Making Measurements and Preparing Solutions

One of the most important laboratory skills is the ability to prepare a solution accurately and precisely. In your previous laboratory experiences most solutions were prepared for you, or the solutions you made did not require an exact or a reproducible concentration. This semester, however, you will prepare many solutions and you will need to do so with appropriate accuracy and precision. This is not a difficult task; it just requires patience and attention to detail.

One of this experiment's goals is to prepare three solutions, cleverly labeled Solution A, Solution B, and Solution C. To do so you will need to complete some stoichiometric calculations, carefully measure out several reagents using a balance, a volumetric pipet, or a graduated cylinder, and bring these reagents to known final volumes in volumetric flasks. After preparing these solutions you will combine them with a fourth solution (which is provided to you) and observe the resulting reaction. If you prepare your solutions carefully, you will see an interesting result. Before preparing these solutions you first will evaluate the accuracy and the precision of various methods for measuring volume.

Skills Emphasized In This Lab

By completing this lab you will become more comfortable with the following skills:

- measuring volume with appropriate accuracy and precision
- summarizing data using basic statistics, such as means, standard deviations, and confidence intervals
- performing the stoichiometric calculations associated with preparing solutions
- preparing solutions
- maintaining an electronic record of your work

Preparing for Lab

Before your come to lab read the following essays, which are available on the course website: "Accuracy, Precision and Analytical Measurements," "The Art of Measuring Mass," and "The Art of Measuring Volume." In addition, read the instructions for preparing the three solutions and complete all necessary calculations. Complete the appropriate sections of your electronic laboratory notebook, including all necessary calculations.

Part A. Evaluating the Accuracy and Precision of Glassware for Measuring Volume

As noted in the essay on "Measuring Volume," there are many ways to measure the volume of a liquid reagent, each with its own inherent accuracy and precision. Generally, the more accurately or more precisely you need to know the volume the more time it takes to make the measurement. For this reason you should carefully think about your need for accuracy and precision when selecting glassware.

In the first part of today's lab you will investigate the accuracy and the precision of the equipment available to you by dispensing 5 mL of water, measuring its mass, and converting that mass to its corresponding volume using water's density, the value for which is a function of temperature. Working with your partners, determine the accuracy and the precision of the following approaches to dispensing 5 mL of water: a disposable dropper, a 10-mL graduated cylinder, and a 5-mL volumetric pipet. The general procedure is described here when using a 10-mL graduated cylinder:

1. Obtain approximately 500 mL of deionized water, record its temperature, and use the table at the end of this document to find its density at this temperature.
2. Dry a 100-mL beaker and record its exact weight in grams.
3. Dispense 5 mL of water into the beaker using a 10-mL graduated cylinder. Weigh the beaker and the water, and determine the mass of water dispensed.

4. Repeat Step 3 for a total of at least five trials, adding each new aliquot of water to your beaker.
5. Calculate the mean, the standard deviation, the relative standard deviation, and the percent error (assuming a true value of 5 mL). Be sure to think about the number of significant figures that you can reasonably report.

Repeat this procedure for the disposable dropper and the 5-mL volumetric pipet, beginning each with an empty beaker. When using the disposable dropper, assume the "rule of thumb" that 1 mL is equivalent to 20 drops. Create a spreadsheet to record your data and to handle the necessary calculations, including finding means, standard deviations, relative standard deviations, confidence intervals, and percent errors. Be sure to save this file and to identify in your lab notebook the spreadsheet's name. Include a brief analysis of your results in your notebook. As a part of this analysis, identify the equipment that was the most accurate, the least accurate, the most precise, and the least precise.

Part B. Preparing the Solutions

Solution A is 50 mL of 0.23 M $KBrO_3$, also known as potassium bromate. Weigh out the correct amount of $KBrO_3$, transfer it to a 50-mL volumetric flask, and dilute to volume with deionized water. Transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Solution B is 50 mL of a mixture containing 0.31 M $CH_2(COOH)_2$, also known as malonic acid, and 0.059 M KBr, which is potassium bromide. Using the general approach used to prepare Solution A, add both solids to the volumetric flask and dilute to volume with deionized water. Transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Solution C is 50 mL of a mixture consisting of 0.019 M cerium (IV) and 2.7 M H_2SO_4, or sulfuric acid. Begin by preparing 100 mL of 2.7 M H_2SO_4 using the available solution of 42.3% w/w H_2SO_4. This concentration unit, which may be less familiar to you, is a weight-to-weight percent (100.0 g of the solution contains 42.3 g of H_2SO_4). The density of 42.3% w/w H_2SO_4 is 1.25 g solution/mL solution. Using a graduated cylinder, measure out the correct volume of 42.3% w/w H_2SO_4 and slowly add it to a 100-mL volumetric flask that already contains approximately 25 mL of deionized water. Dilute to volume with deionized water and mix thoroughly.

Cerium is obtained using cerium (IV) ammonium nitrate, $Ce(NH_4)_2(NO_3)_6$, which is available as a solid. Using the general approach used to prepare Solution A, use your solution of 2.7 M H_2SO_4 to transfer the $Ce(NH_4)_2(NO_3)_6$ to the 50-mL volumetric flask and dilute to volume using your 2.7 M H_2SO_4. When complete, transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Did You Prepare the Solutions Carefully?

Now comes the moment of truth! Obtain a clean 250-mL beaker, a magnetic stirrer, and a stir bar. Place the stir bar in the beaker and place the beaker on the magnetic stirrer. Pour all of *Solution A* and all of *Solution B* into the beaker and adjust the stir rate to produce a small vortex (A little solution tornado!). If the solution develops an initial amber color, then wait until it turns colorless before proceeding; this is not a common occurrence. When the solution is colorless, add, at the same time, all of *Solution C* and 3 mL of the already prepared *Solution D* (measured using a graduated cylinder). Examine the resulting reaction for several minutes, observing the interesting and spectacular results. You will have no trouble deciding if your solutions were prepared carefully. Be sure to have your instructor verify your success and to record your observations in your notebook.

Cautions

There are no cautions for this lab other than the normal respect for chemicals.

Waste Disposal

The reaction mixture can be flushed down the drain with plenty of water.

Lab Report

Your electronic laboratory notebook and associated data files serve as your report for this lab. Be sure to place copies of both in your group's Google Drive folder. These documents are due at the end of lab.

Density of Water as a Function of Temperature

Temperature (^oC)	Density(g/mL)
15	0.9991026
16	0.9989460
17	0.9987779
18	0.9985986
19	0.9984082
20	0.9982071
21	0.9979955
22	0.9977735
23	0.9975415
24	0.9972995
25	0.9970479
26	0.9967867
27	0.9965162
28	0.9962365
29	0.9959478
30	0.9956502