25. Qualitative Analysis 2

First you will run a series of classification tests on a set of known compounds. The compounds are each representative of a particular functional group class. In this experiment you will observe how each class of compounds behaves in each test. You will compare these results with the results for the unknown in the second part of the experiment.

Your task will be to identify the functional group class the unknown belongs to. It is best to do the knowns and the unknown together to save time.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter in the manual, the lecture notes on the Chemistry Department web site, and pages 435-447 and 762-798 in Williamson. Do the first parts of your notebook writeup.

A student who has prepared for the Qualitative Organic Analysis experiments should be able to:

1. Given structures of compounds, identify the functional groups present, and give examples of compounds containing given functional groups.
2. Name when given the structure, and give the structure when supplied with the name, of common organic compounds used in CHEM 2230L and CHEM 2240L. (See the lecture notes on the Chemistry Department web site for a detailed list).
3. Given the structures of known compounds, predict the results of the following solubility tests: Water, and litmus test of any resulting solution; 5% aqueous NaOH, aq. $NaHCO_3$; 5% aq. HCl; and concentrated H_2SO_4. Also, given results from these tests, identify the functional groups present in the molecules.
4. Given the structures of known compounds, predict the results of the following tests: Br_2/CCl_4 test, $KMnO_4$ (cold) test, Tollens' test, Jones (chromic acid) test, ferric chloride test, 2,4-dinitrophenylhydrazine (2,4-DNP) test. Also, given results from one or more of these tests, identify the functional group(s) present in the molecules tested. Test results may be given simply as positive or negative, or the results may be described as they are seen in the lab (example: Which of the following compounds gives a purple solution in the presence of $FeCl_3$?)
5. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment, and give the role of each (reactant, solvent, catalyst, etc.).
6. Identify and explain safety considerations for this experiment. (Why is

the Tollens' reagent destroyed after the day's experiments?)
7. Perform the day's experiments safely and successfully.

Quizzes given after this experiment has been performed may also include:

8. Given the starting materials, predict the products (both organic and inorganic) of the reactions that give rise to the following tests: NaOH solubility, $NaHCO_3$ solubility, HCl solubility, Br_2/CCl_4 test, $KMnO_4$ (cold) test, Tollens' test, Jones (chromic acid) test, 2,4-dinitrophenylhydrazine (2,4-DNP) test.

Compounds to be Tested

In the procedures below, the phrase "test compound" is used. In the second experiment, the test compound is the unknown. In the first experiment, the test compounds are benzyl alcohol, 2-propanol, ***tert***-butyl alcohol, benzaldehyde, acetophenone, phenol, propanoic acid, and benzoic acid.

Do every test on every compound with the following exceptions: Do only the solubility and sodium bicarbonate tests on benzoic acid. Only do the Tollens' test on compounds that give a positive 2,4-DNP test. In the second experiment, you may identify the functional group of the unknown before you do all of the tests. If you do, you should still complete all of the tests.

TEST PROCEDURES

Water Solubility and Litmus Test

Add 2 drops of the test compound to 1 mL of deionized water in a test tube or a reaction tube. (For benzoic acid use the amount of acid that fits on the tip of your microspatula.) Shake the test tube and observe. If two layers remain, the compound is probably insoluble. However, if you believe that some of it dissolved, classify it as slightly soluble. If there is only one homogeneous layer, classify the compound as soluble. Use caution in interpreting the solubility results because compounds with lower molecular weight in many classes are water soluble even when higher molecular weight compounds are not. Test any compounds that dissolve in water with blue litmus paper and observe any color changes that occur.

The leftover solutions may be diluted and flushed down the drain.

Jones Test

Do not use ethanol to clean test tubes prior to running this test. Ethanol gives a positive Jones test. Add one or two drops of the test compound to 0.5 mL of acetone in a reaction tube. Add one drop of Jones' reagent to this solution. A positive test is the formation of a green color within five seconds of addition of the reagent. Be careful handling Jones' reagent, which is a very strong acid and a powerful oxidizing agent. It also contains chromium, a toxic heavy metal.

Leftover solutions should be placed in the chromium waste container in the hood.

2,4-Dinitrophenylhydrazine (2,4-DNP) Test

Do not use acetone to clean test tubes prior to running this test. Acetone gives a positive 2,4-DNP test. Dissolve one or two drops of the test compound in ten drops of ethanol. Add 15 drops of the 2,4-DNP reagent to the solution and allow it to stand for fifteen minutes. A yellow to red precipitate is a positive test.

Leftover solutions should be placed in the non-halogenated organic solid waste containers in the hood.

Tollens' Test

Do this test only on compounds that give positive results on the 2,4-DNP test. Use a clean shell vial for each test. Add ten drops of Tollens' reagent to the vial. Add one drop of the test compound to the vial and allow it to stand. Do not heat, stir, or agitate the vial. A positive test is the formation of a silver mirror on the inside wall of the vial within a few minutes. (If the vial is not clean on the inside, a black precipitate may form instead.)

Excess Tollens' reagent must be destroyed immediately or it may form explosive compounds. As soon as you finish with the tests, pour the contents of all the shell vials into the beaker of nitric acid in the hood. Rinse the vials with water and add the rinse water to the same beaker. Dispose of the empty used vials in the broken glass container.

Ferric Chloride Test

Add twenty drops of a 0.5% aqueous solution of iron(III) chloride to a test tube. Add one drop of the test compound. Immediate formation of a purplish blue color is a positive test.

The leftover solution may be diluted and poured down the sink.

Sodium Bicarbonate Test

Add one or two drops of the test compound to one milliliter of 10% aqueous sodium bicarbonate solution in a reaction tube. (For benzoic acid use the amount of acid that fits on the tip of your microspatula.) A positive test is the generation of bubbles of carbon dioxide gas as the compound dissolves. Watch closely for bubbling. Remember that there are only one or two drops being tested so there won't be many bubbles.

The leftover solution may be diluted and poured down the sink.

CLEANUP

Details are given above for each procedure. As usual, all used Pasteur pipettes are placed in the broken glass box.

POST-EXPERIMENT ASSIGNMENT

If this is the last experiment of the semester, your instructor may have you complete and turn in a combined notebook writeup and lab report before leaving lab. If not, write the lab report and have it ready to turn in by the beginning of the next lab or by the date that your instructor designates. If it is not the last experiment of the semester, prepare for the qualitative analysis portion of the next quiz. Otherwise, prepare for the final exam.