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Iodine-mediated regioselective synthesis of vinyl triflones from styrenes with CF₃SO₂Na

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An unexpected regioselective synthesis of vinyl triflones was developed. This iodine-mediated C—H triflylation of styrenes with CF₃SO₂Na occurred at room temperature affording various vinyl triflones, which could be used for the preparation of other CF₃SO₂-containing compounds.

The wide application of fluorinated compounds in pharmaceuticals, agrochemicals, and materials¹ have resulted in increasing interest in fluorine-containing groups and fluorinating methods.² Among various fluorine-containing groups, the trifluoromethanesulfonyl (triflyl, SO₂CF₃, Tf) group is the strongest electron-withdrawing group with high lipophilicity.³ The CF₃SO₂-containing compounds are frequently used as structural units in bioactive compounds,⁴ catalysts or ligands,⁵ and advanced functional materials.⁶ In this context, substantial efforts have been devoted to the development of new synthetic methods of CF₃SO₂-containing compounds,^{2f} especially for aryl trifluoromethanesulfones (triflones).⁷ In contrast, much less attention has been paid in the preparation of vinyl triflones, despite these compounds are attractive in organic synthesis and pharmaceutical industry. Normally, vinyl triflones are prepared by transformation from alkyl triflones⁸ or alkynyl triflones (Scheme 1a).⁹ The intramolecular triflyl rearrangement, reported by the groups of Okauchi, Maas, and ours,10 provides an alternative approach to vinyl triflones (Scheme 1b). However, the direct incorporation of triflyl into alkenes has never been reported.

Pd- or Cu-catalyzed cross-coupling reactions of vinyl halides, vinyl boronic acids, or alkynes with sulfinate salts provides a routine and direct approach to vinyl sulfones.¹¹ However, the use of CF_3SO_2Na as a nucleophilic coupling partner remains to be a challenge, because the strong electron-withdrawing character of the triflyl group³ reduces its nucleophilicity.¹²



Recently, a variety of transition-metal-free protocols,^{13,14} especially iodine-catalyzed/mediated direct sulfonylation of alkene and alkyne derivatives¹⁴ have been developed for the efficient synthesis of vinyl sulfones. Inspired by these achievements, we were dedicated to exploring the direct triflylation of alkenes under transition-metal-free conditions. Herein, we report our progress in the iodide-mediated regioselective synthesis of vinyl triflones from styrenes with CF₃SO₂Na (Scheme 1c). To the best of our knowledge, this work represents the first example of direct C—H triflylation of alkenes.

We embarked on our studies with the repetition of the iodine-mediated synthesis of vinyl sulfones^{14e} by the reaction of 1-methyl-4-vinylbenzene **1a** and PhSO₂Na (Scheme 2a).



Scheme 2. I₂-mediated synthesis of vinyl sulfones

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triflones

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After obtaining vinyl sulfone **2a** in 86% yield, we then decided to switch PhSO₂Na to CF₃SO₂Na for the analogous synthesis of vinyl triflones (Scheme 2b). In contrast to the high yield of vinyl sulfone **2a**, the conversion of **1a** to vinyl triflone was low. Noteworthily, a novel unknown vinyl triflone **4a**, not the expected product **3a**,¹⁵ was obtained.

The above interesting result encouraged us to optimize the reaction conditions for improving the yield of vinyl triflone 4a (Table 1). First, a variety of iodine sources were screened. It was found that molecular iodine was the most effective (entry 1). NIS or PhI(OAc)₂/KI gave lower yields, while ICl could not promote this reaction (entries 2-4). Considering the fact that HI would be generated from the reaction system, the addition of a base should benefit this reaction. Indeed, higher yield of 4a was observed in the presence of an organic or inorganic base (entries 5-8). The reaction performed with two equivalents of NaHCO₃ gave the best result (entry 8). Subsequently, the effect of catalytic metal salt was evaluated (entries 9-12). Among the common metal salts, CuI could result in slightly higher yield (entry 9). Other metal salts, such as FeCl₂, were ineffective. Further screening of copper salts revealed CuCN was optimal, giving 4a in 57% yield (entry 12). Finally, different solvents including DCE, THF, DMF, and DMSO were investigated (entries 13-16). However, none of them gave better result than that of CH₃CN. It was noteworthy that lowering or elevating reaction temperature also resulted in lower yields (entries 17 and 18).

Table 1. Optimization of reaction conditions^a

	Y >> + c∈ si	metal (10 mol %)	(i	\sim
	. 01300	base, s	solvent		
	1a (3.0 e		12 11	/	4a
entry	iodine source	Base	metal	solvent	yield ^b (%)
1	l ₂	_	_	CH₃CN	18
2	NIS	—	—	CH₃CN	14
3	PhI(OAc)₂/KI	—	—	CH₃CN	11
4	ICI	—	—	CH₃CN	0
5	I ₂	DBU	—	CH₃CN	20
6	I ₂	NaOAc	—	CH₃CN	22
7	I ₂	K ₂ CO ₃	—	CH₃CN	37
8	l ₂	NaHCO ₃	—	CH₃CN	40
9	I ₂	NaHCO ₃	Cul	CH₃CN	45
10	I ₂	NaHCO ₃	FeCl ₂	CH₃CN	33
11	I ₂	NaHCO ₃	CuTc	CH₃CN	38
12	I ₂	NaHCO ₃	CuCN	CH₃CN	57
13	I ₂	NaHCO ₃	CuCN	DCE	trace
14	I ₂	NaHCO ₃	CuCN	THF	7
15	l ₂	NaHCO₃	CuCN	DMF	16
16	I ₂	NaHCO ₃	CuCN	DMSO	28
17 [°]	I ₂	NaHCO ₃	CuCN	CH₃CN	41
18^d	I ₂	NaHCO ₃	CuCN	CH₃CN	28

iodine source (3.0

^{*a*}Reaction conditions: **1a** (0.2 mmol), CF_3SO_2Na (0.6 mmol), iodine source (0.6 mmol), base (0.4 mmol), metal salt (0.02 mmol), solvent (1.0 mL), under air, rt, 12 h. ^{*b*}Yields determined by ¹⁹F NMR spectroscopy using trifluoromethoxybenzene as an internal standard. ^{*c*}50 ^{*o*}C. ^{*d*}O ^{*o*}C.

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^a Reaction conditions: 1 (0.5 mmol), CF_3SO_2Na (1.5 mmol), I_2 (1.5
mmol), NaHCO3 (1.0 mmol), CuCN (0.05 mmol), CH3CN (2.0 mL),
under air, rt, 12 h, isolated yields.

With the optimized reaction conditions in hand, the substrate scope of this protocol was then examined. As shown in Scheme 3, a range of styrenes (1a-1o) participated in the reaction giving vinyl triflones 4a-4o in moderate yields along with the recovery of the starting materials in low yields. In general, the electron-donating substituent such as alkyl, alkoxyl, or aryl group was essential for this transformation. The aromatic fluoride (1m), chloride (1n), and bromide (1o) were tolerated under the standard reaction conditions, thus providing opportunities for additional transformations. In the cases of styrenes bearing strong electron-withdrawing substituents including ester, nitro, and cyano, low conversion was obtained. Unfortunately, neither the sterically hindered ortho-substituted styrenes nor the unactivated alkenes were suitable substrates. It was noteworthy that benzofuran- or thiophene-derived styrenes (1p, 1q) were applicable to the reaction, affording the vinyl triflones 4p and 4q in moderate yields. The structure of product 4h was further confirmed by HMQC and DEPT135 NMR spectra (see ESI).

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Scheme 4. Transformation of compound 4g



Scheme 5. Gram-scale preparation of 10 from 1g



The hitherto unknown vinyl triflones **4** are compounds with great potential for further synthetic manipulation, notably as the excellent Michael addition acceptors. For instance, the reaction of vinyl triflone **4g** with different *C-*, *O-*, *N-*, and *S*-nucleophiles afforded the addition products **5-10** in good yields, respectively (Scheme 4). These compounds are formal difunctionalized products from styrene **1g** with the simultaneous introduction of one triflyl group and the other functional group. In fact, the gram-scale preparation of **10** could be performed directly from styrene **1g** (10 mmol) without the purification of intermediate **4g** (Scheme 5).

To gain insight into the reaction mechanism, the radical trap were experiments performed by adding 2,2,6,6tetramethylpiperidinyloxy (TEMPO) or 2,6-di-tert-butyl-4methylphenol (BHT) to the reaction mixture. Surprisingly, only trace of the desired product was detected in the presence of TEMPO, whereas BHT had no obvious effect on the reaction yield. We surmised that the detrimental effect of TEMPO might arise from its oxidation ability.¹⁶ Though we could not firmly preclude the radical pathway, we were more inclined towards a cationic reaction mechanism accordingly to our experimental results and other related works.¹⁷ As shown in Scheme 6, the electrophilic addition of iodine or CF₃SO₂I, in situ generated from the reaction of CF_3SO_2Na and I_2 , onto styrenes 1 gave a three-membered cyclic iodonium ion intermediate 11. Subsequent nucleophilic attack on 11 by CF₃SO₂Na in a Markovnikov fashion afforded iodotriflylated product 12. At last, spontaneous elimination of HI took place yielding vinyl triflones 4.14a,b,e,g-i However, the exact role of CuCN in this reaction remains unclear.



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Scheme 6. Proposed reaction mechanism

In conclusion, we have developed an iodine-mediated direct C—H triflylation of styrenes with CF_3SO_2Na . This reaction offers a new route for the regioselective synthesis of vinyl triflones, which can undergo Michael addition by different nucleophiles to afford a series of CF_3SO_2 -containing compounds. Further investigations of the mechanism and the application of this method are ongoing in our laboratory.

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