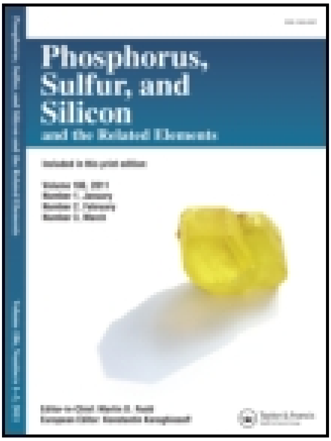

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Synthesis of Thioacrolein S-Oxide and Thioketene S-Oxide using FVT

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Synthesis of Thioacrolein *S*-Oxide and Thioketene *S*-Oxide using FVT

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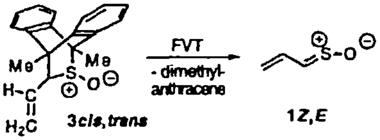
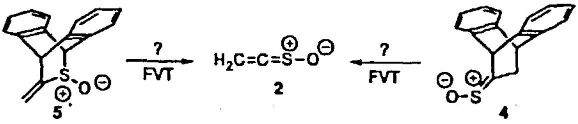
FVT was used to synthesize thioacrolein *S*-oxide and thioketene *S*-oxide. These reactive species were characterized at low temperature by IR and/or NMR spectroscopies.

INTRODUCTION

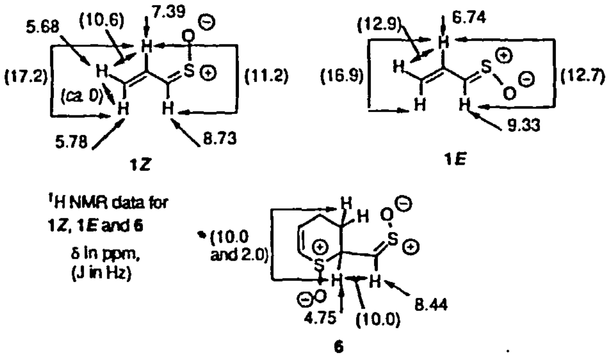
In order to synthesize thioacrolein *S*-oxide **1** and thioketene *S*-oxide **2**, we prepared the retro Diels-Alder precursors **3**, **4** and **5**.





THIOACROLEIN *S*-OXIDE[1] :

FVT of the vinylic sulfoxide **3** at 600°C quantitatively gave 9,10-dimethylanthracene. Volatile products were trapped at -196°C and analyzed by NMR spectroscopy. The ^1H NMR and ^{13}C NMR spectra were in good agreement with the structure of thioacrolein *S*-oxide. They revealed the formation of two isomeric sulfines. The ratio ***1Z*/*1E*** was 78/22. When the sample was allowed to stand at room temperature, the sulfine signals slowly disappeared and a new product was mainly formed. Signals observed by ^1H NMR and ^{13}C NMR were tentatively assigned to the dimeric structure **6**.

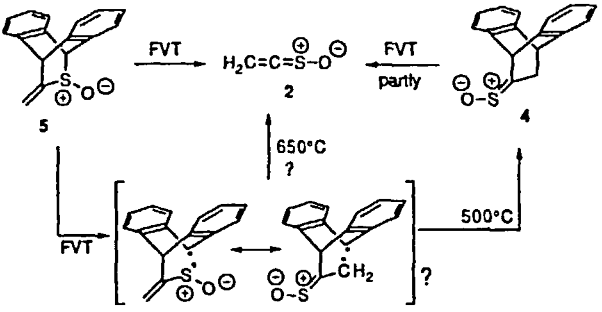


1Z **1E** **6**

^{1}H NMR data for 1*Z*, 1*E* and 6
δ in ppm,
(J in Hz)

THIOKETENE *S*-OXIDE[2] :

The major product obtained by FVT of precursor 4 was the corresponding ketone (60% yield estimated by NMR) resulting from sulfur extrusion. However some anthracene was also obtained and bands at 1700 and 1040 cm^{-1} were observed in the IR spectrum of the volatile products. These bands were tentatively assigned to thioketene *S*-oxide. Furthermore the same IR bands were observed after FVT of the second precursor 5 at 650°C. Interestingly when this precursor was thermolyzed at lower temperature (500°C) formation of the sulfine 4 was noticed. We propose that this rearrangement occurs *via* homolytic cleavage of the dibenzylic C-S bond.



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[1] N. Pelloux-Léon, R. Arnaud, J.-L. Ripoll, P. Beslin, Y. Vallée, *Tetrahedron Lett.*, 38, 1385–1388 (1997).

[2] N. Pelloux-Léon, F. Minassian, J. Levillain, J.-L Ripoll, Y. Vallée, *Tetrahedron Lett.*, 38, 4813–1816 (1998).