



# Synthesis of Alkynes from Aldehydes and Ketones via $\alpha$ -Substituted N-Tosyl Hydrazones

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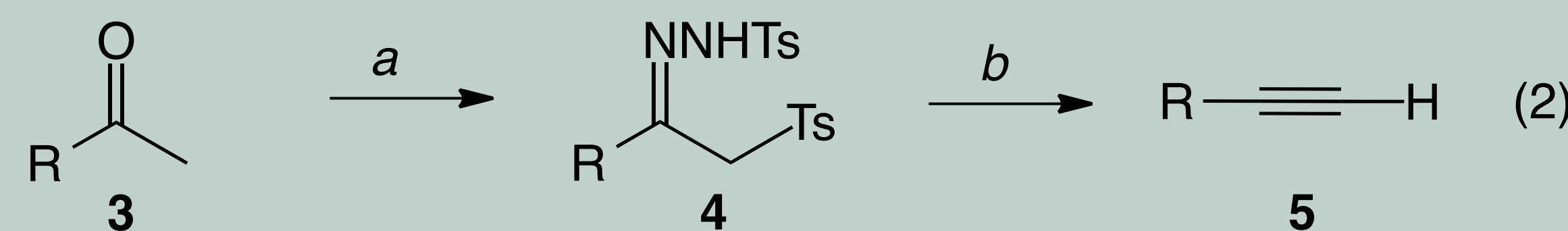
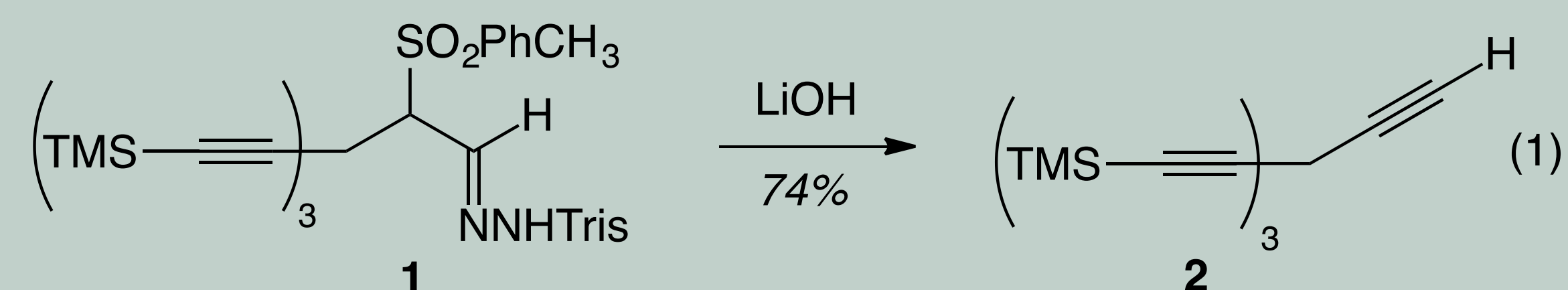


## Abstract

Alkynes are desirable functional groups due to their versatility in conversion to other functional groups as well as their use in metal mediated coupling reactions. Both internal and terminal alkynes can be formed from ketones and aldehydes via  $\alpha$ -substituted N-tosyl hydrazone intermediates in good yields. The versatility and breadth of the reaction is being investigated using variously substituted aldehydes and ketones. The research describes the synthesis of the  $\alpha$ -substituted N-tosyl hydrazones and the elimination reaction to form the alkynes.

## Introduction

In the synthesis of tetraethynylmethane **2** the last alkyne was introduced through the elimination of a sulfone and N-trisylhydrazone (eq 1).<sup>1</sup> As with the synthesis of **2** incorporation of the final alkyne in our synthesis of tetraethynyladamantane has proven challenging. This research seeks to determine scope and versatility of the reaction.



a. 1. LDA, 2. TsCl, 3. H<sub>2</sub>NNHTs b. LiOH

**3a** R = -Ph

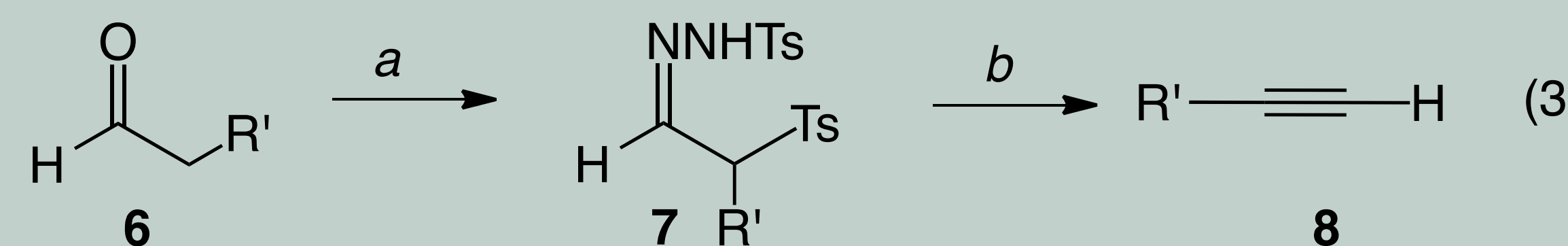
**4a** 27%

**5a** not isolated

**3b** R = -C(CH<sub>3</sub>)<sub>3</sub>

**4b** 27%

**5b** not isolated

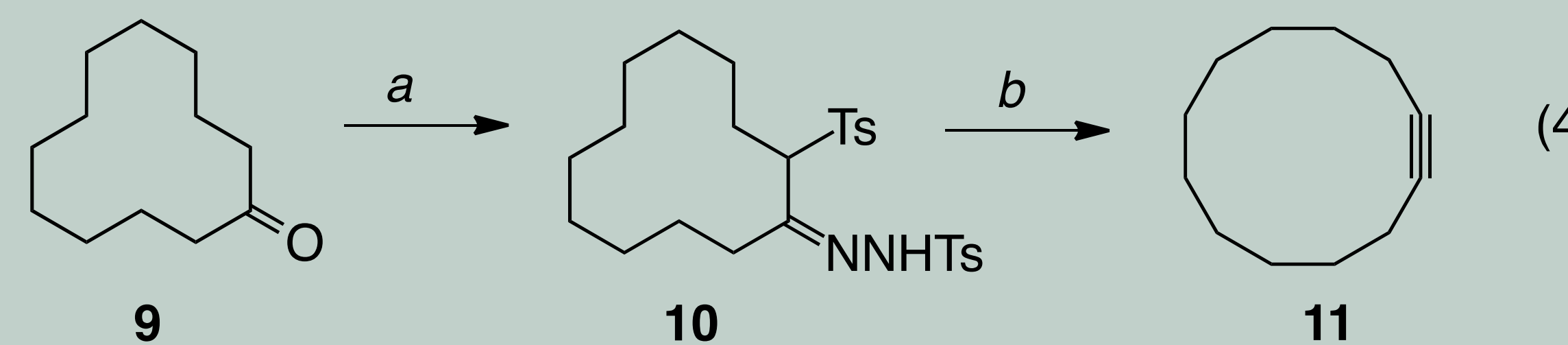


a. 1. LDA, 2. TsCl, 3. H<sub>2</sub>NNHTs b. LiOH

R' = -(CH<sub>2</sub>)<sub>5</sub>CH<sub>3</sub>

68%

89%



a. 1. LDA, 2. TsCl, 3. H<sub>2</sub>NNHTs b. LiOH

## Discussion

- Addition of the  $\alpha$ -tosyl group proved more challenging than anticipated. Crude <sup>1</sup>H NMR indicated the presence of product, but isolation of the product by column chromatography was elusive.
- Due to the difficulty in obtaining pure products, the reactions were carried through using crude reaction products. Hence, no isolated yields are reported.
- Phenylacetylene and 1-heptyne showed IR stretches at 2254 cm<sup>-1</sup> and 2253 cm<sup>-1</sup> respectively.
- One problem may be the volatility of the products. Future work will include trapping the resultant alkyne with a silyl protecting group.

## Conclusion

The reaction shows promise as an alternative method of producing alkynes. Further research will focus on isolation and purification of the intermediates and products.

## Acknowledgements

Funding for this project was provided by NH-INBRE grant.

<sup>1</sup> Feldman, K. S.; Kraebel, C. M.; Parvez, M. *J. Am. Chem. Soc.* **1993**, 115, 846.