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.

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. In addition, the adamantane compound has been incorporated into polyacrylates, polysteres, and polycarbonates increasing their thermal stability and glass transition temperatures. 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 Carboxyl Chloride

\[
\text{Reaction: } \text{COOH} + \text{Cl}_2 + \text{CCl}_4 \rightarrow \text{COCI}_2 + \text{COCl} \\
\text{IR Peaks (cm}^{-1}\text{): } 1795.07 (\text{Cl-C=O})
\]

Conversion of Carboxyl Chloride to Methyl Ester

\[
\text{Reaction: } \text{COCI}_2 + \text{CH}_3\text{OH} \rightarrow \text{COCH}_3 + \text{HOCI} \\
\text{IR Peaks (cm}^{-1}\text{): } 1737.08 (\text{C=O}); 1238.03 (\text{C-O})
\]

Conversion of Methyl Ester to Primary Alcohol

\[
\text{Reaction: } \text{COCH}_3 + \text{LiAlH}_4 \rightarrow \text{OH} + \text{CH}_3\text{LiAlH}_3 \\
\text{IR Peaks (cm}^{-1}\text{): } 3222.48 (-\text{OH})
\]

Conversion of Primary Alcohol to Aldehyde

\[
\text{Reaction: } \text{OH} + \text{PCC} \rightarrow \text{CHO} + \text{H}_{2}\text{O} \\
\text{IR Peaks (cm}^{-1}\text{): } 1719.28 (\text{C=O})
\]

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.

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