26. Qualitative Analysis 2

This experiment uses a series of wet chemistry analytical tests to determine the functional group(s) present in a series of known and an unknown compounds. The known 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, and from these comparisons deduce the functional groups contained within the unknown material. The exact chemical structure of the unknown will not be able to be determined by this set of tests, only the functional group present will be identified. Some of the unknown compounds may be identical to the known compounds (~25%), most are not.

The unknown sample will be tested alongside the known samples to save time and to facilitate accurate comparisons.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter in the manual, the experimental notes on the Organic Chemistry web site, and relevant pages in Klein. Complete the Qualitative Analysis Prediction datasheet. This datasheet will be used to assign your grade for the notebook portion of this experiment.

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 Organic Chemistry web site for a detailed list).
3. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment.
4. If given a structure, predict the results from any of the tests being carried out today.
5. Give the expected observations for a positive and negative result of any of the tests being carried out today. The tests include solubility, pH, ferric chloride, sodium bicarbonate, Jones, Tollens’, 2,4-dinitrophenylhydrazine, and iodoform.
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. If given a series of test results, identify the functional group present in the test compound.
9. Given the starting materials, predict the products (both organic and inorganic) of the reactions that give rise to the following tests: NaHCO₃ solubility, Tollens’ test, Jones (chromic acid) test, and 2,4-dinitrophenylhydrazine (2,4-DNP) test.

10. Understand the chemistry behind each test.

**Safety Considerations**

The known compounds include phenol and propanoic acid. Phenol is toxic and may be absorbed through the skin. Propanoic acid is corrosive. Some of the unknown chemicals are also toxic and corrosive. Be very careful when handling chemicals. Wear gloves. Wipe up all spills immediately. Keep chemicals away from skin. If accidental contact is made, wash immediately with copious amounts of soap and water. Wash hands thoroughly at end of lab.

**Compounds to be Tested**

The known test compounds are benzyl alcohol (1° alcohol), 2-propanol (2° alcohol), tert-butyl alcohol (3° alcohol), phenol (phenol), acetophenone (methylketone), cyclohexanone (ketone, not methyl ketone), benzaldehyde (aldehyde) and propanoic acid (carboxylic acid). Each student will also receive one unknown compound. Each unknown possesses at least one of the functional groups. Most of the unknown compounds are different than the known compounds.

Each student will perform every test on every compound with the following exceptions: Only do the Tollens’ test on compounds that give a positive 2,4-DNP test, only do iodoform test on compounds that give a negative Tollens’s test (and a positive 2,4-DNP).

This is a time intensive lab and it is very important to work quickly and efficiently during the allotted 2 hours and 50 minutes. Your instructor may allow students to share the testing of the known compounds. Each student is responsible for the testing and identification of their own unknown.

An effective testing method is to first set up a series of 10 labeled test tubes. This includes the 8 known compounds and, if working with partners, the 2 unknown compounds. Subject all 10 of the test tubes to one analysis. Record your full observations (except for your partner’s unknown result). Do not simply state “+” or “-” or “no reaction”. Properly dispose of the contents, wash all test tubes with soap and water and repeat for the next analysis.

The analyses can be done in any order with the exception of Tollens’s and iodoform. These two must be done sequentially after 2,4-DNP. It may be prudent to set up the 2,4-DNP test, allow to react for the 15 minutes, and during this time, gather another set of test tubes and do another test. Do not waste time, waiting for students around a test to clear. Instead jump to another test. **If you waste time during the class period, you will not finish. Time management during this experiment is very important.**

The reagents and waste containers for each of the different tests will be set up in a different location. Be sure to use the correct reagents and dispose of the waste in the appropriate container.
EXPERIMENT

Write your unknown number down in your notebook. Wear gloves for entire experiment. If material gets on gloves, throw away and get clean pair. If chemicals (especially Jones and 2,4-DNP) touch skin, immediately wash with copious amounts of soap and water.

Water Solubility

Place 1 mL of deionized water into a clean test tube. Add 2 drops of test compound. Shake vigorously and observe. If one homogenous layer results the compound is soluble. If two distinct layers result, or “oil drops” remain, the material is insoluble. Something between the two should be classified as slightly soluble. Most compounds with ≤ 4 carbons are soluble. Most compounds with ≥7 carbons are insoluble. Compounds with 5 and 6 carbons may or may not be soluble. Test any compounds that dissolve in water with blue litmus paper and observe any color changes that occur.

Waste from solubility tests can be diluted and washed down the drain.

Litmus Test

Test the pH of the solution from all compounds which were soluble in water using blue litmus paper. Acidic compounds are carboxylic acids and phenols. Basic compounds are amines. The rest of the commonly encountered functional groups are neutral.

Sodium Bicarbonate Test

Place approximately 1 mL of sodium bicarbonate solution in a test tube. Add 2 drops of test compound. Observe closely. A positive test will result in the formation of small carbon dioxide gas bubbles. The presence of oil droplets is not a positive result, this simply indicates low solubility. Watch closely for bubbling. Remember that there are only one or two drops being tested so there won’t be many bubbles and they will depart the solution rapidly.

Sodium Bicarbonate solution is baking soda water. It is innocuous. Waste from this test can be diluted and washed down the drain.

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. A positive result is a rapid formation of a red-blue-purplish color.

Ferric chloride is innocuous. Waste from ferric chloride tests may be diluted and washed down the sink.

Jones Test

_Do not use ethanol to clean test tubes before running this test._ Ethanol gives a positive Jones test. Add one or two drops of the test compound to approximately 0.5 mL (10 drops) of acetone in a test tube. Add one drop of Jones' reagent. Shake. Note color changes.
Hexavalent chromium (Cr⁶⁺) as found in Cr₂O₃ is a known carcinogen and a strong oxidizing agent. The solvent is concentrated sulfuric acid which will cause severe burns. Be very careful handling Jones' reagent.

Leftover solutions need to be placed in the chromium waste container located in the hood.

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

*Do not use acetone to clean test tubes before 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 in test tube. Add 15 drops of the 2,4-DNP reagent, shake and allow it to stand for fifteen minutes. Note color and consistency changes. (Can do other tests while waiting for this test to complete.)

2,4-DNP is a known mutagen. Sulfuric acid is the solvent and will cause severe burns. Leftover solutions should be placed in the designated waste container 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 2-3 drops of the test compound to the vial and allow it to stand for 10 minutes. 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 (If the vial is not clean or if vial has been agitated 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 waste beaker containing nitric acid in the back of 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.

Caution: Silver nitrate will react with skin and turn it a dark brown black. This color will come off as the skin cells are replaced in 3-10 days. The spots are not harmful.

**Iodoform test**

Do this test only on compounds that give a positive result on the 2,4-DNP test **AND** negative results on the Tollens’ test.

In a test tube dissolve 2-3 drops of test compound in 2 mL of water. Add 1mL of 3M NaOH. Slowly add 1 mL of iodine solution. Shake. Note any solid that may form. This may be a precipitate or a suspension.

Waste from this test should be placed in segregated waste container within the hood.

Caution: I² is corrosive and will cause severe burns. NaOH is corrosive and will cause burns.

**CLEANUP**

Ensure your waste from each of the qualitative analysis tests has been disposed of properly. Thoroughly wash test tubes with soap and water.
Wipe your work area clean with a damp sponge. Wash hands thoroughly with soap and water.

**POST-EXPERIMENT ASSIGNMENT**

Complete datasheet and turn into instructor within specified time. Cap unknown. Ensure unknown number is legible, if not use indelible ink pen to rewrite number on vial. Return vial containing unknown to instructor. This same unknown will be used next week for Qualitative Analysis 3.

Prepare for the Qualitative Analysis 2 portion of the next quiz.

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