1,5-AND 1,3-PHOTOCYCLIZATION REACTIONS OF 8-SUBSTITUTED-1,2,3,4-TETRAHYDRO-1-NAPHTHALENONES

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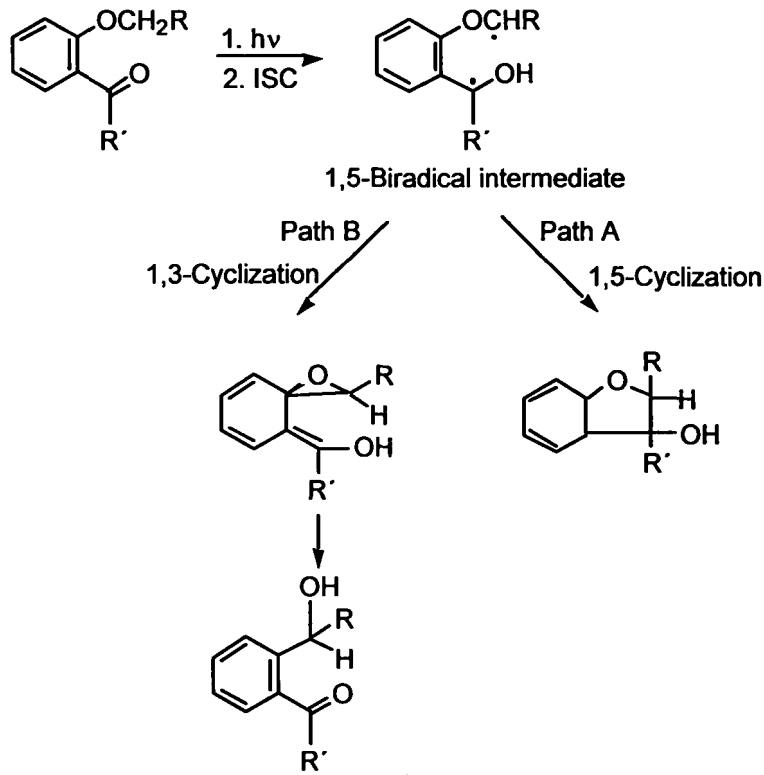
Absract: Photocyclization reactions were carried out on 8-(4-halobenzyloxy)-1,2,3,4-tetrahydro-1-naphthalenones (halogen = Cl and Br in **1a** and **1b** respectively), 8-(4-nitrobenzyloxy)-1,2,3,4-tetrahydro-1-naphthalenone **1c** and 8-oxo-5,6,7,8-tetrahydro-1-naphthyloxyacetonitrile **1d** in benzene. Irradiation of **1a-b** afforded rearranged naphthyl alcohols (1,3-cyclization products) **4a-b** and naphtho[1,8-*bc*]furanol derivatives (1,5-cyclization products) **2a-b**. On the other hand, irradiation of **1c-d** under the same condition afforded only 1,5-cyclization producsts **2c-d** in each case. Different behaviours for the photoreaction of **1a-b** and for **1c-d** is attributed to the stability of the 1,5-biradical intermediates formed by δ-hydrogen abstraction. Substituent effects in cyclization step of 1,5-biradicals are discussed along with reaction pathways.

Introduction:

The importance of the benzofurane ring system in synthetic product with pharmacodynamic applications explain the large number of papers published. For examples, naphthofurans prepared by photocyclization[1] or thermal[2] method showed anti-tumor cell growth through interactions with DNA in *vitro* , benzofurans substituted with benzoyl group at 3-position[3-4] showed angiotropic, antiinflammatory, fibronolytic and coronary vasodilator effects.

Among the numerous way for preparation of furane derivatives, photocyclization reactions of *o*-substituted aromatc carbonyl compounds are used to synthesis these compounds. The first example of photocyclization to prepare benzofurane was reported by Pappas *et al.* They prepared *cis*- and trans-benzofuranols by irradiation of 2-benzyloxybenzaldehyde in acetonitrile[5].

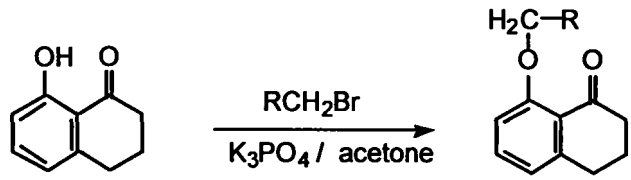
In general, photocyclization reactions of carbonyl compounds proceed *via* 1,5-biradical intermediates formed through δ-hydrogen abstraction by the excited carbonyl group as shown in Scheme 1[6-11] . The 1,5-biradicals can undergo 1,5-cyclization to dihydrobenzofuranols (path A) or 1,3-cyclization to spiroenols (path B)[6-7,12] which rearrange to the corresponding 2-acylalcohols or their hemiacetals. Preferance for path A or path B depends on the type of substituents R and R′. For example, when benzophenones (R′=Ph) are used as starting materials, 1,5-cyclization occurs to give dihydrobenzofuranols[6-7,13]. However, when benzaldehydes (R′=H) and acetophenones (R′=Me) are employed, 1,3-cyclization competes with 1,5-cyclization to afford rearranged products[12]. Changing R from alkyl group to electron withdrawing ethoxycarbonyl or cyano group, 1,5-cyclization occurs predominantly[12]. In this paper, we report synthesis of furanol derivatives using photocyclization of 8-substituted-1,2,3,4-tetrahydro-1-naphthalenones **1a-d**. Substituent effects on the cyclization of 1,5-biradical intermediates are also discussed (Scheme 1).



Scheme 1

Results and Discussion

8-Substituted-1,2,3,4-tetrahydro-1-naphthalenones **1a-d** for photocyclization reactions were prepared by the reactions of 8-hydroxy-1,2,3,4-tetrahydro-1-naphthalenones with *p*-chlorobenzyl chloride *p*-bromobenzyl bromide, *p*-nitrobenzyl bromide or bromoacetonitrile in presence of a base. The results are outlined in Figure 1 and Table 1.



1a, R= *p*-Cl-C_6H_4
1b, R= *p*-Br-C_6H_4
1c, R= *p*-$NO_2C_6H_4$
1d, R= CN

Figure 1

Table 1. Synthesis of α-Tetralone Derivatives 1a-d

Starting material [a]	Reagent	Base	Solvent	Time (minutes)	Product	Yield (%)
N		K_3PO_4	Acetone	240	**1a**	89
N	Cl-C$_6$H$_4$-CH_2-Cl	K_2CO_3	Acetone	180	**1b**	67
N		K_2CO_3	Acetone	180	**1c**	60
N	Br-C$_6$H$_4$-CH_2-Br	K_3PO_4	Acetone	240	**1d**	44
	O_2N-C$_6$H$_4$-CH_2-Br					
	$BrCH_2CN$					

[a] N is 8-hydroxy-5,6,7,8-tetrahydro-1-naphthalenone.

Irradiation of six-membered cyclic ketones **1a-b** R= *p*-Cl-C_6H_4, *p*-Br-C_6H_4) with high-pressure mercury lamp in benzene afforded naphtho[1,8-*bc*]furanols **2a-b** (4% and 3%, respectively) along with their dehydrated products **3a-b**, 6% yield in each case and rearranged naphthyl alcohols **4a-b** are also isolated in 87% and 85% yields, respectively. Compounds **3a-b** would be formed by dehydration of **2a-b** during isolation procedures after irradiation. Though, *cis*- and *trans*-isomers were possible for **2a-b**, only *trans*-isomer was formed in each case, showing stereoselectivity in the cyclizaiton step.

The stereochemistry of **2a** was assigned to be *trans* by comparison with *cis*-isomer obtained by irradiation of **1a** in acetonitrile[14] using anisotropic effect. The ^{1}H nmr spectra showed that, the methylene group at C_3 in **2a** deshielded C_2-H by anisotropic effect. A large difference in steric bulkiness between hydrogen and substituent at C_2 and between methylene and hydroxyl groups in 1,5-biradicals **6** would produce high stereoselectivity for *trans*-isomer, that is, sterically favorable isomer is produced selectively. The stereochemistry, for **2b** is not clear.

On the other hand, irradiation of **1c-d** under the same condition afforded only naphtho[1,8-*bc*]furanols **2c-d** and no rearranged alcohols were obtained. The naphthofuranol **2c** was produced in 70% yield and its stereochemistry is not clear. In the case of **1d**, a mixture of *cis*- and *trans*- isomer was isolated (33%). *Cis*- and *trans*-isomers with regard to cyano and hydroxyl groups of naphtho[1,8-*bc*]furanol **2d**. The *Cis: trans* ratio was 1:44 judging from the ^{1}H nmr spectra in which the methylene group at C_3 in **2d** deshielded C_2-H in **2d** at the *trans* position by an anisotropic effect [14-19]. The results are shown in Figure 2 and Table 2.

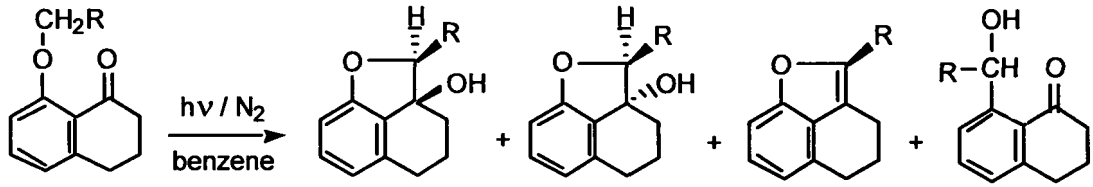
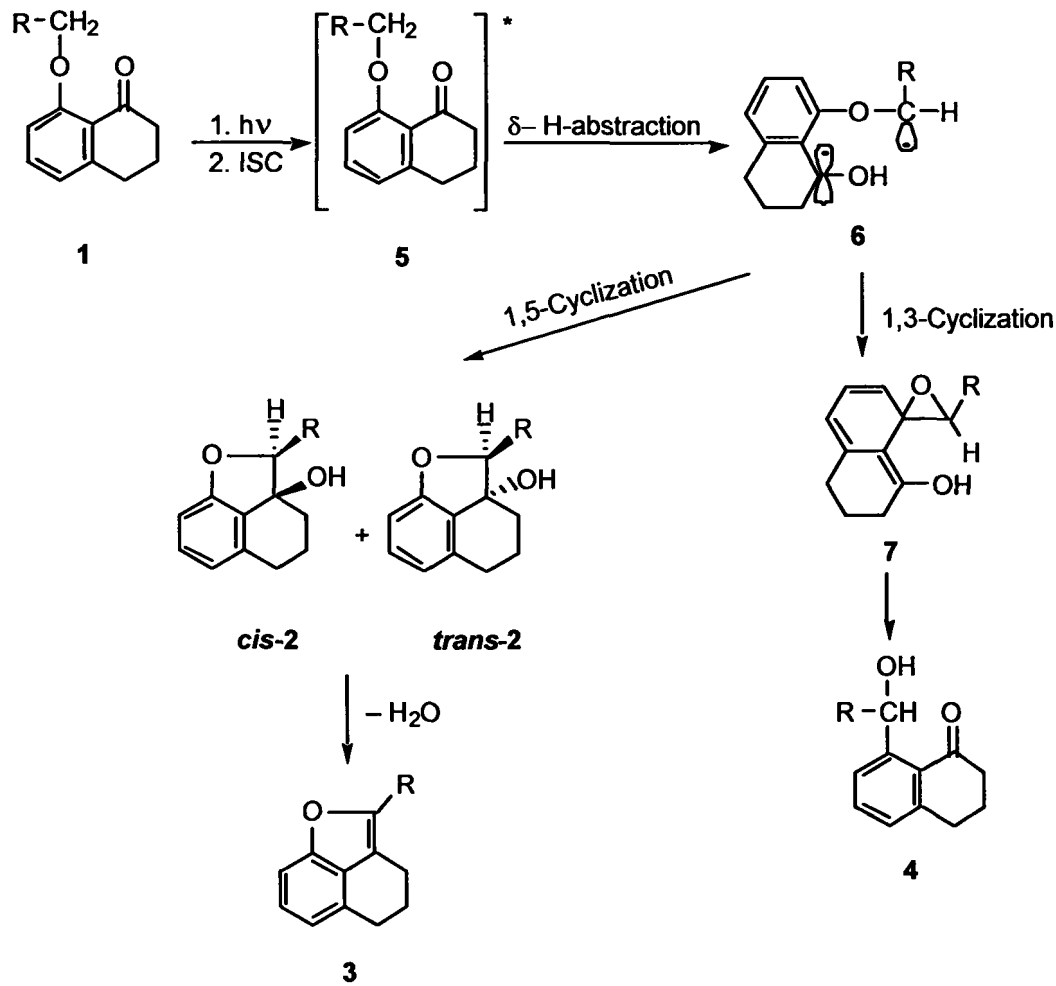


Figure 2

All of our results would be explained by intramolecular cyclization of 1,5-biradical intermediates formed by δ-hydrogen abstraction. The mechanism on this type of photoreaction have been well studied[7,11]. The mechanistic pathways of products formation are summarized in scheme 2.



Scheme 2

Table 2. Photocyclization Reactions of α-Tetralone Derivatives 1a-d [a]

Starting material	R	Irradiation Time (minutes)	Product yields %		
			2	3	4
1a	*p*-Cl-C_6H_4	30	4	6	87
1b	*p*-Br-C_6H_4	40	3	6	85
1c	*p*-NO_2-C_6H_4	30	70	–	–
1d	CN	45	33 [b]	–	–

[a] A benzene solution (500 ml) of **1a-d** (2.00 mmoles) was irradiated after deoxygenation by bubbling nitrogen gas for 1 hour. [b] *Cis-* and *trans-* isomers with regard to cyano and hydroxyl groups of naphthofuranol **2d**.

Irradiation of **1** produces (n, π*) excited triplet state **5** after intersystem crossing process (ISC). The carbonyl group of **5** abstracts δ-hydrogen to give 1,5-biradical **6** which can undergo two competing cyclization reactions. For example, 1,5-intramolecular cyclization of **6** produces furanols **2** which can undergo dehydration on silica gel during isolation procedure to give **3**. On the other hand 1,3-cycliztion is necessary for the formation of rearrranged naphthyl alcohols **4a-b** from **1a-b**. The possible intermediates for rearranged alcohol formation are the spiroenols **7** which were initially suggested by wagner *et al.*[6,20] 1,5- or 1,3-Cyclization for the 1,5-biradical **6** formed by δ-hydrogen abstraction could be explained by the conformation of the six-membered cyclic ketones and substituent effects. In six-membered ring ketones, the dihedral angle between the carbonyl group and benzene ring [21,22] is small. The *p*-orbital in the 1,5-biradicals **6** formed by δ-hydrogen abstraction would be nearly parallel to the π-orbitals of the benzene ring. Rotation by about 90° [6] around the Ar-C bond is necessary for furane ring formation. However, such a rotation reduces benzylic conjugation between the *p*-orbital and benzene ring and accordingly causes strain in the six-membered ring. Therefore, spirocyclization of **6** to **7** occurs predominantly. Spiroenols **7** afford rearranged alcohols **4** by cleavage of the ether linkage. However, such a spirocyclization would be inhibited by the presence of electron withdrawing substituent. For example when R=*p*-$NO_2C_6H_4$ or CN group, the 1,5-biradical intermediates **6** formed do not give rearranged products through spirocyclization[6]. This is due to stabilization of the biradicals formed by push-pull resonance[23-26] between the electron withdrawing group (NO_2 or CN group in **1c** or **1d** respectively) and the naphthyloxy oxygen atom. Therefore, the biradicales **6** become are not reactive enough to make epoxide with benzene ring.

Experimental :

The melting points are uncorrected. Column chromatography was performed on silica gel (Wakogel C-200). Ether refers to diethyl ether. Dry benzene for photoreactions was prepared by distillating over calcium hydride. Photoreactions were carried out with 400-W high-pressure mercury lamp (Riko UVL-400 HA) with pyrex filter. The ir spectra were determined on a Hitachi Model 270-30 IR spectrometer. The ^1H and ^{13}C nmr spectra were determined at 200 Mhz and 50 Mhz on a Varian Gemini 200 FT NMR spectrometer, using tetramethylsilane as the internal standard.

8-(4-Chlorobenzyloxy)-1,2,3,4-tetrahydro-1-naphthalenone 1a:

Compound **1a** was obtained as colorless crystals from benzene-hexane, mp 126-127°, identical with an authentic sample[14] in the ir and nmr spectra.

8-(4-Bromobenzyloxy)-1,2,3,4-tetrahydro-1-naphthalenone 1b:

A mixture of 8-hydroxy-1,2,3,4-tetrahydro-1-naphthalenone[27] (3.0 g, 18.5 mmoles), *p*-bromobenzylbromide (4.7

g, 18.6 mmoles), tripotassium phosphate (7.8 g, 36.7 mmoles) and acetone (30 ml) was stirred and refluxed for 180 minutes. After removal of the insoluble materials by filteration the acetone was evaporated. The residue was chromatographed and eluted with benzene to give **1b** (4.1 g, 67%) as colorless crystals, mp 130-131° from benzene-hexane; ir (potassium bromide): 1673 cm^{-1} (Ar-CO); ^1H nmr (deuteriochloroform): δ 2.04 (tt, J=6 and 6 Hz, 2H, 3-H_2), 2.68 (t, J=6Hz, 2H, 2-H_2 or 4-H_2), 2.93 (t, J = 6Hz, 2H, 2-H_2 or 4-H_2), 5.11 (s, 2H, OCH_2), 6.81 (d, J = 8Hz, 1H, 5-H or 7-H), 6.83 (d, J=8Hz, 1H, 5-H or 7-H), 7.22-7.57 (m, 5H, 6-H and *p*-Br-Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 23.2 (t), 30.4 (t), 41.3(t), 69.7(t), 112.0(d), 120.7(d), 123.2(s), 128.3(d), 128.7(d), 133.7(s), 133.9(d), 136.1(s), 147.1 (s), 160.0 (s), 196.1(s).

$C_{17}H_{15}BrO_2$ requires: C, 61.63; H, 4.53. Found: C,61.57; H,4.49.

8-(4-Nitrobenzyloxy)-1,2,3,4-tetrahydro-1-naphthalenone 1c:

Compound **1c** (60%) was obtained as colorless crystals from benzene-hexane in a manner similar to the synthesis of **1b**, mp 141-142°; ir (potassium bromide): 1550 cm^{-1} (NO_2), 1675 cm^{-1} (Ar-CO); ^1H nmr (deuteriochloroform): δ 2.07 (tt, J=6 and 6Hz, 2H, 3-H_2), 2.64 (t, J=6Hz, 2H, 2-H_2 or.4-H_2), 2.99 (t, J=6Hz, 2H, 2-H_2 or 4-H_2), 5.14 (s, 2H, OCH_2), 6.81 (d, J=8Hz, 1H, 5-H or 7-H), 6.84 (d, J=8Hz, 1H, 5-H or 7-H), 7.23-7.54 (m, 5H, 6-H and *p*-NO_2–Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 22.6 (t), 30.7 (t), 41.2 (t), 71.1 (t), 112.0 (d), 120.4 (d), 123.3 (s), 128.2 (d), 128.8 (d), 133.7 (s), 133.9 (d), 136.4 (s), 147.6 (s), 162.3 (s), 196.4(s).

$C_{17}H_{15}NO_4$ requires: C,68.69; H,5.05; N,4.71. Found: C,68.72; H,4.94; N,4.66.

8-Cyanomethoxy-1,2,3,4-tetrahydro-1-naphthalenone 1d:

Compound **1d** was obtained as colorless crystals from benzene-hexane, mp 78-79°, identical with an authentic sample[14] in the ir and nmr spectra.

General Procedure for Photocyclization of Ethers 2a-d

A benzene solution (500 ml) of the starting material (2.00 mmoles) was deoxygenated by bubbling nitrogen gas for 1 hour and then irradiated by a high pressure mercury lamp. The irradiation was stopped when the ether almost disappeared. After irradition the benzene was evaporated. The residue was chromatographed and eluted with benzene-ether to give a variety of products.

***Trans* 2-(*p*-chlorophenyl)-2a,3,4,5-tetrahydro-2H-naphtho[1,8-bc]furan-2-ol 2a:**

compound **2a** was obtained as colorless crystals from benzene-hexane, mp 145-146°; ir (potassium bromide): 3290 cm^{-1} (OH); ^1H nmr (deuteriochloroform): δ 2.11 (tt, J=6 and 6Hz, 2H, 4-H_2), 2.73-2.88 (m, 2H, 3-H_2 or 5-H_2), 3.04 (t, J=6Hz, 2H, 3-H_2 or 5-H_2), 5.72 (broad s , 2H, OH and 2-H), 6.81-7.32 (m, 3H, 6-H, 7-H and 8-H), 7.41-7.52 (m, 4H, *p*-Cl-Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 18.6 (t), 24.5 (t), 30.9 (t), 74.2 (s), 95.2 (d), 107.6 (d), 119.2 (d), 128.3 (d), 128.5 (d), 129.6 (s), 129.8 (d), 133.8 (s), 134.5 (s), 136.4 (s), 158.7 (s).

$C_{17}H_{15}ClO_2$ requires: C, 71.20; H,5.27. Found: C,71.14; H,5.29;.

2-(*p*-Bromophenyl)-2a,3,4,5-tetrahydro-2H-naphtho[1,8-bc] furan-2a-ol 2b:

Compound **2b** was obtained as colorless crystals from benzene-hexane, mp 156°; ir (potassium bromide): 3300 cm^{-1} (OH); ^1H nmr (deuteriochloroform): δ 2.14 (tt, J=6 and 6Hz, 2H, 4-H_2), 2.75-2.83 (m, 2H, 3-H_2 or 5-H_2), 2.99 (t, J=6Hz, 2H, 3-H_2 or 5-H_2), 5.11 (s, 1H, OH), 5.62 (s, 1H, 2-H), 6.83-7.39 (m, 3H, 6-H, 7-H and 8-H), 7.40-7.51(m, 4H, *p*-Br-Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 18.7 (t), 24.6 (t), 31.2 (t), 73.9 (s), 95.3 (d), 107.5 (d), 119.3 (d), 128.4 (d), 128.5 (d), 130.1 (s), 130.3 (d), 133.9 (s), 135.1 (s), 136.4 (s), 158.8 (s).

$C_{17}H_{15}BrO_2$ requires: C,61.63; H,4.53. Found: C,61.59; H,4.46.

2-(*p*-Nitrophenyl)-2a,3,4,5-tetrahydro-2H-naphtho[1,8-bc]furan-2a-ol 2c:

Compound **2c** was obtained as colorless crystals from benzene-hexane, mp 178°; ir (potassium bromide): 3330 cm^{-1} (OH^-), ^1H nmr (deuteriochloroform): δ 2.17 (tt, J=6 and 6Hz, 2H, 4-H_2), 2.72-2.86 (m, 2H, 3-H_2, or 5-H_2), 3.11 (t, J=6Hz, 2H, 3-H_2 or 5-H_2), 5.19 (s, 1H, OH), 5, 68 (s, 1H, 2-H), 6.82-7.40 (m, 3H, 6-H, 7-H and 8-H), 7.34-7.54 (m, 4H, *p*-NO_2-Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 19.1 (t), 24.7 (t), 31.2 (t), 74.3 (s), 65.6 (d), 107.4 (d), 119.3 (d), 128.6 (d), 128.7 (d), 130.4 (s), 130.6 (d), 133.9 (s), 135.4 (s), 136.5 (s), 159.2 (s).

$C_{17}H_{15}NO_4$ requires: C,68.69; H,5.05; N,4.71. Found: C,68.71; H,5.11; N,4.77.

***Cis*-2-cyano-2a,3,4,5-tetrahydro-2H-naphtho[1,8-bc]furan-2a-ol *cis* 2d:**

Compound ***cis*-2d** was obtained as colorless crystals from benzene-hexane, mp 113-114°, identical with an authentic sample[14] in the ir and nmr spectra.

***Trans*-2-cyano-2a,3,4,5-tetrahydro-2H-naphtho[1,8-bc]furan-2a-ol *trans* 2d:**

Compound ***trans*-2d** was obtained as coloreless crystals from benzene-hexane, mp 116-117°, identical with an authentic sample[14] in the ir and nmr spectra.

2-(*p*-chlorophenyl)-4,5-dihydro-3H-naphtho[1,8-bc]furane 3a:

Compound **3a** was obtained as a colorless oil, identical with authentic sample[14] in the ir and nmr spectra.

2-(*p*-Bromophenyl)-4,5-dihydro-3H-naphtho[1,8-bc]furane 3b:

Compound **3b** was obtained as colorless oil, ^1H nmr (deuteriochloroform): δ 2.01 (tt, J=6 and 6 Hz, 2H, 4-H_2), 2.85 (t, J=6Hz, 2H, 3-H_2 or 5-H_2), 3.01 (t, J=6Hz, 2H, 3-H_2 or 5-H_2), 6.90-7.43 (m, 5H, 6-H, 7-H, 8-H and *p*-Br-Ph-H_2), 7.51-7.72 (m, 2H, *p*-Br-Ph-H_2): ^{13}C nmr (deuteriochloroform): δ 22.7(t), 24.4 (t), 26.9 (t), 107.2 (d), 113.7(s), 119.5(d), 125.4 (d), 126.9 (d), 128.2 (s), 128.9 (d), 129.7 (s), 129.9 (s), 133.2 (s), 146.3 (s), 153.1(s).

$C_{17}H_{13}BrO$ requires: C,65.18; H,4.15. Found: C,64.93; H,4.11.

8-[(1-Hydroxy-1-(4-chlorophenylmethyl)]-1,2,3,4-tetrahydro-1-naphthalenone 4a:

Compound **4a** was obtaind as a colorless oil, identical with authentic sample[14] in the ir and nmr spectra.

8-[(1-Hydroxy-1-(4-bromophenylmethyl)-1,2,3,4-tetrahydro-1-naphthalenone 4b:

Compound **4b** was obtained as a colorless oil; ir (neat): 3995 (OH), 1670 cm^{-1} (Ar-CO); ^1H nmr (deuteriochloroform): δ 1.82-2.31 (m, 2H, 3-H_2), 2.45-2.66 (m, 2H, 2-H_2 or 4-H_2), 2.83-3.11 (m, 2H, 2-H_2 or 4-H_2), 5.18 (d, J = 6Hz, 1H, OH), 6.09 (d, J=6Hz, 1H, *p*-Br-PhC*H*OH), 7.07-7.69 (m, 7H, 5-H, 6-H, 7-H and *p*-Br-Ph-H_4); ^{13}C nmr (deuteriochloroform): δ 22.4 (t), 30.8 (t), 40.6 (t), 73.4 (d), 127.8 (d), 128.1 (d), 128.2 (d), 128.9 (d), 131.3 (s), 132.5 (s), 133.1 (d), 141.9 (s), 145.6 (s), 146.6 (s), 202.1 (s)

$C_{17}H_{15}BrO_2$ requires: C,61.63; H,4.53. Found: C,61.72; H,4.61.

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