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COMMUNICATION

I_2-Catalyzed N-sulfonylation of Sulfoximines with Sulfinates in Water at Room Temperature

Wenting Zheng,[a] Mingchao Tan,[a] Lu Yang,[c] Ratnakar Reddy Kuchukulla,[b] Lihong Zhou,[b] Qingle Zeng*[a]

Abstract: In the presence of catalytic amounts of I_2 and H_2O_2, oxidative coupling of NH-sulfoximines and arylsulfinates in water at room temperature under air has been developed. This protocol for synthesis of *N*-sulfonyl sulfoximines has merits including using water as a green and cheap solvent, requiring no metal catalyst or additive, mild conditions, a safe and simple operation, good yields, 100%ee product, and a wide substrate scope.

Since the discovery of the first sulfoximine compound, methionine sulfoximine (Figure 1), in 1950,[1] many sulfoximine derivatives with extensive bioactivities have been identified.[2] However, pesticide Sulfoxaflor, which has recently been approved by the United States Environmental Protection Agency (EPA) for distribution on the US market, is the first drug or pesticide containing a sulfoximine group to be approved for market (Fig. 1).[3] This progress regarding the safety of sulfoximine compounds will promote further research on this class of drugs and agricultural chemicals. Furthermore, sulfoximine derivatives can be used as chiral auxiliaries,[4] chiral ligands,[5] and organocatalysts in organic synthesis.[6] Therefore, more convenient synthetic methods for sulfoximine derivatives are in high demand. A facile synthetic approach to *N*-sulfonyl sulfoximines, a class of basic sulfoximine derivatives, would be beneficial for the study of sulfoximine derivatives.

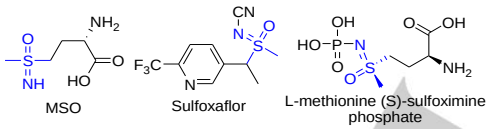

Figure 1. Some sulfoximine compounds.

The main methods for the synthesis of *N*-sulfonyl sulfoximines are as follows: (i) The sulfonylation of sulfoximines, involving the transformation of sulfoximines into their salts using a strong base, followed by reaction with TsCl (62% yield, 96% optical purity) (Scheme 1(a));[7] (ii) the oxidation of *N*-tosyl sulfilimines by *m*-chloroperoxybenzoic acid (55% yield, 98% optical purity)[7,8] or peroxyacetic acid under ruthenium tetroxide catalysis (Scheme 1(b));[9] (iii) the reaction of sulfoxides with TsN_3 under catalysis by copper powder (38%–65% yield, 96% optical purity) (Scheme 1(c));[7,10] (iv) the transformation of $ArSO_2NH_2$ into stable nitrene species $Ar'I{=}NSO_2Ar$ by hypervalent iodine(III) reagents $Ar'IX_2$, such as iodobenzene diacetate ($PhI(OAc)_2$) and iodosylbenzene (PhI=O), under catalysis by some transition metal salts, which then react with sulfoxides to afford the desired *N*-arylsulfonyl sulfoximines (organic sulfides can also be oxidized *in situ* to the corresponding sulfoxides) (Scheme 1(d));[11] (v) the $CuCl_2$-catalyzed imination of sulfoxides and sulfides using *N*-fluorobenzenesulfonamides (Scheme 1(e));[12] and (vi) a similar metathesis reaction of sulfoxides and *N*-tosyl sulfilimines at a high temperature of 190 °C under catalysis by copper metal powder, which affords the desired *N*-sulfonyl sulfoximine products accompanied by reduced sulfide side-products (Scheme 1(f)).[13] Nitrene species are generally recognized as the reactive intermediates involved in methods (iii)–(vi) above. These methods have some disadvantages, such as the use of moisture-sensitive metal sodium, explosive tosyl azide, toxic TsCl, stoichiometric and potentially explosive *m*-chloroperoxybenzoic acid or peroxyacetic acid, transition metal catalysts (such as Ru, Cu, Fe, and Ag), and/or a low atom economy (such as when using hypervalent iodine(III) reagents and peroxy acids), which do not conform with the principles of green chemistry.[14]

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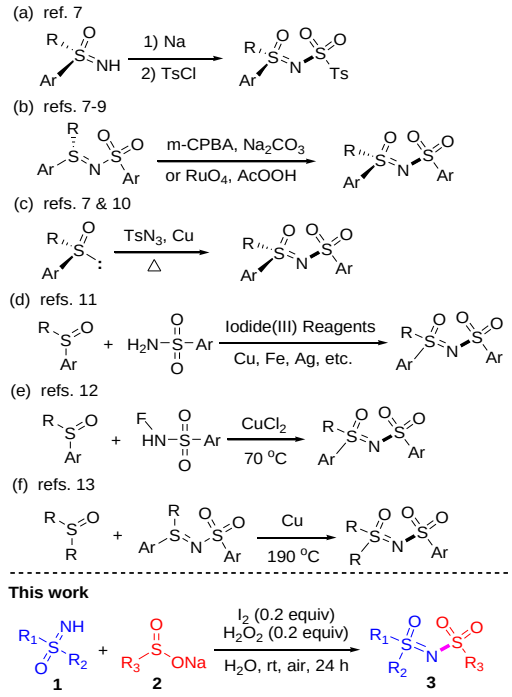


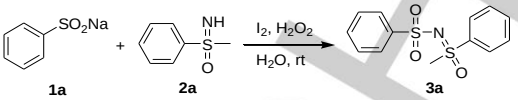
Scheme 1. Synthesis of N-sulfonyl sulfoximines.

Sodium arylsulfinate has also been reported to react with iodine (I_2) to form a sulfonyl iodide, followed by a radical reaction or N–H bond cleavage and S–N bond formation to afford the desired product.[15] Recently, iodine-mediated[16] or -catalyzed[17] reactions have become a hot research topic. A variety of experiments have confirmed that iodine can effectively replace transition metals as the catalyst in some organic reactions.[18] Furthermore, the use of abundant, safe, and economical water as the reaction solvent has attracted increasing attention in organic synthesis.[19]

As part of our ongoing studies on organosulfur chemistry,[20] carbon–heteroatom bond formations,[21] and green chemistry,[22] we have developed a green synthetic method for *N*-sulfonyl sulfoximines. In the presence of catalytic amounts of I_2 and H_2O_2, the *N*-sulfonylation of sulfoximines by alkylsulfinates in water under air at room temperature was achieved. Our protocol has many advantages over reported methods, such as improved green-chemistry merits (no transition metal catalyst, 0.2 equiv. of I_2 as catalyst, 0.2 equiv. of H_2O_2, and air as a green oxidant), simple conditions (room temperature), and easy and safe operation (no dangerous chemicals, such as sodium, no toxic chemicals, and no explosive azides).

Initially, we used sodium phenylsulfinate (**1a**) and methyl phenyl sulfoximine (**2a**) as model substrates to optimize the reaction conditions, with the results summarized in Table 1. Reactions using CuI and $CuBr_2$ as catalysts in different solvents were first tested, with no *N*-sulfonyl sulfoximine product detected (Table 1, entries 1–4). Fortunately, using *N*-bromosuccinimide (NBS) as a stoichiometric reagent in various solvents and at different temperatures afforded *N*-phenylsulfonyl methyl phenyl sulfoximine **3a** in low to moderate yields (Entries 5–8). Among different conditions, the NBS-mediated reaction in PEG 400 at room temperature gave the highest yield of 50% (Entry 7). A higher temperature of 60 °C diminished the NBS-mediated reactions (Entries 6 and 8 vs. 5 and 7).

Table 1. Optimization of reaction conditions.[a]



Entry	Catalyst (equiv)	Solvent	Temp (°C)	Time (h)	Yield (%)
1	CuI (0.1)	H_2O	90	10	0
2	CuI (0.1)	PEG_{400}	90	10	0
3	CuI (0.1)	CH_3OH	60	10	0
4	$CuBr_2$ (0.1)	DMSO	100	20	trace
5	NBS (1)	Dioxane	rt	20	42
6	NBS (1)	Dioxane	60	20	35
7	NBS (1)	PEG_{400}	rt	20	50
8	NBS (1)	PEG_{400}	60	20	37
9	I_2 (0.2)	PEG_{400}	rt	20	25
10[b]	I_2 (0.2)	PEG_{400}	rt	24	30
11[b]	I_2 (0.2)	THF	rt	24	0
12[b]	I_2 (0.2)	Toluene	rt	24	0
13[b]	I_2 (0.2)	H_2O	rt	24	81
14[b]	I_2 (0.2)	H_2O	60	24	<10
15	I_2 (0.5)	H_2O	rt	24	82

[a] Reaction conditions: **1a** (1 mmol), **2a** (1 mmol), I_2 (0.2 mmol), H_2O (1 mL) at room temperature (rt) for 24 h under air, unless otherwise mentioned. Isolated yields. [b] With H_2O_2 (0.2 mmol) as an oxidant.

Given that NBS may release toxic Br_2, molecular iodine was adopted to replace NBS. In order to find a greener process, a catalytic amount of I_2 (0.2 equiv) was used for this reaction, but low yield was obtained (Entry 9). Addition of an equivalent H_2O_2 to I_2 increased the yield of this reaction (Entry 10). Interestingly, the effect of solvents are dramatic (Entries 10-13). Non-protic solvents such as THF and toluene gave no product (Entries 11-12), perhaps because sodium phenylsulfinate (**1a**) cannot dissolve completely in these organic solvents. In order to increase the solubility of sodium phenylsulfinate (**1a**), water was adopted as solvent (Entry 13). Surprisingly, water afforded the highest yield of 81% among the above screened conditions (Entry 13). Due to the rapid consumption of I_2 and H_2O_2, higher temperature sharply cut down the yield (Entry 14). When the amount of I_2 was increased to 0.5 equiv and no H_2O_2 was added (Entry 15), the yield was 82%, which was marginally higher than that with 0.2 equiv I_2 and 0.2 equiv H_2O_2 (Entry 13). However, molecular iodine is somewhat expensive, and reducing the amount of I_2 is preferred for the isolation and purification process and our environment. Therefore, 0.2 equiv I_2 and 0.2 equiv H_2O_2 was chosen for the optimized condition.

As shown in Table 2, various sodium arylsulfinates reacted smoothly with methyl phenyl sulfoximine under the optimized conditions, giving the corresponding sulfonamides in moderate to good yields. Sodium benzenesulfinates with electron-donating alkyl groups, such as 4-methyl and 4-*tert*-butyl (**1b** and **1c**), afforded **3b** and **3c** with similar yields to that of **1a**. Electron-withdrawing groups on the sodium benzenesulfinates were not

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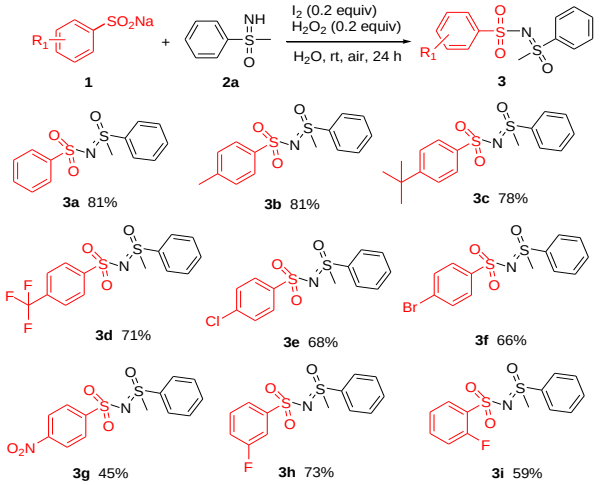
beneficial to the reaction (**3d**–**3i**). For *para*-substituents, weaker electron-withdrawing abilities led to higher yields of the desired products (**3d**–**3f** vs. **3g**), which might be due to stronger electron-withdrawing groups (**3g**) making the reaction intermediates unstable. Owing to steric hindrance, sodium benzenesulfinate bearing an *ortho*-fluoro group gave a lower yield compared with that bearing a *meta*-fluoro group (**3i** vs. **3h**).

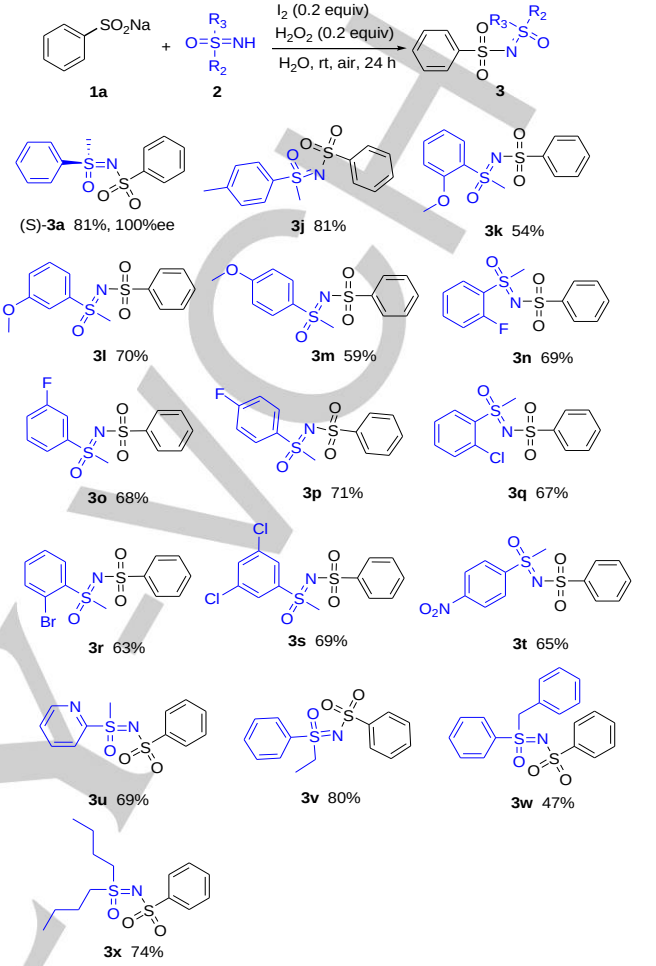
Table 2. Reaction of various substituted sodium phenylsulfinates with methyl phenyl sulfoximine



Next, we evaluated the reactions of various sulfoximines with sodium benzenesulfinate, as summarized in Table 3. Overall, the *N*-phenylsulfonylation of various sulfoximines proceeded smoothly, affording moderate to high yields of the products (**3a** and **3j**–**3x**).

Sulfoximines display chirality when two different groups are attached to the S atom. Enantiomeric purity is very important for chiral molecules, such as chiral drugs, natural products, agricultural chemicals, and perfumes.[23] Pleasingly, no racemization occurred during this process when enantiopure (*S*)-methyl phenyl sulfoximine was used, namely, affording (*S*)-*N*-phenylsulfonyl methyl phenyl sulfoximine ((*S*)-**3a**) with 100%ee and in the same yield as that obtained for the racemic compound.

Table 3. Reaction of various sulfoximines with sodium phenylsulfinate



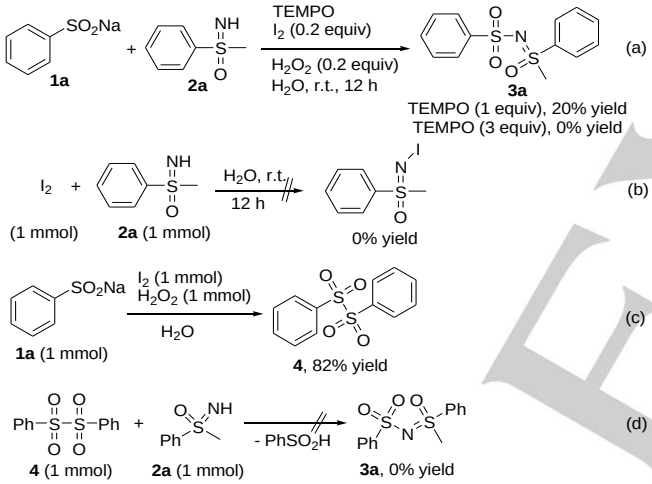
For aryl methyl sulfoximines with electron-donating groups on the benzene rings, weak electron-donating alkyl groups, such as methyl groups, gave the corresponding products (**3j**) in similar yields to that of the model substrate (**2a**). Meanwhile, strong electron-donating methoxy groups were disadvantageous for the reaction (**3k**–**3m**). Among the methoxy-substituted methyl phenyl sulfoximines, the *meta*-substituted substrate afforded the highest yield (**3l** vs. **3k** and **3m**).

The electron-withdrawing ability of substituents on aryl methyl sulfoximines seemed to have no major effect on the reaction yield (**3n**–**3t**), with even the sulfoximine bearing the strongly electron-withdrawing nitro group affording a 65% yield. All yields were in the range of 63%–71%. Interestingly, *ortho*-, *meta*-, and *para*-fluoro substituents had a marginal effect on the yield (**3n**–**3p**). Perhaps owing to the increase in bulk from F to Cl and then Br, the yields of substrates with these halo substituents decreased correspondingly (**3n**, **3q**, and **3r**). Notably, heteroaryl methyl sulfoximine was also tolerated in this reaction, affording a good yield (**3u**).

Ethyl sulfoximine (**3v**) achieved a similar yield to that of methyl sulfoximine (**3a**). Benzyl phenyl sulfoximine only afforded a moderate yield (**3w**), perhaps owing to the steric hindrance of the benzyl group. Interestingly, dialkyl sulfoximine and sodium

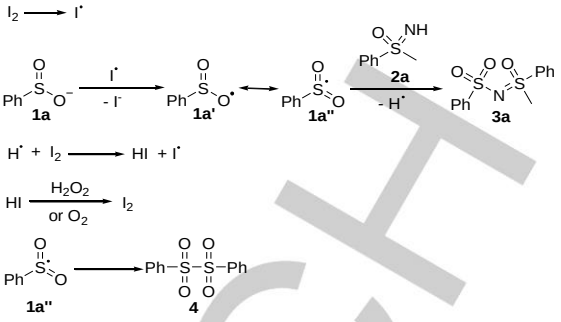
phenylsulfinate achieved *N*-phenylsulfonylation smoothly, affording a good yield (**3x**).

To obtain a better understanding of the mechanism, a control experiment was conducted using classical radical scavenger 2,2,6,6-tetramethylpiperidine *N*-oxide (TEMPO). With 1 equiv. of TEMPO, the reaction did not reach completion, perhaps because not only H_2O_2, but also air, might slowly restore molecular iodine over a long period (12 h). Excess TEMPO (3 equiv.) completely inhibited the reaction of methyl phenyl sulfoximine with sodium phenylsulfinate (Scheme 2, eq. a). This showed that this reaction involved free radicals. Next, we attempted to identify the radical intermediate involved. The reaction of I_2 and sulfoximine gave no product (Scheme 2, eq. b), and compound *N*-iodosulfoximine has never been reported, indicating that it might not be stable. Finally, under the optimized conditions without the addition of methyl phenyl sulfoximine **2a**, sodium benzenesulfinate self-coupling product **4** was obtained in 82% yield, providing analytical data consistent with that previously reported (Scheme 2, eq. c).[24] However, self-coupling product **4** did not react with methyl phenyl sulfoximine **2a** to give target product *N*-phenylsulfonyl methyl phenyl sulfoximine **3a** (Scheme 2, eq. d).



Scheme 2. Control reactions

Based on the above experimental results and some related reports,[25] we postulate a reasonable mechanism as shown in Scheme 3. Sodium phenylsulfinate exists in the form of phenylsulfinate anion in water. The phenylsulfinate anion (**1a′**) interacts with iodine to form a phenylsulfinyl radical (**1a**), which is converted into its tautomer phenylsulfonyl free radical (**1a″**). The phenylsulfonyl radical (**1a″**) react with methyl phenyl sulfoximine (**2a**) to give the desired product (**3a**) and to release H• radical, which promptly reacts with I_2 to form an HI and to restore I• radical. H_2O_2 or oxygen in air will oxidize HI into I_2. If there is no methyl phenyl sulfoximine (**2a**), the phenyl sulfonyl radicals will undergo a self-coupling reaction to form diphenyl disulfone (**4**).



Scheme 3. A proposed mechanism

In sumary, we have developed an oxidative coupling N–S bond formation process using NH-sulfoximines and sodium alkylsulfinates to synthesize *N*-sulfonyl sulfoximines in moderate to good yields. This oxidative coupling reaction was carried out in water under air at room temperature, providing a simple, practical, and green method for preparing *N*-sulfonyl sulfoximine derivatives with good functional group tolerance. A plausible mechanism was also proposed.

Experimental Section

A 25 mL test tube equipped with a magnetic stirrer was charged with sodium phenylsulfinate (**1a**, 164.16 mg, 1.0 mmol), methyl phenyl sulfoximine (**2a**, 155.22 mg, 1 mmol), H_2O_2 (0.2 equiv) and I_2 (50.8 mg, 0.2 mmol), and H_2O (2 mL). The reaction mixture was stirred at room temperature for 24 h. Then the reaction was quenched with sodium thiosulfate, and the resulting mixture was extracted with ethyl acetate (3 × 20 mL), and the combined organic layer was dried over anhydrous $MgSO_4$, filtered, and then condensed using a rotary evaporator. The residue was purified by column chromatography on silica gel with a mixture of petroleum ether and ethyl acetate as an eluent to afford the desired product *N*-phenylsulfonyl methyl phenyl sulfoximine **3a**.

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Keywords: oxidative coupling • green chemistry • sulfonylation • sulfoximines • sulfinates

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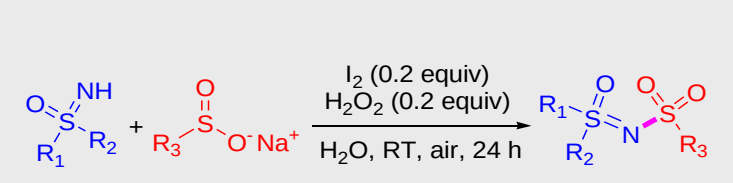

Mild N-sulfonylation of sulfoximines: In the presence of catalytic amount of I_2 and H_2O_2, N- sulfonyl sulfoximines were synthesized from NH-sulfoximines and sodium alkyl- and aryl-sulfinates in water under air at room temperature. Chirality of NH-sulfoximines are perfectly kept in products.

*Wenting Zheng, Mingchao Tan, Lu Yang, Lihong Zhou, Ratnakar Reddy Kuchukulla, Qingle Zeng**

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I_2-Catalyzed N-sulfonylation of Sulfoximines with Sulfinates in Water at Room Temperature