

Preparation of *N,N*-Diethyl-*m*-toluamide (DEET) and its Identification by IR and NMR Spectroscopy

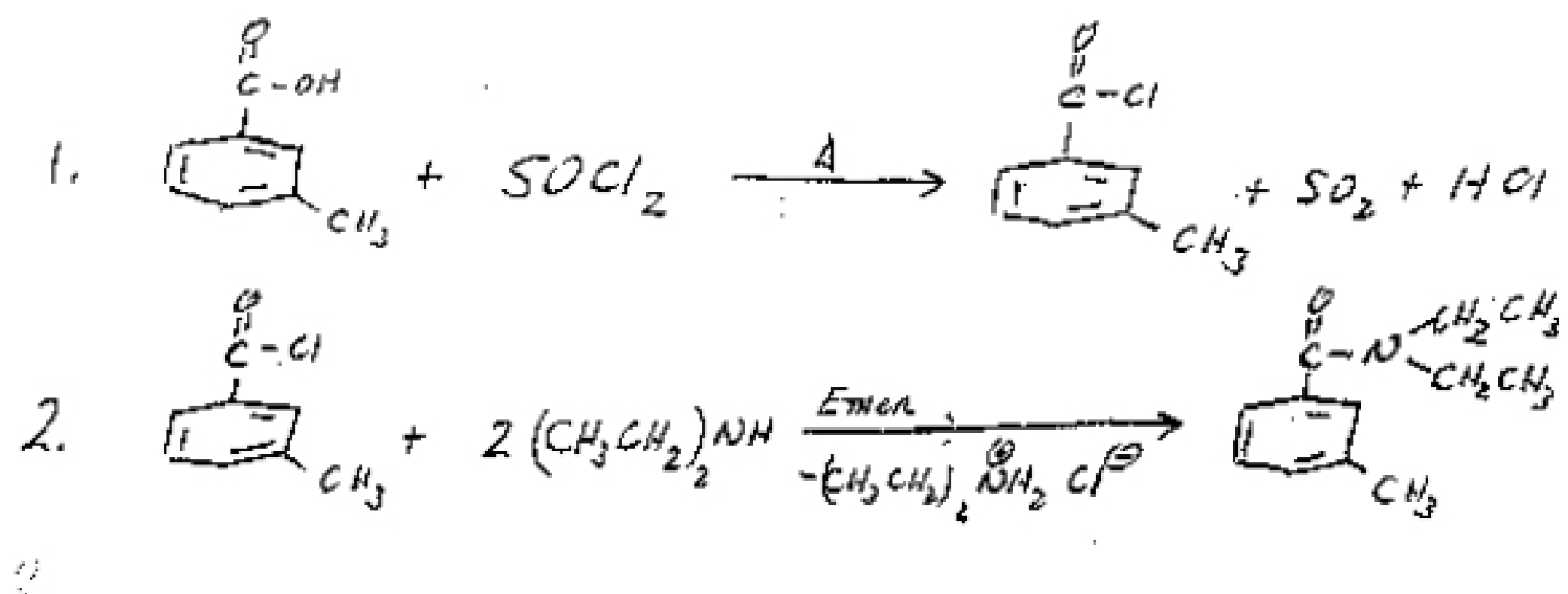
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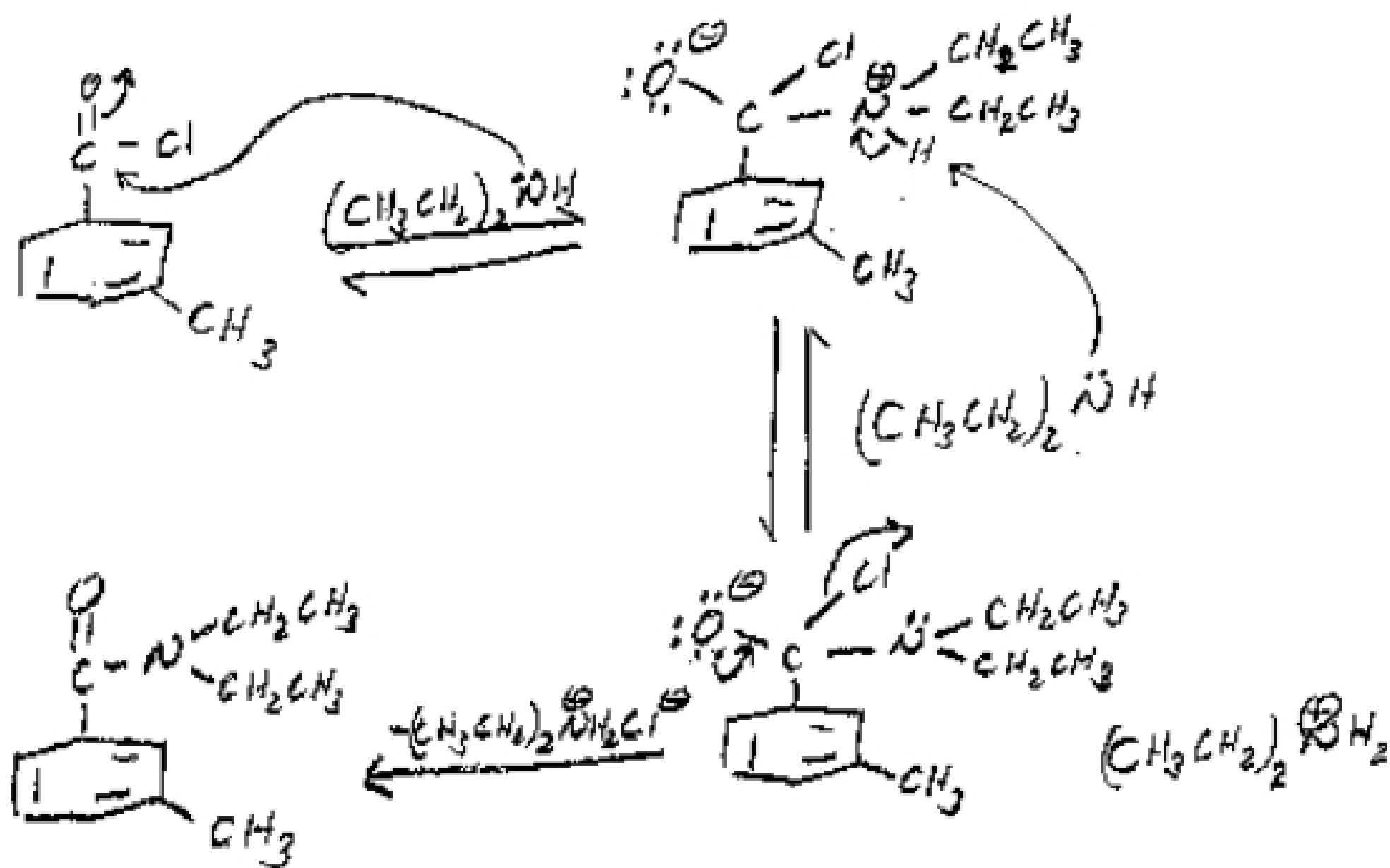
INTRODUCTION

N,N-Diethyl-*m*-toluamide (DEET) is the active ingredient in many insect repellants.¹ In this experiment, DEET will be prepared from *m*-toluic acid through the intermediate acid chloride and then identified by its IR and proton NMR spectra. The overall reaction is a series of two acyl substitutions and is shown in Scheme 1 below:



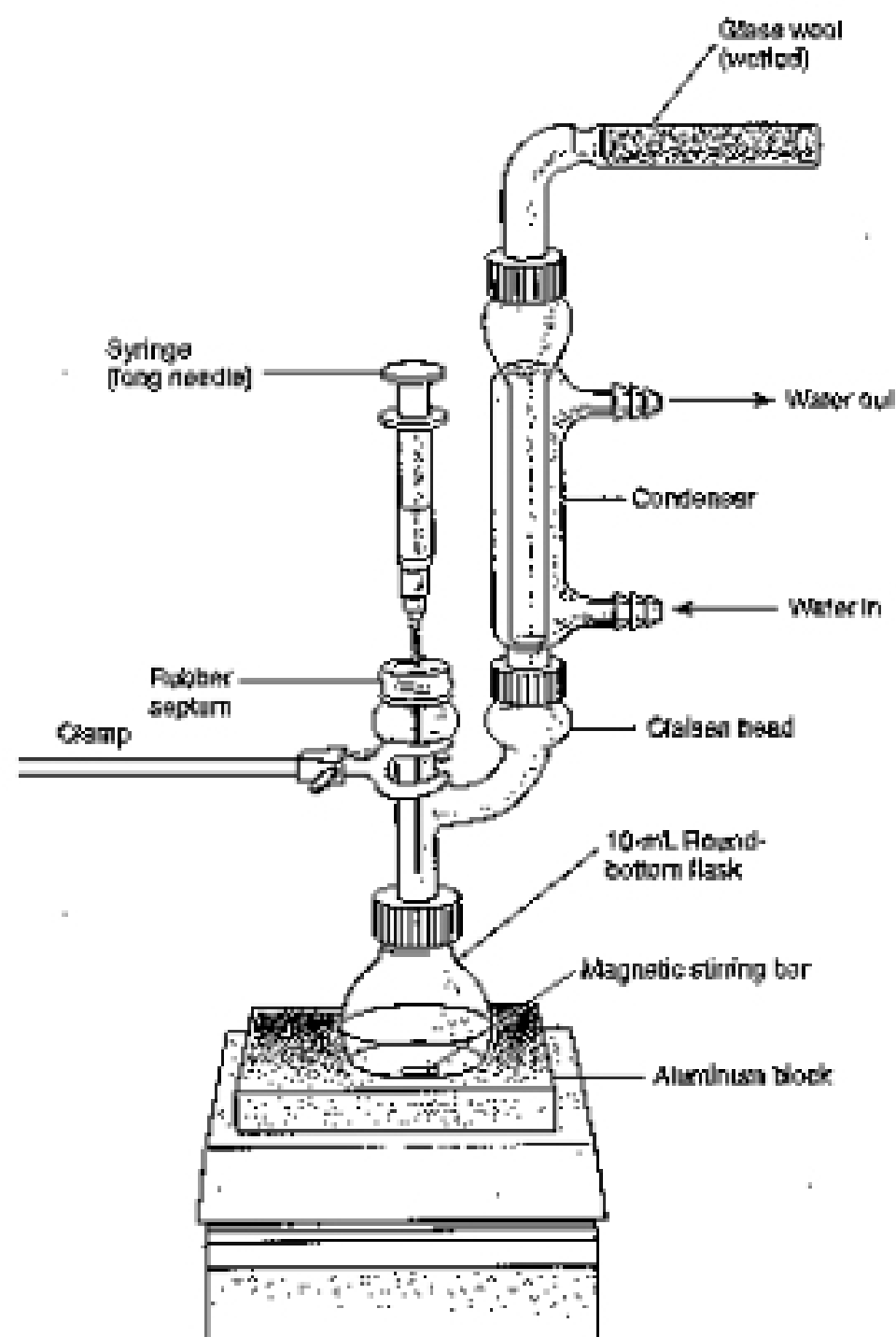
Scheme 1

Carboxylic acids can be converted to acid chlorides by treatment with a compound such as phosphorus trichloride (PCl_3) or thionyl chloride (SOCl_2).² The reactive acid chloride can then be treated with a primary or secondary amine to give the amide along with HCl, which reacts with the excess amine to give an alkylammonium chloride salt. The mechanism of this process is shown on the following page in Scheme 2:



Scheme 2

To carry out this reaction, the apparatus shown in Figure 1 will be assembled. **The apparatus must be dry,** since thionyl chloride will react with water to give sulfur dioxide and hydrogen chloride.⁴



Apparatus for Experiment 45. Note: A long syringe needle is recommended.

Figure 1³

m-Toluic acid and thionyl chloride will be charged into the reaction flask (USE A HOOD!), then boiled gently to effect the formation of the acid chloride. The acid chloride will then be cooled to room temperature and dissolved in *anhydrous* ether. Ice-cold diethylamine will be added as a solution in anhydrous ether to form the amide. The resulting ether mixture will be washed with aqueous sodium hydroxide (to remove any excess acid chloride), then washed with hydrochloric acid (to remove any excess diethylamine). The resulting ether solution of DEET will be dried and evaporated to yield the crude product, which will then be purified by column chromatography to afford pure DEET. The percent yield from *m*-toluic acid will be determined and the product will be analyzed by transmission infrared spectroscopy (IR) as a neat sample using NaCl plates⁵ to confirm its structure by (1) looking at the major absorptions and comparing them to a correlation table⁶ and (2) by comparing the spectrum to that of an authentic sample. The major IR absorption is expected to be the amide carbonyl at $1640 - 1700 \text{ cm}^{-1}$.⁶ The product will also be analyzed by ¹H-NMR spectroscopy as a solution in CDCl₃. The ¹H-NMR chemical shifts are expected to be a multiplet at δ 7 – 8 for the four aromatic protons, a singlet around δ 2 – 3 for the three protons on the benzylic methyl group, a quartet at δ 2 – 3 for the four CH₂ protons in the ethyl groups, and a triplet at around δ 0.5 – 1 for the six CH₃ protons in the ethyl groups.⁷