CHE 175 Unknown Checklist
Spring 2006

Student name: _____

Unknown #: _____

_____Name of unknown:

_____Structure of unknown:

_____CAS registry number: _____

_____Description of unknown:

solid: _____ liquid _____ color: _____

_____Solubility _____H_2O _____6M NaOH _____6M HCl

_____Measured physical property:

Initial MP _____ Final MP _____
Initial BP _____ Final BP _____

_____2,4-DNP test _____positive _____negative

_____Tollens test _____positive _____negative

_____Beilstein test _____positive _____negative

_____Iodoform test _____positive _____negative

_____Ferric chloride test _____positive _____negative

_____MS with analysis

_____IR with analysis

_____^{1}H NMR with analysis

Identification of an Unknown Organic Compound

Introduction

An important component of organic chemistry involves the identification and characterization of the structures of compounds. Although you can determine the structure of a compound by spectroscopic methods (MS, IR and NMR), physical properties and confirmatory tests for functional groups are recorded because they remain essential components of the identity of compounds.

In this experiment, you will receive a vial containing one gram of a liquid or solid organic compound. You will perform solubility tests on your unknown, test your compound for specific functional groups, and obtain and analyze spectroscopic data on it. These data will help you to identify the structure and IUPAC name of your unknown.

Compound Purification

You should assume that the compound is impure. Before you conduct any test on the unknown, you will need to purify it. If your unknown is a liquid, distill it and record its boiling point; if your unknown is a solid, recrystallize it and record its melting point. After you purify the unknown, be sure to **get the TA to sign your notebook next to your measured boiling point or melting poin*t***. If your notebook does not have the TA's signature indicating that you purified your sample, you will **lose 5 points** from your lab report. In your lab report, you must compare your boiling or melting point measurement to the actual measurement reported for your unknown.

I highly recommend that you **set aside 0.3 grams of the impure sample** so that you will have some of the unknown left to test if your purification goes wrong. You will not be given more of the compound if you spill it or run out of it.

After you have purified your unknown, you will conduct the tests (solution and spectroscopic) described next. It is not necessary to collect your data in the order below. Please use the checklist above to keep track of the tests as you conduct them.

Solubility Tests

Solubility tests are done in the sequence: water, then base, and then acid. If your compound is soluble in water, you do not need to check for solubility in the basic or acidic solutions.

Solubility in Water: Add 3 drops or 20 mg of the unknown to a small test tube. Add 1 mL of deionized water to the test tube. Thoroughly mix the solution then set it aside for 1 minute. If two layers form, or if the mixture is cloudy, the unknown is insoluble in water. If a transparent solution results, the unknown is soluble in water. Record your observations.

If the unknown is soluble in water, test a drop of the solution on a piece of litmus paper. Record the pH response as acidic, basic, or neutral. Low molecular-weight (<5 carbons) organic compounds are soluble in water:

- carboxylic acids have pH < 7
- amines have pH > 7
- aldehydes, ketones and alcohols have pH ≈ 7

Solubility in 6M NaOH: If the unknown is insoluble in deionized water, test its solubility in basic solution, by mixing 3 drops or 20 mg of the unknown in 1 mL of 6M NaOH. Record your observations. Solubility in 6M NaOH is a positive test for acids.
Solubility in 6M HCl: If the unknown is insoluble in deionized water and basic solution, test its solubility in acidic solution by mixing 3 drops or 20 mg of the unknown in 1 mL of 6M HCl. Record your observations. Solubility in 6M HCl is a positive test for bases.

2,4-Dinitrophenylhydrazine (2,4-DNP) Test for Aldehydes and Ketones

Place 2 – 3 drops of the liquid unknown or 20 mg of the solid unknown on a watch glass. If the unknown is a solid, dissove it in 5 drops of ethanol. Add 7 – 8 drops of the 2,4-DNP reagent to your unknown sample. Stir with a glass rod and let the mixture stand for up to 15 minutes. The formation of a yellow, orange, or red precipitate is a positive test for the presence of aldehydes or ketones. The 2,4-DNP products of aromatic or highly conjugated aldehydes and ketones tend to be orange to red; other aldehydes and ketones tend to give yellow 2,4-DNP products. Record your observations.

Tollens Test for Aldehydes

Tollens reagent: thoroughly rinse a clean test tube with 10% sodium hydroxide. Place 2 mL of a 5% silver nitrate solution in the test tube, and add a drop of 10% sodium hydroxide. Add 2% ammonia solution, drop by drop, with constant shaking, until the precipitate of silver oxide just dissolves.

Add one drop or a few crystals of unknown to the freshly prepared Tollens reagent. Gently heat the test tube in a steam bath if no reaction is observed immediately. The formation of a silver mirror or black precipitate is a positive test for the presence of an aldehyde. Record your observations.

Beilstein Test for Halides

Heat the loop end of a copper wire in a Bunsen burner flame, and allow it to cool. Dip the wire into a small sample of the unknown. Use the Bunsen burner to heat the wire again (in the outer part of the flame). First, the unknown will burn then a green flame will form if the unknown contains a halogen. Record your observations.

Iodoform Test for Methyl Ketones

Add four drops or 40 mg of unknown to a test tube. Add 5 mL of dioxane, and mix until the unknown dissolves. Add 1 mL of 10% NaOH solution, and then slowly add the iodine-potassium iodide solution with shaking until a **slight** excess causes the dark color of iodine to persist. Heat the mixture to 60˚C. Formation of a yellow solid (iodoform) is a positive test for the presence of a methyl ketone. Record your observations.

Ferric Chloride Test for Phenols

Under a hood, place 4 – 5 drops (40 mg) of the unknown in a small test tube. Dissolve the unknown in 1 mL of ethanol. Add 1 – 2 drops of 2.5% ferric chloride solution and mix. Watch the solution closely because the color change may disappear quickly. A color change from yellow (the color of the ferric chloride) to blue, green, violet, or red is a positive test for phenol. Record your observations.

Mass Spectrum

Determine the molecular weight of the compound. The molecular weight should help you to determine the molecular formula and its degree(s) of unsaturation. Assign fragmentation peaks, especially the base peak, in the mass spectrum of the unknown.

Infrared Spectrum

Please sign up to obtain an IR spectrum of your unknown. Please be ready to prepare your IR sample at least 5 minutes before your IR time.

If the unknown is a liquid, place 2 – 3 drops on an IR salt plate. If your liquid runs off the IR plate when it is turned on its edge, wipe the excess liquid along the edge with a Kimwipe. Record the IR spectrum, and assign peaks of the functional groups present in your compound.

If the unknown is a solid, dissolve 20 mg of it in methylene chloride and place a few drops of the solution on an IR salt plate. Use an air hose to evaporate the methylene chloride. You should have a thin coating of your solid on the IR plate. Record the IR spectrum, and assign peaks of the functional groups present in your compound.

^{1}H NMR Spectrum

Please sign up to obtain a ^{1}H NMR spectrum of your unknown. Please bring your prepared sample to the NMR lab at least 3 minutes before your NMR time.

If the unknown is a liquid, dip a Pasteur pipette into your unknown sample until the amount of sample in the pipette tip is ~1/3 inch. Carefully place this pipette into an NMR tube. Use a clean pipette to add ~0.4 mL $CDCl_3$ to the first pipette; this will transfer the unknown to the NMR tube. Remove the pipette from the NMR tube. Securely cap the tube and gently mix the solution.

If the unknown is a solid, use the NMR tube to scoop a few crystals (~20mg) of your unknown inside. Place a Pasteur pipette into your NMR tube. Use another pipette to add ~0.4 mL $CDCl_3$ to the first pipette; this will prevent contamination of the NMR solvent for those using the solvent after you. Remove the pipette from the NMR tube. Securely cap the tube and gently mix the solution.

Lab Report and Identification of the Unknown

Based on your analysis of all your data, identify your unknown by its structure, IUPAC name and CAS registry number. The CAS registry number is the personal identification number given to a compound that can be found on the internet, in a chemical catalog that sells the compound, or on the compound's MSDS.

Please refer to the Lab Rubric for details on what your lab report should include. Your introduction should provide a general description of the analytical techniques used to characterize your unknown. You should cite at least two references (source(s) for your introduction and sources(s) for the melting point and CAS registry number of your unknown). As usual, please attach a copy of the lab rubric to the top of your lab report.