Asymmetric Dearomatization Hot Paper

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Asymmetric Dearomatization of β-Naphthols through an Amination Reaction Catalyzed by a Chiral Phosphoric Acid**

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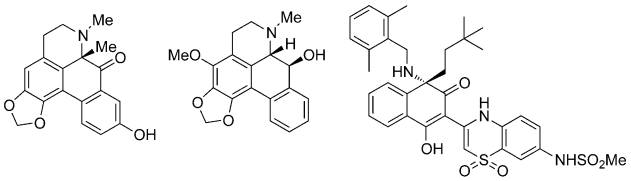
Abstract: *A highly efficient catalytic asymmetric dearomatization of naphthols by means of an electrophilic amination reaction catalyzed by chiral phosphoric acid is presented. This protocol provides a facile access to functionalized β-naphthalenone compounds with a chiral quaternary carbon center in excellent yields and enantioselectivity (up to 99% yield, up to 96% ee).*

The catalytic asymmetric dearomatization reaction (CADA) has emerged as a powerful organic transformation for the construction of complex molecules from relatively simple aromatic compounds.[1] Phenols are readily available, cheap, and versatile.[2] The asymmetric dearomatization of phenols and their derivatives provides chiral multifunctionalized cyclic enones, which frequently appear as a structural core in natural products.[3] In view of this synthetic utility, significant efforts have been devoted to the development of dearomatization reactions of phenols, including metal- and hypervalent-iodine-mediated oxidative dearomatization approaches,[4] transition-metal-catalyzed alkylative and arylative dearomatization,[5] and phase-transfer chiral-anion-catalyzed fluorinative dearomatization reactions.[6] Despite these elegant contributions, the catalytic asymmetric dearomatization of phenol and its derivatives is still in its infancy, and novel catalytic dearomative processes are highly desirable.

In the meantime, the enantioselective electrophilic α-amination of carbonyl compounds represents an attractive approach to construct optically active α-amino carbonyl scaffolds. Among the electrophilic amination reagents utilized, azodicarboxylates are preferred due to their high reactivity and subsequent versatile transformations.[7,8] In continuation of our efforts in exploring catalytic asymmetric dearomatization reactions, we envisioned that a chiral phosphoric acid might catalyze the asymmetric dearomative amination of substituted naphthols with azodicarboxylate. This dearomatization protocol could easily construct enantioenriched α-amino-β-naphthalenone skeletons with a chiral quaternary carbon center; such structural motifs are important components of various biologically active natural products and therapeutic reagents (Figure 1).[9]



(–)-*N*-methylguattescidine guatterine hepatitis C virus polymerase inhibititor

Figure 1. Examples of natural products and biologically active compound containing an α-amino-β-naphthalenone core.

We began our study by testing the reaction of 1,3-dimethyl-2-naphthol (**2a**) with diethyl azodicarboxylate (DEAD) under chiral phosphoric acid (**1**) catalysis.[10] To our delight, in the presence of 10 mol% (*S*)-**1a**, the reaction between **2a** and DEAD in toluene at room temperature proceeded smoothly to afford the desired product **4a** in 92% yield and 80% *ee* (entry 1, Table 1). Further evaluation of chiral phosphoric acids with different substituents and backbones indicated that all the reactions proceeded smoothly to form the desired dearomative product **4a** with variable enantioselective control (entries 1–6, Table 1). Notably, catalysts **1a**, **1c**, and **1f** provided product **4a** with the absolute configuration opposite to that induced by catalysts **1b**, **1d**, and **1e** (for details, see the Supporting Information). Among the catalysts screened, (*S*)-BINOL-derived **1e** and (*R*)-SPINOL-derived **1f** afforded comparable results in terms of yield and enantioselectivity (81% *ee*, 82% *ee*, entries 5 and 6, Table 1). It should be noted, the desired product was obtained in excellent yield but with no enantioselective control when the extremely bulky catalyst **1g** was employed. Screening of solvents was next carried out. Among the solvents evaluated, *o*-xylene and CCl_4 were found to be best. For instance, product **4a** with 87% *ee* was obtained in either *o*-xylene or CCl_4 when catalyst (*S*)-**1e** was utilized (entries 8 and 9, Table 1). Gratifyingly, excellent enantioselectivity was achieved in *o*-xylene and CCl_4 (95% *ee* and 96% *ee*, respectively) when the catalyst was switched from (*S*)-**1e** to (*R*)-**1f** (entries 10 and 11, Table 1) (for complete optimization of the reaction conditions, see the Supporting Information).

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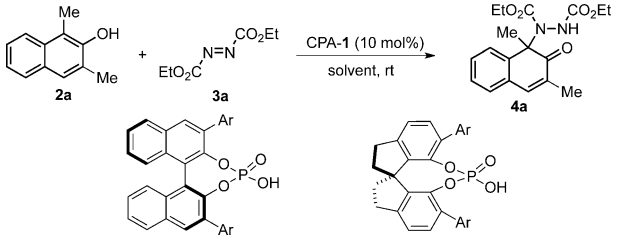
Table 1: Optimization of reaction conditions.[a] CPA = chiral phosphoric acid.

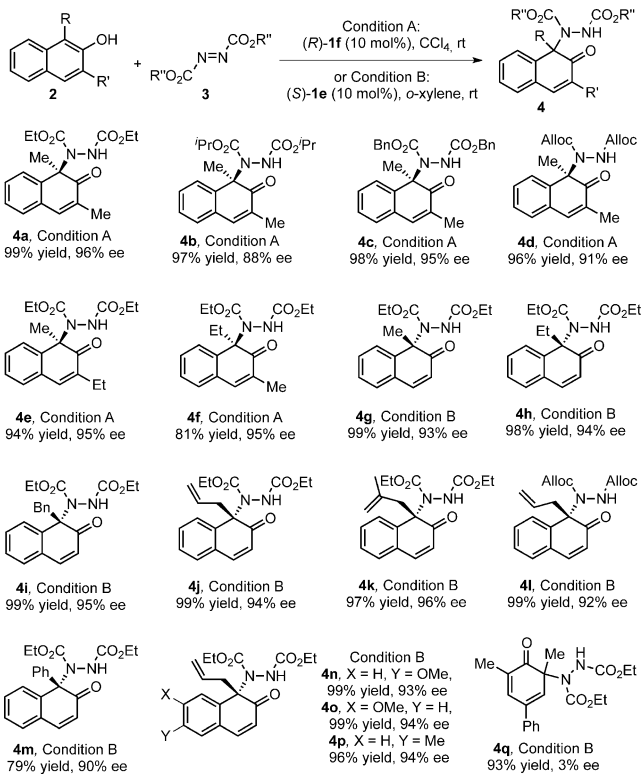


(*S*)-BINOL-CPA
1a, Ar = 3,5-$(CF_3)_2$-C_6H_3
1b, Ar = 2,4,6-(^{i}Pr)$_3$-C_6H_2
1c, Ar = 1-naphthyl
1d, Ar = 9-phenanthyl
1e, Ar = 9-anthryl

(*R*)-SPINOL-CPA
1f, Ar = 3,5-$(CF_3)_2$-C_6H_3
1g, Ar = 2,4,6-(^{i}Pr)$_3$-C_6H_2

Entry	1	Solvent	*t* [h]	Yield [%][b]	*ee* [%][c]
1	**1a**	toluene	4	92	−80
2	**1b**	toluene	4	97	25
3	**1c**	toluene	17	86	−10
4	**1d**	toluene	2	98	79
5	**1e**	toluene	2	98	81
6	**1f**	toluene	10	96	−82
7	**1g**	toluene	24	96	0
8	**1e**	*o*-xylene	1	98	87
9	**1e**	CCl_4	3	99	87
10	**1f**	*o*-xylene	36	99	−95
11	**1f**	CCl_4	36	99	−96

[a] Reactions were performed with **2a** (0.1 mmol), **3a** (0.15 mmol), and **1** (10 mol%) in 2.0 mL solvent. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.



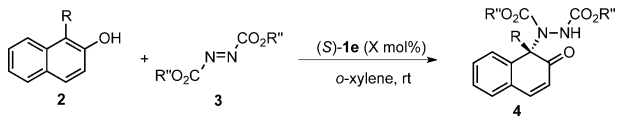
Scheme 1. Substrate scope.

Using the optimized reaction conditions (Conditions A: 10 mol% (*R*)-**1f**, 1.5 equiv **3**, room temperature, CCl_4), we next explored the scope of the dearomatization reaction. The results are summarized in Scheme 1. Firstly, the effect of the substituents on the azodicarboxylate was investigated by testing their reactions with 1,3-dimethyl-2-naphthol (**2a**). Different substituted (Et, *i*Pr, Bn, and allyl) azodicarboxylates were all well tolerated, and their corresponding dearomatized products were obtained in excellent yields and enantioselectivity (**4a–d**, 96–99% yield, 88–96% *ee*). With DEAD as the amination reagent, various substituted 2-naphthols were evaluated. In the presence of 10 mol% **1f**, both 1-methyl-3-ethyl-2-naphthol **2e** and 1-ethyl-3-methyl-2-naphthol **2f** were suitable substrates, affording the desired products **4e** and **4f** in excellent yields and enantioselectivity (81–94% yield, 95% *ee*). Moreover, 2-naphthols bearing no substituent at the 3 position, challenging substrates in asymmetric dearomatization reactions,[4i,5d] were also well tolerated. Product **4g** could be obtained in excellent yield and enantioselectivity under the following reaction conditions: Conditions B: 10 mol% (*S*)-**1e**, 1.5 equiv **3**, room temperature, *o*-xylene (99% yield, 93% *ee*, Scheme 1).

We further evaluated the effect of the substituents of the 2-naphthols by testing the reactions between 1-substituted 2-naphthols and azodicarboxylates under conditions B. Different aliphatic groups such as ethyl, benzyl, and allyl as well as 2-methylallyl could be well tolerated, and the corresponding products were obtained in excellent yields and enantioselectivity (**4h** to **4l**, 97–99% yield, 92–96% *ee*). To our delight, aryl-substituted substrate **2m** could be smoothly converted to the corresponding dearomative product **4m** in 79% yield and 90% *ee*. In addition, different groups on the aromatic ring of 2-naphthol such as 6-OMe, 7-OMe, and 6-Me were also well tolerated; the dearomatized products were generated smoothly with no erosion of yield and enantioselective control (**4n** to **4p**, 96–99% yield, 93–94% *ee*). It should be noted, with catalyst (*S*)-**1e** in *o*-xylene, **4d** was isolated with 82% *ee*, and the absolute configuration of **4d** was opposite to that obtained by using catalyst (*R*)-**1f**. After a single recrystallization of **4d** with 82% *ee*, enantiopure **4d** (> 99% *ee*) was obtained. The absolute configuration of **4d** obtained under conditions B was established as *S* by an X-ray crystallographic analysis.[11] It is noteworthy that the substituted phenol substrate was also suitable for this amination reaction, affording the corresponding dearomatized product smoothly in excellent yield in almost racemic form (**4q**, 93% yield, 3% *ee*).

Encouraged by the high efficiency of this asymmetric amination process, we further examined reactions with low catalyst loading. These results are summarized in Table 2. When the catalyst loading was decreased from 10 mol% to 0.05 mol%, comparable results were achieved for the reaction of **2g** with DEAD (96–99% yield, 90–93% *ee*, entries 1–5, Table 2). However, a further decrease of the catalyst loading to 0.01 mol% led to a drop in reactivity and enantiocontrol (90% yield, 70% *ee*, entry 6, Table 2). With 1 mol% of (*S*)-**1e**, the reactions of substrates **2h**, **2j**, and **2k** could proceed smoothly to provide the corresponding prod-

Table 2: Reaction with low catalyst loading.[a]

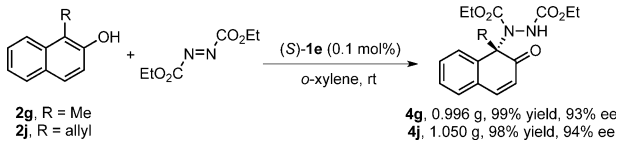


Entry	**2**, R	*X* [mol%]	Time	**4**, yield [%][b]	*ee* [%][c]
1	**2g**, Me	10	4 h	**4g**, 99	93
2	**2g**, Me	5	4 h	**4g**, 99	93
3	**2g**, Me	1	4 h	**4g**, 99	93
4	**2g**, Me	0.1	5 h	**4g**, 98	93
5	**2g**, Me	0.05	3.5 d	**4g**, 96	90
6	**2g**, Me	0.01	7 d	**4g**, 90	70
7	**2h**, Et	1	3.5 h	**4h**, 99	93
8	**2j**, allyl	1	1.5 h	**4l**, 99	92
9	**2k**, 2-methylallyl	1	4 h	**4k**, 97	96

[a] Reactions were performed with **2**, 1.5 equiv **3**, (*S*)-**1e** (*X* mol%) in *o*-xylene. [b] Yield of isolated product. [c] Determined by HPLC analysis on a chiral stationary phase.

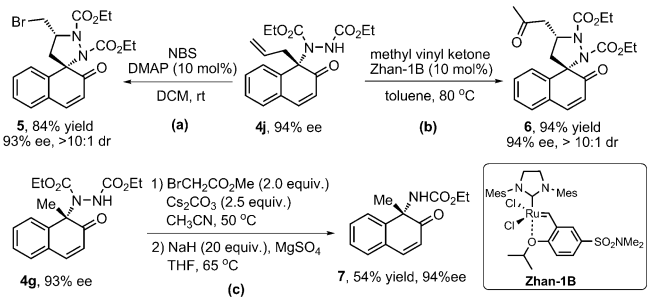
ucts in nearly quantitative yield and excellent *ee* (97–99% yield, 92–96% *ee*, entries 7–9, Table 2).

To evaluate the practicality of this catalytic process, gram-scale reactions of **2g** and **2j** were carried out. As shown in Scheme 2, products **4g** and **4j** could be obtained in excellent yield and enantioselectivity (93-94% *ee*) when 0.1 mol% of (*S*)-**1e** was utilized. The reaction under conditions A with 0.1 mol% catalyst **1f** also proceeded well to afford the corresponding product in excellent yield but with decreased enantioselectivity (62% *ee* for **4a**).



Scheme 2. Gram-scale reactions.

To further demonstrate the synthetic utility of this methodology, several transformations of the product were carried out. For instance, when product **4j** (94% *ee*) was subjected to electrophilic bromination conditions, an intramolecular bromoamination cyclization proceeded smoothly to provide spirocyclic compound **5** in 84% yield with good stereochemical integrity and high diastereoselectivity



Scheme 3. Transformation of the products. NBS = *N*-bromosuccinimide, DMAP = 4-dimethylaminopyridine, DCM = dichloromethane.

(93% *ee*, > 10:1 d.r., Scheme 3a). Furthermore, a cross-metathesis/aza-Michael addition cascade was also successfully carried out in the presence of 10 mol% Zhan-1B and 4 equivalents of methyl vinyl ketone; spirocyclic compound **6** was obtained in 94% yield without the loss of enantiomeric purity (94% *ee*, Scheme 3b).[12] Treatment of product **4g** (93% *ee*) with methyl bromoacetate/cesium carbonate in acetonitrile followed by N–N bond cleavage with sodium hydride afforded carbamate **7** in 54% yield with complete retention of enantiomeric purity (94% *ee*, Scheme 3c).[13]

In summary, we have developed the first catalytic asymmetric dearomatization of naphthols by means of an electrophilic amination reaction catalyzed by a chiral phosphoric acid. This protocol provides a facile and highly efficient access to functionalized β-naphthalenone compounds with an enantioenriched chiral quaternary carbon center in excellent yields and enantioselectivity. In addition, the combination of large-scale reactions, low catalyst loading, mild reaction conditions, and useful transformations of the product would warrant the synthetic utility of this methodology.

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[11] For more information of the crystallographic data of (*S*)-**4d**, see the Supporting Information. CCDC 1014270 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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