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## Phosphorus, Sulfur, and Silicon and the Related Elements

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

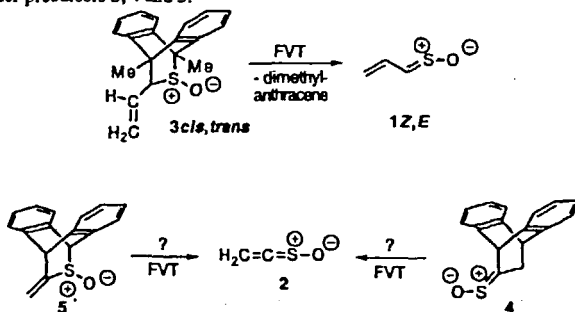
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FVT was used to synthesize thioacrolein *S*-oxide and thioketene *S*-oxide. These reactive species were characterized at low temperature by IR and/or NMR spectroscopies.

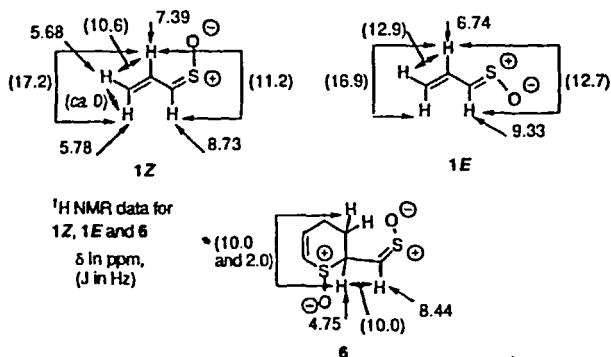
### INTRODUCTION

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



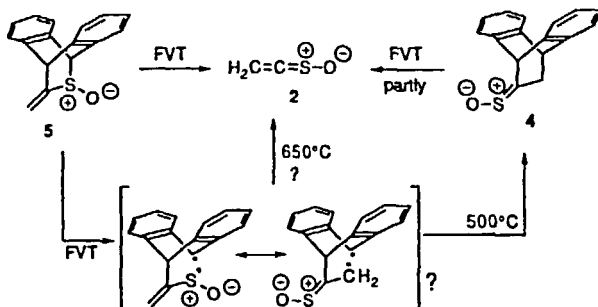
### THIOACROLEIN *S*-OXIDE<sup>1</sup>:

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 <sup>1</sup>H NMR and <sup>13</sup>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 <sup>1</sup>H NMR and <sup>13</sup>C NMR were tentatively assigned to the dimeric structure 6.



### THIOKETENE S-OXIDE<sup>2</sup>:

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<sup>-1</sup> 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.



### References

- [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).