

EXPERIMENT 3 - Separation & Identification of Two Unknowns

You will be assigned a unique mixture of two unknowns – a basic compound (amine) and an acidic compound (carboxylic acid or phenol). This is a dry lab. Each student will independently devise a procedure for separating these compounds before lab and solve the structure using provided IR, ^1H NMR, and ^{13}C NMR spectra.

Separation of Acids and Bases by Extraction

Acid-base extraction is possible because one component will be more soluble in either organic or aqueous solution at certain pH ranges. Changing the pH creates ionic conjugate acids or bases, which are water-soluble. The non-ionized organic compounds left behind are more soluble in organic solvents than in water.

We use this principle to our advantage in the separation of acidic and basic compounds. Treating such a mixture with aqueous base converts acidic compounds to salts that travel to the aqueous layer as they are more soluble; separating layers thus separates the compounds. Acidification of the basic aqueous extract converts the salt into the protonated acid form which, because this form is insoluble in water, can be filtered if it is a solid or extracted with an organic solvent if it is a liquid. Similarly, organic bases (amines) can be extracted with aqueous acid, then isolated by basifying the acidic extract.

Separation and isolation of each component

Separation of the components follows the classical extraction procedure with dilute acid and base. After extracting three times with a dilute aqueous solution, back-extract the aqueous solution with the organic solvent—some non-ionized compounds have appreciable water solubility and back-extracting will assure a more efficient separation of the acidic or basic component from the others. The ionized compound in aqueous solution will need to be neutralized with a concentrated acid or base then extracted three times with the organic solvent.

When the un-ionized form is regenerated from the aqueous solution, it is best to extract the aqueous mixture with the organic solvent even if the component is a solid. Filtration is often an inefficient isolation procedure due to partial solubility and mechanical material loss.

Notebook Preparation: Procedure

Students will describe how to separate the unknowns with a step-wise written procedure, flow chart, and diagram in the notebook. Students must have this procedure approved by the TA before being given unknown spectra. Include all glassware, including size. An inventory of solvents, solutions, and volumes is as follows. It may not be necessary to use each solution below. Other equipment and materials such as drying agents, pH paper, should be included in your procedure as well.

Organic solvent: ethyl acetate, extract with 30 mL portions.

Basic solutions: 1 M NaOH, extract with 25 mL portions; 3 M NaOH, add drop-wise

Acidic solutions: 1 M HCl, extract with 25 mL portions; Concentrated HCl, add drop-wise

Neutral solution: saturated NaCl (brine), use 10 mL to wash combined organic extracts

Clean up & Safety – include within the procedure

- Transfer any unused or to be discarded basic aqueous layer to the container labeled “Aqueous waste - basic.”
- Transfer any unused or to be discarded acidic aqueous layer to a container labeled “Aqueous waste - acidic.”
- The contents of the rota-vap and any unused or to be discarded organic layer should be disposed of in the container labeled “Organic solvent waste.”
- Dispose of the unused unknowns in the bag labeled “Unknowns-waste.”
- Dispose of any Pasteur pipet, filter paper, drying agent in the container labeled “Solid waste.”

EXPERIMENT 3 REPORT GUIDELINES

- Detailed procedure for separation of an acid and base
- Structural determination of two unknowns via IR and NMR spectroscopy
 - o Unknown A Results & Discussion
 - o Unknown B Results & Discussion
- Read and apply writing style / guidelines on 146A website
- Include the unknown number as a Header – appears at the top of every page.
- The structures of both unknowns should be provided at least twice in the report (abstract & results sections). Look up the name/structure online. Use common name if applicable.

Exp 3 Report Formatting & Organization Guidelines

Abstract – one paragraph summary of the experiment in this order:

(1) purpose – “The purpose of this experiment was to…”

(2) methods – general methods for separation and analysis

(3) main results – distinctive IR stretch(es) and NMR peak(s) for the unknown acid (UNK A) and unknown base (UNK B) in separate sentences

(4) conclusion - state the identities of the two compounds (acceptable names).

Include the structures of both compounds below (ChemDraw) – no Figure heading.

Procedure - Your audience for this section is an intro-level (CHEM 8M) student.

Incorporate the written procedure in complete sentences (paragraphs, bullet points, or numbered) with the flow chart and diagrams. Use ChemDraw to make the flow chart and diagrams. Number these as figures and give short, descriptive titles. Refer to each figure parenthetically in the written procedure. You are encouraged to go the extra mile by including photos of experimental setups, additional safety notes, and pro-tips or potential sources of confusion.

Results - Your audience for this section is a fellow 146A student and TAs.

IR – State one or two stretch(es) indicative of the functional group(s) in the solved structure. Include a table with functional group, bond, expected, and observed wavenumbers for each IR active bond in the solved structure.

NMR – State only a few signals in the ^1H and ^{13}C NMR spectra – those that were pivotal in your structural assignments. The ^1H NMR table should include the assignment (letter corresponding to labeled structure), observed chemical shift, expected chemical shift (use predictor tool or calculate values of solved structure), integration, and splitting. The ^{13}C NMR table should include the assignment, observed chemical shift, and expected chemical shift.

Discussion – Re-state the methods used for structural determination of the unknown. Your analysis should be critical, showing how you deduced the structure of your unknown. Include the complete interpretation of IR and ^1H and ^{13}C NMR with the chemical shifts, integration, and splitting, comparison with expected values and assignments (which bonds gave rise each IR band in the table and to which Hs each NMR signal belongs?). Students should refer to tables from the results section and peaks within (ex. the 3H singlet at 2.0 ppm was assigned to signal A) rather than reproducing the entire table and structure. Conclude with a statement of the methods used and the unknown name.

References – follow guidelines on 146A website

- References should be cited as endnotes – superscripted number goes after punctuation at the end of the sentence. The endnotes are listed in the References section. If a reference is listed at the end of the report, it needs to actually be used and cited in the text.

Exp 3 Determination of Unknowns
Draft

Name _____

Editor _____

CHEM 146A GRADING RUBRIC

SECTION	EDITOR GENERAL COMMENTS And/or leave comments within the body of text Reference p. 33 frequently!	POINTS ASSIGNED for future reference, not necessarily for editor's use!
LAB REPORT		
ABSTRACT One paragraph, six sentences max: Purpose, methods, main results, and conclusions.		/ 20
PROCEDURE Written procedure, flow chart, diagrams; appropriate level of detail for audience (intro ochem student)		/ 50
RESULTS Each main result, including the final structure, is briefly stated using complete sentences; clearly labeled and organized IR & NMR tables Limited interpretation, just the facts!		/ 50
DISCUSSION Briefly re-state methods & main results in the context of logical, easy-to-follow structural elucidation. Pertinent IR signals and all NMR signals addressed		/ 40
REFERENCES Proper citations are used as end-notes and references are properly formatted		/ 10
NEATNESS AND ORGANIZATION Proper grammar, order, and format per instructor announcements and guidelines in lab manual.		/ 10
REASONABLE DRAFT Solid attempt at the report		/ 10
EDITING Thorough analysis of partner's report, referencing lab manual; assistance with format & sentence structure without re- writing their report!		/ 10
LAB REPORT TOTAL		/ 200

Exp 3 Determination of Unknowns Name _____
Revised Final Report

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