Synthesis of Mono-Substituted 1-Ethynyladamantane

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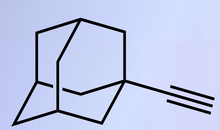
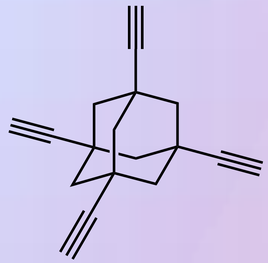
ABSTRACT

The model system for the synthesis of 1,3,5,7-tetraethynyladamantane was completed in five steps with an undetermined overall yield. The monosubstituted 1-ethynyladamantane was characterized by IR spectroscopy and qualitative analysis. Upon optimization of the overall yield, this will be applied to the synthesis of the tetrasubstituted adamantane.



Particular Goal:
1-Ethynyladamantane



Future Goal:
1,3,5,7-Tetraethynyladamantane

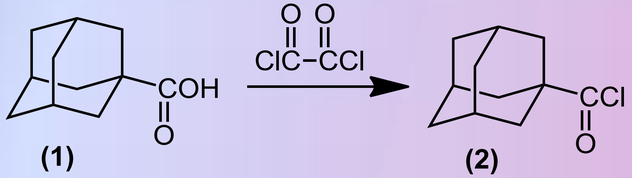
INTRODUCTION

The goal of this particular research project is to synthesize a terminal alkyne, 1,3,5,7-tetraethynyladamantane, via a five step reaction process. We began with the synthesis of 1-ethynyladamantane, the monosubstituted form, as a model system.

Terminal alkynes are useful intermediates in organic synthesis because they are able to undergo numerous reactions including electrophilic addition, nucleophilic addition, hydroboration, hydration, reduction, and organometallic coupling reactions.[1] In addition, the adamantane compound has been incorporated into polyacrylates, polyesters, and polycarbonates increasing their thermal stability and glass transition temperatures.[2] Based on the usefulness of both the adamantane compound and terminal alkynes, the ability to synthesize 1,3,5,7-tetraethynyladamantane more efficiently would be convenient for future reactions and research.

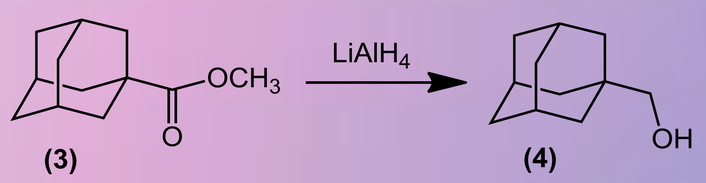
RESULTS

Conversion of Carboxylic Acid to Carbonyl Chloride



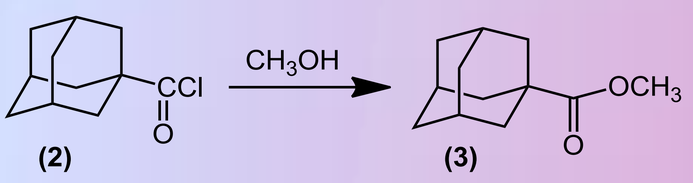
IR Peaks (cm^{-1}): 1795.07 (Cl-C=O)

Conversion of Methyl Ester to Primary Alcohol



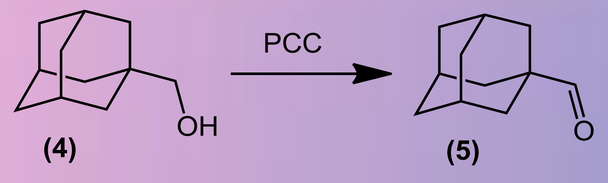
IR Peaks (cm^{-1}): 3222.48 (-OH)

Conversion of Carbonyl Chloride to Methyl Ester



IR Peaks (cm^{-1}): 1737.08 (C=O); 1238.03 (C-O)

Conversion of Primary Alcohol to Aldehyde



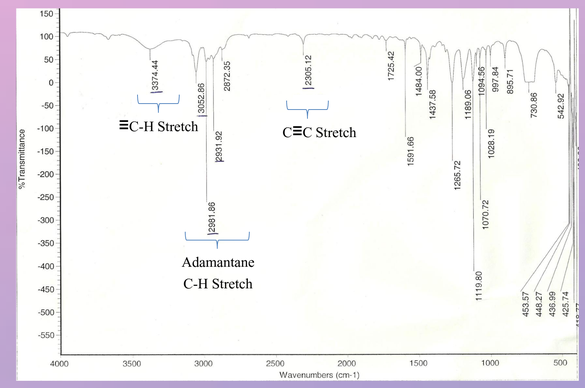
IR Peaks (cm^{-1}): 1719.28 (C=O)

References

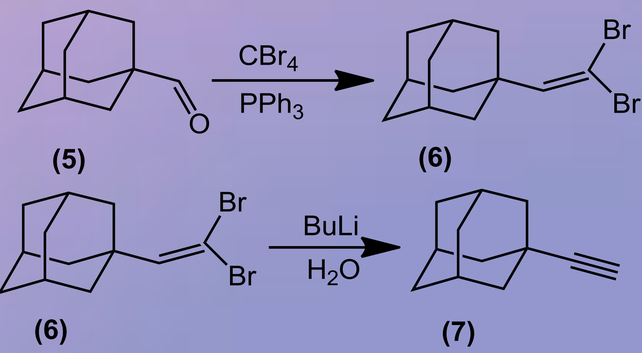
[1]Brown, William; Foote, Christopher; Iverson, Brent; Anslyn, Eric. Infrared Spectroscopy. *Organic Chemistry*, 5; Brookes/Cole Cengage Learning: Belmont, CA; 2005; 277-8.

[2]Malik, A.A; Archibald, T.G.; Baum, K.; Unroe, M.R. Thermally Stable Polymers Based on Acetylene-Terminated Adamantanes. *Journal of Polymer Science: Part A: Polymer Chemistry* **1992**, 30, 1747-1754.

INFRARED SPECTRUM OF 1-ETHYNYLADAMANTANE



COREY FUCHS REACTION



(6) – IR Peaks (cm^{-1}): 1588.11 and 731.12 (=CBr_2 stretch)

(7) – IR Peaks (cm^{-1}): 3374.44 (≡C-H stretch); 2305.12 (C≡C stretch)

Baeyer Test and Bromine Test: Positive for alkene or alkyne

Iodine Test: Positive for alkyne

CONCLUSIONS

The proposed synthesis shows great promise as completion of all five steps was accomplished for the monosubstituted product. Each step was confirmed by IR spectroscopy. Upon optimization of yield for the reaction intermediates and the final product, the proposed reaction sequence will be applied to the synthesis of 1,3,5,7-tetraethynyladamantane.

ACKNOWLEDGEMENTS

I would like to thank Dr. Carolyn Weinreb for all of her guidance and patience during this research project. I would also like to thank Mark St. Peter for his work on this research project in the spring of 2011.