.174	-0.258	120.844	-0.262
6	59.912	-0.243	-0.243	C_5	127.929	-0.213	128.054	-0.186
				C_6	126.548	-0.152	126.855	-0.141
				C_7	125.819	-0.197	126.385	-0.135
				C_8	125.667	-0.152	125.747	-0.171
				C_9	126.456	-0.288	126.081	-0.171
				C_{10}	134.152	-0.273	134.232	-0.141
				C_{12}	20.643	-0.166	129.102	-0.156
				C_{13},C_{17}			129.937	-0.217
				C_{14},C_{16}			128.616	-0.277
				C_{15}			134.184	-0.247

[a] Chemical shifts are expressed in ppm; Solvent DMSO-d_6; [b] At room temperature; [c] - sign indicates shielding of carbon atom; [d] Numbering of carbon atoms are the same as given in Scheme 1.

Table 3 shows the ^{13}C-NMR chemical shifts for β-CD, **3**, **4** and their β-CD complexes. The significant changes observed in the chemical shifts of the various carbon atoms suggest the formation of strong complexes of **3** and **4** with β-CD. With **4**-β-CD complexes, as in the case of compounds **1** and **2**, significant shielding is present. However, it is perplexing to observe that only with **3**-β-CD complexes, is there considerable deshielding of carbon atoms. It is relevant to note that a recent excellent review[18] on NMR studies of CD complexes also points out the difficulty in interpretation of ^{13}C-NMR screening changes mainly due to the pronounced sensitivity of carbon shielding towards even minor conformational distortions.

IR Spectra. Complex formation is also evidenced from IR studies of **2** and **4** and their complexes with β-CD. It is observed that the aromatic C-H stretching frequency at 3000 cm^{-1} is shifted to 2900 cm^{-1} in the complex with enhanced intensity. The carbonyl group of the free naphthyl esters around 1730 cm^{-1} is shifted to 1740 cm^{-1} in the complex indicating that the C=O group has a decreased mesomeric interaction with the aryl ring in the complex. It is also observed that the C=C stretching frequencies of the aryl rings decrease in intensity.

X-ray diffraction studies. The X-ray powder diffraction patterns for β-CD, **2**, **4**, **2**-β-CD and **4**-β-CD complexes as well as their physical mixture are also indicative of complex formation. The sharp peaks of β-CD (2θ) in the region of 8.6, and 12.3 were missing in the **2**-β-CD and **4**-β-CD complexes. Peaks at 10.3, 17.2 and 22.9 in **2** were shifted to 10.8, 17.7 and 21.7 in the complexes, with the β-CD peaks at 10.4 and 17.3

merging with them. However, a physical mixture of **2** and β-CD exhibited the peaks without any change.

Table 3. 400 MHz ^{13}C-NMR chemical shifts (ppm) of β-CD, 3, 4 and their β-CD complexes[a,b]

β-CD		Δδ[c] in			carbons of 3		carbons of 4	
carbons	(δ)	3-β-CD	4-β-CD	carbon atoms[d]	(δ)	3-β-CD (Δδ)[c]	(δ)	4-β-CD (Δδ)[c]
1	102.018	+0.035	-0.227	C=O	169.417	+0.213	169.723	-0.046
2	72.419	+0.080	-0.167	C_1	118.522	+0.106	118.624	-0.076
3	72.905	+0.262	-0.031	C_2	148.227	+0.076	148.208	-0.076
4	81.648	+0.019	-0.258	C_3	121.664	+0.167	121.569	-0.076
5	71.933	+0.217	-0.060	C_4	130.984	+0.091	129.750	-0.076
6	59.912	+0.125	-0.167	C_5	127.447	+0.319	127.656	-0.076
				C_6	125.762	+0.198	125.819	-0.046
				C_7	126.658	+0.192	127.428	-0.061
				C_8	127.447	+0.107	126.669	-0.061
				C_9	133.352	+0.076	134.031	-0.046
				C_{10}	129.269	+0.136	128.946	-0.061
				C_{12}	20.891	+0.121	129.325	-0.060
				C_{13},C_{17}			131.010	-0.076
				C_{14},C_{16}			128.824	-0.076
				C_{15}			133.272	-0.076

[a]Chemical shifts are expressed in ppm; Solvent DMSO-d_6; [b]At room temperature; [c]+ sign indicates deshielding and - sign indicates shielding of carbon atoms; [d]Numbering of carbon atoms are the same as given in Scheme 1.

Also in the case of **4**, the peaks of β-CD at 8.6, 10.4, 12.3, 17.3 and 26.9 and peaks of **4** at 12.3, 13.1 and 23.3 were missing in the **4**-β-CD complex. β-CD peaks at 10.4, 20.7 and 25.2 were changed to 10.7, 21.1 and 26.1 respectively in the complex and the peak of **4** at 23.3 was changed to 23.8. But physical mixture showed both β-CD and **4** peaks that were unchanged.

Host - Guest ratio & dissociation constants. Complex formation was also evidenced from the determination of host - guest ratio by a gravimetric method using a reported procedure[10] (Table 4). The values are less than unity with all the guests suggesting that in addition to a 1 : 1 complex, small amounts of 2 : 1 complexes are also formed. Dissociation constants (K_D), for the complexes of **1-4** with β-CD were estimated from UV spectral measurements at room temperature by using the Benesi-Hildebrand equation.[19] The values are low suggesting the formation of stronger complexes between the substrates and the CD.

Table 4. Host-guest ratio and K_D values of β-CD complexes of 1-4

	1	**2**	**3**	**4**
H : G ratio	1 : 0.82	1 : 0.61	1 : 0.97	1 : 0.97
λ_{max} (nm)	277	272	279	270
K_D (M)[a]	1.4×10^{-3}	1.4×10^{-3}	1.8×10^{-3}	8.0×10^{-4}

[a] Measured in 1% methanol - 99% water mixture at room temperature from absorption measurements.

(b) Photo-Fries rearrangements of naphthyl esters:

The results of irradiation of **1** and **2** in isotropic media and as their β-CD complexes in aqueous solution and in the solid state are presented in Table 5. Irradiation of **1** in homogeneous solution yields 2-acetyl-1-naphthol (**6**) as the major product along with 4-acetyl-1-naphthol (**8**) and 1-naphthol. However, when an aqueous solution of **1**-β-CD complex is irradiated, the formation of 1-naphthol (**5**) is totally suppressed. In addition to 2- and 4- isomers, some unknown products are also formed. An increase in the amount of CD improves the yield of the 2-isomer. The suppression of cleavage product (**5**) is due to the inability of the radical pair to move apart, when it is held more tightly inside β-CD cavity. In contrast, when the β-CD solid complex of **1** is photolysed, exclusive formation of **6** is observed.

Table 5. Percentage yield of products in the photo-Fries rearrangement of 1and 2[a]

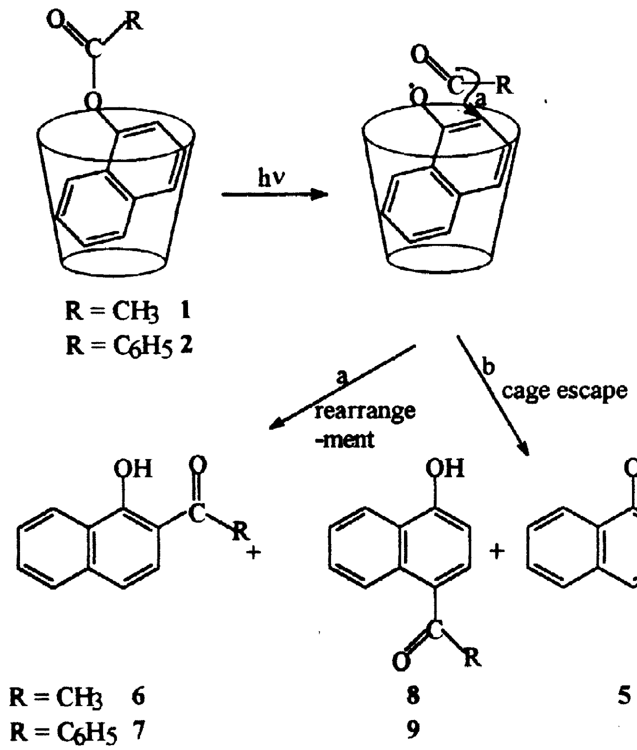
Medium	photoproducts from 1					photoproducts from 2					
	total yield	rearranged products		cleavage products		total yield	rearranged products		cleavage products		
		6	**8**	**5**	X[b]		**7**	**9**	**5**	**19**	X[b]
Hexane	30	24	4	2	--	30	21	8	1	--	--
Methanol	30	21	6	3	--	31	19	9	3	--	--
β-CD-water (1:1)[c]	49	28	10	--	11	40	23	10	1	2	4
β-CD-water (5:1)	45	29	6	--	10	55	39	3	1	2	10
β-CD-solid (1:1)	15	15	--	--	--	20	20	--	--	--	--

[a] Analysed by GC; error limit ± 5 %, [b] unidentified products, [c] ratio of CD to guest in the complex

Photolysis of **2** in an isotropic medium yields a mixture of 2-benzoyl-1-naphthol (**7**) and 4-benzoyl-1-naphthol (**9**) with a small amount of cleavage product **5**. When an aqueous solution of **2**-β-CD complexes (1 : 1 and 1 : 5) is irradiated, along with the mixture of **7** and **9**, trace amounts of **5** and benzoic acid (**19**) are also formed. An increase in the amount of CD improves the yield of **7**, while that of **9** decreases. When the solid 1:1 complex of **2**-β-CD is irradiated, formation of the 2-isomer is exclusive.

This remarkable regioselectivity is attributed to the formation of an inclusion complex (Scheme 2), in which the β-CD moiety encircles the 4-position of the naphthyl ring and there is hindrance for the attack of the acetyl radical at that site. Subsequently attack is preferred at the exposed 2-position to yield the 2-isomer. The reduced yield of conversion in the solid state irradiation is due to the recombination of the initially formed acetyl and naphthoxy radicals to regenerate the starting material, when they are trapped inside the CD cavity.

Results of the irradiation of **3** and **4** in isotropic media and their β-CD complexes in aqueous solution are presented in Table 6. Irradiation of **3** in isotropic medium results in the formation of 1-acetyl-2-naphthol (**11**) and 3-acetyl-2-naphthol (**13**) along with small amount of 2-naphthol (**10**). Photolysis of an aqueous solution of **3** within β-CD (1 : 1 and 1 : 5 complexes) also gives **11** as the major product and as the host concentration is increased, the formation of **11** also increases. When **3**-β-CD solid complex is irradiated, quantitative formation of the 1-isomer is observed.



R = CH_3 **1**
R = C_6H_5 **2**

R = CH_3 **6** **8** **5**
R = C_6H_5 **7** **9**

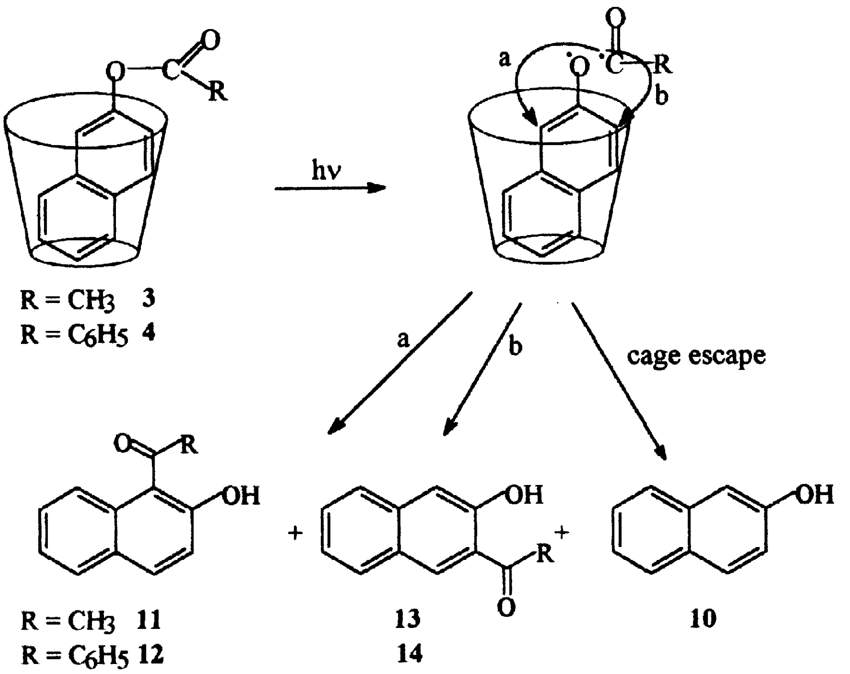
Scheme 2

Table 6. Percentage yield of products in the photo-Fries rearrangement of 3 and 4[a]

Medium	photoproducts from 3					photoproducts from 4							
	total yield	rearranged products		cleavage products		total yield	rearranged products				cleavage products		
		11	13	10	X[b]		12	14	16	18	10	19	X[b]
Hexane	30	24	4	2	--	30	20	2	3	1	4	--	--
Methanol	30	15	5	6	4	30	16	2	3	2	7	--	--
β-CD-water (1:1)[c]	49	24	7	12	7	57	33	4	--	--	1	1	18
β-CD-water (5:1)	50	29	10	9	2	59	31	3	3	--	1	4	17
β-CD-solid (1:1)	20	20	--	--	--	37	35	1	1	--	--	--	--

[a] Analysed by GC; error limit ± 5 %, [b] unidentified products, [c] ratio of CD to guest in the complex

Ester **4** upon irradiation in isotropic solvents like methanol and hexane gives 1-benzoyl-2-naphthol (**12**) as the major product with small amounts of isomeric 3-, 6-, and 8-benzoyl-2-naphthols. A trace amount of 2-naphthol is also formed. When the β-CD complexes of **4** (both 1 : 1 and 1 : 5) are irradiated, the yield of other isomers are reduced and the 1-isomer is observed as the major product with improved yield. When the solid complex of **4**-β-CD is irradiated, 1-benzoyl-2-naphthol (**12**) is nearly the only product.



Scheme 3

With esters **3** and **4** there is a possibility of the formation of both 1- and 3-isomers as there are two *ortho*-positions available for the attack of the benzoyl radical. However, the complexation with β-CD (Scheme 3) protects the molecule in such a way that the 1-position is exposed, while the 3-position is inaccessible. This is even more significant in solid phase irradiation, where the molecules are tightly packed. This results in quantitative conversion to one of the *ortho*-isomers. The predominant formation of one of the *ortho*-rearranged products (**11** and **12**) is in accordance with odd-electron densities[17] in various carbon atoms of the naphthoxy radical. It is also likely that electronic factors play a major role compared to steric aspects in controlling the product selectivity in the case of 2-naphthyl esters.

EXPERIMENTAL

Materials

β-Cyclodextrin (Aldrich) was used as received. 1- and 2-naphthyl acetates and benzoates were prepared and recrystallized by a reported procedure.[20] Double distilled water was used thoroughout the study. Infrared spectra (IR) of β-CD, guests, their β-CD complexes and physical mixtures were recorded in KBr/neat, between 200-4000 cm^{-1} using a SHIMADZU IR spectrometer. ^{1}H- and ^{13}C-NMR spectra of β-CD complex were recorded in DMSO with a JEOL GSX-400 NMR spectrometer using tetramethylsilane (TMS) as internal standard. Some of the ^{1}H-NMR spectra were recorded in a 90 MHz PERKIN-ELMER (R32) spectrometer. The X-ray diffraction patterns of the powdered samples were measured in the region of 0-57°, employing Cu

K_α radiation (1.54 Å) from MABE JOEL MODEL JDX-830 powder diffractometer.

Preparation of β-CD complexes

To a saturated solution of β-CD in water, an equimolar amount of substrate dissolved in methanol was added and stirred for 24 hours at room temperature. The resultant white crystalline precipitate was filtered, washed with diethyl ether to remove any uncomplexed substrate and dried in an air oven for 3 hours at 60 °C. The dried white crystalline powder was used for further studies.

Spectral studies

A stock solution of the ester ($1x10^{-2}$ M) was prepared by weighing a known amount of the substrate and dissolved in 100 % methanol (HPLC grade). 0.1 mL of this stock solution was taken and then the respective volume of β-CD ($1x10^{-2}$ M-stock solution in water - freshly prepared) was added and stirred for 24 hrs. Absorption spectra were recorded to calculate the equilibrium constant for the formation of the complexes. UV absorption spectra were recorded with a UV/VIS JASCO 7800 double beam spectrophotometer. Optical densities were monitored at appropriate wavelengths ranging from 200-400 nm in aqueous medium at room temperature. The host-guest ratio was estimated as per the reported procedures.[10]

Photolysis of complexes

An aqueous solution of β-CD complexes or homogeneous solutions were irradiated in a quartz tube using a multilamp photoreactor (SAIC, India) fitted with eight (each 8 W) 254 nm medium pressure mercury lamps after purging with nitrogen gas for 30 minutes. Irradiations were carried out for 5 hours. Solid β-CD complexes were irradiated using the same reactor in a quartz cell for 100 hours.

Analysis of the reaction mixture

The percentage of conversion and product distribution were obtained by analysing with Gas Chromatography (Netel Chromatograph, India) equipped with a flame ionization detector and a 2 feet length, 0.5 mm inner diameter stainless steel column containing SE-30 (10 %) or OV1 column packed with a methyl silicone stationary phase with nitrogen as a carrier gas. Reaction products were also identified by co-injection of authentic samples prepared separately.

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