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Graphical Abstract

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$Bi(OTf)_3$ catalyzed synthesis of acyclic β-sulfanyl ketones via a tandem Meyer-Schuster rearrangement / conjugate addition reaction

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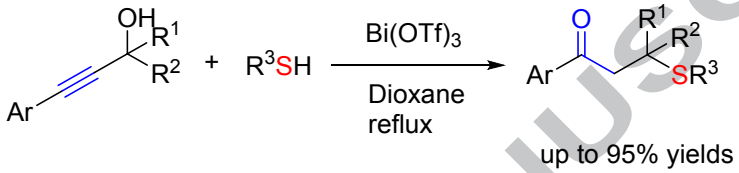
Yuan Wang [a], Yan Yin [a,*], Qinglin Zhang [a], Wanyong Pan[a], Huifeng Guo [a], Keke Pei[a]
[a]*School of Chemical and Environmental Engineering, Shanghai Institute of Technology, 100 Hai Quan Rd., Shanghai, 201418, P. R. China.*



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[a]*School of Chemical and Environmental Engineering, Shanghai Institute of Technology, 100 Hai Quan Rd., Shanghai, 201418, P. R. China*

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* Corresponding author. Tel.: +1-021-608-7220; fax: +1-021-608-7230; e-mail: yinyan@sit.edu.cn

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ABSTRACT

A new strategy to prepare acyclic β-carbonyl thioethers from propargyl alcohols and sulfur nucleophiles is reported. The investigation of the reaction substrates scope indicated that primary 3-aryl propargyl alcohols and thiols underwent the transformation smoothly. The reaction probably proceeded a $Bi(OTf)_3$-catalyzed tandem Meyer-Schuster rearrangement of 3-aryl propargyl alcohol, followed by a thiol Michael conjugate addition of thiols to in situ generated α, β-unsaturated ketones. The method was 100% atom economic, high-yielding, and easy to handle, making it a valuable method for the construction of β-carbonyl sulfides.

1. Introduction

Acyclic β-carbonyl thioether is an useful building block in organic synthesis,[1] and widely exists in natural products,[2] drugs,[3] beverages,[4] and foods.[4] Usually, this moiety is obtained through thiol Michael addition of α, β-unsaturated ketones and sulfur-containing nucleophiles.[5] Nucleophilic substitution of mercaptan to β-carbonyl halides under basic conditions afforded an alternative route to prepare β-carbonyl thioether.[6] Gill reported the synthesis of sulfides through β-acylethylation with ketonic Mannich bases.[7] Wen's group developed a novel acid-promoted direct cross-coupling reaction of methyl ketones and DMSO to prepare β-acyl allylic methyl sulfides in 2011.[8] Hou et al. reported an amine-catalyzed one-pot synthesis of thioethers directly from carboxylic acids, thioureas, and Michael acceptors in 2015.[9] Although methods to construct β-carbonyl thioether are available, the development of new synthetic methods for this particular structure remains an important and challenging task for organic chemists.

Previously, our group reported the synthesis of acyclic β-aminoketones from 3-aryl propargyl alcohols and nitrogen nucleophiles, which probable proceeded a $Fe(OTf)_3$ catalyzed Meyer-Schuster rearrangement of 3-aryl propargyl alcohols, followed by an intermolecular hydroamination between nitrogen nucleophiles and α,β-unsaturated ketones. This research also showed that only primary 3-aryl propargyl alcohols and sulfonamides underwent the transformation smoothly (Figure 1, Scheme A).[10] As we all know sulfur is highly nucleophilic, we guessed the replacement of sulfonamides with sulfur alcohols would allow the formation of acyclic β-carbonyl thioethers from 3-aryl propargyl alcohols (Figure 1, Scheme B). Herein, the scopes of propargyl alcohols and sulfur nucleophiles were investigated, which provided an 100% atom economic and starting materials readily available method for the synthesis of acyclic β-carbonyl thioethers.

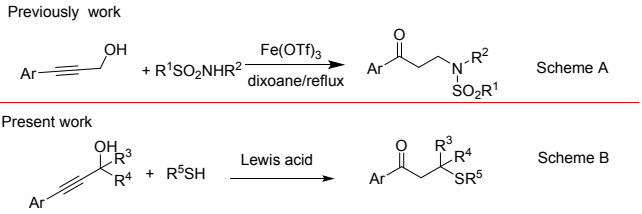


Figure 1. Conversion of 3-aryl propargyl alcohols

2. Results and discussion

A series of 3-aryl substituted propargyl alcohols (**1a-1m**, **1n**, and **1p**) were prepared through a Pd^{2+}-catalyzed Sonogashira coupling reaction of Ar-X and commercial available prop-2-yn-1-ols (Figure 2, Scheme A).[11] 1,3-Diphenyl-prop-2-yn-1-ols (**1o**, **1q**, and **1r**) were formed from the nucleophilic attack of phenylacetylene towards phenyl carbonyl compounds (Figure 2, Scheme B).[12]

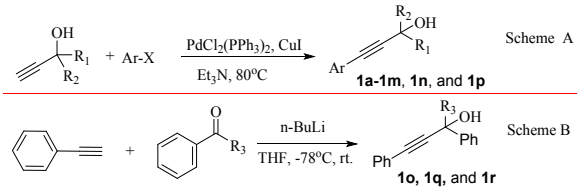
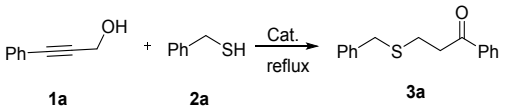


Figure 2. Synthesis of **1**.

To efficiently prepare acyclic β-carbonyl sulfides, we screened reaction conditions using 3-phenylprop-2-yn-1-ol (**1a**) and phenylmethanethiol (**2a**) as the starting materials (**Table 1**). When 15 mol% TfOH, $Fe(OTf)_3$, $FeCl_3$, and $Cu(OTf)_2$ were used as the catalyst, 54%–78% moderate yields of 3-(benzylthio)-1-phenylpropan-1-one (**3a**) were achieved after refluxing in

dioxane for 8 h (Entries 1−4). The complete disappearance of **1a** was detected by TLC after 30 h using 15 mol% AgOTf as the catalyst, and a 62% yield was obtained (Entry 5). When 15 mol% $Bi(OTf)_3$ was added, the yield of **3a** reached 93% in 2 h with dioxane as the solvent (Entry 6). Reaction solvents were tested with 15 mol% $Bi(OTf)_3$ as the catalyst, the reaction mixtures were quite complex probably due to the side reactions between **2a** and the solvents when (trifluoromethyl)benzene and DCE were used as the solvents,[13] and 45% and 38% yields were observed, respectively (Entries 7 and 8). Reaction with toluene gave only a 15% yield, and the unconsumed **1a** was recovered (Entry 9). The investigation of catalyst loading indicated that increasing the amount of $Bi(OTf)_3$ from 15 mol% to 20 mol% caused no effect on the yields compared entry 10 with 6, but decreasing the catalyst loading to 10 mol% lowed the reaction rate and yield (Entry 11 vs. 6). A **1a**:**2a** ratio of 1 gave lower yields over longer reaction times because a small amount of **2a** was oxidized to 1,2-dibenzyldisulfane (Entry 12 vs. 6). Increasing the **1a**:**2a** ratio to 1.2 produced same 93% yield to **1a**:**2a** ratio of 1.1 (Entry 13 vs. 6). No reaction occurred between **1a** and **2a** without catalyst (Entry 14). 2-Methylene-1,5-diphenylpentane-1,5-dione was separated after refluxing the mixture of **1a** in dioxane for 5 h (Entry 15).[14] The optimized reaction conditions for further studies were 15 mol% $Bi(OTf)_3$ as the catalyst, the 1.1 ratio of propargyl alcohol and sulfur nucleophiles as starting materials, and dioxane as the solvent.

Table 1. Optimization of the reaction conditions[a]



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

Entry	Catalyst	Solvent	Catalyst loading (mol%)	Ratio of **2a**:**1a**	Time (h)	Yield of **3a** (%)[b]
1	TfOH	Dioxane	15	1.1	8	54
2	$Fe(OTf)_3$	Dioxane	15	1.1	8	60
3	$FeCl_3$	Dioxane	15	1.1	8	62
4	$Cu(OTf)_2$	Dioxane	15	1.1	8	78
5	AgOTf	Dioxane	15	1.1	30	62
6	$Bi(OTf)_3$	Dioxane	15	1.1	2	93
7	$Bi(OTf)_3$	(Trifluoromethyl) benzene	15	1.1	2	45
8	$Bi(OTf)_3$	DCE	15	1.1	2	38
9	$Bi(OTf)_3$	Toluene	15	1.1	2	15[c]
10	$Bi(OTf)_3$	Dioxane	20	1.1	2	93
11	$Bi(OTf)_3$	Dioxane	10	1.1	5	86
12	$Bi(OTf)_3$	Dioxane	15	1	3	85
13	$Bi(OTf)_3$	Dioxane	15	1.2	2	93
14	-	Dioxane	-	1.1	2	NR[c]
15	$Bi(OTf)_3$	Dioxane	15	-	5	NR

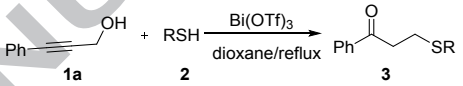
[a] Reaction conditions: **1a** (0.2 mmol), solvent (1mL), reflux.

[b] Isolated yield after flash chromatography.

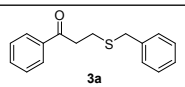
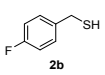
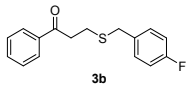
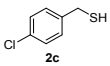
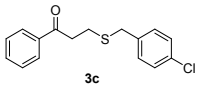
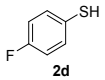
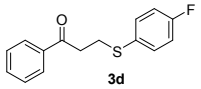
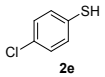
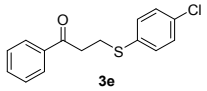
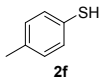
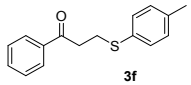
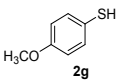
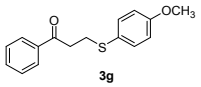
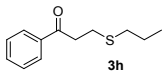
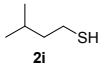
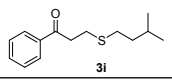
[c] The unconsumed **1a** was recovered.

Previously, we found the reaction with amide and aniline as the nitrogen nucleophiles could not give the corresponding acyclic β-aminoketones because of low nucleophilicity,[10] so we investigated the scope of commercially available sulfur-containing nucleophiles. The transformations of thiols, including phenylmethanethiols, benzenethiols, and aliphatic thiols, to β-carbonyl thioethers were fast and high-yield (**Table 2**). However, replacing thiols with urea did not produce the desired products after 16 h (data not shown here). **2b** with F and **2c** with Cl at 4 position of the benzyl ring produced similar yields to **2a** after refluxing 5 h (90% yield for **2b** and 95% yield for **2c** vs 93% for **2a**. Entries 2 and 3 vs Entry 1). Benzenethiols with F, Cl, CH_3, and OCH_3 at the para-position of the phenyl ring gave the corresponding β-carbonyl thioethers in good yield after 5 h (Entries 4-7), but the yields of substrates with electron-donating groups were lower than with electron-withdrawing groups because more thiols **2** were converted to disulfides. Aliphatic thiols **2h** and **2i** were submitted the investigation, both of them gave the expected products in 92% yields (Entries 8 and 9).

Table 2. Scope of the sulfur-containing nucleophiles[a]



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

Entry	Thiol	Product	Time(h)	Yield (%)[b]
1	 **2a**	 **3a**	2	93
2	 **2b**	 **3b**	5	90
3	 **2c**	 **3c**	5	95
4	 **2d**	 **3d**	5	89
5	 **2e**	 **3e**	5	90
6	 **2f**	 **3f**	5	83
7	 **2g**	 **3g**	5	70
8	 **2h**	 **3h**	5	92
9	 **2i**	 **3i**	3	92

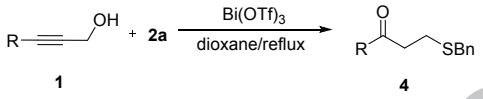
[a]Reaction conditions: **1a** (0.2 mmol), RSH (0.22 mmol), $Bi(OTf)_3$ (0.03 mmol), dioxane (1 mL), 110 ^{O}C

[b]Isolated yield after flash chromatography.

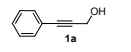
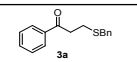
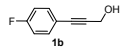
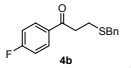
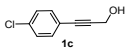
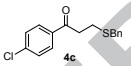
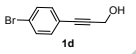
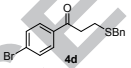
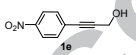
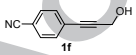
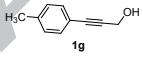
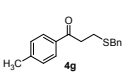
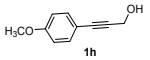
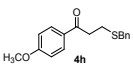
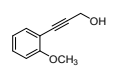
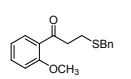
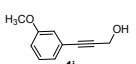
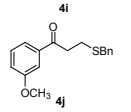
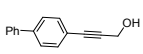
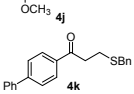
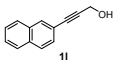
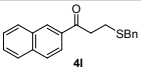
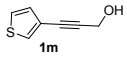
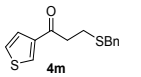
Yanada reported that only 3-phenylprop-2-yn-1-ol derivatives with electronic donating grousp on the phenyl ring underwent $Bi(OTf)_3$-catalyzed tandem Meyer-Schuster rearrangement and

intramolecular 1,4-addition to the resulting vinyl ketone.[14] We then explored the scope of propargyl alcohols with **2a** as the sulfur nucleophile (**Table 3**). Propargyl alcohols (**1a**-**1m**) with different electron densities on the carbon-carbon triple bond were prepared according to **Scheme A** in **Figure 2** by changing the aryl groups α to the triple bond. The substituents at the 4-position of the phenyl ring had obvious effects on the reaction times and yields (Entries 1-11). **1b** with fluorine, **1c** with chlorine, and **1d** with bromine finished the conversation in good yields after refluxing longer times compared with **1a** (83% for **1b** in 5 h, 69% for **1c** in 20 h, and 78% for **1d** in 16 h, entries 2-4). It is worth noting that the reaction of **1b** proceeded faster than **1c** and **1d** probably because of the complex interactions existing in **1b**.[15] However, there were no transformation for substrates with very electron-withdrawing groups (**1e** with NO_2 and **1f** with CN), and the starting materials were recovered (Entries 5 and 6). **1g** with methyl group showed similar reactivity to **1a** (Entry 7). Methoxy groups at para or ortho positions of the phenyl ring were favorable for the reaction rate because of the higher electron density of carbon-carbon triple bond, and **4h** and **4i** were achieved in high yield after 0.5 h (Entries 8 and 9). Changing the aromatic group at the carbon-carbon triple bond from phenyl group to biphenyl, naphthyl, and thienyl groups did no cause pronounced effects on the reaction, and **4k**-**4m** were separated in excellent yields (Entries 11-13). However, the expected products were not detected when the Ar groups in **1** were replaced by methyl group or ethyl group.

Table 3. Scope of propargyl alcohols **1**.[a]



1 **4**

Entry	Alcohol	Product	Time(h)	Yield (%)[b]
1	 **1a**	 **3a**	2	93
2	 **1b**	 **4b**	5	83
3	 **1c**	 **4c**	20	69
4	 **1d**	 **4d**	16	78
5	 **1e**	-	72	NR[c]
6	 **1f**	-	72	NR[c]
7	 **1g**	 **4g**	2	85
8	 **1h**	 **4h**	0.5	85
9	 **1i**	 **4i**	0.5	92
10	 **1j**	 **4j**	3	86
11	 **1k**	 **4k**	2	90
12	 **1l**	 **4l**	2	90
13	 **1m**	 **4m**	2	95

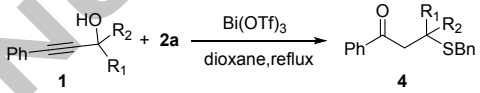
[a]Reaction conditions: **1a** (0.2 mmol), RSH (0.22 mmol), $Bi(OTf)_3$ (0.03 mmol), dioxane (1 mL), 110 ^{O}C.

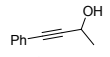
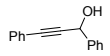
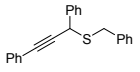
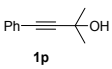
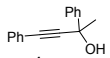
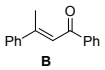
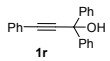
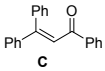
[b]Isolated yield after flash chromatography.

[c]The unconsumed **1a** was recovered.

After investigating primary propargyl alcohols, we turned our attention to secondary and tertiary propargyl alcohols. The results showed that all propargyl alcohols with bulky group α to -OH (**1n**-**1r**) completely disappeared within 1.5 h under the optimized condition (Table 4). The alcohols **1n**-**1q** produced β-carbonyl thioethers in 30%-45% yields, and unexpected by-products, such as benzyl(3-phenylprop-2-yn-1-yl)sulfanes and α,β-unsaturated ketones, were separated (Entries 1-4). When **1r,** with two Ph groups α to -OH, gave only 1,3,3-triphenylprop-2-en-1-one in 90% yield via Meyer-Schuster rearrangement (Entry 5).

Table 4. Scope of 3-phenyl propargyl alcohols **1**.[a]



Entry	Alcohol	Time (h)	Yield of **4** (%)[b]	By product
1	 **1n**	1.5	45	complex
2	 **1o**	1	40	 **A** 45% yield
3	 **1p**	1	40	complex
4	 **1q**	1	30	 **B** 40% yield
5	 **1r**	1	-	 **C** 90% yield

[a]Reaction conditions: **1a** (0.2 mmol), RSH (0.22 mmol), $Bi(OTf)_3$ (0.03 mmol), dioxane (1 mL), 110 ^{O}C.

[b]Isolated yield after flash chromatography.

Combining the knowledge of the transformation of propargyl alcohols into α,β-unsaturated ketones (Table 4),[14,16] and the separation of dimeric diketone, we proposed a possible mechanism for the present $Bi(OTf)_3$-catalyzed transformation of propargylic alcohols (Figure 3). Propargylic alcohols easily formed intermediate α,β-unsaturated ketones in the presence of catalytic amounts of $Bi(OTf)_3$, which underwent a nucleophilic attacked by thiols to afford β-carbonyl thioethes. In alterative, self-coupling of intermediate α,β-unsaturated ketones led to dimeric diketone.

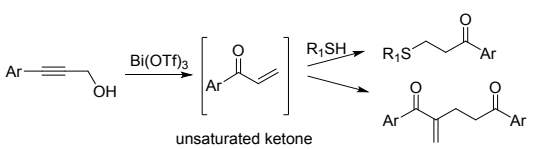


Figure 3. Proposed mechanism

3. Conclusion

In summary, we developed a $Bi(OTf)_3$-catalyzed preparation of acyclic β-carbonyl sulfides[17] from 3-aryl propargyl alcohols and thiols, which probably proceeded by tandem Meyer-Schuster rearrangement of propargyl alcohols, followed by the nucleophilic attack of thiols on α,β-unsaturated ketones. The starting material was easy to obtain, the experimental process was simple. Few by-products, high yields, and 100% atomic utilization rate make it a potentially attractive method for the synthesis of acyclic β-carbonyl sulfides. Reactions with propargyl alcohols as nucleophiles were currently underway in our lab and will be published in due course.

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Supplementary data

Supplementary data associated with this article can be found in the online version at http://dx.doi.org.

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17. Typical procedure: To a mixture of propargyl alcohol **1** (0.2 mmol) in dioxane (1 mL), $Bi(OTf)_3$ (0.03 mmol) and nucleophile **2** (0.22 mmol) were added. The mixture was stirred at refluxing temperature until **1** disappeared monitored by TLC. Then the reaction was quenched by saturated $NaHCO_3$ solution and extracted with ethyl acetate. The combined organic phases were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give a residue. Finally the residue was purified through column chromatography on silica gel (200-300 mesh) with hexane/EtOAc as eluent to afford the products **3a-3i**, **4b-4d**, and **4g-4q**. 3-(benzylthio)-1-phenylpropan-1-one (**3a**): 92% yield; yellow oil; ^{1}H NMR (500 MHz, Chloroform-d) δ 7.92 – 7.86 (m, 2H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.35 – 7.29 (m, 4H), 7.24 (s, 1H), 3.77 (s, 2H), 3.17 (t, *J* = 7.5 Hz, 2H), 2.84 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 198.30 , 138.38 ,136.60 , 133.23 , 128.87 , 128.64 , 128.58 , 128.03 , 127.08 , 38.75 , 36.92 , 25.88 . IR (KBr, cm^{-1}) 3726, 3647, 3470, 2957, 2918, 1683, 1596, 1560, 1492, 1448, 1378, 1019, 979, 748, 695, 491.

Highlight:

A new strategy to prepare acyclic β-carbonyl thioethers

Primary 3-aryl propargyl alcohols and thiols underwent the transformation smoothly

The method was 100% atom economic, high-yielding, and easy to handle