Looking for PCBs in Water
or
Can PCBs Wash out of Landfills and Contaminate Ground Water?

by Jonathan Gutow, Spring 1999. Revised 4/01, 11/02, 11/03

Introduction

One of the big points of contention with the PCB clean up of the Lower Fox River is that people do not want PCB contaminated sediment placed in their local landfill. The primary argument they give is that the PCBs will leach out and contaminate their ground water. Because water that leaches through modern landfills must be collected and processed to remove contaminants this is unlikely to be a problem. However, even if all the water that leaches through a landfill is not collected, PCBs are not likely to be carried into the ground water in measurable amounts. The reason is that PCBs are insoluble in water. They preferentially stick to the soil sediment. In this lab you will demonstrate this to yourself as well as get to use one of the common techniques for detecting specific organic compounds.

The method you will be using is called gas chromatography, GC for short. In a GC a small amount of sample containing the chemicals you want to detect are mixed with a gas stream flowing through a pipe packed with a powder. The powder is often a ground up glass (sand or silica). Each of the particles of powder are coated with an organic substance (often a wax derived from petroleum products). Some compounds stick to this wax more strongly than others. The better the compound sticks to the powder the longer it takes to travel through the GC pipe. As the gas flows out of the pipe it is passed across a hot filament and then a sensitive electronic thermometer. The carrier gas (He in our case) has a lower thermal conductivity than the organic molecules we are detecting so the temperature measured by the thermometer increases when organic molecules are coming out. The relative thermometer temperature is recorded versus the time since the sample was injected. Each type of organic molecule in the sample comes out at a different time and shows up as a peak in the trace. The bigger the peak the more of that compound that was in the sample.

Since you cannot inject water or solids into a GC we will be extracting the organic molecules from the water and the sediment by mixing the water and the sediment with hexanes. The hexanes we will use are a mixture of all the isomers of C_6H_{14}. At room temperature hexanes are a transparent colorless liquid. Hexanes are nonpolar organic molecules. Nonpolar organic molecules like PCBs dissolve readily in hexanes. Thus when you mix hexanes with something that contains nonpolar organic molecules they move into the hexanes. The hexanes can then be separated and analyzed for what they contain. Water does not dissolve in hexanes because it is polar.

We will use biphenyl as a model for PCBs since it is considered less toxic. Remember PCBs are just biphenyl with some of the hydrogen atoms replaced with chlorine atoms. Instead of

real dirt we will be using a mixture of charcoal (pure carbon), sand and $CaCO_3$ to avoid seeing peaks from all the other organic molecules that end up in real soil from rotting plants and animals.

Procedures

I. Preparation of samples

1. Obtain 4 plastic 1 mL Eppendorf centrifuge flasks. Using a permanent marker or tape label them with 1-4 and your initials on the top.

2. Put "synthetic sediment" into flask #3 until the pointy bottom part is about 2/3 full. To this add a few crystals of biphenyl. Then fill the flask to near the top with deionized water. Seal the flask. Shake it vigorously for about one minute. Put your initials on the side of the tube using permanent marker and bring it to the instructor to be centrifuged so that the sediment will settle out.

3. Dispense 1 mL of pure hexanes from the hexanes bottle on the stock bench into flask #1. Seal the top and be careful not to contaminate this sample with anything. This is your reference for pure hexanes.

4. Put one small to medium size (~1 mm across) crystal of biphenyl into flask #2. Dispense 1 mL of hexanes from the stock bottle into the same flask. Seal the flask and shake it to dissolve the crystal. You will use this sample to determine what a signal from biphenyl looks like on the GC.

5. When flask #3 is done being centrifuged carefully open the top and use a clean plastic tuberculin syringe to transfer the clear water at the top into flask #4.

6. Dispense 1 mL of hexanes into both flasks #3 and #4. Seal them and shake well for about 1 minute. It is very important to free the sediment at the bottom of #3 in the shaking process otherwise it will not mix with the hexanes.

7. Return Flask #3 to the instructor to be centrifuged again.

II. Detecting biphenyl using a Gas Chromatograph (GC)

You will be using a small high accuracy syringe to measure out small amounts of sample for injection into the GC. Before each time you use this syringe you should clean it by carefully sucking up a full syringe worth of wash hexanes (in the beaker provided) and squeezing it into another waste beaker. Repeat the process for a total of three rinses.

1. The GCs will be warmed up before class begins. You should check that the temperatures of the various parts of the GC are proper before using them. The detector should be at 200-250 ˚C. The injection port should be at 200-250˚C. The column/oven temperature should be at 180-220 ˚C. If the temperatures are wrong please inform your instructor. It is also important that these temperatures stay relatively constant during your four runs. Also check that the attenuation is set to 2 and that the detector is turned on. THE TIME FOR SAMPLES TO COME OUT VARIES AMONG GCs. ALL YOUR TRACES SHOULD BE FROM THE SAME INSTRUMENT!!!!

2. **In the interest of saving time this step will be done as a demonstration by the instructor.** We will be using the column attached to port A. Carefully open flask #1 and pull 10 μL of the pure hexanes into a clean GC syringe. Push the **Start** button on the chart recorder/integrator. Insert the needle into port A. Wait for about 1 second. **Smoothly** push the syringe plunger down. Wait for about 1 second. Remove the syringe. The chart recorder should rapidly go off scale as the hexanes come through. After the hexanes pass through the column the trace on the chart recorder should return to near baseline. Allow the sample to run for 6 minutes. Push the **stop** button on the chart recorder. This is the kind of data a sample with no biphenyl in it will yield. **In the interest of saving time this will be done as a demonstration by the instructor.**

3. **In the interest of saving time this step will also be done as a demonstration by the instructor.** We will be using the column attached to port A. Carefully open flask #2 and pull 10 μL of the hexanes into a clean GC syringe. Push the **Start** button on the chart recorder/integrator. Insert the needle into port A. Wait for about 1 second. **Smoothly** push the syringe plunger down. Wait for about 1 second. Remove the syringe. The chart recorder should rapidly go off scale as the hexanes come through. Allow the sample to run for 6 minutes. Push the **stop** button on the chart recorder. This is the kind of data a sample with a small amount of biphenyl in it will yield. **In the interest of saving time this will be done as a demonstration by the instructor.**

4. We will be using the column attached to port A. Carefully open flask #3 (hexanes + biphenyl contaminated sediment) and pull 10 μL of the hexanes into a clean GC syringe. Push the **Start** button on the chart recorder/integrator. Insert the needle into port A. Wait for about 1 second. **Smoothly** push the syringe plunger down. Wait for about 1 second. Remove the syringe. The chart recorder should rapidly go off scale as the hexanes come through. Allow the sample to run for 6 minutes. Push the **stop** button on the chart recorder.

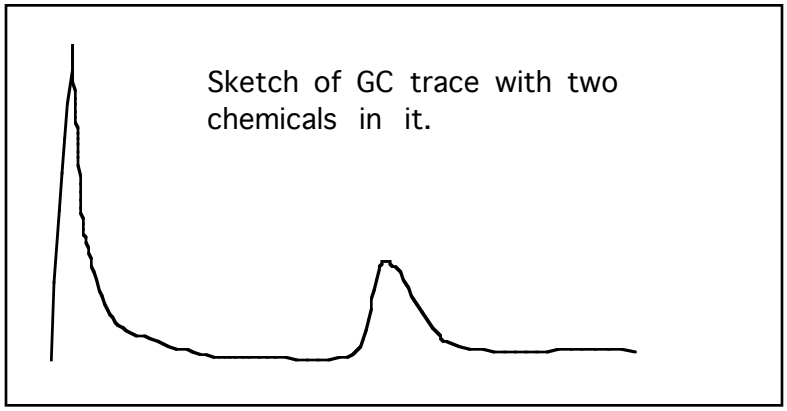
5. We will be using the column attached to port A. Carefully open flask #4 (hexanes + water that may have biphenyl in it) and pull 10 μL of the hexanes floating on top of the water into a clean GC syringe. Push the **Start** button on the chart recorder/integrator. Insert the needle into port A. Wait

for about 1 second. **Smoothly** push the syringe plunger down. Wait for about 1 second. Remove the syringe. The chart recorder should rapidly go off scale as the hexanes come through. Allow the sample to run for 6 minutes. Push the **stop** button on the chart recorder.

6. Dispose of all the sealed sample flasks in the beaker provided. Do not dump out their contents.

III. Interpretation of your results or how to read a GC trace.

You should have 4 GC traces: pure hexanes, hexanes spiked with biphenyl, hexanes mixed with water that might have biphenyl in it and hexanes that were mixed with sediment containing biphenyl. Use the first two traces to figure out what time hexanes come out and what time biphenyl comes out. By examining the last two traces (#3 and #4) determine whether biphenyl (an analog of PCBs) can be transferred to water from sediment. Below is a cartoon of a typical GC trace from a two component mixture.



Name_____ **Date**_____

Partner_____ **Section**_____

Staple properly identified copies of your GC traces to this data sheet. Indentify which trace is for which sample and which peak in each trace is from what compound.

IV. Questions

1. Do you see any evidence from your experiment that biphenyl (our stand-in for PCBs) can dissolve in water? Explain your answer.

2. Do you believe there is a significant risk of PCBs in landfills contaminating ground water? Why?