1. Transfer and Extraction Techniques

1.1. Competent Chemist Rating: *"Ethyl Ester's Excellent Adventure"*

Techniques Checklist:

- Extraction and Washing ❑
- Careful transfer of solutions without loss of material ❑
- Solvent drying and concentration ❑
- Melting point determination ❑
- Nuclear Magnetic Resonance (NMR) spectrometer operation ❑

Pre-lab Discussion and Required Reading:

- Extraction : Zubrick Ch. 15, LLP Ch. 10
- Theory of extraction: Zubrick Ch. 37
- Melting point determination : Zubrick pp. 87-92
- NMR theory and operation : Zubrick Ch. 35, LLP Ch. 15.2

Digital Lab Techniques Manual:

- 5. Reaction Work-Up I: Extracting, Washing & Drying
- 6. Extraction Work-Up II: Using the Rotavap

Equipment:

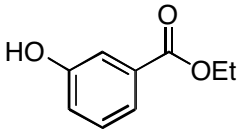
- Graduated Cylinder (100-mL)
- Separatory funnel (125-mL)
- Erlenmeyer flasks (2x250-mL)
- Beaker (150-mL)
- Round-bottomed flask (100-mL)
- NMR tube
- Funnel
- Filter Paper
- Rotary evaporator

Goal:

To manipulate and purify a known amount of a contaminated sample and to record its ^{1}H NMR spectrum, all with minimal loss of material.

Experimental Outline:

• You will receive a vial containing 100 mg of ethyl 3-hydroxybenzoate contaminated with triethylamine. You will also receive four different ^{1}H NMR spectra: one of the mixture in your vial, and one each of pure ethyl 3-hydroxybenzoate, triethylamine, and diethyl ether.



Ethyl 3-Hydroxybenzoate
MP 72–74 °C

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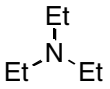

Triethylamine

• Dissolve your sample in 50–75 mL of ether in a separatory funnel.
• Remove the amine by extraction with a 10% HCl solution.
• Continue with a standard aqueous work-up, including an ether back-extraction - *see Extraction and Washing Guide*.
• Remove the solvent by rotary evaporation to a constant weight, and obtain a mass.
• Take a ^{1}H NMR spectrum of the compound and compare to the other spectra.
• Recombine the NMR sample with the remainder of the purified sample.
• Remove the solvent for the final time to a constant weight.
• Obtain a mass and a melting point.

Helpful Hints:

• When removing solvent with the rotary evaporator, make sure the receiving flask is cold and the water bath is warm. Otherwise, your product will never solidify.
• If you have trouble getting your product to solidify, try adding a few milliliters of methylene chloride to your flask and returning it to the rotary evaporator.

Results:

• To obtain your "CC Rating" in Transfer and Extraction Techniques, you must end with at least 90 mg of ethyl 3-hydroxybenzoate. Additionally, this material must be of adequate purity as determined by ^{1}H NMR analysis. This means that the spectra should show only negligible amounts of impurities as judged by the professor and TA. In addition, the purified material should melt over no more than three degrees, with the lower range beginning no lower than 69 °C and the upper range ending no higher than 73 °C. This material must also be submitted to the TA for possible weight and melting point confirmation measurements.