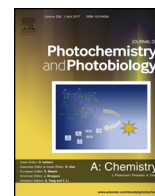




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Diastereoselective photocycloaddition reactions of 2-naphthalenecarboxylates and 2,3-naphthalenedicarboxylates with furans governed by chiral auxiliaries and hydrogen bonding interactions



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ABSTRACT

By using chiral auxiliaries and hydrogen bonding interactions, we have developed diastereoselective photocycloaddition of 2-naphthalenecarboxylates and 2,3-naphthalenedicarboxylates with furan derivatives. In photoreactions of (*l*)-menthyl 2-naphthalenecarboxylate with furan and 3-furanmethanol, respective maximum 48% and 40% diastereomeric excesses (d.e.) are observed. In photoreactions of di-8-phenyl-(*l*)-menthyl 2,3-naphthalenedicarboxylate with 3-furanmethanol, maximum 67% d.e. is obtained. Use of solvents of low polarity, low temperatures and low furan concentration leads to increased diastereoselectivities. Variable-temperature (VT) NMR and fluorescence quenching studies indicate that hydrogen bonding interactions between the carbonyl oxygen of naphthalenecarboxylic acid esters and the OH group in 3-furanmethanol take place in both the ground and excited states. The results of computational studies show that geometries of C₂ symmetric naphthalenedicarboxylate reactants are important in governing the high diastereoselectivity in the photoreactions of 2,3-naphthalenedicarboxylates.

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1. Introduction

Photocycloaddition reactions between unsaturated compounds such as alkenes, alkynes and arenes have been extensively studied and used for synthesis of various polycyclic compounds and natural products [1–8]. The nature of short lived exciplexes, which serve as reactive intermediates in these processes, control the efficiencies, and regio-, chemo- and stereo-selectivities of the photocycloaddition reactions [9,10]. As a result, π – π , dipole [11–13] and hydrogen bonding interactions [14–27] in the exciplexes, as well as structural constraints arising in intramolecular systems

[28–34] and in microcavities [35,36] have been used to govern the nature of these processes.

Photocycloadditions of cyanonaphthalenes with furan illustrate how dipole moments of exciplexes govern product distributions. For example, photoreaction of 1-cyanonaphthalene with furan generates predominantly an *endo*-[4+4] photocycloadduct [37–44], whereas the corresponding reaction of 2-cyanonaphthalene with furan produces mainly a cage product [39,41,42,44–46]. In an earlier study, we also demonstrated how dipole moments of 2-cyanonaphthalene derivatives influence the efficiencies of cage compound forming photoreactions with furan [47]. In addition, we showed that intramolecular photoreactions of cyanonaphthalene-furan linked compounds [48] and hydrogen bonding interactions in intermolecular photoreactions of cyanonaphthalenes with 3-furanmethanol [49] control the efficiencies and regioselectivities. The current study was designed to gain insight into how the stereoselectivities of photocycloadditions can be controlled by using chiral auxiliaries and hydrogen bonding interactions. As described below, we report the results of photoreactions of naphthalenecarboxylic acid esters bearing chiral auxiliaries with hydroxy-substituted furans.

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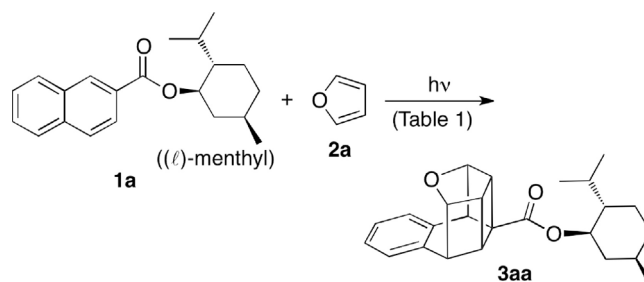
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2. Results and discussion

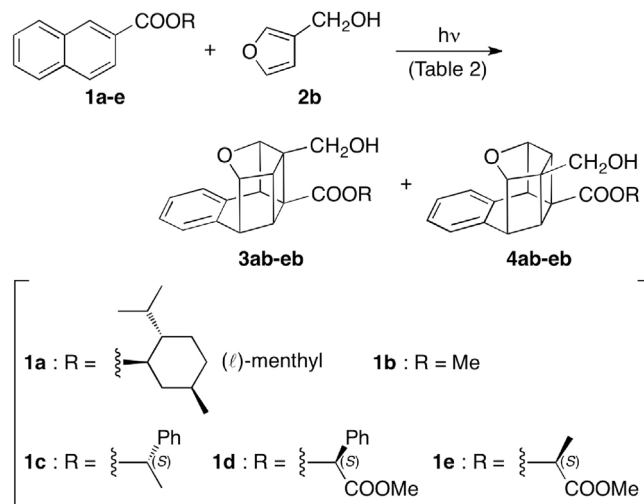
In the first phase of this investigation, the photocycloaddition reaction of the 2-naphthalenecarboxylate **1a**, possessing a (*l*)-menthylloxycarbonyl chiral auxiliary group, with furan (**2a**) was investigated focusing on its diastereoselectivity (Scheme 1, Table 1). Solutions containing **1a** and varying concentrations of **2a** in various solvents in Pyrex vessels, under an argon atmosphere and at varying temperatures were irradiated using a high-pressure mercury lamp. ¹H NMR and/or HPLC analysis of the crude product mixtures showed that photoreactions promote the cycloaddition to produce cage compound **3aa** in >90% yields with the diastereomeric excesses (d.e.) shown in Table 1. Pure **3aa** was obtained by concentration of the photolysates in vacuo followed by silica gel column chromatography.

Studies of solvent effects (entries 1–6) show that the highest d. e. (39%) is observed when the least polar solvent, pentane, is employed and that reactions in aromatic solvents such as benzene and toluene result in low d.e. values. When furan (**2a**) itself is used as solvent, a lower d.e. (23%) is obtained (entry 7). Dependence of the d.e. on the concentration of **2a** in pentane shows that an increase in the concentration of **2a** leads to a decrease in d.e. (entries 1, 8–10). A study of temperature effects (r.t. to –70 °C) on d. e. showed that lowering the temperature leads to an increase in d. e. (entries 11–13). Finally, we found that the highest d.e. (48%) is attained when the reaction is carried out at –70 °C using [**2a**] = 100 mM (entry 14).

The effect of the chiral ester group of 2-naphthalenecarboxylate on diastereoselectivities of photocycloaddition reactions with 3-furanmethanol (**2b**) was explored next. These photoreactions generate regioisomeric cage products **3** and **4** as mixtures of diastereomers (Scheme 2, Table 2). Owing to spectral overlap issues, the d.e. of only **4** can be determined by using ¹H NMR analysis (Fig. S1). Photoreaction of (*l*)-menthyl ester **1a** with **2b** in benzene produces **3ab** and **4ab** in >90% total yield (entry 1) and a **3ab/4ab** ratio of 1.20. The d.e. of **4ab** was determined to be 23%. In comparison, photoreaction of the simple methyl ester **1b** with **2b** produces **3bb** and **4bb** in >99% total yield and a **3bb/4bb** ratio of 1.40 (entry 2). However, reactions of esters containing other chiral auxiliaries such as (*S*)-1-phenylethyl (**1c**) and (*S*)-methyl mandelate derivative (**1d**) proceed to form the respective regioisomeric adducts **3** and **4** in <10% yields. Finally, methyl *D*-lactate derivative **1e** reacts with **2b** to form the corresponding regioisomers **3eb** and



Scheme 1. Photocycloaddition of (*l*)-menthyl 2-naphthalenecarboxylate (**1a**) with furan (**2a**).



Scheme 2. Photocycloaddition of 2-naphthalenecarboxylates **1a–e** with 3-furanmethanol (**2b**).

4eb in high total yield (>90%) and in a high **3eb/4eb** ratio of 2.16 (entry 5). However, the d.e. of **4eb** is low (15%) compared with that of **4ab** (entry 1). The results of a study of solvent effects, using pentane, hexane, cyclohexane, ethyl acetate and acetonitrile (entries 6–10), show that the highest d.e. for formation of regioisomer **4ab** (38%) occurs when the reaction is conducted in cyclohexane (entry 8). The temperature effect on photoreactions of

Table 1
Photocycloadditions of (*l*)-menthyl 2-naphthalenecarboxylate (**1a**) with furan (**2a**).^a

Entry	Solvent	ϵ^b	[2a] (mM)	Temperature (°C)	d.e. of 3aa ^c (%)
1	Pentane	1.8 ^d	50	r.t.	39
2	Cyclohexane	2.0 ^e	50	r.t.	36
3	Ethyl acetate	6.0 ^f	50	r.t.	19
4	Dichloromethane	8.9 ^f	50	r.t.	20
5	Benzene	2.3 ^f	50	r.t.	18
6	Toluene	2.4 ^e	50	r.t.	17
7	Furan	2.8 ^g	50	r.t.	23
8	Pentane	1.8 ^d	100	r.t.	36
9	Pentane	1.8 ^d	1000	r.t.	28
10	Pentane	1.8 ^d	2000	r.t.	28
11	Pentane	1.8 ^d	500	r.t.	29
12	Pentane	1.8 ^d	500	–20	32
13	Pentane	1.8 ^d	500	–70	35
14	Pentane	1.8 ^d	100	–70	48

^a 300 W high-pressure mercury lamp, Pyrex vessel, [**1a**] = 5 mM, conversion = 15–40% (¹H NMR and/or HPLC analysis), conversion yield >90% (GLC analysis).

^b Dielectric constant.

^c Determined by ¹H NMR and/or HPLC analyses.

^d Data from Ref. [50].

^e Data from Ref. [51].

^f Data from Ref. [52].

^g Data from Ref. [53].

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