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Synthesis of *o*-Sulfinylanilines from *N*-Alkyl Sulfoximines and Arynes

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ABSTRACT: *N*-Alkyl sulfoximines react with *in-situ* generated arynes under mild conditions providing *o*-sulfinylanilines in good yields. The transformation is characterized by a broad substrate scope and a good functional group tolerance. The structure of a reaction product was confirmed by single crystal X-ray diffraction.

Aryne chemistry has widely been applied in constructing valuable organic molecules.[1] Among the products, those with a sulfur/nitrogen skeleton play a prominent role.[2-6] According to the number of reactants, the synthetic methods can be divided into two categories. The first includes two-component reactions where arynes insert into S–N bounds of compounds such as trifluoromethanesulfinamides,[2] sulfenamides[3], and sulfilimines.[4] The second category encompasses three-component reactions of arynes, sulfur compounds, and amines.[5] A recent example of the former category with relevance for our findings is a two-component reaction of arynes with *N*H-sulfoximines reported by

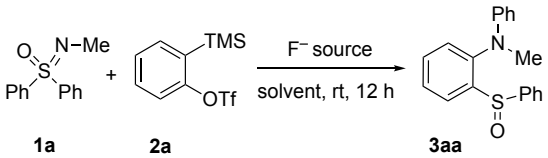
Hosoya and co-workers in 2017.[6a] While *S*-alkyl-*S*-aryl derivatives led to *N*-arylated products,[6] *S,S*-diarylsulfoximines afforded *o*-sulfinylanilines. However, the yields of the latter products remained modest and overall, the substrate scope was limited. Expecting a more uniform product formation, we started wondering about reactions between arynes and *N*-alkylated sulfoximines. If the latter reacted analogously as their *N*H-counterparts, one could also gain knowledge of potential degradation pathways of such compounds.[7] The results of this study are described here.

N-Alkyl sulfoximines can be prepared by various routes.[8,9] Due to their relevance in drug development,[10] we decided to focus our initial attention on the use of *N*-methyl derivatives.[11] In general, such compounds are stable, and only a few degradation pathways by S–C and S=N cleavage reactions are known.[12,13]

For the initial reactivity study and the optimization of the reaction conditions (on a 0.1 mmol scale), an equimolar mixture of *N*-methyl sulfoximine **1a** and *o*-silylaryl triflate **2a** in acetonitrile was treated with 2 equiv of CsF and stirred for 12 h at ambient temperature. To our delight, a selective reaction occurred leading to a 50% yield of the formal sulfinylamination product **3aa** (at 54% conversion of **1a** as determined by ^{1}H NMR spectroscopy using an internal standard, Table 1, entry 1). At a reaction temperature of 60 °C, the yield of **3aa** increased to 74% at 84% conversion of **1a** (Table 1, entry 2). Changing MeCN to THF, DCM, DCE, or toluene as solvents led to very low (conversions of **1a** and) yields of **3aa** (at ambient temperature, Table 1, entries 3-6). Next, the fluoride source was altered. Pleasingly, with a combination of KF/18-crown-6 instead of CsF, a 70% yield of **3aa** was achieved even at ambient temperature (Table 1, entry 7). Thus, heating was not required. Increasing the ratio of **1a** and **2a** from 1.0:1.0 to 1.0:1.5 and 1.0:2.0 raised the

yield from 70% to 81% and 84%, respectively (Table 1, entries 7-9). Under the latter condition, **1a** was fully consumed, and isolating the product by column chromatography afforded **3aa** in 81% yield (Table 1, entry 9). Hence, the optimized condition for this reaction involved the use of 2.0 equiv of both **2a** and KF/18-crown-6 in acetonitrile for 12 h at ambient temperature.

Table 1. Optimization of the Reaction Conditions[a]



1a **2a** **3aa**

entry	base	solvent	conversion of **1a** (%)[b]	yield of **3aa** (%)[b]
1	CsF	MeCN	54	50
2[c]	CsF	MeCN	84	74
3	CsF	THF	25	20
4	CsF	DCM	<5	N.D.
5	CsF	DCE	<5	N.D.
6	CsF	toluene	<5	N.D.
7	KF/18-crown-6	MeCN	71	70
8[d]	KF/18-crown-6	MeCN	93	81
9[e]	KF/18-crown-6	MeCN	100	84 (81)

[a]Reaction conditions: Use of 0.1 mmol of **1a**, 0.1 mmol of **2a**, and 0.2 mmol of fluoride source. [b]Yields of **3aa** and conversions of **1a** were determined by ^{1}H NMR analysis of the crude mixture with mesitylene as internal standard. The yield of **3aa** isolated by column chromatography is shown in parentheses (entry 9). N.D. = no desired product. [c]Performed at 60 °C. [d]Use of 0.15 mmol (1.5 equiv) of **2a**. [e]Use of 0.2 mmol (2.0 equiv) of **2a**.

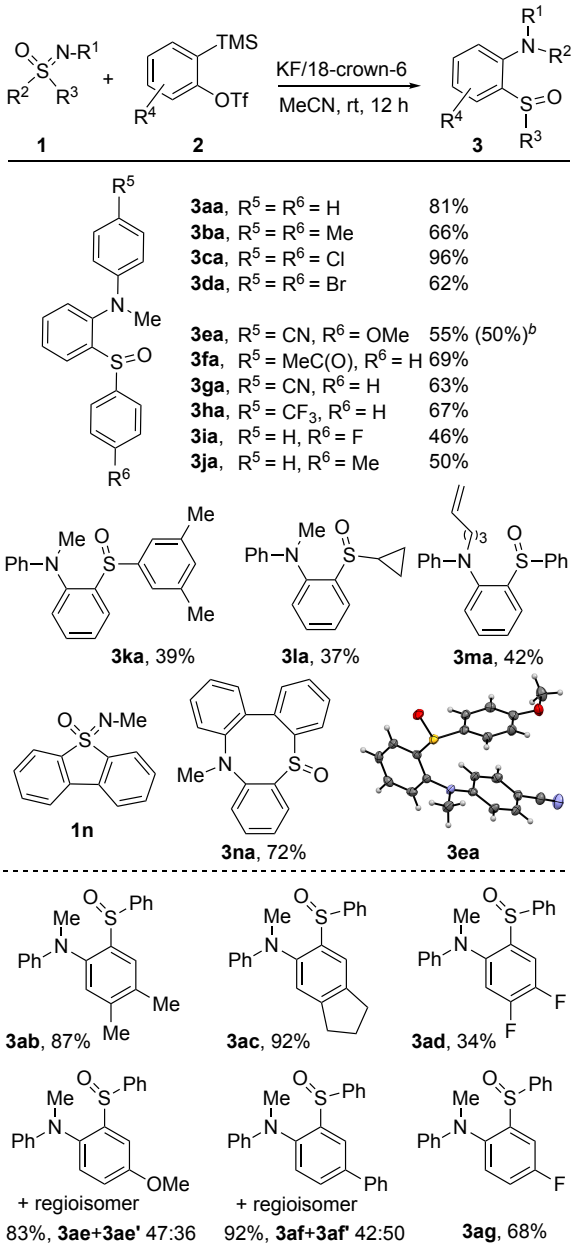
Under the optimized conditions, various substrate combinations were tested. The results are shown in Scheme 1. In the first series of experiments (leading to products **3aa**-**na**), aryne precursor **2a** was applied in combination with a range of *N*-alkyl sulfoximines **1**. First,

compounds with two identical *S*-aryl groups (R_2 = R_3) and an *N*-methyl substituent were used. The reactions proceeded well affording products **3aa**-**da** in yields ranging from 62% to 96%. Electronic effects induced by para-substituents on the arenes did not appear to play a significant role. Next, the behavior of unsymmetrically substituted *N*-methyl sulfoximines (**1e**-**j**) was studied. Although for those compounds, the yields of the resulting products **3ea**-**ja** were slightly lower (46-69%), an interesting electronic factor was found. In all cases except one, the more electron-rich aryl group retained at sulfur while the aryl with an electron-withdrawing group (or an H) migrated to the aniline nitrogen. The only exception was found in the formation of **3ia**, where for unknown reasons the unsubstituted arene moved to the nitrogen, whereas the 4-fluoro-bearing aryl remained connected to the sulfur atom. The comparably low yield of **3ka** (39%) resulting from the reaction of **2a** with *S*-3,5-dimethylphenyl-*S*-phenyl sulfoximine **1k** could be due to steric reasons. Noteworthy, selective phenyl-to-nitrogen migration was observed for this compound. The same was true for the formation of **3la** stemming from *S*-cyclopropyl-*S*-phenyl sulfoximine **1l**. With 37% the yield of **3la** was only moderate, but importantly, this result showed that also *S*-alkyl-*S*-aryl sulfoximine could be applied. The formation of **3ma** (in 42% yield) revealed that the *N*-substitution was not restricted to methyl groups. Here, a 5-pentenyl substituent was located at this position. Finally, starting from cyclic sulfoximine **1n** a ring expansion occurred leading to eight-membered thiazocine derivative **3na** in 72% yield. Performing the reaction of **1e** with **2a** on a 1 mmol scale, gave **3ea** in 50% yield. The solid-state structure of **3ea** was confirmed by X-ray crystal structure analysis.

In the next series of experiments, various *o*-silylaryl triflates (**2b**-**g**) were tested with *S*,*S*-diphenyl-*N*-methyl diphenyl sulfoximine (**1a**) as co-substrate. Those results are shown in Scheme 1 too. Now, electronic effects induced by substituents became apparent. Products

stemming from reactions with electron-rich arynes led to higher yields than those with electron-poor partners. Thus, for example, **3ab**, **3ac**, and **3ae**/**3ae'** were obtained in yields of 87%, 92%, and 83%, respectively. In contrast, **2d** and **2g** with electron-withdrawing

Scheme 1. Substrate Scope and X-Ray Crystal Structure of 3ea[a]



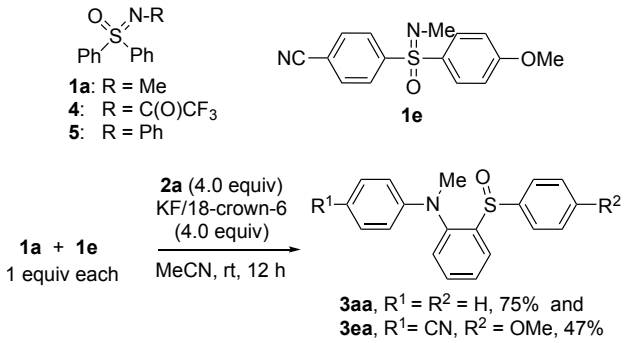
[a]Reaction conditions: **1** (0.1 mmol), **2** (0.2 mmol), KF/18-crown-6 (0.2 mmol). The yields refer to the amounts of products isolated by column chromatography. [b]For **3ea**, yield of a reaction on a 1 mmol scale in parentheses.

fluoro groups afforded the corresponding products in only 34% (for **3ad**) and 68% (for **3ag**) yield. Unsymmetrical arynes **2e** and **2f** led to mixtures of isomeric products **3ae**/**3ae'** and **3af**/**3af'** in good yields but disappointingly low regioselectivities. In contrast, and to our surprise, fluoro-substituted **2g** behaved differently, affording *o*-sulfinylaniline **3ag** in 68% yield as single regioisomer.

Further, control experiments were conducted to comprehend the details of the reaction. The importance of the *N*-alkyl group was revealed by applying sulfoximines **4** and **5** with non-aliphatic *N*-substituents (Scheme 2). Neither of the two substrates reacted emphasizing the importance of the *N*-alkyl substituent. This effect could not be compensated by an *N*-trifluoracetyl or *N*-phenyl group.

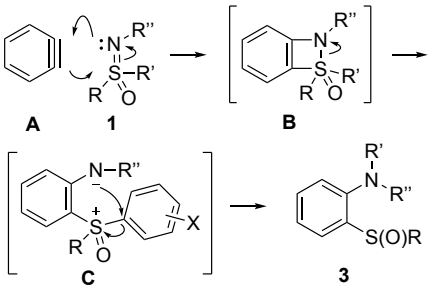
Next, a crossover experiment was carried out. Hence, a mixture of an equimolar amount of **1a** and **1e** was reacted with 4 equiv of **2a** and 4 equiv of the fluoride source. As a result, only **3aa** and **3ea** were observed in yields that were in line with the former experiments (75% for **3aa** and 47% for **3ea**). None of the potential cross-over products was detected suggesting that the aryl migrations from the sulfoximine sulfur to the aniline nitrogen proceeded in an intramolecular manner.

Scheme 2. Control Reactions



According to the experimental results and reported literatures,[4,6,14] a possible reaction path as shown in Scheme 3 can be proposed. Two points are important here: First, the product formation is intramolecular as shown by the cross-over experiments, and second, the aryl migration has a pronounced migratory preference. Thus, in the depicted scenario, *in-situ* formed aryne **A** (from **2**) reacts with sulfoximine **1** by a [2+2] cycloaddition-like pathway affording four-membered ring intermediate **B**. Subsequent S–N bond cleavage gives **C** which leads to product **3** by aryl migration. Thus, the process is intramolecular, and the high selectivity in the aryl migration is a result of a beneficial stabilizing effect of the negative charge by the arene bearing an electron-withdrawing group X.

Scheme 3. Suggested Reaction Path



In summary, we studied reactions of *N*-alkyl sulfoximines with *in-situ* formed arynes leading to *o*-sulfinylanilines. The product formation can be explained by a multistep reaction sequence initiated by a formal [2+2] cycloaddition between the S–N and C–C units of the starting materials. A subsequent bond-breaking process followed by a highly selective intramolecular relocation of an electronically preferred S-aryl group provides the products in good yields.

ASSOCIATED CONTENT

Data Availability Statement

The data underlying this study are available in the published article and its online Supporting Information.

The Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/.....

Experimental procedures, characterization data, NMR spectra for new compounds (PDF)

FAIR data, including the primary NMR FID files, for compounds **1a**-**n**, **2b**-**g**, **3aa**-**na**, **3ab**-**ag**, **4**, and **5**.

Accession Codes

CCDC 2229956 (for **3ea**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interests.

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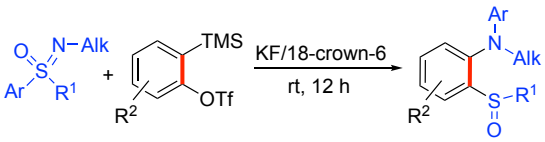

broad substrate scope

up to 96% yield