An Enigmatic Chemical Conundrum!

Throughout the semester you will use your skills as a critical reader, a careful observer, and a logical thinker to solve problems in the laboratory. The first experiment of the semester will help you develop these abilities.

Pre-Lab Assignment: Developing Good Questions

An important part of doing science is learning to ask good questions. Carefully read through this laboratory handout and identify at least two questions for which the answers will help you complete this experiment; for example, you might ask: "What is the brown precipitate that forms when you add NH_3 and NH_4NO_3 to Fe^{3+}?" or "What is the most common roll in craps?". Write your questions in the section of your notebook for pre-laboratory work. We will answer these questions at the beginning of lab, so be prepared to share your questions.

Background

In the summer of 2012, faculty and students in the Classical Studies department excavated the site where Minshall Hall once stood (Minshall Hall was demolished in the early 1970s following completion of the Percy L. Julian Science and Mathematics Center; the site now is the location of Hoover Hall). Among the artifacts uncovered during this excavation was an old safe with a strange combination lock. Found with the safe were papers that belonged to one of DePauw's notoriously mischievous chemistry professors and a box that contained four well-sealed vials of solutions. Among these papers were the following four notes scrawled in nearly illegible handwriting:

- Combination for Safe: cobalt (II) chloride, iron (III) sulfate, nickel (II) sulfate, copper (II) chloride
- The sum of anions is that of the most common roll in craps.
- Each solution doth have but one anion.
- The code for iron (III) chloride, or $FeCl_3$, is XYYY.

The four well-sealed solutions were labeled as follows:

Babbling Beverage: 3, 5, 6 Confusing Concoction: 3, 4, 6

Deflating Draught: 1, 2, 5 Felix Felicis: 1, 2, 4

It seems likely that the safe's combination consists of four sets of numbers, with each set of numbers a string of 2–5 digits that codes for a simple inorganic compound, such as iron (III) chloride, $FeCl_3$, where the numbers represent ions. The single digits that accompany the four well-sealed solutions presumably represent the six ions that make up these solutions: copper(II), nickel(II), cobalt(II), iron(III), chloride, and sulfate.

Procedure

To discover the contents of the safe, you must break the strange code and match each ion to one of the numbers 1–6, and then determine the four sets of numbers that correspond to the safe's combination. Samples of the four oddly named solutions are available in lab, along with some additional reagents (see below). Work with your partner to identify the ions in each solution, assign each ion to a single digit, and use these digits to find the safe's combination. When you think you know the combination, check with your instructor. If your combination is correct, you will receive a portion of whatever is inside the safe; if your combination is not correct, however, then you must continue to work on this problem until you crack the code. Make sure that you record incorrect guesses in your lab notebook and explain why they were incorrect. Be careful: to receive the highest grade you must discover the correct combination on your first try.

Chemical Reactivity Resources

To help you determine which ions are in each solution, you will test the solutions using a variety of reagents. The following two tables summarize the chemistry of the six possible ions with these reagents. Use a clean test tube for each test and follow the directions carefully. Precipitates (solids) are identified as ppt; all other results give the color of the solution. The reagent dmg is dimethylglyoxime. Entries in a **bold** font are so distinctive in color that they are particularly useful for making a positive identification. Remember, these are tests for a solution with a single cation or anion; the "well-sealed" solutions are mixtures of ions.

Table 1: Reactivity of the two anions.

anion	to 20 drops of solution, add 2 drops of 6 M HNO_3 and 5 drops of 0.1 M $AgNO_3$	to 20 drops of solution, add 2 drops of 6 M HNO_3 and 5 drops of 0.1 M $Ba(NO_3)_2$
Cl^-	**white ppt**	nothing
SO_4^{2-}	nothing	**white ppt**

Table 2: Reactivity of the four cations.

cation	color of original solution	to 20 drops of solution, add 14 drops of 7 M NH_3 and 6 drops of 4 M NH_4NO_3	to the previous solution, add 5 drops of 0.1 M dmg (see note below)	to 20 drops of new solution, add 5 drops of KSCN
Fe^{3+}	yellow	**brown ppt**	nothing	**blood-red**
Co^{2+}	pink	pale red-brown	brown	pink
Ni^{2+}	faint green	pale purple	**red-pink ppt**	faint green
Cu^{2+}	light blue	**dark blue**	dark blue	pale green w/ white ppt

Note: If a precipitate forms after you add NH_3 and NH_4NO_3, then you first must remove the precipitate before you add dmg. You can do this by placing your test tube in a centrifuge, balancing the centrifuge with an identical test tube that contains an equal amount of water, and briefly spinning the test tubes. Use a pipet to remove the overlying solution from the precipitate with a pipet, transfer it to a clean test tube, and proceed with the addition of dmg.

Waste Disposal

Dispose of your solutions in the appropriately labeled waste container in the hood.

Lab Report

Complete the report form for this experiment and turn it in by the beginning of the next lab period.

By the Way

There really was an excavation of one corner on the site of Minshall Hall Alas, a mysterious safe was not among these artifacts.