

A CONVENIENT SYNTHESIS OF SELENOLSULFONATES FROM THE OXIDATION OF
SULFONHYDRAZIDES WITH BENZENESELENINIC ACID

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Abstract The reaction of sulfonylhydrazides with benzeneseleninic acid furnishes selenolsulfonates in high yield.

In connection with our current studies of the "selenosulfonation" of olefins¹, we required a convenient method for preparing selenolsulfonates 1. A limited number of these compounds has been previously reported, usually from the reaction of areneselelenyl bromides with sodium arenedisulfonates². We recently observed that the oxidation of N-acylhydrazines with commercially available benzeneseleninic acid (2) provides an efficient synthesis of selenolesters³. We now report that this technique may be extended to the preparation of diverse selenolsulfonates from the corresponding sulfonylhydrazides by the following process



Typically, a dichloromethane solution of the sulfonylhydrazide (20 mmol) was added over 15-30 min to a suspension of benzeneseleninic acid (20 mmol) in dichloromethane. A vigorous reaction with gas evolution was observed. The pale yellow solution was evaporated to dryness under reduced pressure and the solid residue crystallized from methanol to afford the corresponding selenolsulfonate in a high state of purity.

The products listed in the accompanying Table include variously substituted areneselelenolsulfonates 1a-f as well as phenyl methaneselenolsulfonate 1g. Clearly, the above method provides an efficient and convenient synthesis of this interesting class of selenenyl pseudo-halides.

Table

Preparation of Selenolsulfonates RSO_2SePh (1)^a

<u>Entry</u>	<u>R</u>	<u>Isolated Yield (%)</u>	<u>m p</u>
1a	Phenyl	88	56-58 ^{ob}
1b	p-Tolyl	92	80 ^{oc}
1c	Mesityl	100 ^d	75-76 ^o
1d	p-Methoxyphenyl	83	79-80 ^o
1e	m-Nitrophenyl	82	125-127 ^o
1f	2,4-Dinitrophenyl	68	147-150 ^o
1g	Methyl	65	88-90 ^o

(a) Products were characterized by C, H, N, S analysis as well as by their ir, nmr and mass spectra. (b) Lit.^{2a} m p. 51^o (c) Lit ⁴ m.p. 77-79^o. (d) The hydrazide and an equimolar amount of triphenylphosphine⁵ were added to 2 molar equivalents of 2, the product was isolated by preparative tlc

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References and Notes

1. T G. Back and S. Collins, following paper.
- 2 (a) T. Austad, Acta Chem. Scand., **A**, 30, 479, (1976). (b) O. Foss, J. Am Chem Soc., **69**, 2236, (1947).
- 3 T.G Back and S Collins, Tetrahedron Letters, 2661, (1979)
- 4 D.H R. Barton, M R Britten-Kelly and D Ferreira, J C.S Perkin I, 1090, (1978).
- 5 The simultaneous generation of PhSeOH in the reaction mixture by the reaction of 2 with triphenylphosphine provides increased yields of selenolesters from N-acylhydrazines.³ This tactic is generally not recommended in the preparation of selenolsulfonates as the inconvenience of chromatographic removal of triphenylphosphine oxide outweighs the slightly improved yields.

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