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# A Cation-Directed Enantioselective Sulfur-Mediated Michael/Mannich Three-Component Domino Reaction involving Chalcones as Michael Acceptors

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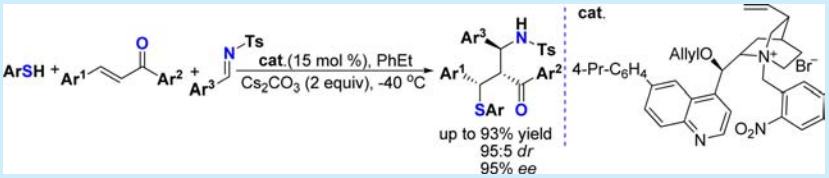
Supporting Information




**ABSTRACT:** A new approach has been developed for an asymmetric sulfur-mediated three-component intermolecular Michael/Mannich domino reaction using chalcones as Michael acceptors. This reaction is catalyzed by chiral quaternary ammonium salts derived from modified quinine and provides facile access to complex sulfur-containing compounds with three contiguous stereogenic centers in yields of up to 93%, with 95:5 *dr* and 95% *ee*. These compounds were further elaborated to give the equivalent of a chiral aza-Morita−Baylis−Hillman reaction involving chalcones and azetidines bearing four chiral centers.

Chiral sulfur-containing frameworks are present in a large number of bioactive natural products and synthetic pharmaceuticals, and considerable research efforts have therefore been devoted to the development of efficient methodologies for the stereoselective construction of S−C bonds.[1] These efforts have culminated in the development of a number of successful methods, including the two-component catalytic asymmetric sulfa-Michael addition of $\alpha,\beta$-unsaturated compounds (e.g., ketones,[2] amides,[3] esters,[4] nitroolefins,[5] sulfones and sulfonates[6]) and sulfa-Michael addition-triggered two-component domino asymmetric reactions,[7] as well as several other similar reactions.[8] Despite significant progress in this area, very few multicomponent sulfa-mediated asymmetric intermolecular domino reactions have been established involving three or more components. In fact, to the best of our knowledge, there have only been three reports in the literature concerning the development of three-component sulfa-initiated asymmetric domino (sulfa-Michael/Michael) reactions.[9] In 2005, Prof. Jørgensen presented the first sulfa-triggered multicomponent domino conjugated addition/amination reactions by using secondary amine as the catalyst.[9a] Subsequently, Melchiorre reported the primary amine catalyzed sulfa-Michael/amination reaction of $\alpha$-branched $\alpha,\beta$-unsaturated aldehyde.[9b] Recently, asymmetric sulfa-Michael/Michael reaction was also achieved by using a secondary amine catalyst.[9c] Thus, developing novel multicomponent sulfur-mediated asymmetric intermolecular domino reactions is highly desirable to provide facile access to versatile sulfur-containing chiral compounds with potentially interesting biological activities. As part of our ongoing programs toward the construction of complex sulfur-containing chiral compounds, we became very interested in the development of novel three-component intermolecular asymmetric sulfa-Michael/Mannich domino reactions.[10]

Chiral phase-transfer catalysts have been shown to be extremely useful in asymmetric transformations, where the stereocontrol is mainly dependent on the electrostatic attraction of two oppositely charged species.[11] Despite the asymmetric domino reactions representing an extremely powerful strategy for the rapid construction of complex molecular scaffolds in a stereocontrolled fashion,[12a] precedent has not yet been reported for asymmetric cascade reactions in the presence of a chiral phase-transfer catalysts. The lack of success in this area could be attributed to the fact that the ion-pairing interactions of these catalytic systems are inherently less directional than those of organocatalysts, where the stereoseletivity is mainly governed by covalent or H-bonding interactions. With this in mind, we endeavored to extend the application of chiral phase-transfer catalysts in asymmetric domino reaction. Herein, we report our preliminary results for the three-component intermolecular asymmetric sulfa-Michael/Mannich domino reactions using chiral phase transfer as catalysts, resulting in high yield with excellent diastereo- and enantioselectivities.

The reaction of **3a** with **4a** and **5a** was initially selected as a model reaction for the optimization of the reaction conditions. Large amounts of the products resulting from the two-component reactions of **3a** with **4a** and **3a** with **5a** were observed during the reaction, although preliminary optimization of the reaction conditions led to the formation of the desired product **6aa** in 69% yield with 89:11 *dr* and 12% *ee* when the reaction was

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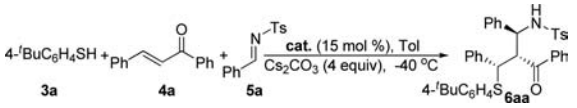
**Table 1. Catalyst Screening in the Asymmetric Sulfa-Michael/Mannich Domino Reaction**




| entry[a] | cat. | yield (%)[b] | *dr*[c,d] | *ee* (%)[d] |
|---|---|---|---|---|
| 1 | **1a** | 69 | 89:11 | 12 |
| 2 | **1b** | 83 | 94:6 | 79 |
| 3 | **1c** | 67 | 94:6 | 79 |
| 4 | **1d** | 83 | 94:6 | 84 |
| 5 | **1e** | 69 | 89:11 | 44 |
| 6 | **1f** | 72 | 93:7 | 80 |
| 7 | **1g** | 49 | 91:9 | <5 |
| 8 | **1h** | 81 | 94:6 | 84 |
| 9 | **1i** | 55 | 90:10 | 26 |
| 10 | **1j** | 65 | 92:8 | 50 |
| 11 | **1k** | 45 | 90:10 | <5 |
| 12 | **2a** | 82 | 95:5 | 88 |
| 13 | **2b** | 82 | 95:5 | 88 |
| 14 | **2c** | 82 | 94:6 | 86 |
| 15 | **2d** | 82 | 95:5 | 89 |
| 16 | **2e** | 64 | 95:5 | 60 |
| 17 | **2f** | 78 | 95:5 | 88 |
| 18 | **2g** | 83 | 95:5 | 89 |
| 19 | **2h** | 83 | 95:5 | 90 |
| 20 | **2i** | 83 | 95:5 | 89 |
| 21 | **2j** | 76 | 95:5 | 88 |
| 22 | **2k** | 80 | 95:5 | 89 |

[a]Reactions were carried out with **3a** (0.1 mmol), **4a** (0.1 mmol), **5a** (0.15 mmol), cat. (15 mol %), and $Cs_2CO_3$ (0.4 mmol) in 1.0 mL of toluene at −40 °C for 5 days. [b]Isolated yield. [c]Ratio of the major diastereomer to the total of the other three. [d]Determined by chiral HPLC.

catalyzed by **1a** in the presence of cesium carbonate as base at −40 °C in toluene (Table 1, entry 1). Inspired by this result, we proceeded to investigate the use of a series of alternative phase transfer catalysts (Figure 1), and the results of these experiments are summarized in Table 1.

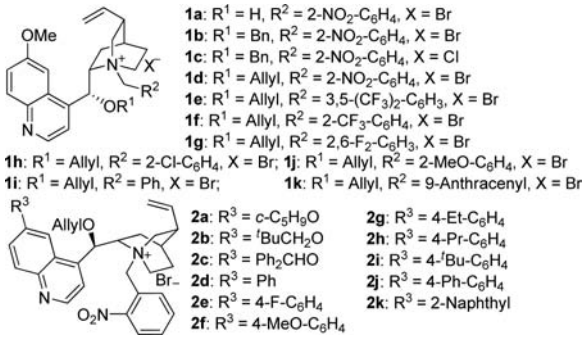



**Figure 1.** Phase-transfer catalysts used in this study.

The use of the benzylated phase-transfer catalyst **1b** led to a significant increase in the yield (83%) and stereoselectivity (94:6 *dr* and 79% *ee*) of the multicomponent reaction (Table 1, entry 2). However, changing the counterion from bromide to chloride (**1c**) led to a decrease in the reactivity, and the yields were reduced from 83% to 67% with the same good stereoselectivity (Table 1, entry 3). Changing the benzyl ether of the catalyst to allyl ether (**1d**) led to an increase in the enantioselectivity from 79% *ee* to 84% *ee*. It is well-known that the electronic and steric hindrance effects of the substituents on the aryl ring at $R^2$ of phase-transfer catalysts derived from quinine can have a significant influence on the performance of the catalyst. With this in mind, catalysts (**1e**−**1k**) incorporating various substituents on the aryl ring of $R^2$ were screened in this reaction. In general, catalysts bearing electron-withdrawing substituents (**1f**, **1h**) gave good yields and stereoselectivities (Table 1, entries 6 and 8). In contrast, catalysts with no substitution at $R^2$ (**1i**) or those bearing an electron-donating substituent at this position (**1j**) gave inferior catalytic performances (Table 1, entries 9 and 10). Both **1e** and **1k** provided only low levels of stereocontrol, which was attributed to the poor steric interaction between the substrate and the catalyst (Table 1, entries 5 and 11). Catalyst **1g** gave an almost racemic mixture of products, which might be attributed to the unfavorable anionic repulsion between the sulfur anion and F.[11f] The catalysts were further modified to improve its performance, and the MeO− moiety on the quinoline ring of **1d** was converted to *c*-$C_5H_9O$ (**2a**), $^t$BuCH$_2$O (**2b**), and Ph$_2$CHO (**2c**). These changes had very little impact on the reactivity or diastereoselectivity, but the enantioselectivity increased slightly from 84% *ee* to 88% *ee* (Table 1, entries 4 and 12−14). When Ph was installed as $R^3$ (**2d**), the enantioselectivity increased to 89% *ee* (Table 1, entry 15). Based on this result, we proceeded to investigate a series of catalysts (**2e**−**2k**) bearing a 4-substituted phenyl moiety as $R^3$ (Table 1, entries 16−22). The best result was achieved using **2h**, which gave the desired product in 83% yield, 95:5 *dr*, and 90% *ee* (Table 1, entry 19). Several other catalysts were also screened in this reaction, although they all performed less effectively than **2h** (see Supporting Information (SI)).

With the optimal catalyst in hand, we have optimized the reaction conditions (see SI), including the reaction temperature, catalysts loading, and the solvents. It was found that the best result was achieved when PhEt was used as the solvent at −40 or −45 °C in the presence of 10 or 15 mol % catalyst loads. The influence of several other parameters on the outcome of the reaction was also evaluated (see SI), including the type and amount of base, the molar ratio of the substrates, the concentration of the reaction, and the nature of the thiophenol nucleophile. The results of these experiments showed that the reaction proceeded smoothly when the ratio of **3a**:**4a**:**5a** was 1:1:1.5, the concentration of the reaction was 0.05 M, $Cs_2CO_3$ (2.0 equiv) was used as the base, and **3a** was used as the thiophenol.

With the optimized conditions in hand, we proceeded to investigate the scope of the reaction using a variety of different chalcones (Table 2). Pleasingly, the reaction exhibited broad substrate scope. In general, better enantioselectivities were achieved for chalcones bearing electron-donating substituents on their $Ar^1$ aromatic ring compared with those bearing electron-withdrawing substituents (Table 2, entries 8−12 vs 2−7). The substituent pattern of the $Ar^1$ ring was found to have a significant influence on the outcome of reaction, with 2-substituted systems giving much lower levels of reactivity and enantioselectivity. For example, when $Ar^1$ was 2-$FC_6H_4$, the desired product was formed in only 46% yield with 81% *ee*. In contrast, the corresponding substrates where $Ar^1$ was 3-$FC_6H_4$ and 4-$FC_6H_4$ gave much higher yields and stereoselectivities (Table 2, entries 2−4). Substituents at $Ar^2$ were also well tolerated, with the desired products being formed in good yields (Table 2, entries 13 and 14). The scope of reaction was then further expanded to include a range of imines (Table 3). Good enantioselectivities were



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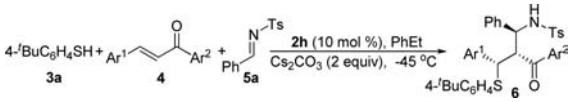
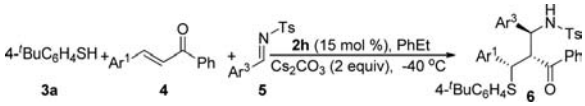
**Table 2. Scope of Chalcones Applied in the Asymmetric Domino Reaction**




| entry[a] | $Ar^1$, $Ar^2$ | $t$ (d) | yield of **6** (%)[b] | $dr$[c,d] | $ee$ (%)[d] |
|---|---|---|---|---|---|
| 1 | Ph, Ph | 7.5 | **6aa**, 73 | 98:2 | 92 |
| 2 | 2-$FC_6H_4$, Ph | 6.5 | **6ba**, 46 | 94:6 | 81 |
| 3 | 3-$FC_6H_4$, Ph | 5.5 | **6ca**, 80 | 96:4 | 91 |
| 4 | 4-$FC_6H_4$,Ph | 5.5 | **6da**, 90 | 95:5 | 90 |
| 5 | 4-$ClC_6H_4$, Ph | 5.5 | **6ea**, 75 | 94:6 | 88 |
| 6 | 4-$BrC_6H_4$, Ph | 6 | **6fa**, 80 | 91:9 | 90 |
| 7 | 4-$CF_3C_6H_4$, Ph | 6 | **6ga**, 76 | 93:7 | 86 |
| 8 | 3-$MeC_6H_4$, Ph | 6.5 | **6ha**, 90 | 94:6 | 91 |
| 9 | 4-$MeC_6H_4$, Ph | 10 | **6ia**, 90 | 95:5 | 93 |
| 10 | 3-$MeOC_6H_4$, Ph | 7.5 | **6ja**, 87 | 92:8 | 92 |
| 11 | 4-$MeOC_6H_4$, Ph | 6 | **6ka**, 74 | 94:6 | 99 |
| 12 | 4-$^iPrC_6H_4$, Ph | 6.5 | **6la**, 54 | 98:2 | 95 |
| 13 | Ph, 4-$MeC_6H_4$ | 7 | **6ma**, 70 | 97:3 | 94 |
| 14 | Ph, 4-$NO_2C_6H_4$ | 6 | **6na**, 71 | 88:12 | 63 |

[a]Reactions were carried out with **3a** (0.1 mmol), **4** (0.1 mmol), **5a** (0.15 mmol), **2h** (10 mol %), and $Cs_2CO_3$ (0.2 mmol) in 2.0 mL of PhEt at −45 °C. [b]Isolated yield. [c]Ratio of the major diastereomer to the total of the other three. [d]Determined by chiral HPLC.

**Table 3. Investigation of the Scope of Various Imines in the Asymmetric Domino Reaction**



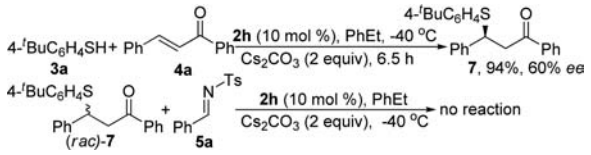

| entry[a] | $Ar^1$, $Ar^3$ | $t$ (d) | yield of **6** (%)[b] | $dr$[c,d] | $ee$ (%)[d] |
|---|---|---|---|---|---|
| 1 | 4-$MeC_6H_4$, 2-$MeC_6H_4$ | 4 | **6ib**, 36 | 95:5 | 91 |
| 2 | 4-$MeC_6H_4$, 3-$MeC_6H_4$ | 6 | **6ic**, 90 | 91:9 | 93 |
| 3 | 4-$MeC_6H_4$, 4-$MeC_6H_4$ | 8 | **6id**, 57 | 94:6 | 91 |
| 4 | 4-$MeC_6H_4$, 3-$MeOC_6H_4$ | 6 | **6ie**, 93 | 95:5 | 95 |
| 5 | 4-$MeC_6H_4$, 3-$FC_6H_4$ | 6 | **6if**, 63 | 95:5 | 90 |
| 6 | 4-$MeC_6H_4$, 4-$FC_6H_4$ | 6 | **6ig**, 93 | 94:6 | 92 |
| 7 | 4-$MeC_6H_4$, 4-$ClC_6H_4$ | 8 | **6ih**, 78 | 97:3 | 94 |
| 8 | 4-$MeC_6H_4$, 4-$BrC_6H_4$ | 10 | **6ii**, 65 | 96:4 | 94 |
| 9 | 4-$MeC_6H_4$, 2-napththyl | 7 | **6ij**, 57 | 97:3 | 94 |
| 10 | 4-$MeC_6H_4$, 4-$CF_3C_6H_4$ | 7 | **6ik**, 74 | 93:7 | 92 |
| 11 | 3-$MeC_6H_4$, 3-$MeOC_6H_4$ | 7 | **6he**, 80 | 93:7 | 93 |
| 12 | 3-$MeC_6H_4$, 3-$MeC_6H_4$ | 6 | **6hc**, 73 | 92:8 | 91 |
| 13 | 3-$MeOC_6H_4$, 3-$MeC_6H_4$ | 10 | **6jc**, 78 | 92:8 | 90 |
| 14 | 3-$MeOC_6H_4$, 3-$MeOC_6H_4$ | 8 | **6je**, 87 | 93:7 | 93 |

[a]Reactions were carried out with **3a** (0.1 mmol), **4** (0.1 mmol), **5** (0.15 mmol), **2h** (15 mol %), and $Cs_2CO_3$ (0.2 mmol) in 2.0 mL of PhEt at −40 °C. [b]Isolated yield. [c]Ratio of the major diastereomer to the total of the other three. [d]Determined by chiral HPLC.

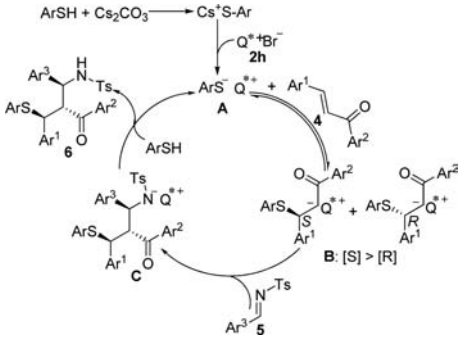
achieved irrespective of the presence of electron-donating or -withdrawing substituents on the aromatic ring of the imines (Table 3, entries 1−14). The substituent pattern also influenced the reactivity, and the best yields were obtained using meta-substituted imines (Table 3, entry 2 vs 1 and 3).

The absolute configuration of the products was determined and deduced by X-ray crystallographic analysis of **11** (see SI and Scheme 3). Control experiments have shown that an enantioselectivity of only 60% *ee* was achieved for the first step in the formation of the addition product of **7** under otherwise identical conditions. When racemic **7** was treated with **5a** under the same catalytic conditions, no reaction was observed (Scheme 1). Based on these results, we have proposed a mechanism for this

**Scheme 1. Control Experiment Results**



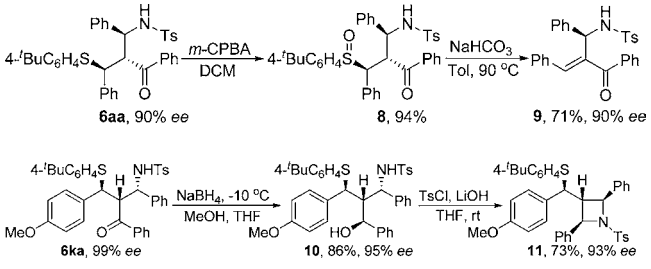

**Scheme 2. Proposed Mechanism for the Formation of Compound 6**




cascade-type reaction,[11a−e] which is shown in Scheme 2. The sulfur anion resulting from the deprotonation of the thiophenol with a base would undergo an ion exchanged reaction with catalyst **2h** to give **A**, which would take part in a Michael addition with **4** from its *Si*-face to furnish intermediate **B**.[11d,e] This step would be reversible, and with the *S* configuration of **B** favoring the nucleophilic addition to the imine **5** from its *Si*-face to form **C**, thus there is existence of a dynamic kinetic resolution in this process. Intermediate **C** would then abstract a proton from ArSH to give product **6**.

To demonstrate the potential utility of the products of this reaction in terms of ability to be further elaborated, **6aa** was oxidized to sulfoxide **8** with *m*-CPBA and further treated with sodium bicarbonate to yield **9**, which represents the chiral equivalent of an aza-Morita−Baylis−Hillman (MBH) reaction of chalcone; however, this target is unavailable by the direct asymmetric aza-MBH strategy until now. Subsequent reduction of the product **6ka** to **10**, followed by a cyclization reaction, gave the sulfur-containing azetidine **11** with four chiral centers (Scheme 3).

**Scheme 3. Derivatization of the Products**



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In summary, we have developed a chiral phase-transfer catalyst-mediated asymmetric three-component intermolecular sulfa-Michael/Mannich domino reaction. The chiral acyclic sulfur-containing compounds with multiple contiguous stereogenic centers were obtained in excellent diastereo- and enantioselectivities. Those products could be facilely transformed to the unprecedented available equivalents via the direct asymmetric aza-Morita–Baylis–Hillman (MBH) reaction of chalcone. Moreover, the chiral azetidine with four chiral centers was also synthesized via the reduction and substitution reactions. This method provides easy access to complex sulfur-containing chiral compounds with potentially biological activities.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b01833.

Experimental procedures and detailed characterization data of all new compounds (PDF)
X-ray crystal details for **11** (CIF)
X-ray crystal details for (±)-**6ka** (CIF)

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### Author Contributions

[†]R.D. and B.Z. contributed equally.


### Notes

The authors declare no competing financial interest.

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