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Hypervalent Iodine in Synthesis XVIII: Synthesis of Selenosulfonates Using A One Pot Reaction of Diaryl Diselenides, Sodium Sulfinates and [Bis(trifluoroacetoxy)iodo] benzene

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Abstract: A one pot reaction to synthesis selenosulfonates is reported.

Selenosulfonates have attracted much attention in organic synthesis recently. They can react with olefins, allenes, acetylenes and diazomethane through electrophilic and free radical additions,[1] all of which have been named "selenosulfonations".

In 1947, Foss synthesized the selenosulfonates first using o-nitrobenzeneselenenyl bromide with sodium sulfinates[2]. Untill 80's some other methods for the preparation of the selenosulfonates became available, such as the oxidation of sulfonhydrazides or sulfinic acid with benzeneseleninic acid[1,3] and the reaction of sulfonhydrazides with selenenyl halides or trihalides[1]. Owing to the utility of the selenosulfonates in synthesis, there is still the need for exploring more convenient approaches to these compounds. In the present paper, we would like to report a new procedure for the preparation of selenosulfonates from diaryl diselenides, sodium sulfinates and [bis(trifluoroacetoxy)iodo]benzene (BTIB) in a one pot reaction.

In the course of the study of the hypervalent iodine in synthesis, we found that the Se-Se bond in diaryl diselenides can oxidatively be cleaved by BTIB to form selenenyl trifluoroacetate[4]. Considering the sodium sulfinates are good nucleophilic reagents and do not react with diaryl diselenides or BTIB in general conditions, we assumed that a one pot reaction of them for preparing selenosulfonates is possible.

In fact, when BTIB was added to the mixture of sodium sulfinates and diaryl diselenides in methylene chloride, the desired products were formed readily[5]. The results were summarized in Table 1.

Table 1. preparation of selenosulfonates

$$RSO_2Na + ArSeSeAr \xrightarrow[CH_2Cl_2]{PhI(OCOCF_3)_2} RSO_2SeAr$$

Entry	R of sodium sulfinate	Ar of diaryl diselenide	Yield of $RSO_2SeAr^{a,b}$%	mp^c, (lit mp)(℃)
1	$p-CH_3C_6H_4$	Ph	81	76−78, (77−79[6])
2	$p-CH_3C_6H_4$	$p-CH_3C_6H_4$	74	106−108
3	$p-CH_3C_6H_4$	$p-ClC_6H_4$	75	98−100
4	Ph	Ph	81	55−56, (55−58[1])
5	Ph	$p-CH_3C_6H_4$	80	55−57, (54[7])
6	Ph	$p-ClC_6H_4$	76	80−82, (75−77[7])
7	$p-ClC_6H_4$	Ph	83	97−99, (99−100[3])
8	$p-ClC_6H_4$	$p-CH_3C_6H_4$	81	144−145
9	$p-ClC_6H_4$	$p-ClC_6H_4$	68	146−149

a: Isolated yield. b: Satisfactory microanalysis obtained for unknown compounds: C±0.21; H±0.18.
c: All mp were uncorrected.

Our method can avoid some toxic reagents or seperated steps from diaryl diselenides in preparing selenosulfonates.

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[4] Previously, benzeneselenenyl trifluoroacetate was generated in situ from benzeneselenenyl bromide and silver trifluoroacetate in benzene: Reich, J., *J. Org. Chem.* **1974**, 39, 248; or from diphenyl diselenide and benzeneseleninic anhydride in the presence of trifluoracetic anhydride: Kutateladze, A. G.; Kice, J. L.; Kutateladze, J. G.; Zefirov, N. S. *J. Org. Chem.* **1993**, 58, 995.

[5] Typical Procedure: A suspension of the sodium sulfinate (2.0mmol) in CH_2Cl_2 (5mL) containing diaryl diselenide (0.50mmol) was cooled at 0℃ and BTIB (0.55mmol) in CH_2Cl_2(2mL) was added dropwise. The mixture was stirred at room temperature for 45 min. The reaction mixture was then washed with H_2O, dried over $MgSO_4$ and the CH_2Cl_2 was removed in vaccuo. After adding the petroleum ether (bp 30−60℃) and cooling in freezer overnight, the product was filtered and recrystallized in CH_2Cl_2 − Petroleum ether to afford pure compound. All compounds synthesized were characterized by mp, ^1H-NMR, IR.

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