

INITIAL  $\beta$ -LACTAM FORMATION IN THE ADDITION OF N-CHLOROSULFONYL  
ISOCYANATE TO DIPHENYLMETHYLENECYCLOPROPANE

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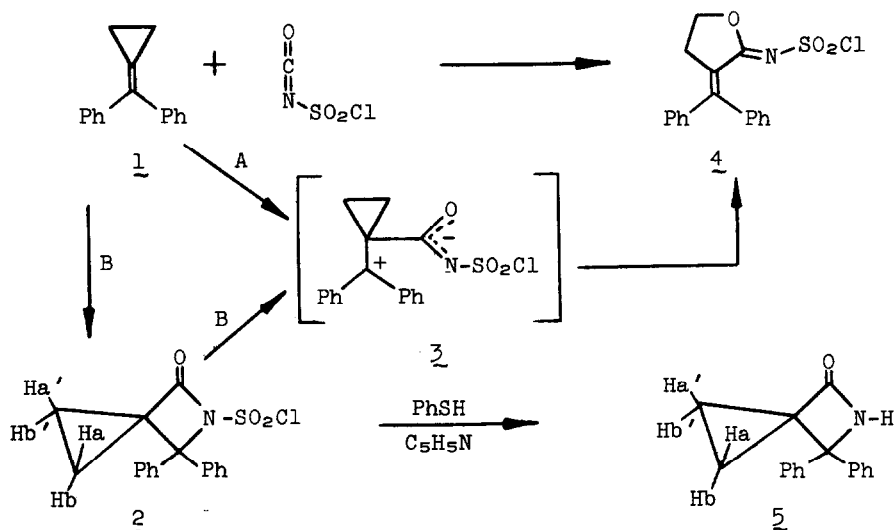
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Recently we reported that the addition of N-chlorosulfonyl isocyanate (CSI) to a vinylcyclopropane proceeded through initial formation of the  $\beta$ -lactam (a  $\pi 2s + \pi 2a$  cycloaddition) which could either be reduced and isolated, or allowed to thermally rearrange through carbon-nitrogen bond heterolysis.<sup>1</sup> It was therefore of considerable interest to note the recent report of Dunkelblum<sup>2</sup> that CSI added to diphenylmethylenecyclopropane (1) to afford the ring-opened iminolactone (4). The proposed mechanism (path A) involved initial formation of dipolar intermediate 3, followed by ring opening of the cyclopropylcarbinyl cation and closure through oxygen. On the basis of our previous work we would have predicted that the addition of CSI to 1 would proceed through initial formation of  $\beta$ -lactam (2) which could then open to 3. Thus a reinvestigation of this reaction was undertaken.

Reaction of CSI with an equimolar amount of 1 in  $\text{CDCl}_3$  occurred rapidly to yield a single product with the following NMR spectrum:  $\delta$  (Hz) 7.33 (s, 10H, aromatic); 1.60 (m, 2H); 1.10 (m, 2H) with the two upfield multiplets as mirror images. Coupled with the carbonyl absorption at  $1828 \text{ cm}^{-1}$  ( $\text{CHCl}_3$ ) this data strongly suggested  $\beta$ -lactam (2) as the initial reaction product. After 24 hours at room temperature, the NMR spectrum now exhibited the peaks

reported<sup>2</sup> for 4

Reaction of CSI with 1 in  $\text{CHCl}_3$  followed immediately by thiophenol/pyridine reduction at  $-30^\circ$ , afforded  $\beta$ -lactam 5 in 45% isolated yield: mp 168-70°. Structural assignment of 5 was based on the following spectral evidence: NMR  $\delta$  (Hz) 7.21 (s with broad base, 11H, aromatic and N-H), 1.26 (m, 2H), 0.91 (m, 2H) (mirror image multiplets); IR ( $\text{CHCl}_3$ )  $\nu_{\text{CO}}$  1767  $\text{cm}^{-1}$ ; mass spectrum  $\text{P}^+$  249 m/e; high resolution mass spectrum 249.1152 ( $\text{C}_{17}\text{H}_{15}\text{NO}$  calcd. 249.1154).



It is therefore evident that the formation of 4 proceeds first through 2 which suffers thermal heterolysis to 3. The formation of 2 in spite of the considerable ring strain involved and high stability of the dipolar intermediate 3 adds to the mounting evidence<sup>3</sup> for a concerted pathway in the CSI addition to olefins.

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#### REFERENCES

- (1) T. J. Barton and R. J. Rogido, *Chem. Comm.*, in press.
- (2) E. Dunkelblum, *Tetrahedron Lett.*, 1551 (1972).
- (3) For evidence of this cycloaddition process see: E. J. Moriconi and W. C. Meyer, *J. Org. Chem.*, **36**, 2841 (1971) and references therein.