Synthesis of N-2-Hydroxyethyltrichloroacetamide

Possible Precursor to Polycarbamate

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Abstract

- ❖ The reaction of hexachloroacetone (HCA) with 2-aminoethanol gave N-2-hydroxyethyltrichloroacetamide as a white solid in high yield.
- ❖ The compound was further reacted with another HCA to produce the corresponding trichloroacetate derivative, $\text{Cl}_3\text{C-C(O)NHCH}_2\text{CH}_2\text{OC(O)CCl}_3$ at room temperature.

- ❖ Both compounds have been spectroscopically characterized, and the later compound will be further reacted with 2-aminoethanol and HCA in sequence to extend the chain which will lead to polycarbamate.

Preparation of N-2-Hydroxyethyltrichloroacetamide

- ❖ N-2-hydroxyethyltrichloroacetamide was prepared from the reaction of 2-aminoethanol and HCA in chloroform at 0°C in high yield as a gel-like liquid which was solidified in hours.

- ❖ The reaction was an exothermic reaction.
- ❖ The trichloroacetamide compound has been spectroscopically characterized by ^1H NMR, ^{13}C NMR, and FT-IR.

Preparation of 2-Trichloroacetamidoethyltrichloroacetate

- ❖ N-2-hydroxyethyltrichloroacetamide was further reacted with HCA to give 2-trichloroacetamidoethyltrichloroacetate in DMSO at room temperature.

- ❖ The amide/ester compound could be also prepared from the reaction of 2-aminoethanol with two equivalents of HCA in THF as an oily liquid, but was solidified in a few hours.

Solvent Effect

- ❖ It was interesting to observe that the reaction between N-2-hydroxyethyltrichloroacetamide and hexachloroacetone (HCA) was not happened in chloroform.

- ❖ However, the same reaction immediately gave disubstituted compound, 2-trichloroacetamidoethyltrichloroacetate, in a polar solvent such as DMSO, acetone or THF.

Introduction

- ❖ The reactions of aliphatic diamines with HCA gave bis(trichloroacetamide) derivatives at room temperature.
- ❖ Although the same reactions with aromatic diamines similarly gave bis(trichloroacetamide) compounds, the reactions required more vigorous condition such as reflux and extended reaction time.
- ❖ Aromatic amines are less reactive than aliphatic amines due to lower nucleophilicity.

^1H NMR: N-2-Hydroxyethyltrichloroacetamide

^1H NMR: 2-Trichloroacetamidoethyltrichloroacetate

CONCLUSIONS

- ❖ N-2-hydroxyethyltrichloroacetamide was prepared from the reaction of 2-aminoethanol and HCA in chloroform at 0°C.

- ❖ N-2-hydroxyethyltrichloroacetamide was further reacted with HCA to give 2-trichloroacetamidoethyltrichloroacetate in DMSO, but the same reaction didn't occur in CHCl_3 .

Introduction

- ❖ Three N-hydroxyphenyltrichloroacetamide derivatives were also prepared by the reaction of hexachloroacetone (HCA) with o, m, or p-aminophenol after refluxing overnight in CHCl_3 or THF (ortho derivative).

- ❖ But no evidence of the hydroxy group activation to produce ester compounds has been observed.

- ❖ The compounds have been spectroscopically and structurally characterized by ^1H NMR, ^{13}C NMR, and x-ray diffraction.

^{13}C NMR: N-2-Hydroxyethyltrichloroacetamide

^{13}C NMR: 2-Trichloroacetamidoethyltrichloroacetate

FUTURE WORKS

- ❖ 2-trichloroacetamidoethyltrichloroacetate prepared will be further reacted with 2-aminoethanol and HCA in sequence to extend the structure which may lead to a polycarbamate.

- ❖ Also, various aminoalcohols such as 2-aminopropanol, 3-aminopropanol will be reacted with HCA to compare the result with the 2-aminoethanol derivatives.

Preparation of Aromatic Polycarbamates

- ❖ Various reactions have been carried out with the three N-hydroxyphenyltrichloroacetamide derivatives to produce polycarbamates through a reflux process in DMSO or DMF, and by using a microwave reactor, however, no reaction has successfully produced polycarbamate yet.

- ❖ Reduced electrophilicity of the carbonyl group may be an obstacle for the reaction to a polycarbamate.

FT-IR: N-2-Hydroxyethyltrichloroacetamide

FT-IR: 2-Trichloroacetamidoethyltrichloroacetate

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