

What you need to do:**Procedure:**

At least 12 clean test tubes will be needed for today's experiment. Students may work in pairs, but each must do all steps of the procedure.

Solubility Testing

This portion of the experiment tests the solubility of the halides. You are provided with NaCl, NaBr and NaI salts. Choose one salt to complete the solubility tests and share data with two other students who complete the tests on the other two salts.

- 1) Add a small amount (the size of a small pea) of the solid salt to a test tube. Note the appearance and other properties in your notebook.
- 2) Now add 2 mL of distilled water and tap the bottom of the test tube gently to dissolve. Record your observations.
- 3) Repeat steps 1 and 2 using hexane as the solvent instead of water.
- 4) Share your results with the other two students completing the data for all of the salts.

NOTE: Any solution that contains hexane should be disposed of in the waste jar under the hood.

The solubility of the halogens will be tested next.

NOTE: If you are not good at describing subtle differences in colors, you may use the crayons provided to make accurate color notes on the two layers of the test tubes for the remaining tests. Simply draw a diagram of the test tube in your notebook and match the color of the experimental layers using the crayons. Remember, sometimes a picture is worth a thousand words.

1) **Testing the solubility of I₂:** Add 1 mL of the aqueous (water) solution of iodine (I₂) to a test tube. Iodine is a solid at room temperature. Can you hear the solid crystals rattling in the bottom of the dropper bottle?

Do not measure the amount in a graduated cylinder. An approximate amount is good enough, and 20 drops is roughly 1mL. Be careful not to spill any of the halogen solutions on your skin or clothes, as it will stain. Record the appearance of the aqueous solution.

2) Now add 1 mL hexane to the test tube used in step 1) and gently tap the bottom of the test tube to mix. Be sure it mixes; it may take a more vigorous shake, but start gently. Don't spill, but do not put your finger over the top and shake. Record all observations of both layers. When your observations are complete, put the solutions in the labeled waste jar in the hood. Rinse the test tube and put the rinsing in the jar too.

Bromine is a liquid at room temperature, and is quite volatile (that is, it evaporates easily). The aqueous solution of bromine (Br₂) is under the hood. Do not take any test tube containing bromine out of the hood. When finished with the bromine solutions, pour them in the waste jar in the hood, and then rinse the test tube with water from a wash bottle.

3) **Testing the Solubility of Br₂:** Using a fresh test tube, add 1 mL of the bromine solution to a test tube. Record the appearance of the aqueous solution. Just as before, add 1 mL of hexane and mix. Record all observations.

Cl₂ is normally a gas at room temperature. Since aqueous solutions of chlorine are not stable for very long (It's the equivalent of a soda going flat due to the loss of CO₂), each student will make a solution of chlorine (Cl₂).

4) **Testing the solubility of Cl₂:** Add 1 mL hexane to a new test tube.

5) Now add 0.5 mL Clorox and 0.5 mL 1 M HCl to the test tube. Do not breathe the bubbles that are given off—this is chlorine gas. Note the color of each layer. When finished, pour the mixture in the waste jar and rinse the test tube.

Electronegativity Testing

Now that baseline observations of what each of the ions and molecules looks like in water and in hexane, this set of reactions will look at all possible combinations of two reactants. This should allow relative electronegativities of the halogens to be confirmed. Remember that you are now doing a reaction, so you must make sure that your solution layers are thoroughly mixed to make sure the reactants come in contact with each other.

- 1) Add 0.5 mL Clorox to 0.5 mL 1M HCl to make $\text{Cl}_2(\text{aq})$, and then add 1 mL hexane and mix. To this, add 1 mL of the water solution of I^- . Mix well and record your observations. Dispose of the solution in the waste jar.
- 2) Repeat using the water solution of Br^- instead of the I^- .
- 3) Add 1 mL of the $\text{Br}_2(\text{aq})$ to 1 mL hexane, mix, and then add 1 mL of the water solution of I^- . Mix well and record all observations. Again, dispose of the solution in the waste jar.
- 4) Repeat using the water solution of Cl^- instead of the I^- .
- 5) Add 1 mL of the $\text{I}_2(\text{aq})$ to 1 mL hexane, mix, then add 1 mL of the water solution of Cl^- . Record all observations. Mix well and record all observations. Again, dispose of the solution in the waste jar.
- 6) Repeat for the water solution of Br^- instead of the Cl^- .

You have now added each of the other halides to each of the halogen solutions. By noting the products of the reaction (or if none occurred) you can organize an experimental scale of the electronegativities of the halogens.

Safety Points

All halogen solutions can be considered somewhat dangerous. Iodine can burn the skin and chlorine and bromine are very corrosive. The volatility of chlorine and bromine increases the danger of inhaling fumes. Avoid all contact with these compounds. If a spill of any of the halogen solutions occurs, call the TA immediately.

Any solution containing bromine must be used only under the hood and should be disposed of in the waste jar. The only solutions that may be disposed of in the sink are the water solutions of the halide salts. All other solutions must be discarded in the waste jar.

Hexane is not considered a toxic substance, but it is an organic solvent, which cannot be disposed of via the sewer. Therefore, all solutions that contain hexane must be disposed of in the waste jar.