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ARTICLE

C-H Bond sulfonylation of anilines with the insertion of sulfur dioxide under metal-free conditions

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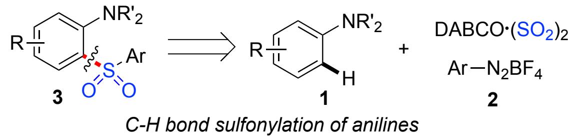
C-H Bond sulfonylation of anilines with the insertion of sulfur dioxide under metal-free conditions is described. 2-Sulfonylanilines are generated in moderate to good yields through a three-component reaction of anilines, DABCO·$(SO_2)_2$, and aryldiazonium tetrafluoroborates under mild conditions. No metal catalyst or additives are needed in this transformation. This direct C-H functionalization is highly efficient, and broad functional group tolerance is observed. A radical process is believed to be involved. In the reaction process, arylsulfonyl radical and tertiary amine radical cation generated in situ from DABCO·$(SO_2)_2$, and aryldiazonium tetrafluoroborate are the key intermediates. Additionally, tertiary amine radical cation acts as the electron carrier through a single electron transfer process.

Introduction

The importance of aromatic amines is known, since aromatic amines are commonly used in organic synthesis.[1] Moreover, aromatic amines are often used as effective targets in the design and synthesis of drugs and bioactive molecules.[2] Although there are numerous methods for the synthesis of aromatic amines, *ortho* C(sp^2)–H bond functionalization of aromatic amines under transition metal catalysis has became a powerful tool for the generation of diverse anilines.[3] Various metal catalysts such as Pd, Rh, Ir, Ru, *etc* have been used to facilitate the C–H bond activation or functionalization of aromatic amines. Recently, photoinduced C-H bond functionalization of aromatic amines through radical process has received continuous interests of chemists.[4] Various functional groups could be installed in the aromatic ring of anilines under mild conditions via radical process. For example, Willis and co-workers reported the sulfonylation of aniline derivatives with sulfinate salts through a photoredox-catalyzed reaction.[4a] Li and co-workers developed an intermolecular C–H bond alkylation of aniline derivatives with α-bromo ketones under visible-light catalysis.[4b] In the transformations, good regioselectivity favoring the *ortho*-position of anilines was observed

As part of a program in our laboratory for the synthesis of sulfonyl compounds via insertion of sulfur dioxide,[5] we are interested in the sulfonylation of anilines from sulfur dioxide through *ortho* C(sp^2)–H bond functionalization. In the past few years, the insertion of sulfur dioxide into small molecules has been recognized as an efficient route for the access to diverse sulfonyl compounds.[6-8] Among the approaches, C(sp^2)–H bond sulfonylation from sulfur dioxide has been developed by using DABCO·$(SO_2)_2$ as the SO_2 surrogate.[8] For instance, Yang and co-workers developed a copper-catalyzed C(sp^2)–H bond sulfonylation under base-free or ligand-free conditions starting from heteroaryls, sulfur dioxide, and aryl halides, providing diaryl sulfones in moderate to good yields.[8a] Direct C-H bond sulfonylation of indoles with the insertion of sulfur dioxide was achieved through a palladium-catalyzed reaction of 1-(pyridin-2-yl)indoles, DABCO·$(SO_2)_2$ and aryldiazonium tetrafluoroborates.[8c] Encouraged by these results, we envisioned that the synthesis of sulfonated anilines via C–H bond sulfonylation with the insertion of sulphur dioxide might be feasible.



Scheme 1. A proposed route for the synthesis of sulfonated anilines via C–H bond sulfonylation

Since anilines could be easily oxidized to radical cation via a single electron transfer (SET) under proper conditions,[4] we conceived that arylsulfonyl radical might be involved, thus accomplishing the synthesis of sulfonated anilines via C–H bond sulfonylation. In our previous report,[5a] we disclosed that arylsulfonyl radical could be easily generated *in situ* from the combination of DABCO·$(SO_2)_2$ and aryldiazonium tetrafluoroborates. Therefore, the preparation of diverse sulfonated anilines might be practicable from a three-

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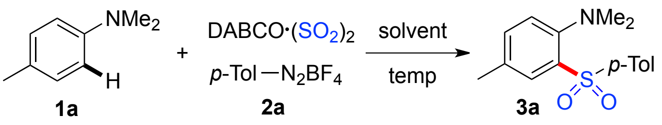
component reaction of anilines, sulfur dioxide, and aryldiazonium tetrafluoroborates (Scheme 1). Thus, we started to explore the feasibility of the C-H Bond sulfonylation of anilines with the insertion of sulfur dioxide through a three-component reaction of anilines, DABCO·$(SO_2)_2$, and aryldiazonium tetrafluoroborates.

Results and discussion

Initially, a model reaction of *N,N*-dimethyl-*p*-toluidine **1a**, DABCO·$(SO_2)_2$ and *p*-methylphenyldiazonium tetrafluoroborate **2a** was evaluated to optimize the reaction conditions. At the beginning, the reaction was performed in THF at 50°C without any catalysts or additives since the sulfonative reactions might occur under catalyst- and additive-free conditions according to our previous works.[6] However, only a trace amount of product was detected (Table 1, entry 1). A similar result was observed when the solvent was changed to 1,4-dixoane, DMF, or DMSO (Table 1, entries 2-4). To our delight, the desired 2-sulfonylaniline **3a** was obtained in 36% yield when the reaction took place in MeCN (Table 1, entry 5). A lower yield (22%) of product **3a** was generated (Table 1, entry 6) when the reaction occurred in 1,2-dichloroethane (DCE). We further explored the reaction at different temperatures. The result was inferior when the reaction was performed at room temperature (Table 1, entry 7). Gratifyingly, the corresponding product **3a** was formed in 49% yield when the reaction proceeded at 65 °C (Table1, entry 8). No better result was obtained when the reaction temperature was changed to 80 °C (Table1, entry 9). A slightly high yield was observed when the amount of DABCO·$(SO_2)_2$ was increased (Table 1, entry 10). Interestingly, the corresponding *N,N*,4-trimethyl-2-tosylaniline **3a** was generated in 90% yield when *N,N*-dimethyl-*p*-toluidine **1a** was added to the reaction mixture dropwisely during 5 min (Table 1, entry 11).

Table 1. Initial studies for the reaction of *N,N*-dimethyl-*p*-toluidine **1a**, DABCO·$(SO_2)_2$ and *p*-methylphenyldiazonium tetrafluoroborate **2a**. [a]

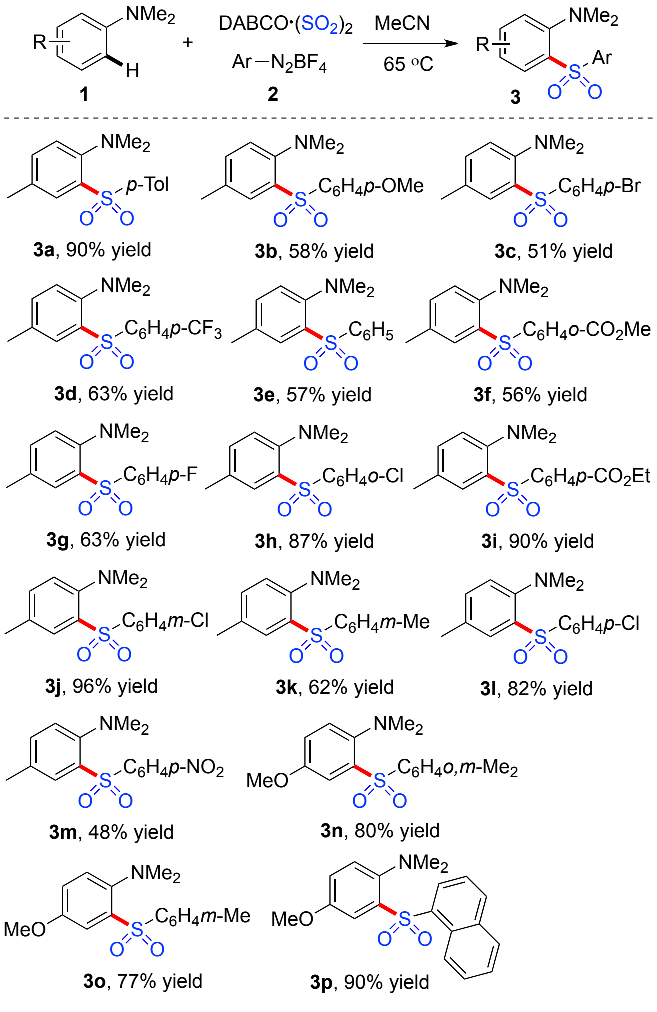


Entry	Temp (°C)	Solvent	Yield (%)[b]
1	50	THF	trace
2	50	1,4-dioxane	trace
3	50	DMF	trace
4	50	DMSO	trace
5	50	MeCN	36
6	50	DCE	22
7	25	MeCN	trace
8	65	MeCN	49
9	80	MeCN	44
10[c]	65	MeCN	53
11[c,d]	65	MeCN	90

[a] Reaction conditions: *N,N*-dimethyl-*p*-toluidine **1a** (0.2 mmol), DABCO·$(SO_2)_2$ (0.2 mmol), *p*-methylphenyldiazonium tetrafluoroborate **2a** (0.3 mmol), solvent (2.0 mL), N_2, 8 h. [b] Isolated yield based on *N,N*-dimethyl-*p*-toluidine **1a**. [c] In the presence of DABCO·$(SO_2)_2$ (0.3 mmol). [d] *N,N*-Dimethyl-*p*-toluidine **1a** was added dropwisely during 5 min.

The scope generality for the C-H bond sulfonylation of anilines with the insertion of sulfur dioxide was then explored. A range of *N,N*-dimethyl anilines **1** reacted with DABCO·$(SO_2)_2$ and aryldiazonium tetrafluoroborates **2** under the optimal reaction conditions, leading to diverse 2-sulfonylanilines in moderate to good yields. The results are shown in Table 2. This three-component reaction was efficient under metal- and additive-free conditions. It is noteworthy that different functional groups including fluoro, chloro, bromo, methoxy, trifluoromethyl, nitro, and ester were all compatible in this transformation. For example, nitro-substituded product **3m** could be afforded in 48% yield, and the ester-containing product **3i** was

Table 2. Scope exploration for the C-H bond sulfonylation of anilines with the insertion of sulfur dioxide [a]



[a] Isolated yield based on *N,N*-dimethyl aniline **1**.

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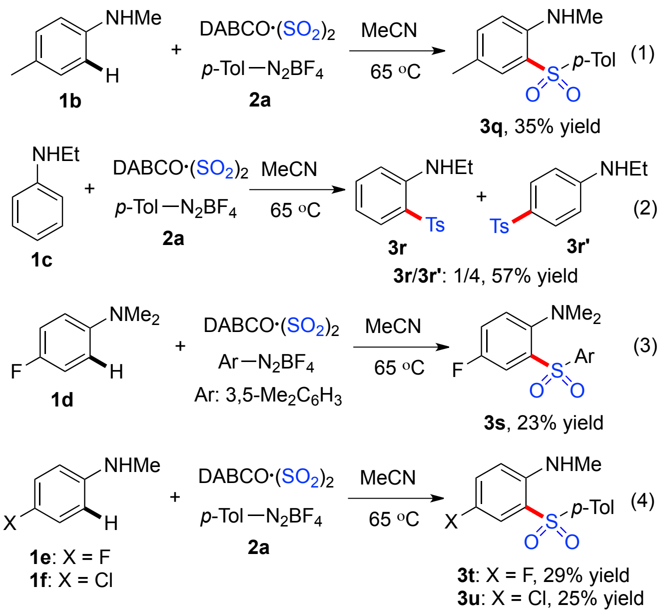
generated in 90% yield. For *N,N*-dimethyl anilines **1** with substitutions on the *para*-position, the C-H bond sulfonylation was exclusively occurred at the 2-position. In some cases, the yields were moderate. Since the presence of copper catalyst might assist the oxidative single electron transfer from radical intermediate to cation intermediate, 10 mol % of CuCl or $CuCl_2$ was added in the reaction of *N,N*-dimethyl-*p*-toluidine **1a**, DABCO·$(SO_2)_2$ and *p*-methoxyphenyldiazonium tetrafluoroborate **2b**. However, no better result was obtained, and the corresponding product **3b** was obtained in 46% and 42% yields, respectively.

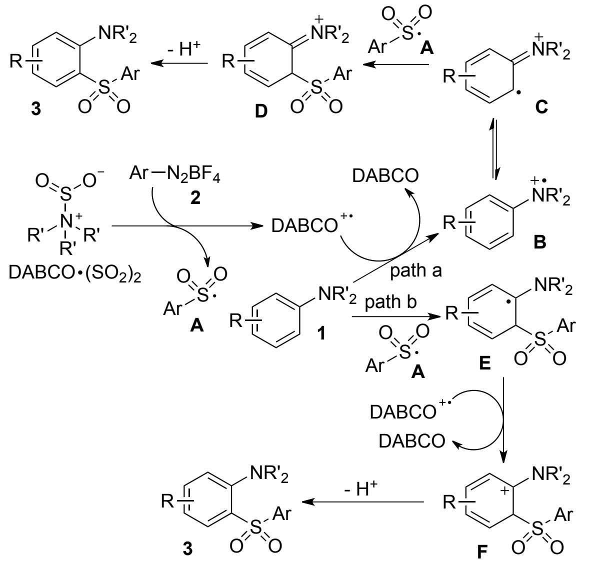
The substrate was further extended to *N*-methyl-*p*-toluidine **1b** and *N*-ethyl aniline **1c** in the reaction of DABCO·$(SO_2)_2$ with *p*-methylphenyldiazonium tetrafluoroborate **2a** (Scheme 2). Compound **3q** was obtained in 35% yield as expected (Scheme 2, eq 1). The formation of a mixture of products **3r** and **3r'** was observed when *N*-ethyl aniline **1c** was employed under the standard conditions (Scheme 2, eq 2). Anilines with electron-withdrawing groups were employed in the reactions as well. For instance, 4-fluoro-*N,N*-dimethyl aniline **1d** reacted with DABCO·$(SO_2)_2$ and 3,5-dimethylphenyldiazonium tetrafluoroborate, giving rise to the desired product **3s** in 23% yield (Scheme 2, eq 3). The corresponding products **3t** and **3u** were afforded in 29% and 25% yield respectively, when 4-fluoro-*N*-methyl aniline **1e** or 4-chloro-*N*-methyl aniline **1f** was used in the reaction of DABCO·$(SO_2)_2$ with *p*-methylphenyldiazonium tetrafluoroborate **2a** (Scheme 2, eq 4).



Scheme 2. Reactions of 4-methylphenyldiazonium tetrafluoroborate **2b**, DABCO·$(SO_2)_2$, with other anilines.

Subsequently, several control experiments were performed to gain more insights into the reaction mechanism. The radical scavenger of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (2.0 equiv) was added to the model reaction of *N,N*-dimethyl-*p*-toluidine **1a**, DABCO·$(SO_2)_2$ and *p*-methylphenyldiazonium tetrafluoroborate **2a** under the standard reaction shown in Table 2. As expected, the desired product **3a** was not detected. Furthermore, $K_2S_2O_5$ as inorganic sulfite or the gaseous sulfur dioxide was used as a replacement of DABCO·$(SO_2)_2$ in the reaction of *N,N*-dimethyl-*p*-toluidine **1a** with *p*-methylphenyldiazonium tetrafluoroborate **2a**. As a result, only a trace amount of product **3a** was observed. This evidence indicated the important role of DABCO·$(SO_2)_2$ in the reaction process.

On the basis of previous reports[4] and the experimental results mentioned above, a plausible mechanism was proposed as shown in Scheme 3. We envisioned that aryldiazonium tetrafluoroborate would react with DABCO·$(SO_2)_2$, giving rise to arylsulfonyl radical **A** and tertiary amine radical cation. Tertiary amine radical cation would go through a single electron transfer (SET) with aniline **1** to generate a radical cation **B**, with the release of DABCO (path a). Then radical cation **B** would undergo tautomerization to produce radical cation **C**, which would couple with arylsulfonyl radical **A** to generate intermediate **D**. The subsequent deprotonation and aromatization would provide the desired sulfonated aniline **3**. Alternatively, arylsulfonyl radical **A** would attack aniline **1** to produce radical **E** (path b). Subsequently, tertiary amine radical cation would be involved through a single electron transfer (SET) leading to cation intermediate **F**. Followed by deprotonation would produce the final product **3**.



Scheme 3. Plausible mechanism for the C-H bond sulfonylation of anilines with the insertion of sulfur dioxide

Conclusions

In conclusion, we have described a C-H bond sulfonylation of anilines with the insertion of sulfur dioxide under metal-free conditions. 2-Sulfonylanilines are generated in moderate to good yields through a three-component reaction of anilines, DABCO·$(SO_2)_2$, and aryldiazonium tetrafluoroborates under mild conditions. No metal catalyst or additives are needed in this transformation. This direct C-H functionalization is highly efficient, and broad functional group tolerance is observed. A radical process is believed to be involved. In the reaction process, arylsulfonyl radical and tertiary amine radical cation generated *in situ* from DABCO·$(SO_2)_2$, and aryldiazonium tetrafluoroborate are the key intermediates. Additionally, tertiary amine radical cation acts as the electron carrier through a single electron transfer process.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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