

Qualitative Analysis 1

Qualitative analysis is the identification of unknown compounds from experimental data, which is something you have done more than once already in this class. In this experiment you will use several of the techniques you have already learned to identify a liquid unknown compound. You will measure the boiling point and the refractive index (RI) and obtain an infrared (IR) spectrum of your unknown. You will identify the unknown substance from the data you obtain.

PRE-EXPERIMENT ASSIGNMENT

Review the material in for Refractive Index, Infrared Spectroscopy and fractional distillation within this manual. Reread the experiment notes on Refractive Index, Infrared Spectroscopy and Fractional Distillation found on the Organic chemistry Web page.

SAFETY

Most of the unknown compounds used in this experiment are extremely flammable. Do not have any open flames or arcing sparking devices in proximity to the chemicals. All of the compounds have some mild toxicity if ingested.

Hot sand baths appear cool. Caution should be used when working around hot surfaces.

A student who has prepared for the Qualitative Analysis 1 experiment should be able to:

Fulfill the objectives given before the experiments on fractional distillation, refractive index and infrared analysis. Complete the first seven portions of your notebook.

1. Know how to read a RI scale and correct for water offset.
2. Know the theory behind RI.
3. Understand the distillation process including reflux, theoretical plates, and boiling points of pure materials and mixtures.
4. Know how to properly assemble and carryout a distillation.
5. Know how to properly analyze a sample using IR.
6. Know how to analyze an IR spectrum and assign functional groups present and absent.
7. Be able to draw the chemical structure if given the name of the possible unknown compounds. Be able to provide a proper name if given the structure.

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

8. If given IR, RI and boiling point data, know how to choose the correct identity of an unknown.

PROCEDURE

You will be given an unknown compound. Write the number down in your notebook. Warning, the liquid contained within can dissolve the number on the outside of the shell vial. You will analyze this sample by RI, IR and a boiling point. The procedure you used previously for refractive index and infrared will be followed. Either you can rewrite the procedure in your notebook, or state the page in your notebook on which those procedures may be found. You have not previously obtained a boiling point, but the procedure you will follow is very similar to carrying out a fractional distillation, without the copper mesh. The sample contains a pure compound instead of a mixture of two.

For the distillation place the entire sample in the long necked round bottom flask. As the sample is being distilled, note the temperature of each drop. Continue distilling until a stable distillation temperature range can be determined. It is not mandatory to distill the entire sample. Note the stable temperature range over which the sample distilled. The experimentally obtained boiling point is usually a bit lower than the literature value. Typically the boiling point is not further than 10°C away from the literature value. This deviation can increase as the literature boiling point increases.

Analyze your sample on an Infrared Spectrometer. Obtain the spectrum, note the five important regions in your notebook. This will determine what functional groups are and are not present in your compound.

Take a Refractive Index of your compound. Correct for machine offset. Compare your corrected refractive index with the possible compounds. Typically a properly measured refractive index will not deviate from the literature value by any more than 0.0040.

Compare the B.P., IR, and RI of your compound to the list of possible unknown compounds provided. It may be beneficial to first eliminate those compounds which would absolutely not give the data obtained. Often the RI data must be used to decide between two possible compounds. Select the composition of your unknown. Write down the name and structure of the compound in your lab notebook.

CLEANUP

Discard used Pasteur pipettes in the broken glass disposal box. Rinse IR salt plates with acetone and return them to the envelopes within jar. Place jar in desiccator. Rinse refractometer plates with ethanol and wipe clean with kim-wipe. Place used kim-wipes in trash can. Place excess unknown in the appropriate non-halogenated liquid organic waste container in the hood. Boiling chips should be disposed of in trash can. Rinse the scintillation vial

used to collect distilled sample with acetone, place upside down in the box on the front bench to dry by the next class.

POST-EXPERIMENT ASSIGNMENT

Finish Datasheet. Tear out notebook pages. Trim edges, staple and turn into instructor. Prepare for the final exam.