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Graphical Abstract



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Electrochemical radical arylsulfonylation/semipinacol rearrangement sequences of alkenylcyclobutanols: Synthesis of β-sulfonated cyclic ketones

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ABSTRACT

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Keywords: Sulfonylation Electrochemical oxidation Semipinacol rearrangement Alkenyl alcohols Electrochemical oxidative radical sulfonylation/semipinacol rearrangement sequences of alkenylcyclobutanols have been developed. The reaction proceeds in an undivided electrochemical cell equipped with platinum plate electrodes employing sodium iodide as a redox catalyst and a supporting electrolyte. This approach is environmentally benign by using shelf-stable arylsulfonyl hydrazides as arylsulfonyl radical precursor and electrons as oxidizing reagents. The present protocol offers a facile way to prepare β -sulfonated cyclic ketone derivatives.

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great importance Sulfone compounds are of in pharmaceutical, agrochemical, and material industries because of their biological and chemical properties.¹ Therefore, the development of novel and practical methods to construct sulfone derivatives has been a subject of intense study.2 The addition of sulfonyl radicals to unsaturated carbon-carbon bonds represents a general route to sulfone synthesis. Recently, sulfonyl hydrazides, economical and shelf-stable reagents, have emerged as valuable sulfonyl radical sources via various oxidation processes.3,4 Recently, several groups reported the syntnesis of β -funtionalized ketones by radical addition and 1,2-rearrangement sequences of allylic alcohol derivatives with various radicals including aryl, acyl, alkyl, difluoromethyl, trifluoromethyl, phosphoryl, and amine radicals.^{5,6} Narasaka and our groups reported the sulfonylation reactions and pinacol-type rearrangements of 1vinyl cyclic alcohols with sodium 2-naphthalenesulfinate promoted by cerium (IV) tetrabutylammonium nitrate⁷ or arylsulfonyl hydrizides catalyzed by potassium iodide and peroxide.⁸ Although thies approaches are moderately satisfied, new arylsulfonylation and semipinacol rearrangement sequences via 1,2-alkyl migration of alkenylcyclobutanols is highly desired.

In the past few years, electrochemistry has emerged as an attractive approach for organic chemists to introduce chemical functionality into organic molecules owing to its environmentally benign and practical nature. Synthetic electrochemistry could achieve the redox reactions without of the assistance of transition-metal catalysts or toxic reagents due to use of electron as oxidizing or reducing reagents.⁹ To the best of our knowledge,

electrochemical oxidative arylsulfonylation and ring expansion sequences of alkenylcyclobutanols has not been reported yet. We envisioned the transformation of the alkenylcyclobutanols to the β -sulfonated cyclopentanones by electrochemical catalyzed oxidative sulfonylation/semipinacol rearrangement sequence with sulfonyl hydrazides as sulfonyl radical precursor.

As part of the research program related to redox reaction and cyclization sequences, we recently reported the intramolecular redox reaction¹⁰ and radical addition/ring expansion reaction of alkenes with several radical sources under redox conditions.^{6c-6g,8} In this paper, we report electrochemical catalyzed oxidative arylsulfonylation and semipinacol rearrangement sequences via 1,2-alkyl migration of alkenylcyclobutanol derivatives.

To determine optimal reaction conditions for the electrochemical radical sulfonylation/semipinacol rearrangement sequences of alkenylcyclobutanol derivatives, we choosed 1-(1phenylvinyl) cyclobutanol (1a) and p-toluenesulfonyl hydrazide (2a) as the model substrates. The reaction was conducted in an undivided cell with platinum plate $(1.0 \times 1.0 \text{ cm}^2)$ as electrodes under constant current. Initially, potassium iodide was employed as the electrolyte and DMSO as the solvent at a constant current of 7 mA. The radical addition and ring expansion product 3a was obtained in 21% yield after the reaction proceeded at room temperature for 6 h (Table 1, entry 1). Then, we surveyed different kinds of common solvents, such as DMSO, methanol, water, THF and co-solvent of acetonitrile/water and THF/water (Table 1, entries 1-6). It was found that co-solvent of THF/water (1/1) was the best media. Various supporting electrolytes such as potassium iodide, sodium iodide, tetrabutylammonium iodide

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Table1. Optimization of the reaction conditions.^a



Entry	Solvent	(mA)	Electrolyte	(h)	$(\%)^{b}$
1	DMSO	7	KI	6	21
2	MeOH	7	KI	6	45
3	H_2O	7	KI	4	38
4	THF	7	KI	4	6
5	MeCN:H ₂ O(1:1)	7	KI	3	68
6	THF:H ₂ O (1:1)	7	KI	3	78
7	THF:H ₂ O (1:1)	7	NaI	2	85
8	THF:H ₂ O (1:1)	7	TBAI	2	15
9	THF:H ₂ O (1:1)	7	NaBr	2	trace
10	THF:H ₂ O (1:1)	7	KBr	3	trace
11	THF:H ₂ O (1:1)	5	NaI	2	85
12	THF:H ₂ O (1:1)	3	NaI	4	80
13 ^c	THF:H ₂ O (1:1)	-	NaI	6	0
14 ^d	THF:H ₂ O (1:1)	5	NaI	2	63
15 ^e	THF:H ₂ O (1:1)	5	NaI	3	48

^a Reaction conditions: Pt anode, Pt cathode, 1-(1-phenylvinyl)cyclobutanol (**1a**, 0.2 mmol), Ts-NHNH₂ (**2**, 0.6 mmol), electrolyte (0.4 mmol) in solvent (8.0 mL) at room temperature. ^b Isolated yield. ^c No electricity. ^d 0.5 equiv of NaI loading.

(TBAI), sodium bromide, and potassium bromide were screened (Table 1, entries 6-10), and it was found that the iodide ion was necessary for the reaction. Sodium iodide was the most efficient electrolyte for this transformation (Table 1, entry 7). And, current intensity was examined and 5 mA proved to be the optimum constant current for this reaction (Table 1, entries 7 and 11-12). No desired product could be obtained without an electric current (Table 1, entry 13). Finally, the effect of the amount of supporting electrolyte (NaI) was also investigated. The yield of 3a decreased when 0.5 or 0.2 equiv of sodium iodide per mol 1-(1-phenylvinyl) cyclobutanol (**1a**) was used (Table 1, entries 14-15).

With the optimal reaction conditions in hand, we investigated the scope of substrates for the electrochemical radical sulfonylation/semipinacol rearrangement sequences of alkenylcyclobutanol derivatives 1 with sulfonyl hydrazide 2. As shown in Table 2, various alkenylcyclobutanols 1 with electrondonating or electron-withdrawing substituted-aryl and naphthyl groups furnished the corresponding migration products with moderate to high yields (50-89%, Table 2, 3a-3i). Notably, this radical sulfonation/semipinacol rearrangement reaction with alkyl-substituted vinylcyclobutanol, 1-(3-phenylprop-1-en-2yl)cyclobutanol, gave 61% yield of desired product 3j under the optimal reaction conditions. To further examine the scope of this reaction, a range of sulfonyl hydrazides 2 such as benzenesulfonyl hydrazide and 2-naphthylsulfonyl hydrazide, were exposed to the optimal reaction conditions to react with alkenylcyclobutanols 1. It was found that the corresponding products 3k-3p were obtained in moderate to high yields (62-82%, Table 2). Furthermore, 1-(1H-inden-3-yl)cyclobutanol (4), 1-(3,4-dihydronaphthalen-1-yl)cyclobutanol (6), and 1-(1phenylvinyl)cyclopentanol (8) derivatives were also used as



^a Reaction conditions: Pt anode, Pt cathode, alkenyl cyclobutanol **1** (0.2 mmol), ArSO₂NHNH₂ **2** (0.6 mmol), NaI (0.4 mmol) in THF:H₂O (1:1, 8.0 mL) at room temperature. ^b Isolated yield.

substrates in these electrochemical radical sulfonylation/semipinacol rearrangement sequences. It was found that the corresponding products 5, 7, and 9 were obtained in 49% (3.6:1 dr), 47% (2.0:1 dr), and 32% yields (Scheme 1). In order to demonstrate the practicability of this electrochemical sulfonvlation and ring expansion, the gram-scale reaction was explored. As shown in Scheme 2, when 1 - (1 phenylvinyl)cyclobutanol (1a) with *p*-toluenesulfonyl hydrazide (2a) under the modified optimum reaction conditions, the reaction proceeded to afford the desired tosyl-substituted cyclopentanone **3a** on a gram scale with 81% yield (Scheme 2). To illustrate synthetic utility, we also carried reductive removal of the sulfone groups to afford cyclopentanone derivative 10 in moderate yield (Scheme 3).



Scheme 1. Electrochemical radical sulfonylation/ring expansion sequences of 4, 6 and 8.



_____3I _____10 (51% yield)

Scheme 3. Desulfonation of compound 31.

To gain mechanistic insights into this transformation, some preliminary experiments were performed. The reaction was shut down the reactivity without an electric current (Table 1, entry 13). A trace of the product was detected in the presence of a 2,2,6,6-tetramethylpiperidin-1-yloxyl radical scavenger, (TEMPO) (Scheme 4, eq 1). The electrochemical sulfonylation and ring expansion reaction of 1a proceeded to afford a 51% of **3a** when *p*-tolunesulfonyl iodide $(Ts-I)^{11}$ was used instead of **2a** under optimal reaction conditions (Scheme 4, eq 2). In another control reaction, the chemical sulfonylation/semipinacol rearrangement sequences could occur in the presence of ptoluenesulfonyl iodide resulting only 35% product (Scheme 4, eq 3). Fortunately, we have isolated the TsI with trace amount during the reaction of 1a with 2a under optimal reaction conditions. These results imply that TsI is likely to be the intermediate in this transformatiom. And, we carried out cyclic voltammetry (CV) experiments to study the redox potential of the substrates. An oxidation peaks of sodium iodide, 1-(1phenylvinyl)cyclobutanol (1a), and p-toluenesulfonyl hydrazide (2a) in water was observed at 0.54, 1.24, and 1.67 V (see, Supporting Information). The CV studies had shown that iodide anion oxidation to molecular iodine could occur at first reaction step.¹⁰ Therefore, we suppose that sodium iodide acts both as an electrolyte and as a redox catalyst. We propose the reaction

mechanism shown in Figure 1 based on our results and previously reported work. $^{\rm 3-4,10-12}$ Arylsulfonyl





cathode: $H_2O \longrightarrow H_2 + \ThetaOH$

Figure 1. Proposed reaction mechanism.

hydrazide 2 reacts with molecular iodine to generate arylsulfonyl iodide intermediate which is decomposed to give arylsulfonyl radical I.¹² The arylsulfonyl radical I then reacts with 1-(1-arylvinyl)-cyclobutanol 1, yielding intermediate II, which is oxidized on anode to afford the cation III. 1,2-Alkyl migration of cation III leads to a ring expansion that yields the product 3.

In conclusion, we have presented an efficient synthesis of β sulfonated cyclic ketone derivatives via the electrochemical catalyzed oxidative sulfonylation/semipinacol rearrangement sequences of alkenylcyclobutanol derivatives **1** with arylsulfonyl hydrazides **2**. This approach is environmentally benign by using shelf-stable arylsulfonyl hydrazides as arylsulfonyl radical precursor and electrons as oxidizing reagents. The present protocol is an efficient option for synthesizing β -sulfonated cyclic ketone derivatives.

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Supplementary Material

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.tetlet

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Highlights:

• The first electrochemical radical

sulfonylation/ring expansion sequences.

• Environmentally benign protocol by using electrons as oxidizing reagents.

Accepted • Wide substrate scope with moderate to high yields.