

Synthesis of Symmetrical α -Alkyl- and α -Arylalkylbenzyl and Diarylmethyl Ethers by HCl-Catalyzed Intermolecular Dehydration

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Abstract— α -Alkyl, α -arylalkyl-, and α -aryl-substituted benzyl alcohols were converted into the corresponding symmetrical dibenzyl ethers in the presence of a catalytic amount of 10% aqueous HCl both in methylene chloride and under solvent-free conditions. Analogous reactions in dioxane and on heating afforded mainly the corresponding arylalkenes, whereas symmetrical dibenzyl ethers were formed as minor products.

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Alkyl and benzyl ethers having no substituent on the α -carbon atom are widely used in organic synthesis. The synthetic potential of ethers derived from sterically hindered benzyl alcohols such as α -alkyl- and α -arylphenylcarbinols has been poorly explored. However, there are published data indicating that α -substituted symmetrical benzyl ethers could be very useful in the synthesis of practically important and difficultly accessible compounds. For example, Friedel–Crafts benzylation of *o*-xylene with α -methylbenzyl trimethylsilyl ether [1] initially gives very rapidly and almost quantitatively symmetrical bis(α -methylbenzyl) ether which then generates α -methylbenzyl cation necessary for the benzylation of *o*-xylene (Scheme 1).

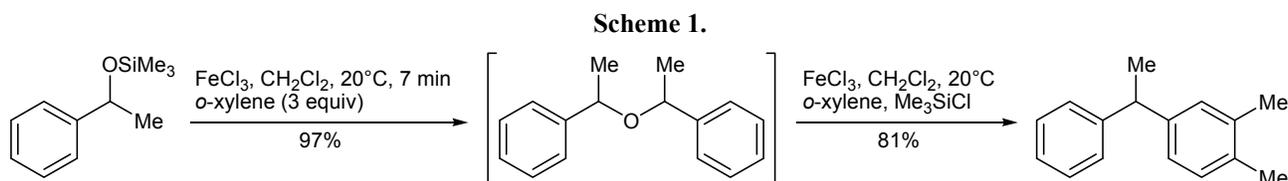
This example suggests that symmetrical α -substituted benzyl ethers specially synthesized by preparative methods can be used to obtain functionally substituted diphenyl- and triphenylmethanes which are practically important for organic chemistry.

Among other examples related to transformations of symmetrical ethers, so-called dismutation (disproportionation) of symmetrical bis(diphenylmethyl) ether A is worth noting (Scheme 2). Ether A is quantitatively

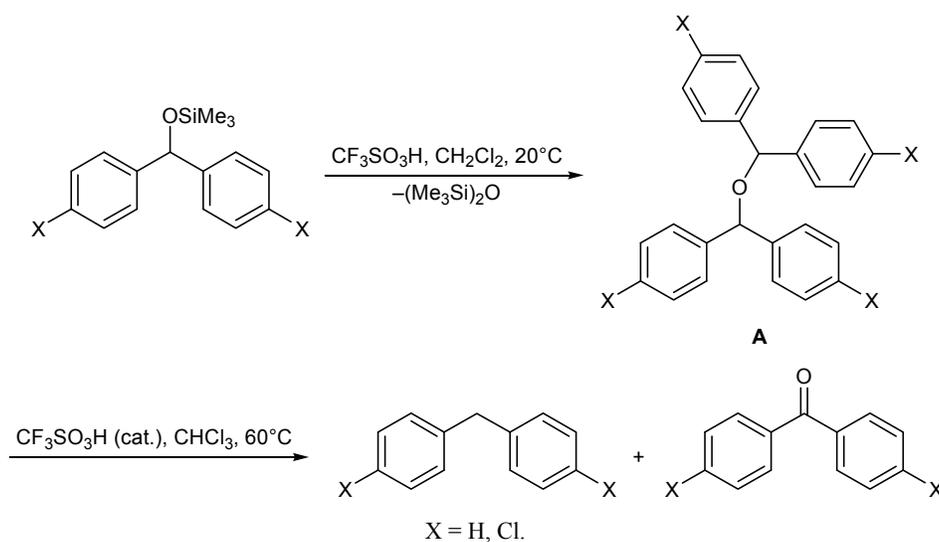
formed under mild conditions from diphenylmethyl trimethylsilyl ether [2], and it then decomposes to diarylmethane (reduction product) and diaryl ketone (oxidation product). Presumably, the use of such transformations of polyphenylated symmetrical ethers for synthetic purposes is not reasonable, but this undesirable process should be taken into consideration while synthesizing target ethers, especially from benzhydrols containing strong electron-donating substituents in the benzene ring (the corresponding ethers undergo disproportionation to diarylmethane and diaryl ketone even at 20°C) [2].

The results of [1, 2] provide a notion of prospects of synthetic application of symmetrical ethers derived from sterically crowded benzyl alcohols and thus stimulate development of methods for the preparation of such ethers.

Preparative procedures for the synthesis of symmetrical α -alkyl- and α -arylbenzyl ethers have been extensively developed over the past 10–15 years. These procedures are based mainly on acid-catalyzed intermolecular dehydration of the corresponding benzyl alcohols and catalytic reactions of their trialkylsilyl



Scheme 2.



ethers. Unlike Brønsted acid-catalyzed (H_2SO_4 , HCl , $\text{CF}_3\text{SO}_3\text{H}$) etherification of alkanols or unsubstituted benzyl alcohols, the above acids were almost not used to catalyze intermolecular dehydration of α -substituted benzyl alcohols. The only example of using trifluoromethanesulfonic acid ($\text{CF}_3\text{SO}_3\text{H}$) as catalyst in the reaction of benzhydrols with hexamethyldisiloxane was reported in [2]. Modified sulfuric acid as Brønsted acid catalyzed intermolecular dehydration of α -alkylbenzyl alcohols and benzhydrols. For instance, Shokrollahi et al. [3] used a carbon-based solid acid (a black powder obtained by high-temperature fusion of naphthalene with sulfuric acid) as catalytic system and presumed that its catalytic properties are determined by the presence of sulfo groups linked to carbon. Silica sulfuric acid was used to catalyze the transformation of diphenylmethyl trimethylsilyl ether to bis(diphenylmethyl) ether [4].

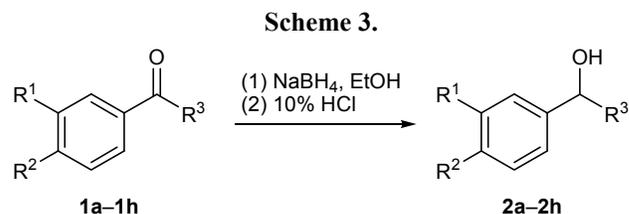
Lewis acids such as ZnCl_2 [5], $\text{BF}_3 \cdot \text{Et}_2\text{O}$ [6], and FeCl_3 [7] were also reported as catalysts in the synthesis of symmetrical α -substituted benzyl ethers, but in most cases etherification reactions were catalyzed by fairly complex catalysts, e.g., $\text{Cu}(\text{OSO}_2\text{CF}_3)_2$ [8], $(\text{MeS})_3\text{PAuCl}-\text{AgNTf}_2$ [9], $\text{CH}_3\text{Rh}(\text{VII})\text{O}_3$ [10], Li_2CuCl_4 [11], $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ [12]. It is quite obvious that the synthesis of symmetrical ethers in the presence of such catalysts [8–12] is more expensive; furthermore, there are problems related to purification of target products from the catalyst and regeneration of the latter for repeated use.

As shown in [13, 14], such halogenating agents as BiCl_3 and BiBr_3 are capable of initiating etherification

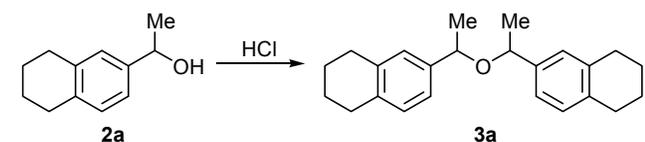
of α -substituted benzyl alcohols. It was presumed that the primary halogenation product, halo-substituted arylalkane reacts with the initial alcohol, affording symmetrical ether in high yield. The authors believed that this reaction may be considered a latent version of the Williamson reaction.

It seems somewhat surprising that nontrivial catalysts were used for the preparation of symmetrical α -substituted benzyl ethers by acid-catalyzed intermolecular dehydration [3–12]. We previously showed [15, 16] that hydrogen chloride, one of the simplest Brønsted acids, quite successfully catalyzes the synthesis of unsymmetrical ethers from benzyl alcohols, benzhydrols, and alkanols. Taking these results into account, we presumed that hydrochloric acid can also be used to catalyze transformation of sterically hindered benzyl alcohols to the corresponding symmetrical ethers. To verify this assumption, we synthesized a series of α -substituted benzyl alcohols by reduction of the corresponding aryl ketones (Scheme 3) and studied their behavior in the presence of HCl .

Initially, etherification of 1-(5,6,7,8-tetrahydronaphthalen-2-yl)ethanol (**2a**) by the action of 10%



$\text{R}^1\text{R}^2 = (\text{CH}_2)_4$, $\text{R}^3 = \text{Me}$ (**a**); $\text{R}^1\text{R}^2 = \text{OCH}_2\text{CH}_2\text{O}$, $\text{R}^3 = \text{Me}$ (**b**), Pr (**c**), *i*- Pr (**d**), PhCH_2 (**e**), Ph (**f**), $4\text{-ClC}_6\text{H}_4$ (**g**), *cyclo*- C_3H_5 (**h**).

Table 1. Etherification of alcohol **2a** by the action of hydrochloric acid

Run no.	Solvent	Temperature, °C	Reaction time, day	Yield of 3a , %	Recovery of 2a , %
1	–	20	40	32	67
2	–	20	75	95	2
3	–	60	0.2	11	88
4	–	60	0.6	48	51
5	–	60	1.2	97	–
6	CH ₂ Cl ₂	20	45	52	47
7	CH ₂ Cl ₂	20	90	88	9
8 ^a	Dioxane	100	0.2	9	36

^a The major product was 6-ethenyl-1,2,3,4-tetrahydronaphthalene (yield 51%).

aqueous HCl was studied. In all experiments, the molar ratio **2a**–HCl was 10:1. The results are collected in Table 1. It is seen that HCl does catalyze intermolecular dehydration of α -substituted benzyl alcohols to the corresponding symmetrical ethers. The etherification of **2a** under mild conditions (20°C) was equally efficient both in the absence of a solvent (run nos. 2, 5) and in methylene chloride which is commonly used as solvent for etherification of alcohols (run no. 7). The long reaction time is compensated by high chemoselectivity of the reaction which afforded symmetrical ether **3a** as the only product. In some cases, the reaction time can be shortened by raising the temperature (Table 1, run no. 5). However, it was not in all cases that elevated temperature accelerated etherification of α -substituted benzyl alcohols. The use of higher boiling solvents (e.g., dioxane) and higher temperatures resulted in reduced chemoselectivity, and mixtures of products were formed where the dehydration product of the initial alcohol predominated.

The conditions ensuring efficient etherification of **2a** (Table 1; run nos. 2, 5) were successfully applied to the synthesis of symmetrical ethers from α -alkyl-, α -arylalkyl-, and α -aryl-substituted benzyl alcohols **2b–2h** (Scheme 4). The data in Table 2 show that the yields of the target symmetrical ethers depend to an appreciable extent on the reaction duration and nature of the R substituent (Table 2; run nos. 4, 5, 11). It is important that the reaction chemoselectivity is retained. In all cases, symmetrical ethers **3b–3h** were formed as mixtures of *threo* and *erythro* isomers which can be separated by chromatography on Al₂O₃ (see Experimental).

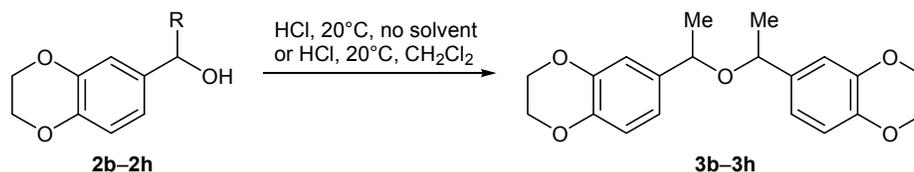
Thus, HCl-catalyzed etherification of α -alkyl-, α -arylalkyl-, and α -aryl-substituted benzyl alcohols under solvent-free conditions or in methylene chloride at 20°C may be regarded as a new, efficient, experimentally simple, and energy-saving preparative method for the synthesis of the corresponding symmetrical ethers.

EXPERIMENTAL

The ¹H NMR spectra were recorded on a Varian VXR-400 spectrometer at 400 MHz using CDCl₃ as solvent; the chemical shifts were measured relative to the residual proton signal of the solvent. The elemental analyses were obtained on a Vario-11 CHN analyzer. The melting points were measured with an Electrothermal 1A9100 digital melting point apparatus. The products were isolated by chromatography on Al₂O₃ of Brockmann activity grade II using diethyl ether–hexane mixtures at different ratios as eluent.

Initial benzyl alcohols **2a–2h** were synthesized by reduction of the corresponding ketones with NaBH₄ [17]. The spectra characteristics of **2b** [18] and **2c–2h** [15] were identical to those reported in the literature.

1-(5,6,7,8-Tetrahydronaphthalen-2-yl)ethanol (2a). Yield 93%. ¹H NMR spectrum, δ , ppm: 1.48 d (3H, CH₃, J = 6.3 Hz), 1.88 m (4H, 6-H, 7-H), 2.58 br.s (1H, OH), 2.82 m (4H, 5-H, 8-H), 4.78 q (1H, CHCH₃, J = 6.3 Hz), 7.02–7.08 m (3H, H_{arom}). Found,

Scheme 4.

R = Me (**b**), Pr (**c**), *i*-Pr (**d**), PhCH₂ (**e**), Ph (**f**), 4-ClC₆H₄ (**g**), *cyclo*-C₃H₅ (**h**).

Table 2. Etherification of benzyl alcohols **2b–2h** by the action of hydrochloric acid at 20°C

Run no.	Alcohol no.	Reaction time, day	Solvent	Yield of symmetrical ether, %	Unreacted alcohol, %
1	2b	20	–	3b (81)	2b (17)
2	2b	20	CH ₂ Cl ₂	3d (74)	2b (25)
3	2c	14	–	3c (88)	2c (11)
4	2c	14	CH ₂ Cl ₂	3c (81)	2c (19)
5	2d	14	CH ₂ Cl ₂	3d (55)	2d (43)
6	2e	14	CH ₂ Cl ₂	3e (71)	2e (28)
7	2f	30	–	3f (97)	–
8	2f	8	CH ₂ Cl ₂	3f (98)	–
9	2g	30	–	3g (94)	2g (5)
10	2g	30	CH ₂ Cl ₂	3g (98)	–
11	2h	6	CH ₂ Cl ₂	3h (51)	2h (47)

%, C 81.43, 81.61; H 8.95, 9.01. C₁₂H₁₆O. Calculated, %: C 81.77; H 9.15.

Etherification of alcohols 2a–2h in the presence of hydrochloric acid. *a.* A mixture of 0.01 mol of alcohol **2a–2h** and 0.3 mL (0.001 mol) of 10% aqueous HCl was kept at 20°C with intermittent stirring for a time indicated in Table 1 or 2. The mixture was treated with 30 mL of diethyl ether, the ether solution was washed with a 2 N solution of NaHCO₃, dried over MgSO₄, and evaporated, and the residue was analyzed by ¹H NMR. The products were isolated by chromatography on Al₂O₃.

b. Alcohol **2a–2h**, 0.01 mol, was dissolved in 15 mL of methylene chloride, 0.3 mL (0.001 mol) of 10% aqueous HCl was added, and the subsequent procedure was the same as in *a.*

6,6'-[Oxydi(ethane-1,1-diyl)]bis(1,2,3,4-tetrahydronaphthalene) (3a). Oily material. ¹H NMR spectrum, δ , ppm: *erythro* isomer: 1.41 d (6H, CH₃, $J = 6.6$ Hz), 1.90 m (8H, 2-H, 3-H), 2.84 m (8H, 1-H, 4-H), 4.21 q (2H, CHCH₃, $J = 6.6$ Hz), 7.03–7.07 m (6H, H_{arom}); *threo* isomer: 1.48 d (6H, CH₃, $J = 6.6$ Hz), 1.88 m (8H, 2-H, 3-H), 2.81 m (8H, 1-H, 4-H), 4.48 q (2H, CHCH₃, $J = 6.6$ Hz), 6.96–7.01 m (6H, H_{arom}). Found, % (for isomer mixture): C 85.85, 85.97; H 8.76, 8.92. C₂₄H₃₀O. Calculated, %: C 86.18; H 9.04.

6,6'-[Oxydi(ethane-1,1-diyl)]bis(2,3-dihydro-1,4-benzodioxine) (3b). Oily material. ¹H NMR spectrum, δ , ppm: *erythro* isomer: 1.35 d (6H, CH₃, $J = 5.9$ Hz), 4.17 q (2H, CHCH₃, $J = 5.9$ Hz), 4.28 s (8H, 2-H,

3-H), 6.76 d.d (2H, H_{arom}, $^3J = 8.5$, $^4J = 1.0$ Hz), 6.83 d (2H, H_{arom}, $^4J = 1.0$ Hz), 6.85 d (2H, H_{arom}, $^3J = 8.5$ Hz); *threo* isomer: 1.44 d (6H, CH₃, $J = 5.9$ Hz), 4.25 s (8H, 2-H, 3-H), 4.44 q (2H, CHCH₃, $J = 5.9$ Hz), 6.77 d.d (2H, H_{arom}, $^3J = 8.5$, $^4J = 1.0$ Hz), 6.79 d (2H, H_{arom}, $^4J = 1.1$ Hz), 6.83 d (2H, H_{arom}, $^3J = 8.5$ Hz). Found, % (for isomer mixture): C 69.72, 69.91; H 6.21, 6.32. C₂₀H₂₂O₅. Calculated, %: C 70.16; H 6.48.

6,6'-[Oxydi(butane-1,1-diyl)]bis(2,3-dihydro-1,4-benzodioxine) (3c). Oily material. ¹H NMR spectrum, δ , ppm: *erythro* isomer: 0.79 t (6H, CH₃, $J = 6.8$ Hz), 1.14 m (2H) and 1.35 m (2H) (CH₂CH₂CH₃), 1.47 m (2H) and 1.75 m (2H) (CH₂CH₂CH₃), 3.94 t (2H, CHCH₂, $J = 6.4$ Hz), 4.28 s (8H, 2-H, 3-H), 6.72 d.d (2H, H_{arom}, $^3J = 8.1$, $^4J = 1.0$ Hz), 6.78 d (2H, H_{arom}, $^4J = 1.2$ Hz), 6.83 d (2H, H_{arom}, $^3J = 8.1$ Hz); *threo* isomer: 0.89 t (6H, CH₃, $J = 6.8$ Hz), 1.29 m (3H) and 1.46 m (1H) (CH₂CH₂CH₃), 1.61 m (2H) and 1.77 m (2H) (CH₂CH₂CH₃), 4.19 t (2H, CHCH₂, $J = 6.4$ Hz), 4.22 s (8H, 2-H, 3-H), 6.61 d.d (2H, H_{arom}, $^3J = 8.1$, $^4J = 1.2$ Hz), 6.70 d (2H, H_{arom}, $^4J = 1.2$ Hz), 6.73 d (2H, H_{arom}, $^3J = 8.1$ Hz). Found, % (for isomer mixture): C 71.98, 72.09; H 7.26, 7.33. C₂₄H₃₀O₅. Calculated, %: C 72.34; H 7.59.

6,6'-[Oxydi(2-methylpropane-1,1-diyl)]bis(2,3-dihydro-1,4-benzodioxine) (3d). Oily material. ¹H NMR spectrum, δ , ppm: *erythro* isomer: 0.62 d and 0.97 d [6H each, CH(CH₃)₂, $J = 7.6$ Hz], 1.84 m [2H, CH(CH₃)₂], 3.54 d (2H, CHO, $J = 7.3$ Hz), 4.28 s (8H, 2-H, 3-H), 6.65 d.d (2H, H_{arom}, $^3J = 8.2$, $^4J = 1.4$ Hz),

6.76 d (2H, H_{arom} , $^4J = 1.4$ Hz), 6.81 d (2H, H_{arom} , $^3J = 8.2$ Hz); *threo* isomer: 0.74 d and 1.01 d [6H each, $\text{CH}(\text{CH}_3)_2$, $J = 7.6$ Hz], 1.98 m [2H, $\text{CH}(\text{CH}_3)_2$], 3.87 d (2H, CHO, $J = 7.3$ Hz), 4.21 s (8H, 2-H, 3-H), 6.51 d.d (2H, H_{arom} , $^3J = 8.2$, $^4J = 1.4$ Hz), 6.57 d (2H, H_{arom} , $^4J = 1.4$ Hz), 6.63 d (2H, H_{arom} , $^3J = 8.2$ Hz). Found, % (for isomer mixture): C 72.01, 72.18; H 7.31, 7.42. $\text{C}_{24}\text{H}_{30}\text{O}_5$. Calculated, %: C 72.34; H 7.59.

6,6'-[Oxydi(2-phenylethane-1,1-diyl)]bis(2,3-dihydro-1,4-benzodioxine) (3e). mp 141–142°C (isomer mixture). ^1H NMR spectrum, δ , ppm: *erythro* isomer: 2.77 d.d (2H, $J = 13.8$, 5.2 Hz) and 2.95 d.d (2H, $J = 13.8$, 8.8 Hz) (CH_2Ph), 4.16 d.t (2H, CHCH_2 , $J = 5.6$ Hz), 4.25 s (8H, 2-H, 3-H), 6.31 d.d (2H, H_{arom} , $^3J = 8.2$, $^4J = 2.0$ Hz), 6.45 d (2H, H_{arom} , $^4J = 2.0$ Hz), 6.65 d (2H, H_{arom} , $^3J = 8.2$ Hz), 7.08 m (4H, H_{arom}), 7.23 m (6H, H_{arom}); *threo* isomer: 2.79 d.d (2H, $J = 13.8$, 5.2 Hz) and 2.99 d.d (2H, $J = 13.8$, 8.8 Hz) (CH_2Ph), 4.23 s (8H, 2-H, 3-H), 4.29 d.t (2H, CHCH_2 , $J = 5.6$ Hz), 6.52 d.d (2H, H_{arom} , $^3J = 8.2$, $^4J = 2.0$ Hz), 6.62 d (2H, H_{arom} , $^3J = 8.2$ Hz), 6.64 d (2H, H_{arom} , $^4J = 2.0$ Hz), 6.97 m (4H, H_{arom}), 7.05 m (6H, H_{arom}). Found, % (for isomer mixture): C 77.26, 77.42; H 5.86, 5.97. $\text{C}_{32}\text{H}_{30}\text{O}_5$. Calculated, %: C 77.71; H 6.11.

6,6'-[Oxydi(phenylmethylene)]bis(2,3-dihydro-1,4-benzodioxine) (3f). mp 122–123°C (isomer mixture). ^1H NMR spectrum, δ , ppm: 4.25 s (8H, 2-H, 3-H), 5.32 s (2H, OCH), 6.83 s (4H, H_{arom}), 6.93 s (2H, H_{arom}), 7.32 m (2H, H_{arom}), 7.34 m (4H, H_{arom}), 7.39 m (4H, H_{arom}). Found, % (for isomer mixture): C 77.39, 77.06; H 5.58, 5.51. $\text{C}_{30}\text{H}_{26}\text{O}_5$. Calculated, %: C 77.24; H 5.62.

6,6'-[Oxydi(4-chlorophenylmethylene)]bis(2,3-dihydro-1,4-benzodioxine) (3g). mp 145–146°C (isomer mixture). ^1H NMR spectrum, δ , ppm: *erythro* isomer: 4.25 s (8H, 2-H, 3-H), 5.24 s (2H, CHO), 6.77 d.d (2H, H_{arom} , $^3J = 8.2$, $^4J = 1.9$ Hz), 6.83 d (2H, H_{arom} , $^3J = 8.2$ Hz), 6.85 d (2H, H_{arom} , $^4J = 1.9$ Hz), 7.28 s (8H, H_{arom}); *threo* isomer: 4.26 s (8H, 2-H, 3-H), 5.25 s (2H, OCH), 6.75 d.d (2H, H_{arom} , $^3J = 8.2$, $^4J = 1.9$ Hz), 6.82 d (2H, H_{arom} , $^3J = 8.2$ Hz), 6.86 d (2H, H_{arom} , $^4J = 1.9$ Hz), 7.29 s (8H, H_{arom}). Found, % (for isomer mixture): C 66.97, 67.06; H 4.38, 4.43. $\text{C}_{30}\text{H}_{24}\text{Cl}_2\text{O}_5$. Calculated, %: C 67.30; H 4.52.

6,6'-[Oxydi(cyclopropylmethylene)]bis(2,3-dihydro-1,4-benzodioxine) (3h). Oily material. ^1H NMR spectrum, δ , ppm: *erythro* isomer: 0.13 m (2H), 0.26 m (2H), 0.42 m (2H), 0.61 m (2H), and 1.14 m (2H) (C_3H_5); 3.24 d (2H, OCH, $J = 6.5$ Hz), 4.25 s (8H, 2-H,

3-H), 6.75–6.84 m (6H, H_{arom}); *threo* isomer: 0.15 m (2H), 0.38 m (2H), 0.45 m (2H), 0.65 m (2H), and 1.19 m (2H) (C_3H_5); 3.83 d (2H, OCH, $J = 6.5$ Hz), 4.26 s (8H, 2-H, 3-H), 6.77–6.86 m (6H, H_{arom}). Found, % (for isomer mixture): C 72.78, 72.87; H 6.42, 6.51. $\text{C}_{24}\text{H}_{26}\text{O}_5$. Calculated, %: C 73.08; H 6.64.

Reaction of alcohol 2a with hydrochloric acid in dioxane. Alcohol **2a**, 0.01 mol (1.76 g), was dissolved in 15 mL of dioxane, 0.3 mL (0.001 mol) of 10% aqueous HCl was added, and the mixture was refluxed for 5 h with stirring. The mixture was cooled, poured into 100 mL of water, and extracted with diethyl ether (2×30 mL). The extract was washed with a 2 N solution of NaHCO_3 and dried over MgSO_4 , the solvent was removed, and the residue was subjected to preparative thin-layer chromatography on Al_2O_3 using diethyl ether–hexane (1:4) as eluent. We isolated 0.81 g (51%) of 6-vinyl-1,2,3,4-tetrahydronaphthalene [^1H NMR spectrum, δ , ppm: 1.92 m (4H, 2-H, 3-H), 2.86 m (4H, 1-H, 4-H), 5.24 d (1H, $\text{CH}_2=$, $J_{\text{cis}} = 10.1$ Hz), 5.77 d (1H, $\text{CH}_2=$, $J_{\text{trans}} = 17.2$ Hz), 6.71 d.d (1H, $\text{CH}=$, $J = 17.2$, 10.1 Hz), 7.06–7.11 m (3H, H_{arom})], 0.15 g (9%) of symmetrical ether **3a** and 0.51 g of unreacted **2a**.

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