Asymmetric Catalysis

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Copper-Catalyzed Intramolecular Desymmetric Aryl C–O Coupling for the Enantioselective Construction of Chiral Dihydrobenzofurans and Dihydrobenzopyrans**

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Abstract: *O-Heterocyclic structures such as 2,3-dihydrobenzofurans are key motifs in many natural compounds and pharmaceuticals. Enantioselective formation of chiral dihydrobenzofurans and analogues was achieved through a copper-catalyzed desymmetrization strategy with a chiral cyclic 1,2-diamine. A broad range of substrates are compatible with this Cu^{I}-diamine catalytic system and afford the desired coupling products with chiral tertiary or quaternary carbon centers in high yields and good to excellent enantioselectivities under mild conditions.*

Oxygen-heterocycles such as 2,3-dihydrobenzofurans and chromans are common structures in a variety of naturally occurring and medicinally relevant compounds (Figure 1).[1–3] Although many catalytic systems employing transition-metal-catalyzed coupling reactions, such as couplings of aryl halides with oxygen nucleophiles catalyzed by Pd[4,5] and Cu[6,7], have been developed for the synthesis of such structures, asymmetric synthesis in this area is still a challenge.[2,8,9]

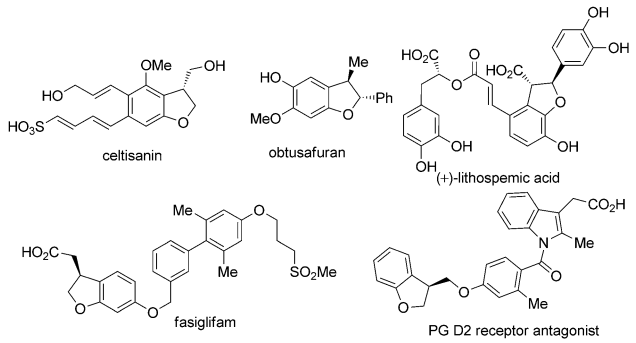

Figure 1. Examples of bioactive natural products or pharmaceuticals with 2,3-dihydrobenzofuran frameworks.

In 2013, we reported the first Pd-catalyzed enantioselective aryl C–O coupling reaction[9a] for the formation of central chirality in chromans through an asymmetric desymmetrization strategy,[10–12] which differentiates the two symmetric hydroxy groups of 2-(2-haloaryl)1,3-diols[13] by intramolecularly reacting with one aryl halide. However, such a method has the following limits to its practical applications: 1) only low to moderate yields were obtained in most cases due to β-H elimination and dehalogenation side reactions; 2) only moderate enantioselectivity was obtained due to the limited availability of chiral ligands; 3) substrates were restricted to those forming six-membered ring chromans. Asymmetric 5-membered cyclization afforded dihydrobenzofurans with only 50% yield and 50% *ee*. Furthermore, substrates were limited to those with tertiary prochiral centers. Substrates with a quaternary prochiral carbon center showed very low reactivity and poor enantioselectivity; and 4) the reaction conditions were relatively harsh.

Although an improved Pd catalytic system was later developed for the asymmetric desymmetrization of 2-(2-halophenoxyl)1,3-diols by employing a SDP(O) ligand,[9b] the efficiency of the system was limited to a narrow range of substrates and still only tertiary chiral carbon centers were formed in these reactions. Thus, it is highly desirable to develop a new catalytic system with a broader substrate scope and with high yield and enantioselectivity.

Copper catalysts have been complementary to Pd catalysts in cross-coupling reactions[14] and copper-catalyzed coupling reactions of aryl halides with alcohols or phenols are important methods for the formation of aryl alkyl ethers or diaryl ethers. Improved protocols for such reactions have been developed in recent years by using appropriate ligands to promote them under relatively mild conditions.[6,7] More importantly, dehalogenation in copper-catalyzed coupling reactions is not as prevalent as in palladium-catalyzed reactions, and this may offer an opportunity to improve the reaction efficiency. However, to the best of our knowledge, only one example of copper-catalyzed enantioselective aryl C–O coupling, generating axial chirality in the intramolecular diaryl ether formation, has been reported[15] and no copper-catalyzed aryl C–O coupling for the enantioselective formation of central chirality has been reported to date. In this communication, we would like to report a copper-catalyzed enantioselective aryl C–O coupling reaction, useful for the formation of 2,3-dihydrobenzofurans and analogues with tertiary and all-carbon quaternary stereochemical centers based on a desymmetrization strategy.

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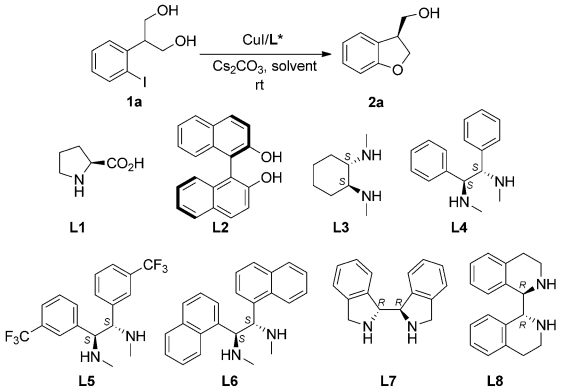
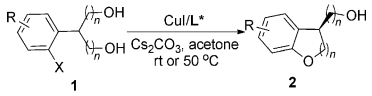
Table 1: Screening reaction conditions.

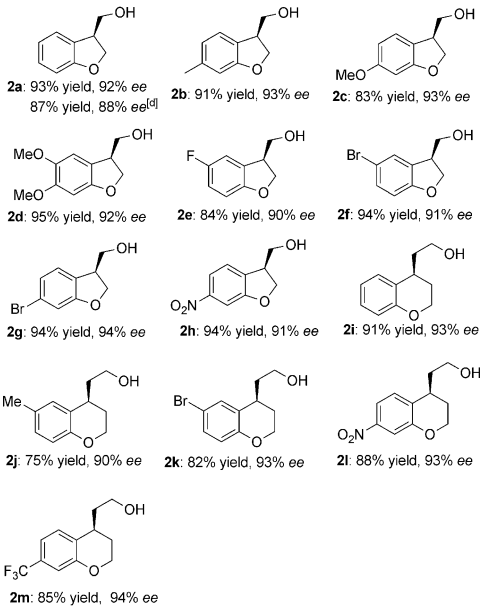


Entry	L*	Solvent	Base	Yield [%][b]	*ee* [%][c]
1	**L1**	MeCN	Cs_2CO_3	13	33
2	**L2**	MeCN	Cs_2CO_3	17	11
3	**L3**	MeCN	Cs_2CO_3	92	80
4	**L4**	MeCN	Cs_2CO_3	91	79
5	**L5**	MeCN	Cs_2CO_3	81	68
6	**L6**	MeCN	Cs_2CO_3	74	79
7	**L7**	MeCN	Cs_2CO_3	91	83
8	**L8**	MeCN	Cs_2CO_3	83	89
9	**L8**	DMF	Cs_2CO_3	86	90
10	**L8**	DMSO	Cs_2CO_3	80	80
11	**L8**	*t*BuOH	Cs_2CO_3	72	80
12	**L8**	toluene	Cs_2CO_3	15	35
13	**L8**	1,4-dioxane	Cs_2CO_3	76	85
14	**L8**	**acetone**	**Cs_2CO_3**	**93**	**92**
15	**L8**	acetone	K_3PO_4	37	85
16	**L8**	acetone	K_2CO_3	6	81

[a] Reagents and reaction conditions: **1a** (0.3 mmol, 1.0 equiv), CuI (0.03 mmol, 10 mol%), ligand (0.045 mmol, 15 mol%), Cs_2CO_3, (0.6 mmol, 2.0 equiv), solvent (1.5 mL), rt, 15 h. [b] Yield of isolated products. [c] Determined by HPLC analysis (Chira AD-H column).

Table 2: Substrate scope for the enantioselective formation of dihydrobenzofurans and chromans with tertiary stereocenters.[a,b,c]





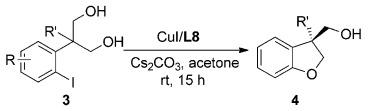
[a] Reagents and reaction conditions: **1** (0.3 mmol, 1.0 equiv), CuI (0.03 mmol, 10 mol%), **L8** (0.045 mmol, 15 mol%), Cs_2CO_3, (0.6 mmol, 2.0 equiv), solvent (1.5 mL), X = I, rt for $n = 1$, 50 °C for $n = 2$, 15 h. [b] Yield of isolated products. [c] Determined by HPLC analysis. [d] X = Br.

Our research was initiated with the model case of the reaction of 2-(2-iodophenyl)propane-1,3-diol (**1a**). As shown in Table 1, three types of chiral ligands, including an N,O-ligand [L-proline (**L1**)], an O,O-ligand [(*R*)-binol (**L2**)], and an N,N-ligand [N^1,N^2-dimethyl-cyclohexamine (**L3**)], were first screened (Table 1, entries 1–3) and **L3** showed much better results than **L1** or **L2** in both the yield and the enantioselectivity. The desired coupling product was obtained in 92% yield and 80% *ee* in MeCN at room temperature with Cs_2CO_3 as the base (Table 1, entry 3).[12] Several other chiral diamine ligands (**L4**–**L6**) were also screened and all led to enantioselectivity that was slightly inferior (Table 1, entries 4–6). The cyclic chiral diamine ligands **L7** and **L8** showed better results in enantioselectivity. **L8** gave the best result with 83% yield and 89% *ee* (Table 1, entry 8). Screening of solvents with the CuI/**L8** catalytic system revealed that a slight improvement of both yield and *ee* value was obtained in DMF and acetone (Table 1, entries 9 and 14). The best results of 93% yield and 92% *ee* were obtained in acetone (Table 1, entry 14). Two other bases K_3PO_4 and K_2CO_3 were also tested but gave inferior results (Table 1, entries 15 and 16). The absolute configuration of **2a** was assigned to be *S* by comparison with reported data.[13]

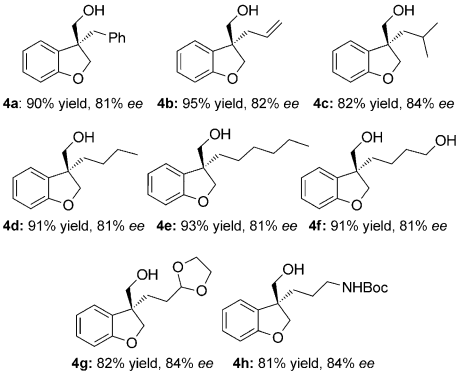
With the optimized conditions in hand, we explored the substrate scope. As shown in Table 2, a variety of 2-iodoaryl 1,3-diols delivered the corresponding coupling products with tertiary chiral centers in high yields and with excellent enantioselectivity. Both electron-donating and electron-withdrawing substituents on the aryl ring were well tolerated. In one example, the bromide substrate **1a′** was tested at room temperature and it produced the desired coupling product in 87% yield and 88% *ee*. Furthermore, the asymmetric cyclization of a class of 1,5-diol substrates also proceeded well at the slightly higher temperature of 50 °C to afford the desired six-membered chromans bearing tertiary chiral carbon centers at the 4-position (**2i**–**m**) in high yields and with excellent enantioselectivity. It should be noted that no dehalogenated by-products were detected in these reactions. The absolute configurations of 2,3-dihydrobenzofurans were assigned by analogy to that of **2a**, whereas the configurations of **2i**–**m** were assigned by comparison with that of **2k**, whose *S* configuration was confirmed unambiguously by X-ray crystallography.[16]

We next turned our attention to the enantioselective formation of quaternary stereochemical centers, especially all-carbon quaternary stereochemical centers, a field which is known to be one of the most challenging in asymmetric synthesis.[17,18] With this copper-catalyzed protocol, various

Table 3: Substrate scope for the enantioselective formation of 2,3-dihydrobenzofurans with quaternary stereocenters.[a,b,c]



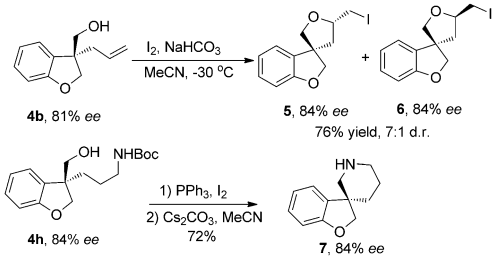
3 4



[a] Reagents and reaction conditions: **3** (0.3 mmol, 1.0 equiv), CuI (0.03 mmol, 10 mol%), ligand (0.045 mmol, 15 mol%), Cs_2CO_3, (0.6 mmol, 2.0 equiv), acetone (1.5 mL), rt, 15 h. [b] Yield of isolated products. [c] Determined by HPLC analysis.

substrates with quaternary prochiral carbon centers were explored. As shown in Table 3, all of the reactions proceeded very well under the same conditions and afforded the desired coupling products in high yields and with all carbon quaternary chiral centers having very good enantioselectivity. Functionalized alkyl groups such as allyl, hydroxy, acetyl, and amine were well tolerated, and may be useful for a variety of transformations. The absolute configuration of **4a** was determined by X-ray crystallography, and the absolute configurations of other products were assigned by comparing them to that of **4a**.

To explore the feasibility of such coupling reactions further, we tried several simple transformations of the products with quaternary stereocenters for the enantioselective formation of spirocyclic compounds. As shown in Scheme 1, the iodo cyclization reaction of **4b** afforded two separable O-spirocyclic compounds **5** and **6** in high yields and 7:1 stereoselectivity.[19] The cyclization of **4h** afforded a hetero-spiro compound **7** in high yield.[20] These spirocyclic structures may be used as important building blocks for further transformations in the synthesis of functional molecules.[21]



Scheme 1. Synthesis of chiral spirocyclic compounds through simple transformations.

In summary, we have discovered a copper-catalyzed, highly enantioselective, intramolecular aryl C–O coupling reaction for the formation of chiral 2,3-dihydrobenzofurans and analogues based on an asymmetric desymmetrization strategy. A wide range of substrates work well with this Cu^I/cyclic diamine ligand catalytic system under mild reaction conditions and afford the O-heterocyclic products in high yields and with high enantioselectivity. All-carbon quaternary stereocenters were also constructed through this strategy and the products were used for the synthesis of some spirocyclic compounds. Further exploration and application of this method in organic synthesis is in progress in our laboratory.

Keywords: asymmetric catalysis · copper · desymmetrization · 2,3-dihydrobenzofuran · O-arylation

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