

ESM 222 Fate and Transport of Pollutants in the Environment

Lab #2: Physicochemical Properties

Report due 04/21/08

Objective:

Understand the behavior of different types of organic compounds as governed by their:

- Volatility
- Solubility
- Density with respect to water

You will also be trained to use the GC/MS for analysis of your samples.

Methods:

There will be two groups working each time block, with two different fume hoods. We will use the following organic compounds:

- Trichloroethylene, TCE
- Toluene
- Acetone
- MTBE
- Dodecane
- A mixture of these five compounds at 20% by volume

Review ahead of time the Material Safety Data Sheets (MSDS) and obtain from the literature the values of the properties you are going to observe, so that you have an idea of what to expect.

These compounds have been chosen given the relatively wide range of properties. We have added a red dye (Sudan IV) so that you can more clearly see the NAPL layer; note that this changes the properties slightly compared to literature values.

Before starting, make a list of the number of vials you are going to need for analysis and label them clearly. I recommend S1-TCE for solubility of TCE by Group 1, V3-Mix for the volatilization of the mixture by Group 3 and so on. Keep a running log of your activities in a notebook so you can later use it to complete your report.

One team member should work on volatilization, another on dissolution and the third person on preparing the mixture and collecting the samples for the GC/MS work.

Rate of Volatilization

- In an empty and clean 100 mL beaker, add exactly 10 mL of organic liquid or the mixture, and note the time with a timer. Use a different beaker for each compound or mixture.
- After 30 minutes empty the remaining organic liquids into separate 10-mL graduated cylinders and measure the loss of liquid volumetrically.
- Then return the remaining organic liquids to their original bottle, except the mixture.
- Rinse the 100 mL beakers and 10-mL graduated cylinders with acetone twice, disposing the acetone/organic mixture in the labeled waste bottle. Leave the beakers and cylinders in the fume hood.

Dissolution

- Fill six clear glass 40-mL vials with deionized (DI) water up to the rim, then pipette out 500 μ L of water using a pipette with a disposable tip.
- Add 500 μ L of each organic liquid or mixture to a different 40-mL "EPA" vial using a pipette with a disposable tip, and close the vial with a cap and septa. Avoid spilling organic liquid; if you do spill some, you should start over otherwise your results will have an unquantifiable error.
- Turn the vial upside down and observe the location of the NAPL layer.
- Manually agitate the vial for about a minute and then leave upside down for 30 minutes. Monitor the dissolution and note the approximate time for disappearance of the NAPL layer. For TCE leave the bottle right side up (why?).
- After 30 minutes, open each 40-mL vial and using a pipette extract 1.5 mL of contaminated water from about the middle of the vial to avoid entraining any residual NAPL. Change the tip for every sample; dispose the tips in the "tip waste" container.
- Place the samples of contaminated water in 2-mL vials for the GC/MS; avoid touching the dispensed liquid or the vial inside

wall with the pipette tip since it could have residual NAPL. Cap the 2-mL vial.

- Empty the contents of the 40-mL vial into the Hazardous Waste container; rinse the vial twice with acetone and empty the residues into Haz. Waste container.
- One person from your team should go with the TA to set up the samples for the GC/MS; in the next few days schedule about an hour to review your data from the GC/MS. You will each have a chance to do this GC/MS analysis throughout the course.

Preparation of the Mixture

(20% by volume, not by mole fraction!)

- 1) To a 40-mL EPA vial with cap & septa, add 2.5 mL of each of the five compounds and close it. Mix it well for about 1 minute.
- 2) Take 50 μ L of mixture with a pipette and add to a 2-mL vial for the GC/MS which already contains 1.5 mL of octane (as a solvent)
- 3) Use 10 mL of the mixture for the volatilization experiment
- 4) Use 500 μ L of the mixture for the dissolution experiment
- 5) Remember to take a sample of the volatilized mixture for analysis after 30 minutes: Take 50 μ L of the volatilized mixture with a pipette and add to a 2-mL vial for the GC/MS which already contains 1.5 mL of octane (as a solvent)
- 6) Give both samples to the TA to run in the GC/MS.
- 7) Dispose of any residual mixture in the Hazardous Waste container marked "Organic Liquids"
- 8) Have someone come back another day to collect the analysis from the GC/MS

Report and Analysis

In a short report prepared by the group, present the following:

1. Percent mass loss
2. Rate of volatilization in g/s for each compound and for the mixture
3. Composition of the volatilized mixture
4. Location of NAPL with respect to water
5. Approximate rate of dissolution (g/s)
6. Concentration of organic in water, compared to maximum solubility

Plot rate of volatilization (dependent variable) against vapor pressure (independent variable) for the 5 compounds in one graph. Does it follow a trend? Where does the mixture fall within the graph? Comment on your results.

Did you achieve full dissolution of the organic liquids in water? If not, how does the concentration compare to your expectations, considering the mass of NAPL you added to the mass of organic dissolved in the aqueous sample? How close were you to the solubility limit (%)? How did the mixture behave compared to the individual compounds? Comment on your results.

We will compare the results from the various groups in a later lab session.