# Toxic Spill Experiment

Let's suppose increased fish death has been noted in the Saint John River, and scientists have to consider the possible causes. There are many possibilities, for example an extremely hot summer could lead to low oxygen levels, or warmer than usual weather could increase bacteria growth. There is of course the possibility that chemicals have been spilled into the water or one of the river's tributaries. Today we will carry out an experiment that deals with heavy metal contamination. Water samples can be taken from the river at various points and tested for different metals. There are several ways to go about this. Initially, one would take water samples in the vicinity of where the dead fish were found and use the most sophisticated instrumentation to find out what the toxin is. Once the metal has been identified one has two options to find out its origin:

1) Take lots of samples from upriver, downriver, and the tributaries, and send them to the lab for analysis. Once the results are known, one can follow a lead until the source is found. However, what if it was only a spill and not a continuous leak? While waiting for results, the spill disappears and one will never know where it came from.
2) Devise simple tests that can be done in the field. Following an increase in concentration of the metal will lead you to its source. This is what we will be doing in today's lab.

The first step is to determine the metal(s) that caused the fish death. Adding a carbonate solution to a river sample might result in a very coloured precipitate telling you which metal you have. Unfortunately, a brightly coloured precipitate will also mask the presence of metals that give white precipitates. We will employ a second set of tests to see what else, if anything, is in the river samples.

Once we have established what causes the fish death we will set up a series of solutions with varying metal concentrations to see if our river samples will show increased metal concentration or not. Using the GPS locations of the samples on a Fredericton map we will find out where the spill occurred. Then it is time for the Department of the Environment to pay a visit to the offending location.

| **Metal Cation (0.01 M)** | **Precipitate Color with carbonate** |
|---|---|
| $Cr^{3+}$ | |
| $Mn^{2+}$ | |
| $Fe^{2+}$ | |
| $Fe^{3+}$ | |
| $Co^{2+}$ | |
| $Ni^{2+}$ | |
| $Al^{3+}$ | |
| $Ag^{+}$ | |
| $Ca^{2+}$ | |
| $Mg^{2+}$ | |
| $Cu^{2+}$ | |

| **Metal Cation** | **Precipitate Color with chloride** |
|---|---|
| $Cr^{3+}$ | |
| $Mn^{2+}$ | |
| $Fe^{2+}$ | |
| $Fe^{3+}$ | |
| $Co^{2+}$ | |
| $Ni^{2+}$ | |
| $Al^{3+}$ | |
| $Ag^{+}$ | |
| $Ca^{2+}$ | |
| $Mg^{2+}$ | |
| $Cu^{2+}$ | |

Procedure

1. In a 50 mL beaker, obtain approximately 10 mL of the 1 M NaCl solution using a graduated cylinder.
2. Add approximately 20 drops of the NaCl solution to a test tube. Add approximately 20 drops of the 0.01 M metal cation solution to the same test tube. Record your observations. Clean the test tube with RO water and do the same for all of the metal ion solutions.
3. In a second 50 mL beaker, obtain approximately 10 mL of the 1 M $Na_2CO_3$ solution using a graduated cylinder.
4. Add approximately 20 drops the $Na_2CO_3$ to a test tube. Add approximately 20 drops of the 0.01 M metal cation solution to the same test tube. Record your observations. Clean the test tube with RO water and do the same for all of the metal ion solutions.
5. Obtain a sample solution from an instructor containing the unknown metal(s).
6. Add 1 mL of the NaCl solution to the sample solution. Is there a precipitate?

7. If you have a precipitate, filter the solid using fluted filter paper and a glass funnel. Collect the filtrate in a test tube. Otherwise pour approximately 2 mL of the solution into a test tube and proceed to step 9.
8. Pour approximately 2 mL of the filtrate in another empty test tube.
9. Add approximately 20 drops of $Na_2CO_3$ to the 2 mL of filtrate. Is there any precipitate? If so, what colour is it? Which metal ion(s) are in the water sample?

<u>Finding the source of metal contamination</u>

1. Using a graduated cylinder, add 10 mL of the 0.001M metal stock solution and add it to a test tube.
2. Using a graduated cylinder, add 2 mL of phenanthroline solution to the same test tube. Mix well.
3. Using a graduated cylinder, add 5 mL of the above solution to another test tube, then add 5 mL of RO water to this test tube. Mix well.
4. Do the above steps until there is hardly any difference in the colour.
5. Line these test tubes up as your calibration curve.
6. Take the river sample labelled #1 and do the same as in steps 1-2. Estimate the concentration.
7. Ask the instructor where the sample came from and request another sample. Continue doing this until you have found the source of contamination.

If the lab is to be performed at UNB we can use spectrophotometers set to 510 nm to record the absorption of the phenanthroline metal complex. Much more precise tracking of concentrations can be made compared to the visual samples.

1. Using a graduated cylinder, add 10 mL of the 0.001M metal stock solution and add it to a test tube.
2. Using a graduated cylinder, add 2 mL of phenanthroline solution to the same test tube. Mix well.
3. Insert the test tube into the spectrophotometer and take a reading.
4. Using a graduated cylinder, measure 5 mL of the above solution and discard the remaining solution into a waste beaker. Rinse the test tube with RO water.
5. Fill the graduated cylinder to the 10 mL mark with RO water. Add its contents to the cleaned test tube. Mix well.
6. Take a reading of the solution in the test tube.
7. Rinse the graduated cylinder with RO water.
8. Go back to step 4 and repeat until the %T is above 92.
9. Take the river sample labelled #1 and do the same as in steps 1-3. Take a reading and estimate the concentration.
10. Ask the instructor where the sample came from and request another sample. Continue doing this until you have found the source of contamination.

## Calibration Curve

| **Metal Concentration** | **%T (Transmittance)** | **A (Absorbance)** |
|---|---|---|
| | | |
| **0.001 M** | | |
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Geographical coordinates (use maps.google.com)

| sample | Coord 1 | Coord 2 | % T | Abs. | Location | Conc. |
|---|---|---|---|---|---|---|
| | | | | | | |
| 1 | 45.953 | -66.625 | | | | |
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