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## One-pot preparation of Julia-Kocienski sulfides and sulfones from alcohols

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#### ARTICLE INFO

## ABSTRACT

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The Julia-Kocienski reaction is a very strong tool for the synthesis of *E*-alkenes 5 from carbonyl compounds and heteroaryl sulfones such as 4 and used extensively in the last decade for the construction of carbon-carbon double bonds present in many natural products (Scheme 1).<sup>1,2</sup> When the sulfone reagents 4 are prepared from thiol 1 and alcohols 2, the corresponding sulfides 3 are generally prepared by Mitsunobu reaction<sup>3</sup> using either DEAD or DIAD along with Ph<sub>3</sub>P.<sup>2b</sup> However, both DEAD and DIAD are expensive and the byproducts (Ph<sub>3</sub>P=O and (NHCO<sub>2</sub>R)<sub>2</sub>) are often difficult to be removed from the desired product.<sup>4</sup> Several approaches similar to Mitsunobu reaction were reported<sup>5</sup> and Lewis acid catalyzed C-S bond formations were also developed.<sup>6</sup> However, some are restricted to benzylic and allylic alcohols and some have similar problems as Mitsunobu reaction does. Since alcohols can be easily transformed to alkyl halides and sulfonates which can be used for alkylation of thiol, sulfides can be prepared by these two steps. In fact, some Julia-Kocienski reagents were prepared by these two steps followed by oxidation.<sup>7</sup> Although Bonini and coworkers reported that the one-pot preparation of propargylic sulfides via either iodide formation or tosylate formation, the yields were not always good.<sup>8</sup> One-pot reactions, where a number of transformations are carried out in a single pot, have attracted considerable attention because they offer significant advantages such as elimination of purification steps, reduction of wastes, time, and costs, and increase efficiency of the process.9,10 Now one-pot synthesis is one of the most powerful synthetic strategies for the environmentally benign reactions. Therefore, we studied one-pot preparation of Julia-Kocienski sulfides and

A method for one-pot preparation of Julia-Kocienski sulfides and sulfones from alcohols and thiols is reported. A variety of primary alcohols were converted to the corresponding mesylates by methansulfonyl chloride and triethylamine in THF. After the reaction is complete, thiol (1 or **10**) and either NaH or *t*-BuOK were added. The Julia-Kocienski sulfides **3**, **9** and **11** were prepared by one-pot two steps procedure from alcohols in 76-96% yields (16 examples). Furthermore, after the sulfide formation, the reaction mixture was neutralized by *p*-toluenesulfonic acid and treated with  $H_2O_2$  and ammonium molybdate in EtOH to give the Julia-Kocienski sulfones **4** in good yields except for *trans*-2-hexen-1-ol.

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Scheme 1. Julia-Kocienski reaction.

sulfones from alcohols and thiols. Since halogenation reactions are generally performed under acidic conditions, we chose mesylate formation from alcohols followed by their reaction with 1 under basic conditions according to Scheme 2. Herein we would like to report our results.



Scheme 2. One-pot preparation of Julia-Kocienski sulfides.

The mesylation reaction of *n*-pentanol 2a was carried out by treating with methansulfonyl chloride (1.2 eq) and triethylamine (1.3 eq) in THF for 3 h to give mesylate 6a in 92% yield (Scheme 3). The yield of 6a did not much change by shortening the reaction time to 1 h and 6a was obtained in 90% yield. The

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obtained mesylate 6a was used for alkylation reaction of thiol 1. The Julia-Kocienski sulfide 3a was obtained in 86% yield when 1 was deprotonated with NaH (1.2 eq) in THF followed by the treatment with 6a (1 eq) for 24 h. When t-BuOK (1.2eq) was used instead of NaH, 94% yield of 3a was obtained. Thus, the sulfide formation can be carried out using either NaH or t-BuOK in THF.



Scheme 3. Preparation of Julia-Kocienski sulfide 3a from 2a.

We next explored the possibility of sequential one-pot preparation of the Julia-Kocienski sulfide 3 (Table 1). After npentanol 2a was treated with methansulfonyl chloride (1.2 eq) and triethylamine (1.3 eq) in THF for 1.5 h, a mixture of 1 (1 eq) and NaH (2.3 eq) in THF was added to the above reaction mixture. The resulting mixture was stirred for 16 h to give sulfide 3a in 86% yield,<sup>11</sup> which is higher than the yield obtained from mesylation (92%) and alkylation of 1 using NaH (86%)  $(92\% \times 86\% = 79\%)$  (entry 1). When alkylation of 1 was performed at higher temperature (50 °C to reflux), the yield dropped to 64% due to partial decomposition of mesylate 6a (entry 2). When NaH was added after the addition of **1** for easier operation, **3a** was obtained in a comparable yield to entry 1 (83%, entry 3). The scope of this reaction was next explored with two other types of alcohols. Although the alkylation of 1 with the mesylate derived from  $\gamma$ -branched citronellol 2b hardly proceeded at room temperature, the reaction at 50 °C gave 3b in 90% yield using 1.3 eq of 1 (entry 5). The alkylation of 1 with the mesylate derived from  $\beta$ -branched cyclohexylmethanol 2c was slow even at 50 °C to give 3c in 60% yield (entry 6). The yield increased to 67% by using t-BuOK (entry 7) and 83% yield was obtained when the mixture was heated under reflux for 6 h (entry 9). The addition of DMF to the THF solution decreased the yield of **3c** (entry 8).

Having optimized the reaction conditions, we next focused our attention on the reaction of the alcohols having functional groups in this one-pot two steps procedure (Table 2). When the alcohol 2d having a t-BuMe<sub>2</sub>SiO group was transformed to its mesylate and then treated with 1 and NaH at 50 °C, 84% yield of sulfide 3d was obtained (entry 1). Although the yield of sulfide 3e was only 56% when 6-tetrahydropyranyloxyhexan-1-ol 2e was treated in a similar manner as entry 1, the yield improved to 87% under reflux (entries 2-3). Tetrahydro-2-furanmethanol 2f was converted to sulfide 3f in 74% yield using t-BuOK by following the procedure of entry 9 in Table 1 (entry 4). Using 1.3 eq of 1, the yield was slightly improved to 76% (entry 5). The alcohol **2g** having a  $\alpha,\beta$ -unsaturated ester group was also transformed to sulfide 3g in 69% yield using NaH (entry 6). The yield was lowered to 46% by using t-BuOK and was improved to 86% using 1.3 eq of 1 under reflux (entries 7-9). Both trans-2-

Table 1. One-pot preparation of the Julia-Kocienski sulfides 3 from alcohols

$\begin{array}{c} \text{RCH}_2\text{OH} & \begin{array}{c} 1 \end{pmatrix} \text{MsCI (1.2 eq), Et}_3\text{N} (1.3 eq) \\ \hline \text{THF, rt, time} \\ 2 \end{pmatrix} \begin{array}{c} \text{N-N} \\ \text{N} $					
entry	2	time	Base (eq)	conditions	yield
1	2a	1.5 h	NaH (2.3)	rt, 16 h	86%
2	2a	1.5 h	NaH (2.3)	reflux, 2 h	64%
3 <sup>b</sup>	2a	1.5 h	NaH (2.3)	rt, 16 h	83%
4	2b	2.5 h	NaH (2.3)	50 °C, 9 h	75%
5°	2b	2.5 h	NaH (2.3)	50 °C, 21 h	90%
6	2c	1 h	NaH (2.6)	50 °C, 15 h	60%
7 <sup>d</sup>	2c	1 h	t-BuOK (2.5)	50 °C, 15 h	67%
8 <sup>e</sup>	2c	1 h	t-BuOK (2.5)	50 °C, 15 h	57%
9	2c	1 h	t-BuOK (2.5)	reflux, 6 h	83%

a) A mixture of 1 and base was added unless otherwise noted. b) Base was added after the addition of 1. c) 1.3 eq of 1 was used. d) 1 wad added after the addition of base. e) THF:DMF = 3.5:1.5.



hexen-1-ol 2h and cis-2-hexen-1-ol 2i were transformed to the allyl mesylates, which reacted with the anion derived from 1 and NaH smoothly. The sulfides 3h and 3i were obtained in 88% and 89% yield, respectively, without isomerization after 3-5 h at

Table 2. One-pot preparation of sulfides 3 from alcohols having functional groups.

1) MsCl (1.2 eq)

$\frac{1) \text{ MsCl (1.2 eq)}}{\text{Et}_{3}\text{N (1.3 eq), THF, 1 h}} \xrightarrow{\text{N-N}} N$				
	<b>2</b> 2)	<b>1</b> (1.0 eq), base conditions	`N´ I Ph	3
entry	2	base (eq)	conditions	yield
1	2d	NaH (2.3)	50 °C, 7.5 h	84%
2	2e	NaH (2.3)	50 °C, 7 h	56%
3	2e	NaH (2.3)	reflux, 7 h	87%
4	2f	<i>t</i> -BuOK (2.5)	reflux, 19 h	74%
5ª	2f	t-BuOK (2.8)	reflux, 19 h	76%
6	2g	NaH (2.3)	50 °C, 5 h	69%
7	2g	t-BuOK (2.5)	50 °C, 5 h	46%
8	2g	NaH (2.3)	reflux, 5.5 h	75%
9 <sup>a</sup>	2g	NaH (2.6)	reflux, 6.5 h	86%
10	2h	NaH (2.3)	rt, 5 h	88%
11	2i	NaH (2.3)	rt, 3 h	89%
12	2j	NaH (2.3)	rt, 2 h	95%
13	2k	NaH (2.3)	rt, 2 h	91%
14	21	NaH (2.3)	rt, 2 h	96%

a: 1.3 eq of 1 was used.



room temperature (entries 10-11). The mesylates derived from benzyl alcohols **2j-2l** are also highly reactive and the corresponding sulfides were obtained in 91-96% yield by the reaction using NaH at room temperature (entries 12-14).

Nicolaou and co-workers reported a total synthesis of Artochamins H, I, and J, which are isolated from *Artocarpus chama* as cytotoxic ingredients.<sup>12</sup> In their synthesis, they used the Julia-Kocienski reaction of sulfone **9**, followed by [3,3] Claisen rearrangement, decarboxylation, and [2,2] cycloaddition (Scheme 4). The alcohol **7** was transformed to the sulfide **8** by Mitsunobu reaction using DEAD and Ph<sub>3</sub>P in 77% yield and then **8** was oxidized by  $H_2O_2$  and ammonium molybdate to give the sulfone **9**. We applied our one-pot two steps procedure to the preparation of **8** from **7**. After **7** was treated with methansulfonyl chloride and triethylamine in THF for 1 h, a THF solution of **1** and NaH was added and stirred at room temperature for 2 h. The sulfide **8** was obtained in 95% yield. Our one-pot two steps procedure has an advantage over Mitsunobu reaction in high yield, simple work-up and low cost of the reagents.



Scheme 4. Preparation of sulfide 8 from 7.

One-pot two steps procedure was applied for the preparation of the 1,3-benzothiazol-2-yl sulfides 11. After an alcohol was treated with methansulfonyl chloride and triethylamine in THF, 10 (1.0 eq) and base were added (Table 3). When n-pentanol 2a was transformed to its mesylate and then treated with 10 and NaH (2.3 eq) at room temperature, sulfide 11a was obtained in 62% yield (entry 1). Using t-BuOK (2.5 eq) instead of NaH, the yield increased to 83% (entry 2). The reaction of cyclohexylmethanol 2c was carried out using t-BuOK under reflux to give 11c in 88% yield (entry 3). Benzyl alcohol 2j was transformed to sulfide 11j at room temperature in 78 and 79% yield using NaH and t-BuOK, respectively (entries 4-5). At 50 °C for 2 h, the yield increased to 91% (entry 6). Thus, 1,3benzothiazol-2-yl sulfides 11 were prepared in high yield by the one-pot procedure from alcohols.

The obtained sulfides **3** were next oxidized to the corresponding sulfones **4** (Scheme 5). When **3a** ( $\mathbf{R} = n$ -Bu) was oxidized by 35% H<sub>2</sub>O<sub>2</sub> aq. (9 eq) and ammonium molybdate<sup>13</sup> (0.1 eq) in ethanol, sulfone **4a** was obtained in 97% yield. In a similar manner, the sulfide derived from *trans*-2-hexen-1-ol **2h** (*trans* only) was oxidized to **4h** as a *trans:cis* = 96:4 mixture in

**Table 3.** One-pot preparation of 1,3-benzothiazol-2-yl sulfides 11from alcohols.



entry	2	base (eq)	conditions	yield
1	2a	NaH (2.3)	rt, 22 h	62%
2	2a	t-BuOK (2.5)	rt, 22 h	83%
3	2c	t-BuOK (2.5)	reflux, 6.5 h	88%
4	2j	NaH (2.3)	rt, 2 h	78%
5	2j	t-BuOK (2.5)	rt, 2 h	79%
6	2j	NaH (2.3)	50 °C, 2 h	91%

61% yield. The sulfide derived from *cis*-2-hexen-1-ol **2i** (*cis:trans* =97.5:2.5) was also oxidized to the corresponding sulfone **4i**. Oxidation occurred rapidly and the yield was 83%, but the obtained sulfone was a mixture of *cis:trans* = 78:22. Unfortunately, the isomerization was further promoted when Na<sub>2</sub>WO<sub>4</sub><sup>7b</sup> was used instead of ammonium molybdate (*cis:trans* = 27:73, 39% yield). This could be explained by the lower Lewis acidity of Mo(6+) compared to W(6+).<sup>15</sup> The benzyl sulfide was oxidized to **4j** in 83% yield.





Finally, we explored the possibility of one-pot preparation of the Julia-Kocienski sulfones 4 from alcohols via mesylates and After *n*-pentanol 2a was treated with sulfides (Table 4). methansulfonyl chloride and triethylamine in THF for 1 h, 1 and NaH was added and stirred for 21 h. Then ethanol, aq. H<sub>2</sub>O<sub>2</sub> (9 eq), and ammonium molybdate (0.1 eq) were added and the resulting mixture was stirred for 23 h to give 4a in 69% yield (entry 1). This yield of our one-pot three-steps procedure from 2a to 4a was lower than that of three separate reactions (92%) (mesylation) x 86% (alkylation using NaH) x 97% (oxidation) = 77%). After alkylation of thiol 1 was completed, the reaction mixture was concentrated under reduce pressure in order to remove THF solvent and the residue was treated in a similar manner as entry 1. Disappointingly, slightly lower 63% yield was obtained (entry 2). When ammonium molybdate was replaced with Na<sub>2</sub>WO<sub>4</sub>, only the sulfide **3a** was obtained in 42% yield instead of the desired 4a (entry 3). When oxone (2KHSO<sub>5</sub>·KHSO<sub>4</sub>·K<sub>2</sub>SO<sub>4</sub>) was used as an oxidant, 4a was obtained in 67% yield after 17 h (entry 4). We next turned our attention to the pH of the reaction mixture. The pH of the oxidation reaction mixture (Scheme 5) was 3, while the final pH in the one-pot three steps procedure was 10 (entry 1 in Table 4). Neutralization may be necessary prior to the oxidation step. We

Table 4. One-pot three steps procedure from 2a to the sulfone 4a.



entry	time1	oxidant (eq) <sup>a</sup>	solvent	time2	yield
1	21 h	H <sub>2</sub> O <sub>2</sub> (9)-A	EtOH	23 h	69%
2	21 h	H <sub>2</sub> O <sub>2</sub> (9)-A	EtOH	23 h	63%
3	22 h	H <sub>2</sub> O <sub>2</sub> (9)-B	MeOH	24 h	0°
4	19 h	Oxone (3)	MeOH-H <sub>2</sub> O	17 h	67%

a) A:  $(NH_{4})_6Mo_7O_{24}$  (0.1 eq), B:  $Na_2WO_4$  (0.1 eq). b) The volatile was removed under reduced pressure before the oxidation step. c) Sulfide **3** was obtained in 42% yield.

searched various acids for neutralization using the conditions of entry 1 in Table 4. The yield of sulfone 4a was as follows: 0% with 1M HCl, 1M NH<sub>4</sub>Cl, H<sub>2</sub>SO<sub>4</sub>, or HNO<sub>3</sub>, 70% with AcOH, and 84% with p-TsOH·H<sub>2</sub>O. After the sulfide formation from npentanol 2a was completed, the reaction mixture was neutralized by p-TsOH·H<sub>2</sub>O (0.5 eq), and then ethanol, 35% H<sub>2</sub>O<sub>2</sub> aq. (9 eq), and (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·H<sub>2</sub>O (0.1 eq) were added to the reaction mixture and stirred for 24 h. The sulfone 4a was obtained in 84% yield, which was higher than both the yield obtained from three separate reactions (77%) and the yield obtained from the one-pot sulfide formation and oxidation reaction ( $86\% \times 97\% =$ 83%) (entry 1 in Table 5). In a similar manner, trans-2-hexen-1ol 2h was transformed to the sulfone 4h (entry 2). The yield was only 35%<sup>16</sup> and this transformation is better to be carried out by the one-pot sulfide formation followed by the oxidation (84% x 61% = 51%). The reaction of benzyl alcohol 2j was also carried out to give the sulfone 4j in 82% yield, which is higher than the yield obtained from the one-pot sulfide formation and oxidation reaction (95% x 83% = 79%) (entry 3).

Table 5. One-pot three steps procedure from 2 to sulfone 4.



In summary, we developed a method for the one-pot preparation of the Julia-Kocienski sulfides and sulfones from alcohols. After a variety of alcohols were transformed to the mesylates by methansulfonyl chloride and triethylamine in THF, thiol (1 or 10) and either NaH or *t*-BuOK were added. The Julia-Kocienski sulfides 3, 9 and 11 were prepared from alcohols in 76-96% yield. Furthermore, one-pot three steps procedure from alcohol to the sulfones 4 was developed. We believe this method is useful for many synthetic chemists.

A typical one-pot preparation of sulfide 3 (entry 1 in Table 1) To a solution of n-pentanol (0.440 g, 5.0 mmol) in THF (20.0 mL) at 0 °C were added methansulfonyl chloride (0.465 mL, 6.0 mmol) and triethylamine (0.906 mL, 6.5 mmol). After the mixture was stirred at RT for 1.5 h, a solution of 5-mercapto-1phenyl-1H-tetrazole (0.891 g, 5.0 mmol) and NaH (0.46 g, 11.5 mmol) in THF (5.0 mL) was added at 0 °C. The resulting mixture was stirred at RT for 16 h. The reaction was quenched with sat. NH<sub>4</sub>Cl aq., and the mixture was extracted with AcOEt (3  $\times$  10 mL). The combined extracts were washed with brine, dried (MgSO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hex:AcOEt = 8:1) to afford **3a** (1.076 g, 86%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.91 (3H, t, *J* = 7.3 Hz), 1.31-1.47 (4H, m), 1.83 (2H, quin, J = 7.3 Hz), 3.40 (2H, t, J = 7.3 Hz), 7.51-7.61 (5H, t)m).<sup>15</sup>

A typical one-pot preparation of sulfone 4 (entry 3 in Table 5) To a solution of benzyl alcohol (0.108 g, 1.0 mmol) in THF (3.0 mL) at 0 °C were added methansulfonyl chloride (0.93 mL, 1.2 mmol) and triethylamine (0.180 mL, 1.3 mmol). After the mixture was stirred at RT for 1 h, a solution of 5-mercapto-1phenyl-1H-tetrazole (0.178 g, 1.0 mmol) and NaH (0.092 g, 2.3 mmol) in THF (2.0 mL) was added at 0 °C. The resulting mixture was stirred at RT for 1 h and treated with p-TsOH·H<sub>2</sub>O (0.095 g, 0.5 mmol) for 15 min. EtOH (3.0 mL), (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·H<sub>2</sub>O (0.123 mg, 0.1 mmol), and 34.5% aq. H<sub>2</sub>O<sub>2</sub> (0.799 mL, 9.0 mmol) were added at 0 °C and the mixture was stirred at RT for 20 h. The reaction was quenched with sat. aq. Na<sub>2</sub>SO<sub>3</sub>, and the mixture was extracted with AcOEt (2  $\times$  8 mL). The combined extracts were washed with brine, dried (MgSO<sub>4</sub>), and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hex:AcOEt = 5:1) to afford 4j (0.246 g, 82%) as a colorless solid. mp = 101-102 °C (recrystallized from hexane-CH<sub>2</sub>Cl<sub>2</sub> (5:1)); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.93 (2H, s), 7.36-7.58 (10H, m).<sup>17</sup>

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# Tetrahedron

# Highlights

- One-pot reaction •
- Preparation of Julia-Kocienski sulfides • from alcohols.
- Julia-Kocienski Preparation of sulfones • from alcohols.
- Useful for alkene synthesis •

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## **Graphical Abstract**

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