

SYNTHESIS OF TRIETHYL PROPARGYL AMMONIUM BROMIDE FROM TRIETHYL AMINE WITH PROPARGYL BROMIDE

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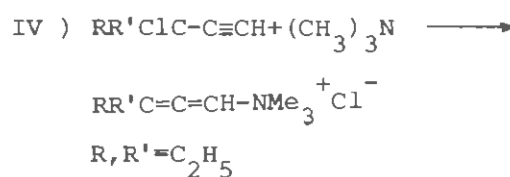
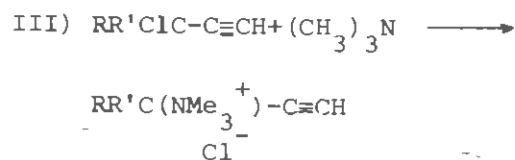
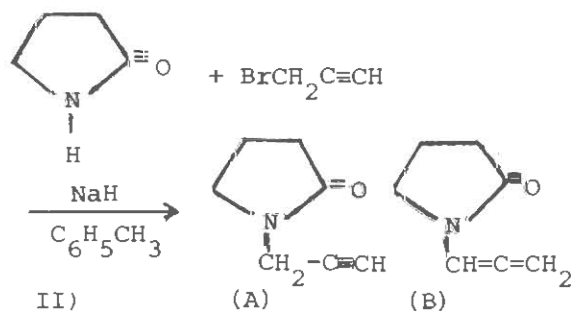
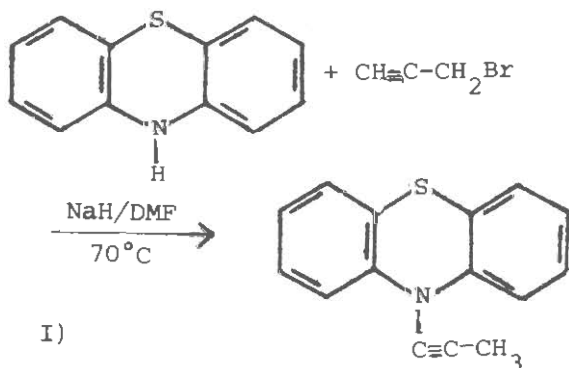
(Approved 18th Dec. 1989)

ABSTRACT

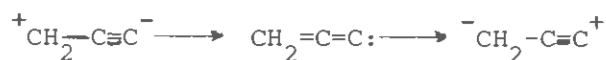
The reaction between triethylamine with propargyl bromide was studied and the structure of triethyl propargyl ammonium bromide was illustrated by ^1H NMR, IR, and Mass spectra.

INTRODUCTION

Most of the time, the exact reaction mechanism of addition of a propargyl halide to an amine, is difficult to predict. This is due to different factors involved e.g. basicity, nucleophilicity, the propargylic compound, solvent, temperature, and the steric hindrances. For example:



Reaction (I) was explained by Hen - nion, et.al [1-3] with the use of a dipolar ion, $^+\text{CH}_2-\text{C}\equiv\text{C}^-$, which has the following resonance forms:



In order to get the product (I), the nitrogen atom from phenothiazine attacks nucleophilically the acetylenic carbon.

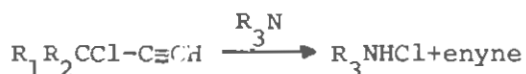
Cho[4] reported that the reaction between 2-pyrrolidone and propargyl bromide, in the presence of a base, would produce compound (A). Other reports contradicted Cho's results and

mentioned (B) as the product [5-6].

In reactions (III) and (IV), Hennion et al. contend that the product may be propargylic or allenic, depending on the sizes of R_1 and R_2 in:



They also claim that the amines with their pK_b about 3.5 to 7.5 are suitable for the addition reactions (III) and (IV). If the basicity is lower, the reaction might be either too slow or it would never happen. And if the basicity of the amine is too high, then the elimination reaction would occur [8]:



EXPERIMENTAL PROCEDURE

After trying different solvents, commercial acetone was chosen as the base solvent for the synthesis. To a mixture consisting of 10.5 ml (75 mmole) of freshly distilled triethylamine, and 40 ml acetone in a 100 ml flask, in an ice water bath, equipped with a magnetic stirrer, 3.77 ml (50 mmole) propargyl bromide was added dropwise.

After addition of the first few drops of propargyl bromide to the flask, a white precipitate was formed and upon completion, the flask was left undisturbed for five hours. Clear crystals were formed. After filtering, the

crystals were washed, two to three times with 5-10 ml dry ether. Recrystallization from acetonitrile gave 98% yield, m.p.: 197-8°C.

Triethyl propargyl ammonium bromide is soluble in polar solvents, e.g. acetonitrile, DMSO, water, and dimethyl formamide. It is insoluble in nonpolar solvents, e.g. ether, acetone, ethyl acetate. Mass spect: $\frac{m}{e}$ 118, 111, 101, 96, 86, 39.

IR spect: 3150 cm^{-1} , 2120 cm^{-1} , 750 cm^{-1}
 1H NMR spect; (in THF): δ 1.1 (t, 12H); 3.6 (q, 6H); 4.3 (d, 3H); 2.8 (t, H). The peak in 3.8 disappears in D_2O .

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