Experiment No. 15 - Identification of Chemical Compounds in Solution

While waiting to start lab, you should pick up:

1-well plate (a glass plate with 9 depressions) {UW}
9-Pasteur pipets with bulbs {UW}
1-1L beaker (to use for waste collection) {on lower shelves by the door|
9- test tubes of unknown samples {on TA's desk}

These will be coded with 2 numbers. Each set will have the same Arabic number, but the 9 in the set will have roman numerals I – IX. You should pick a set of 9 with the same Arabic number, They will be in the same column of the test tube rack.

Examine each sample looking for any distinct differences. Some will appear rather cruddy. Some react with light to change color or form a precipitate; some with air (CO_2) to form a precipitate; some have limited solubility and start as essentially saturated solutions. If a little water evaporates from the solution or the room gets a little cooler, a precipitate may form. Such observations may help in your identification, or at least help serve as evidence to confirm your identification.

Uncork test tubes and place a Pasteur pipet in each. When not in use, always keep each pipet in its test tube of solution so that you do not cross contaminate any solution.

Next, you will test for strong bases and acids. Get three strips each of red and blue litmus paper. Tear each in thirds and lay out on a paper towel. Place a drop of the first sample on a piece of each color litmus paper and observe the results. Repeat with each of the other samples. The test for a strong acid will be quite definitive, but for the strong bases not so much. Solutions of sodium or potassium salts of some anions are quite basic; e.g., carbonate, sulfide, hypochlorite, *i.e.*, the anions of weak acids. You will need further evidence to identify those solutions giving a strong litmus test for bases.

You should have prepared a 9x9 matrix table to summarize the results of the next tests. You will not fill in the diagonal as it represents each sample reacting with itself. You will fill in only half of the remaining table (because the two halves are mirror images of each other, *i.e.*, A added to B is essentially the same reaction as adding B to A.) Place 3 drops of sample I in each of 8 wells (depressions) of the plate. Add 3 drops of II to the first well, 3 drops of III to the second well, 3 drops of IV to the third well, etc, until you have tested I with the other 8 solutions. You are looking for changes that indicate a reaction occurred. The 3 most useful observations are: no reaction (NR) that is, no visible indication that a reaction has occurred; the formation of a new colored solution (not just a lightening of color because of dilution); and the formation of a precipitate (a solid product). A precipitate may appear in different forms. As visible solid particles that may quickly fall out of solution or maybe as only a cloudiness in the mixture which is no longer clear. (Clear is not the same as colorless.) Other indications of a reaction are production of heat (or cooling) and production of a gas. But these two can be difficult to observe, especially when working with such small amounts of solution. Enter each of your observations in

the appropriate cells of the matrix. Clean the well plate. A strong stream of distilled water from your water bottle should be sufficient to wash the reaction results into the waste liter beaker. If a reaction produced a gummy precipitate, you may need to go to the sink and use a brush, maybe even soap. Rinse thoroughly with DI water. Do the second set of tests by putting 3 drops of II into 7 wells and then adding each of the other 7 in turn. Enter your observations, clean the plate and then the next set with III and the other 6. Continue until you have done all the possibilities and filled in all the cells in half of the matrix.

Show your matrix to your TA. The most common deficiency is not indicating the color of the precipitates. The TA will give you the list for your set of unknowns. It will be a list of the 20 possible unknowns with your 9 checked. You are to identify your 9 and label each with the Roman numeral assigned to it. You can use the labeled list as part of the data in your report. Your TA should tell what else may be required.

A good place to start your identification is with the strong acids. If you have only one, then no problem. You can do a confirming test, if you wish. If you have two, you will need to devise a way to identify which is which. Next, you might want to look at the bases. Another possibility is to look at any sample that had reactions with many of the other samples or one that had no or few reactions. Use the solubility rules on page 292 of your lab manual. The reactions discussed on page 296 offer tests that can be used in different combinations to test for many possible unknowns. For example, consider the reaction between KI and KIO_3 in acidic solution. On each side of the benches will be a tray of dropping bottles containing all of the possible unknowns, except water, plus some more concentrated acids and bases. If you think one of your samples is KI, you can test it with known KIO_3 from the tray. Or if you think you have KIO_3, you can test it with known KI from the tray. You can do similar tests with $KClO_3$ instead of KIO_3 or NaBr instead of KI. Or you can check to see if suspected $Na_2S_2O_3$ decolorizes I_2 produced by this reaction. On page 297 are some tests that can be used to differentiate between unknowns that react similarly. If you have questions or doubts, ask either of us.

When you finish, wash the well plate and return to the tray under the window. The Pasteur pipets go in the beaker(s) in the hood. Return the unknown samples to the rack on the TA's desk. Dump the contents of your waste beaker into the jug in the right-hand hood and place it under the hood at your desk.