

IDENTIFICATION of a DRUG

REF: *Aldrich Catalog Handbook of Fine Chemicals*, Aldrich Chemical Co., Inc., Milwaukee, WI
CRC Handbook of Chemistry and Physics, CRC Press, Inc., Boca Raton, FL
Introduction to organic chemistry, W. Brown & T. Poon, Wiley, Hoboken, NJ
The Merck Index, Merck & Co., Inc., Rahway, NJ
The Organic Chem Lab Survival Manual, J. Zubrick, Wiley, Hoboken, NJ

EQUIPMENT: Mel-Temp apparatus incl. temp. display
Buchner funnel
vacuum filter flask + water trap / vacuum tubing
short-stem, glass funnel
equipment in student lab drawers

MATERIALS: impure sample (3-4 grams) of an unknown drug compound
m.p. tubes
filter paper, fluted or "foldable", for gravity filtration
filter paper for Buchner funnels
ethanol (95%)
1% FeCl₃ solution
litmus paper
ice

INTRODUCTION

A few simple tests can often uniquely identify an organic compound from a list of a few compounds. Determining the melting point of a purified compound is a common, simple test. A common simple chemical test for phenols (ref. Brown & Poon, ch. 9) is the ferric chloride test; Fe³⁺ interacts with a phenol group to produce a violet to red complex. Litmus paper can also be used to determine whether a compound is acidic (carboxylic or other acid) or basic (such as an amine) in water.

Your impure drug sample will be one of the following:

- Advil
- acetylsalicylic acid (aspirin)
- methylparaben
- phenacetin
- salicylic acid
- Tylenol

Your task will be to determine the identity of your unknown drug.

PROCEDURE

1. Make a neat, legible table that lists each possible drug; for each compound the table should include: (ref. Zubrick ch. 3)
 - a. systematic (IUPAC) name
 - b. structural formula – especially note if a compound contains a phenol group
 - c. melting point
 - d. pKa or pH of aqueous solutions (if available)
 - e. water solubility information (esp. hot water solubility)

Work with a partner for steps 2-7:

2. Determine the melting point of your impure sample (ref. Zubrick ch. 12).
3. Purify your sample by recrystallization & hot filtration (ref. Zubrick ch. 13).
 - a. Use about 1 gram of your sample and 3-5 mL of 95% ethanol.
 - b. heat the mixture to boiling on a hot plate
 - c. after the drug dissolves, gravity filter the hot solution using fluted filter paper
 - d. rinse about 15 mL of cold, de-ionized water through the filter
 - e. cool the filtrate for 10 minutes at room temperature, and then for 5 minutes in an ice bath
 - f. collect the purified drug by vacuum filtration and wash with a few milliliters of cold water
 - g. allow the crystals to dry in your lab drawer for 1 week
4. Determine whether or not your compound may be acidic:
 - a. place about 0.5 - 1 mL of de-ionized water in a small test tube
 - b. dissolve a small amount of your purified (but possibly still damp) drug in this test tube
 - c. test the solution with blue litmus paper; if blue litmus paper changes to RED, the solution is acidic, and your compound is an acid
5. Perform a simple chemical test to help to determine the identity of your drug:
 - a. place about 0.5 - 1 mL of de-ionized water in a small test tube
 - b. dissolve a small amount of your purified (but possibly still damp) drug in this test tube
 - c. add 1 drop of FeCl₃ solution to the test tube
 - d. a violet to red color confirms the existence of a phenol in your drug
6. NEXT WEEK, determine the melting point of your dry, purified sample.
7. You may repeat any or all of this experiment as necessary to improve your results.
8. **ON OR BEFORE THE DUE DATE FOR THIS REPORT**, turn in your table and a description of how you determined the identity of your drug. This should be neat, legible and contain all your data and conclusions. You may use tables, words, and/or charts to illustrate your thought process.