Chemistry Techniques and Explorations

An Introductory Chemistry Laboratory Manual

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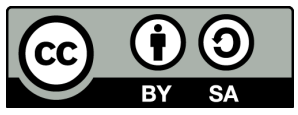

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About the Author

Dr. Daniel R. Albert is an Associate Professor of Chemistry at Millersville University of Pennsylvania. Dr. Albert is an experimental physical chemist who is engaged in research on understanding the impact of gas-liquid interactions on environmental processes. He is also engaged in chemical education research and develops inexpensive instrumentation for targeted applications and educational purposes.

Dr. Daniel R. Albert

Author Acknowledgements

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Goal of Laboratory Manual

The goal of this laboratory manual is to provide opportunities for introductory chemistry students to learn about and practice important techniques that will be utilized throughout their career. These opportunities will allow students to develop as practicing scientists. Another important aspect of becoming a practicing scientist is to develop new experiments in the laboratory. The exploration laboratory experiments provide freedom and time for students to use previously acquired skills toward answering a new question. Experiments identify whether the focus is technique or exploration. It is important to provide flexibility and time for student discussion during the exploration laboratories. All labs are designed for a two-hour laboratory session with the exploration laboratories extending over a two-week period.

Table of Contents

Safety and Record Keeping

Learning to work safely in a laboratory setting while also keeping accurate and appropriate records are key skills that we are developing during this course. Safely working in a laboratory means that we minimize risks for ourselves, other people in the laboratory and building, and for the environment.

Figure 1.1: "Dr. Adah Elizabeth Verder mouth pipetting" by National Institutes of Health (NIH) is marked with Public Domain Mark 1.0.

In first learning about safety in the laboratory we often encounter lists of guidelines for what we should and should not do in the laboratory. These guidelines are to help us build a baseline for understanding and mitigating risk in teaching laboratories with well-established experiments. One of the safety rules is to not “Pipet by Mouth.” What do you think the purpose of this safety rule is?

Safety Rules

Here are some examples of standard safety rules for teaching laboratories. To aid in understanding these rules, you will be discussing with others in the laboratory what you think their purposes are.

- **Never leave chemical containers open.**
- **Label all containers with what is inside of them.**
- **Long hair and loose clothing must be secured.**
- **Closed toed shoes must be worn in the laboratory.**
- **Do not wear laboratory gloves into the hallway.**
- **Laboratory goggles must always be worn.**
- **Know the location of fire extinguishers and emergency exit routes.**
- **Do not work alone in the laboratory.**

RAMP Approach to Safety

Enhancing your understanding of laboratory safety requires preparing for new situations and equipping yourself with ways to ensure safe practices. A commonly used acronym to aide in working safely in the laboratory is RAMP.

R – Recognize

A – Assess

M – Minimize

P – Prepare

Recognize

In order to operate safely, we need to identify potential risks. If we never recognize the risks associated with an experiment then there is no way we can be adequately prepared. Think about the Titanic. The people in charge of safety never fully recognized the risks of hitting an iceberg and therefore were wholly unprepared when that situation arose. So how do you recognize a risk when you haven't performed an experiment before?

In introductory laboratories we will help you recognize new risks that exist when performing new techniques or using new reagents. Chemical labeling under the Global Harmonized System (GHS) and National Fire Protection Agency (NFPA) diamonds are both used to help people working with chemical reagents recognize the associated risks. Look at the GHS symbols and try to guess what risks they are identifying. Check your accuracy by looking at the OSHA Hazard Communication Standard Pictograms.

Figure 1.2: "GHS HAZCOM Safety Labels" by Mpelletier1 is licensed under CC BY-SA 3.0.

Assess

Once you have recognized the risks associated with particular actions in the laboratory, the next step is to assess how working with different materials can be potentially hazardous. GHS symbols and NFPA diamonds are a first step in assessing risks for chemicals, but they usually do not provide enough information to fully assess the risk. Remember: NFPA diamonds and GHS symbols are present to help recognize a potentially risky situation.

Assessing a risk requires us to gather more information. Gathering and interpreting quality information are universally beneficial skills and are often called Information Literacy.

In the chemistry lab there are certain well-established tools to aid in assessing risk. Among the primary tools used are Safety Data Sheets (SDS) or Material Safety Data Sheets (MSDS) that outline the risks and cautions associated with a given materials. Companies that sell chemical reagents are required to provide this information for their products. Places that use hazardous materials are also required to make Safety Data Sheets available to those who encounter those materials.

A Chemical Hazard Risk Evaluation Matrix can be helpful in thinking through the various risks of carrying out a laboratory experiment. Take a look at this Chemical Hazard Risk Evaluation Matrix from SUNY-Stony Brook

Minimize

Once we have established the risks associated with performing an action in the laboratory, the next step is to reduce those risks as much as reasonably possible. We won't ever be able to fully eliminate risks, but we can take measures to decrease the likelihood of serious injuries and accidents. The common steps to minimize risks are as follows:

Replace Hazardous Substances for Less Hazardous Substances.

In the 20th century benzene was commonly used in organic chemistry synthesis but we eventually learned that benzene was quite toxic and carcinogenic. This knowledge led to benzene being replaced with other, less harmful substances.

Use Spaces and Devices Engineered To Minimize Hazards.

Performing chemical experiments in chemical laboratories equipped with good ventilation or fume hoods can decrease the risk of inhaling a particular chemical. Refrigerators used to store chemicals look externally similar to what you would find in your kitchen, but they are specifically designed to be explosion proof. The risk of your milk exploding is really low, but the risk of stored chemical in a laboratory exploding is much greater!

Use Appropriate Personal Protective Equipment (PPE).

Safety goggles help protect your eyes from being exposed to hazardous chemicals. Wearing closed-toed shoes protect your feet from drops and spills. Gloves can protect your hands from being exposed to hazardous substances. These are examples of PPE that minimize risk.

Prepare

While we can minimize risk, we can never eliminate all hazards. Preparing for accidents can aid us in responding appropriately when an accident occurs. By preparing, we not only increase our chances of solving a problem, but we also help ensure that we do not worsen a problem when responding to an accident.

We have emergency measures in place in the laboratory. One example is an eye-wash station that is used if someone gets a hazardous substance in their eye. You might think that this station is redundant because we should all be wearing safety eyewear. You are correct. It is redundant, and that is the point. While we expect that we will not need to use the eyewash because of our minimization of risk with PPE, we are still prepared in case an accident does occur. Other examples of these measures include first aid kits for minor cuts and scrapes, a safety shower for large spills on people, spill kits to clean up spills on surfaces, and a fire extinguisher for small fires.

Laboratory Notebook

The laboratory notebook is where all information about laboratory experiments should be stored. The idea is that the laboratory notebook is a permanent record of all preparation, all observations and data in the laboratory, and all analysis of data after an experiment. The main purpose is to record all that you have done so that you and others can look at the notebook and replicate experiments. Laboratory notebooks are routinely used in court cases to determine if samples were handled properly and who gets awarded patents for new technology.

Guidelines for Maintaining a Laboratory Notebook

- **Table of Contents is kept current with page numbers.**
- **Pages are numbered consecutively and no blank spaces are left in the notebook.**
- **All entries are made in permanent ink at the time of the observation.**
- **Errors are corrected by crossing out with a single line, initialing the correction, and dating when the correction was made.**
- **Procedures for the experiments are well documented.**
- **Notebook is signed and dated to document when data entry begins and ends.**
- **Data and observations are recorded in an organized fashion.**
- **Data are recorded with appropriate units and significant figures.**
- **Notebook is signed by a witness at the end of data collection.**
- **Sample calculations and details of data analysis are provided.**
- **Summary of findings is recorded after the experiment.**

References

American Chemical Society, “Guidelines for Chemical Laboratory Safety in Academic Institutions.” 2016, https://www.acs.org/content/dam/acsorg/about/governance/committees/chemicalsafety/publications/acs-safety-guidelines-academic.pdf Accessed: February 26, 2023.

Separating Substances, Measuring Mass, and Analyzing Data - Technique Laboratory

Introduction for Measurement of Masses

Balances are used to measure masses of objects. Chemists have used balances for centuries to gain key insights into chemical processes and reactions. The balance has long been one of the most important tools in the chemistry laboratory!

Use of Balance

The tare button is the one important button on the balance. By pressing the tare button you "zero" the balance. Zeroing the balance means that whatever mass is currently on top of the balance is set to zero mass.

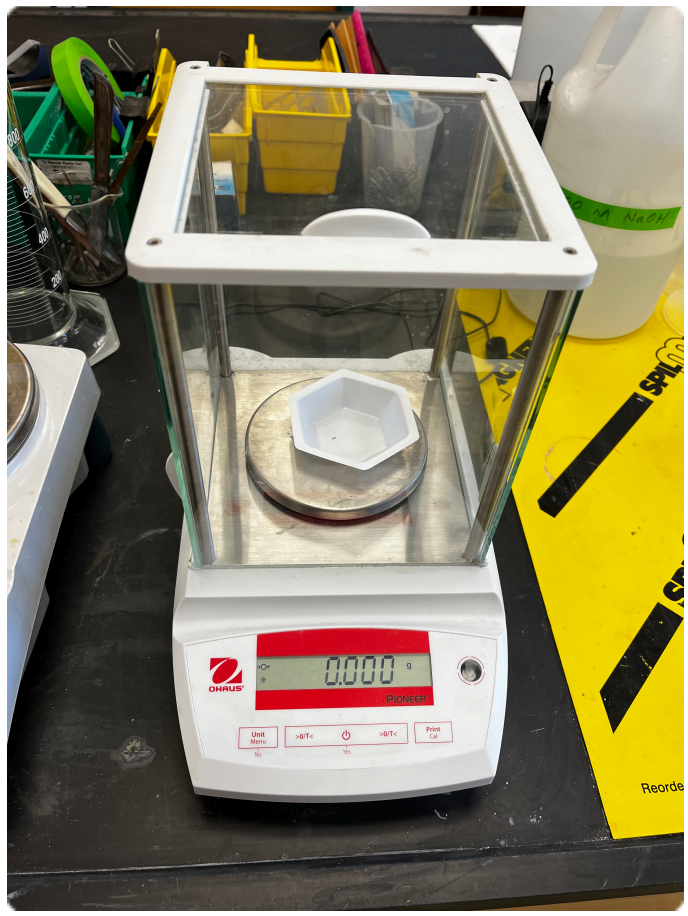

Figure 2.1: Electronic Laboratory Balance

Tips for measuring masses using a top-loading balance:

1. Never put the object being massed directly on the balance. Always use a piece of weighing paper or a container (beaker/flask) to hold the material being massed.
2. Close the balance door/lid to avoid drafts from air currents that will cause readings to fluctuate.
3. All objects being massed must be at room temperature. This avoids buoyancy correction problems due to warm air being less dense than cold air and prevents air currents that will cause readings to fluctuate.
4. Write down all the digits from the balance. If you are asked to mass 2 grams of material, it is perfectly fine if the balance reads 1.956 grams, but you must write down the actual value (1.956 grams) and not just write 2 grams in your laboratory notebook.
5. Clean up any spills immediately using a brush.

Measure Directly

Measuring a mass directly is just like stepping on a scale to determine your weight. This approach is convenient to use when some uncertainty is acceptable for the measurement and when it is easy to separate the material you are massing from its container. The basic procedure for massing directly is as follows:

1. Place something on the balance (weighing paper or a beaker).
2. Tare the balance so it reads zero when the weighing paper or beaker is on the balance or record the mass of the weighing paper/beaker.
3. Place the material to be massed on the weighing paper or beaker and record the mass.

Mass by Difference

Measuring a mass by difference is how I would determine the weight of babies in my house. I would step on the scale while holding the baby to determine my weight and that of the baby. Then I would step on the scale again without the baby. The difference between the two weights is the weight of the baby.

(Mass of Dr. A Holding a Baby) − (Mass of Dr. A) = (Mass of Baby)

Measuring by difference always infers a measurement based on two other measurements. It is used when you want an accurate mass and when it is difficult to separate an object at the end of an experiment.

As an example, say you wanted to measure how many grams of peanut butter are left in a jar. You could try to scrape all the peanut butter out and measure the mass directly, but it would be much easier to determine the mass by difference. If you know the mass of the empty peanut butter jar, then all you need to determine the weight of the peanut butter is the mass of the jar with the peanut butter in it. Then you can take the difference between the masses to determine the mass of peanut butter remaining.

When to Tare?

When taking initial masses of objects, it is often beneficial to think through to the end of the experiment to decide if you will be taking a mass by difference at the end or determining a mass directly. This dictates if you should tare the balance with your container on it or include the mass of the container. For example, if you

are asked to directly mass 5 grams of solid into a beaker you could either tare the balance without the beaker or tare the balance with the beaker present.

If you will take a mass of something in the beaker at a later date, then you should tare the balance before putting the beaker on it. This would lead to recordings in your notebook of something like the following:

Mass of Empty Beaker: 101.568 grams

Mass of Beaker with Solid: 106.237 grams

Mass of Solid: $106.237\ g - 101.568\ g = 4.669\ g$

Alternatively, if you will not take the mass of an object in the beaker later, then you could tare the balance after adding the beaker. This would lead to recordings in your notebook of something like the following:

Mass of Solid: 4.669 grams

It certainly feels easier to just have the one value written down but be careful that you won't need the mass of the beaker later. If you are unsure about whether you will need the mass of the beaker later, then it is best to record its mass and follow the first approach. Although you will need to write down two more numbers and do a little subtraction, it could save you in the end.

Separating a Heterogeneous Mixture and Determining Masses

You will be provided with a mixture that contains sand (SiO_2), table salt (NaCl), and benzoic acid (C_6H_5COOH). Your goal is to separate the mixture into its components and determine the percent composition of each component in the mixture.

Here is some basic information about the properties of sand, table salt, and benzoic acid. We will use these properties to separate the three different solids from each other. The table below lists how many grams of each substance will dissolve in 100 grams of water at various temperatures in degrees Celsius.

Aqueous Solubility (g/100 g water) at Various Temperatures

Substance	0 °C	20 °C	40 °C	80 °C	100 °C
SiO_2	0	0	0	0	0
NaCl	35.6	35.8	36.5	38.1	39.2
C_6H_5COOH	0.17	0.42	1.1	2.7	7.1

In separating the mixture, we will use a few different techniques.

Heating With Bunsen Burner

Bunsen burners are effective ways to heat a sample. There are two adjustable parameters on a Bunsen burner: the gas flow rate and the mixing ratio with air.

To ignite the Bunsen burner, first turn on the gas and then use a striker to ignite the gas. Once the Bunsen burner is lit, the gas flow and mixing ratio are adjusted to produce an efficient flame. An efficient flame has a sharp inner blue cone of combustion.

If your Bunsen burner does not light, first check to make sure you have gas flow. If you are properly turning on the gas and have flow, then check to see if the striker is producing sparks when you use it. Test the striker a few times to make sure you can consistently produce sparks. If you have gas flow and are producing sparks, but the Bunsen burner still won’t light, then you need to adjust the air mixing ratio. Turn the air mixing valve to minimize the amount of air that enters the Bunsen burner. This should allow you to light the burner but then you will need to increase the amount of air to produce a more intense flame.

Safety Considerations for a Bunsen Burner

Bunsen burners can pose serious risks in the laboratory. Probably one of the most obvious risks is that you will use the Bunsen burner to heat objects, and those hot objects can burn you if not handled appropriately. Remember that hot glassware looks identical to cold glassware, so be aware of the temperature of a laboratory tool before handling it.

You also risk igniting other objects. Make sure that paper and other flammable materials are not near the Bunsen burner when lit. This is especially true for flammable liquids. Make sure you are aware of the SDS requirements for the materials you are using on a Bunsen burner. Flammable liquids should never be used in the proximity of a Bunsen burner.

Vacuum Filtration

Filtration is a technique that enables us to separate solids from liquids in the laboratory. The solids become trapped because they are too large to pass through the piece of filter paper, while the liquid travels through the paper.

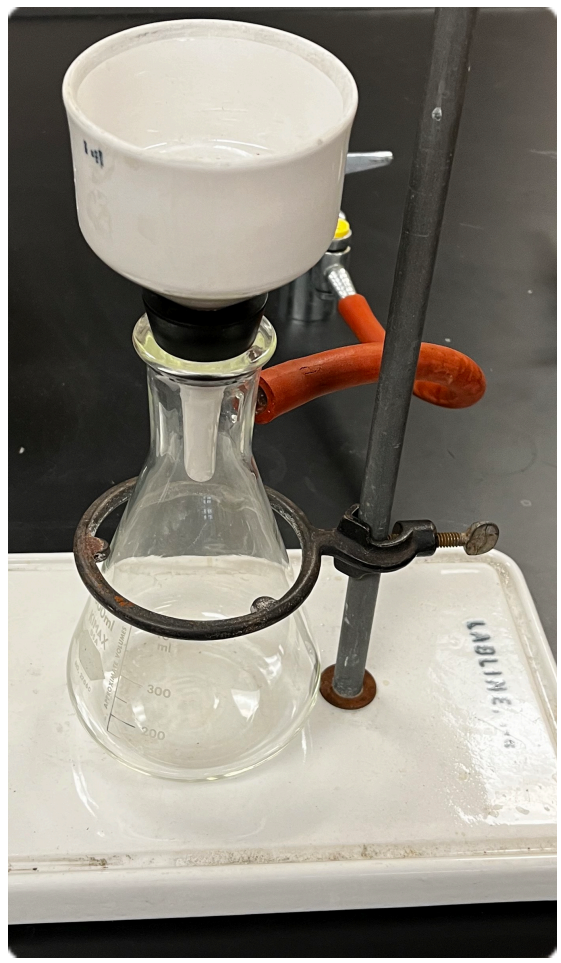

Figure 2.2: Vacuum filtration apparatus. Red tubing is connected to vacuum line. Filter paper is placed in white funnel.

Vacuum filtration uses the same idea while also pulling a vacuum inside the filter flask to increase the flow rate of liquid through the filter paper.

After filtration is completed, all the solid is stuck to the filter paper. It is difficult if not impossible to separate completely the filter paper from the solid at this point.

If you are trying to quantify the mass of the solid, which type of mass measurement technique should you use? Massing by difference is needed when using filter paper.

The solid on the filter paper is usually washed multiple times before being allowed to dry on the filter paper.

Decanting

To decant means to pour off the liquid from a solid.
When you decant, it is important to prevent the solid from being transferred to the new container. It is also helpful to rinse the solid after decanting to make sure the separation is complete.

Experiment

1. You will analyze about 5 grams of the mixture in this laboratory. Make sure to think about what masses you need to record during this process. Remember we are trying to quantify what percentage of the mass is SiO_2, what percentage of the mass is NaCl, and what percentage of the mass is C_6H_5COOH.

2. To the total mixture, add about 50 mL of distilled water and boil the water and slurry mixture while stirring. What components will dissolve in the water? What components will NOT dissolve in the water? Use the solubility table to make this determination. After the water has boiled, decant the liquid into a

new beaker. Think about if you want to take the mass of the new beaker before pouring the liquid into it. Rinse the solid with more boiling distilled water and add that to the new beaker as well.

3. Once the liquid in the new beaker is cool to the touch, place that beaker in an ice bath for 15 minutes to cool it down to zero degrees Celsius. After the beaker is cooled in the ice bath you should see a new solid formed. What component is the solid that forms in the ice bath? Use the solubility properties to determine what components are in the solution and what components are in the solid at this time.

4. Use vacuum filtration to separate the liquid from the solid. Before performing vacuum filtration, make sure to think about what information you will need to determine the mass of the solid and what you will use to wash the solid (either boiling water or ice water).

5. We now have two solids and one liquid. The solid on the filter paper can be dried by continually pulling the vacuum. The solid in the beaker can be dried by gently heating over a flame. The component that is still dissolved in water can be isolated by transferring to a beaker and gently heating to remove the water.

6. When heating to remove the water, use “boiling stones,” so the solution does not splatter. Add a few boiling stones to the solution. Think about whether you will need to know the mass of these stones to determine the mass of the last component.

Safety Considerations

Practice appropriate fire safety protocols for using Bunsen burners and working with hot glassware. Examine SDS sheets for NaCl, SiO_2, and benzoic acid (C_6H_5COOH) to note special safety considerations for working with these materials.

Waste Disposal

Dispose of solid waste in the appropriately labeled solid waste container. Dispose liquid waste in the appropriately labeled liquid waste container.

Introduction for Treatment of Data

A key component of scientific analysis is the repeatability of experiments. We want to get consistent results when trials are repeated and also understand how much variability exists in the measurements. Accomplishing both repeatability and gaining an understanding of how much measurements will vary from one experiment to the next are key to the scientific process.

Accuracy and Precision

Whenever we take measurements we are generally concerned with the quality of those measurements. We define the quality of the measurement using precision and accuracy measures. In your Introductory Chemistry courses at Millersville University, we will define the precision of a measurement using Relative Average Deviation and accuracy using Relative Error.

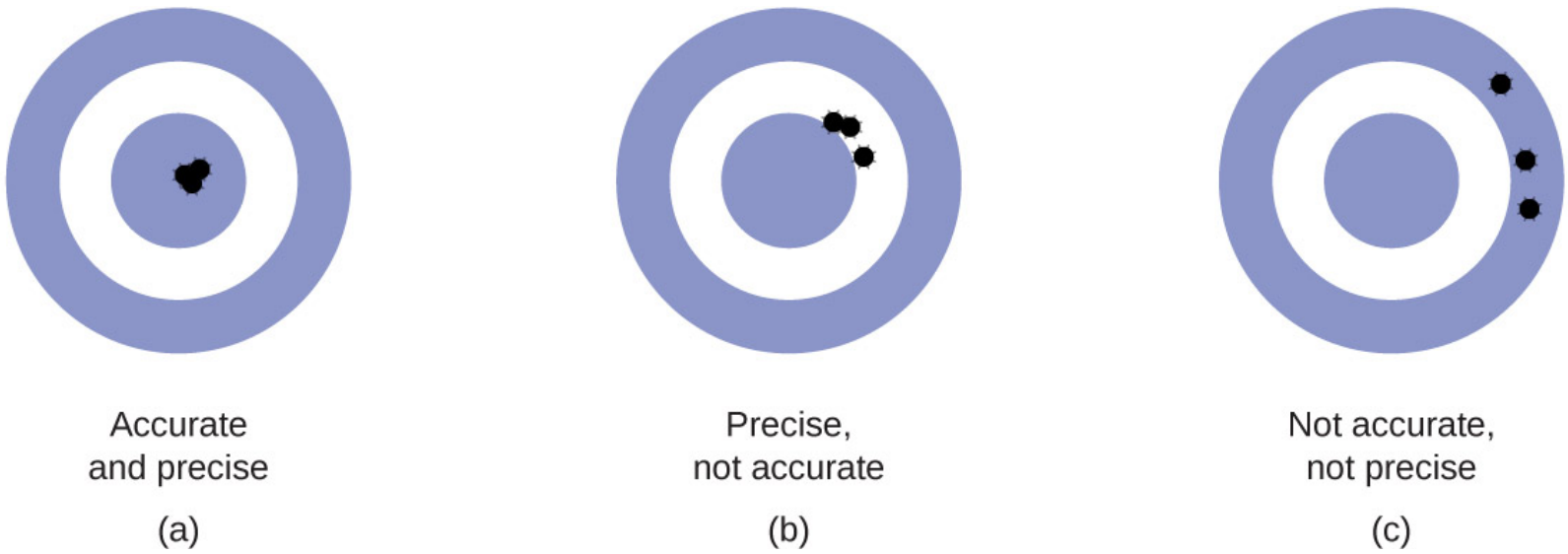

Figure 2.3: 1.6: Measurement Uncertainty, Accuracy, and Precision is shared under a CC BY 4.0 license and was authored, remixed, and/or curated by OpenStax

Accuracy is a measure of how close we are to the "true" value. Sometimes we know the true value, and sometimes we do not know the true value.

Precision is a measure of the repeatability of our measurements. An experiment with high precision means that all the results are close to each other. An experiment with low precision has not very repeatable results.

Average Value

Since we want to understand the repeatability of experiments, we will often repeat an experiment many times. This repetition allows us to see the consistency or lack of consistency in our results. A key result of those many measurements is the average value. An average is found by adding the results from all the trials and dividing by the total number of trials.

Overall results are typically reported as an average value. In addition to the average value, we also want to report the repeatability of the measurements. One way to report repeatability is by using Relative Average Deviation.

Relative Average Deviation

Relative Average Deviation (RAD) is expressed as a percentage and essentially tells us how much repeated measures fluctuate. A RAD of 1% would mean we expect each trial to differ by about 1% from each other. This is effectively the uncertainty associated with our measurement. A smaller RAD means your measurements have higher precision. Here we will use a four-number data set (97, 99, 100, and 103) to demonstrate how to calculate RAD:

Step 1: Calculate the average of all your trials:

$$\frac{97 + 99 + 100 + 103}{4} = 99.8$$

Step 2: Take the absolute value of difference between each trial and the average:

$$|97 - 99.8| = 2.8$$

$$|99 - 99.8| = 0.8$$

$$|100 - 99.8| = 0.2$$

$$|103 - 99.8| = 3.2$$

Step 3: Calculate the average of the four deviations from the average found in Step 2:

$$\frac{2.8 + 0.8 + 0.2 + 3.2}{4} = 1.8$$

Step 4: Calculate the RAD by taking the average of deviations in Step 3, dividing it by the average from Step 1 and multiplying it by 100 to get into a percentage.

$$\frac{1.8}{99.8} \times 100\% = 1.8\%$$

Percent Error

Relative Error is expressed as a percentage and essentially tells us how far our measured value is from a true value. It is calculated by taking the absolute value of the difference between a measured value and a true value. If we measure the density of an aluminum bar to be 2.63 g/mL, but the true density of aluminum is 2.70 g/mL then our Relative Error is 3%:

$$\frac{|2.63 - 2.70|}{2.70} \times 100\% = 2.6\%$$

Pre-Lab Questions

1. What are the safety considerations for using a Bunsen burner?

2. In the SDS sheets for SiO_2, NaCl, and C_6H_5COOH, what are the main hazards associated with these substances? What precautions do you think should be taken when working with these substances?

3. Look at the different steps of the experiment. In what phase is each of the three different components at each step of the procedure? In step 2 of the experiment, what type of mixture will you have before decanting and after decanting?

4. Think about the masses you will need to take during the experiment. Set-up a table for recording data in your laboratory notebook where you will record all those masses. Think about whether you will be measuring directly or measuring by difference for each of the components. If measuring by difference, between what masses will you be taking the difference?

5. What physical property of the mixture components are you using to separate them? What are the laboratory techniques used to separate each of the different components from the various mixtures?

Post-Lab Questions

1. What are the masses of each component?

2. What is the percent mass of each component in the mixture?

3. Collect data about the percent masses from four other groups in the laboratory. Use that data to calculate the average percent mass of each component and the relative average deviation of the mass percent of each component.

4. Your instructor will provide you with the true mass percents of each component. Calculate the percent error of your group average mass percents.

5. Which mass percent was the most and which was the least precise? Which mass percent was the most and which was the least accurate?

6. Explain experimentally why you think the mass percents of certain components were more or less precise or accurate.

References

Dr. Aimee Miller, *CHEM 103 Laboratory Manual*, Millersville University.

Measuring Volumes - Technique Laboratory

Motivation

Many chemical reactions take place in the solution phase with the reacting chemicals dissolved in a liquid solvent. In environmental and biological applications, that solvent is routinely water. In the synthesis of organic compounds for medicines, fabrics, plastics, etc., the solvent is typically something other than water. No matter your primary experimental scientific interests (biology, chemistry, earth sciences, medicine, etc.), you will likely work with liquid samples.

Figure 3.1: "Oxfam engineer tests the water quality" by Oxfam East Africa is licensed under CC BY 2.0.

A convenient and often used measurement for quantifying the amount of a liquid is its volume. This is true for everyday products we use like gasoline, water, or milk, and it is true in the chemistry laboratory, too. In this lab we will explore different techniques and tools for measuring liquid volumes, explore their accuracy and precision, and determine when the different tools will be appropriate.

Cleaning Glassware for Volumetric Measurements

Clean glassware is critical to achieving reliable results in the laboratory. For volumetric measuring devices you are using, it is best to clean them with water first and then to clean with the liquid that you will be using. The primary idea is that a little bit of the liquid always gets left behind on surfaces, so the best thing to do is ensure that the liquid left behind is the liquid you are using in the measurement.

When rinsing glassware for cleaning, you should do multiple small volume cleanings instead of one large volume cleaning. Rinsing a beaker three different times with 10 mL of water each time is much more effective than rinsing once with 30 mL of water. When rinsing make sure to coat all of the walls of the glassware with the rinse liquid.

Volume Measuring Devices

Below are descriptions of some of the common tools in the chemistry laboratory for measuring volumes and tips and tricks for using them.

Beakers And Erlenmeyer Flasks

Beakers and Erlenmeyer flasks are convenient ways to work with liquids in the laboratory because it is easy to add and remove liquids to them. Beakers and Erlenmeyer flasks come in many different sizes (total volumes) and often have markings along the side that indicate additional volumes. For example, a 100 mL beaker might have markings every 10 mL and a main marking at 100 mL.

It is tempting to use beakers and Erlenmeyer flasks to measure a volume because they are always readily available, but the markings on these tools are like the picture below, a “mL inspired volume.”

Figure 3.2: Erlenmeyer Flask: "Erlenmeyer Flasks 1" by biologycorner is licensed under CC BY-NC 2.0.

Figure 3.3: Beaker: "Siphon beaker" by niallkennedy is licensed under CC BY-NC 2.0.

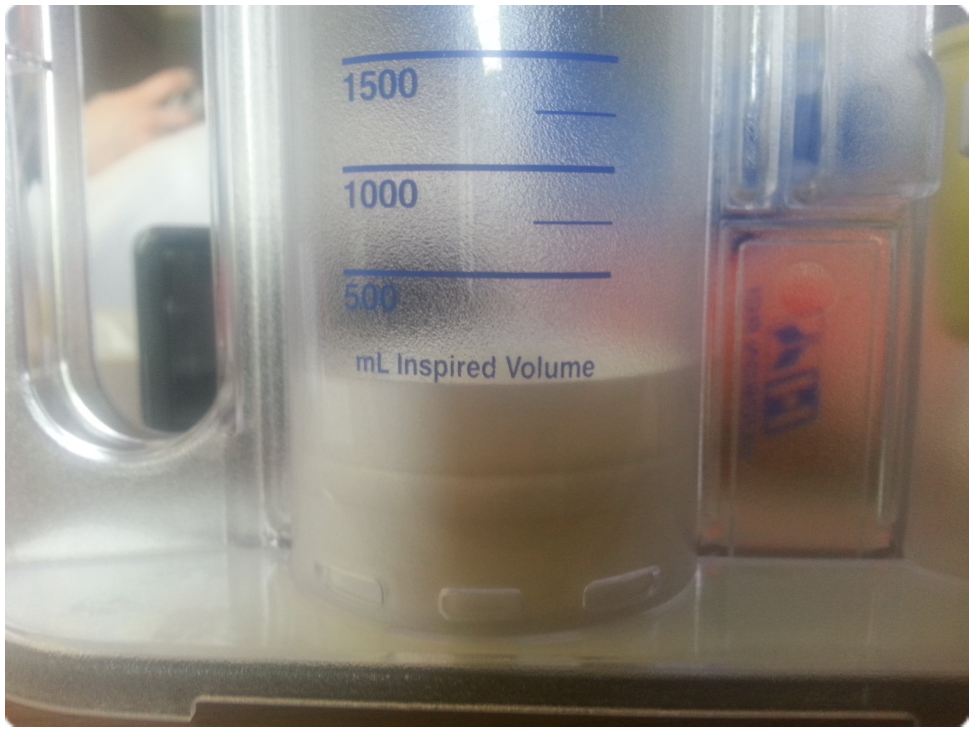

Figure 3.4: Volume measuring container at a hospital.

If you care at all about having a known or consistent volume, avoid the temptation of using the markings on beakers and Erlenmeyer flasks for measurements of volume.

Graduated Cylinders

Graduated cylinders are tubes with marks that indicate specific volumes. Graduated cylinders come in a variety of sizes, and the uncertainty associated with the graduated cylinder depends on the size of the cylinder and the markings. Advantages of graduated cylinders are that they are relatively quick and easy to use and that they also allow for the measurement of any volume within the range of the cylinder. For example, the 100 mL graduated cylinder pictured below could be used to record any volume between 10 and 100 mL.

Figure 3.5: Graduated Cylinder: "Laboratory Equipment - Graduated Cylinder" by eltpics is licensed under CC BY-NC 2.0.

To use a graduated cylinder, you simply fill the cylinder with a liquid and then read the liquid level using the marks on the side of the cylinder. You will notice that the liquid surface is not flat at the top but instead curved as the water adheres to the glass walls of the cylinder. The curved surface of the liquid is called the meniscus. The liquid level is read at the bottom of the meniscus in order to have a consistent measurement standard. Notice that in figure 3.6 the edges of the meniscus are between 19 and 20 mL but the bottom of the meniscus is between 20 and 21 mL.

When you are reading a graduated cylinder, it is critical that you take the time to look closely at the markings and understand the spacing. Not all graduated cylinders are the same, so you can't assume a certain marking pattern. In the picture above each milliliter is marked (19, 20, 21, and 22). In addition, there are nine lines in-between each milliliter value so each of these lines represents one-tenth of a milliliter. When reading the graduated cylinder in the picture above, I see the bottom of the meniscus is in between the 1st and 2nd line after the 20 mL mark. Notice that the bottom of the meniscus is likely to not fall exactly on a marking, which allows us to interpolate the value to get the correct value along with an estimate of the uncertainty.

Figure 3.6: "Buret" by photobunny is licensed under CC BY-NC-ND 2.0.

My thought process for reading the cylinder in figure 3.6 is first to note that the value is somewhere between 20 and 21 mL, then I go a step further and see it is between 20.1 and 20.2 mL, and then finally I make my determination of the recorded value by estimating the last digit. It looks as if it is closer to 20.2 mL than 20.1 mL, so I would read it as 20.18 mL. Someone else might come along and have a slightly different estimation of the last digit, maybe they think that it is 20.16 mL or 20.19 mL. This slight disagreement is fine and gives an approximation of the uncertainty of the measurement. I would record this value with four significant digits since my last recorded digit should be the one that has uncertainty.

An important error to avoid in volume measurement readings is parallax error. This error occurs because the bottom of the meniscus is not in the exact same location as the markings. The bottom of the meniscus is in the center of the measuring device and the markings are on the walls of the device. You will get an incorrect reading that has parallax error if your eye level is above or below the meniscus. Always read volumes with your eye at the same height as the meniscus to ensure you don't have parallax error. This will often mean you need to lift up the measuring device or reposition your body in order to be at eye level with the meniscus.

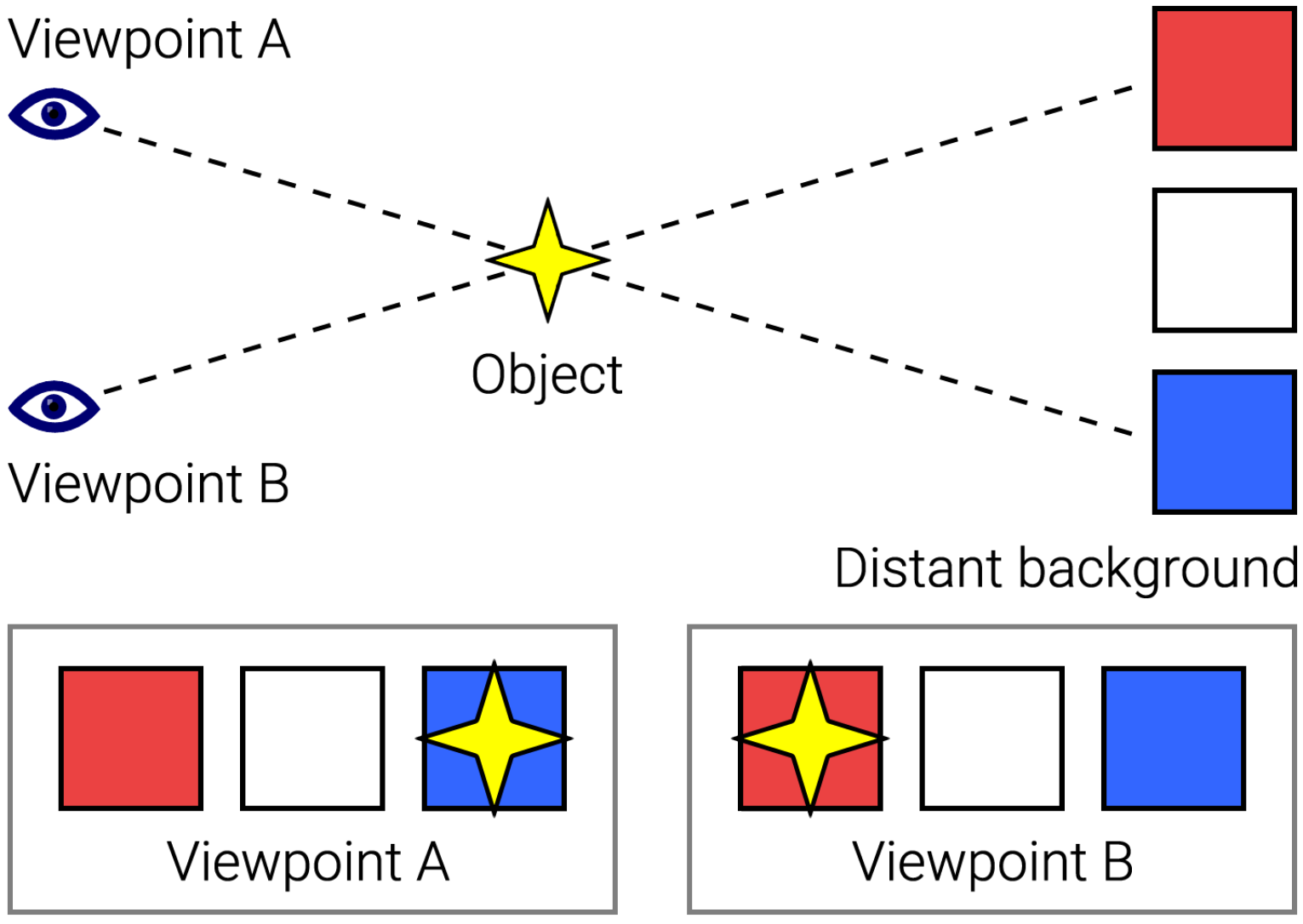

Figure 3.7: Parallax Example.png by JustinWick is licensed under CC BY-SA 3.0.

Reminders for graduated cylinders:

1. Pay attention to carefully interpret the markings.
2. Avoid parallax error by having the meniscus at eye level.
3. Estimate the last digit to appropriately reflect the uncertainty of the measurement.

Volumetric Pipets

Unlike graduated cylinders, volumetric pipets are designed to deliver only a specific volume of liquid. If 10 mL of liquid is needed, a 10 mL volumetric pipet is used, but if 25 mL of liquid is needed, then a completely different 25 mL volumetric pipet is used. A volumetric pipet is a specialist in that it can deliver only the one specific volume that it was designed to deliver. A volumetric pipet has only one mark on it. That mark is the line for the volume of liquid the pipet was designed to deliver.

Figure 3.8: Various sizes of volumetric pipets.

A pipet bulb is used to draw liquid into the pipet from a separate container (typically an Erlenmeyer flask or beaker). To use a volumetric pipet:

1. Place the tip of the pipet into the liquid.
2. Take the pipet bulb not attached to the pipet and squeeze it.
3. Attach the squeezed bulb to the top of the pipet.
4. Slowly release the bulb while making sure to keep the tip of the pipet submerged in the liquid. If the tip of the pipet becomes dislodged from the liquid, air will be introduced into the pipet and potentially make a big mess to clean-up.
5. Continue to allow the liquid to rise in the pipet until it rises above the mark but has not reached the pipet bulb. It is important to keep liquid from entering the pipet bulb.
6. Remove the pipet bulb and place a finger (thumb or pointer finger work best) on top of the pipet to keep the liquid level constant in the pipet.
7. Slowly release pressure on your finger so that the liquid level slowly drops.
8. Get the bottom of the meniscus to the mark (make sure you read it at eye level to avoid parallax error!). This can take some practice. If you go past the line, you will need to use the pipet bulb again to draw more liquid into the pipet.

9. Take the pipet out of the liquid and wipe off the outside of the pipet.

10. Place the tip of the pipet into a new container, release the pressure from your finger, and allow the liquid to flow down the wall of the container.

11. You will notice that there is a small amount of liquid remaining in the tip of the pipet. Leave that small amount of liquid in the pipet. The small amount of liquid is supposed to be there as the pipets are designed as TD (to deliver) pipets and calibrated knowing that the small amount of liquid gets left behind.

Volumetric Flasks

Volumetric pipets are designed to deliver a specific volume, whereas volumetric flasks are designed to contain a specific volume. Like volumetric pipets, volumetric flasks are designed for only one specific volume, so if you want a different volume, then you need to use a different volumetric flask.

When filling a volumetric flask, you need to be careful when the liquid approaches the line because you will need to start completely over once it passes the line. It is helpful to use a dropper to control the last bit of liquid added to ensure that it is not filled above the line.

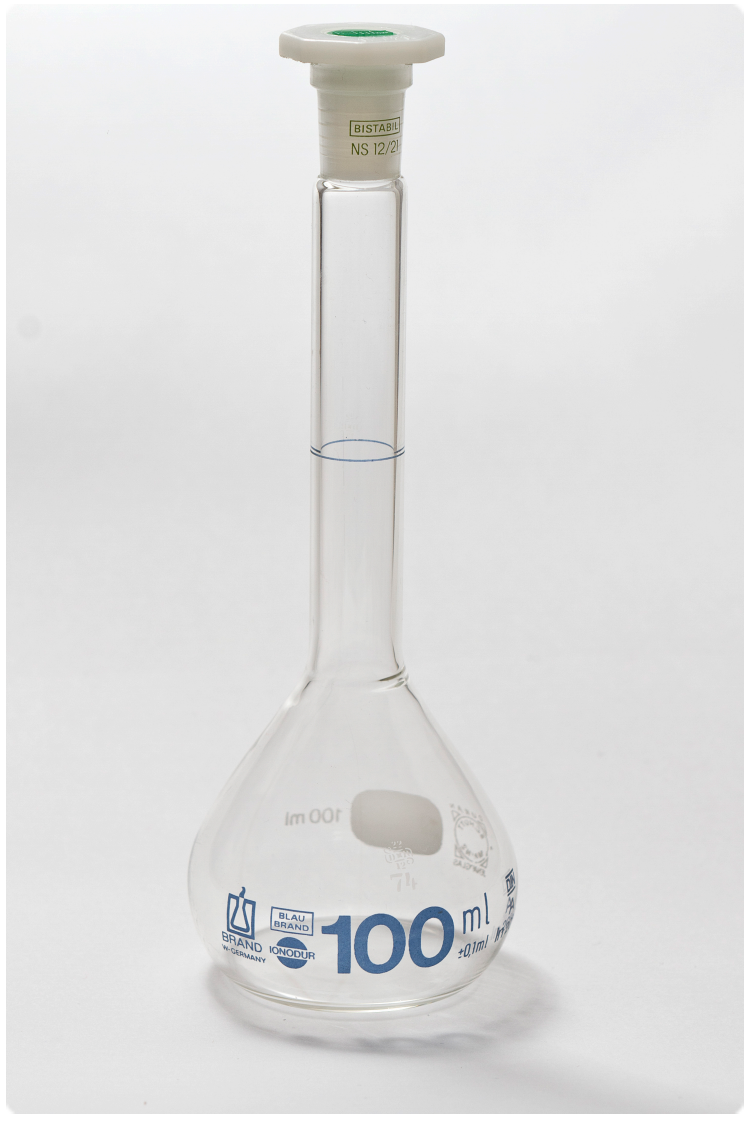

Figure 3.9: Volumetric Flask: "File:Brand volumetric flask 100ml.jpg" by Lucasbosch is licensed under CC BY-SA 3.0.

Practice Technique for Graduated Cylinder, Volumetric Pipet, and Volumetric Flask

Before we start comparing the different measuring tools, we will practice and demonstrate to our lab partners that we can appropriately apply these techniques.

For the graduated cylinder add a random amount of liquid and record the volume. Have everyone at your lab bench do the same without communicating. Now read the volumes on all the graduated cylinders. Compare your recorded results with the other people in your lab group to ensure you have consistent results. Have the lab bench sign off in your notebook that you properly can use this skill.

Add exactly 25 mL of liquid to your graduated cylinder. Have a lab partner check that you have appropriately added 25 mL. Have the lab bench sign off in your notebook that you properly can use this skill.

Demonstrate the appropriate use of a volumetric pipet to your lab bench. Have the lab bench sign off in your notebook that you properly can use this skill.

Demonstrate the appropriate use of a volumetric flask to your lab bench. Have the lab bench sign off in your notebook that you properly can use this skill.

Determine Accuracy and Precision of Measurement Devices

Goal: Determine the accuracy (relative error) and precision (relative average deviation) for measuring 25 mL of water using four different tools: beaker, graduated cylinder, volumetric pipet, and volumetric flask.

A common way to calibrate volume-measuring devices is to measure the mass of a liquid that has a well-established density. Water is commonly used since it is readily available, and the density is well-established. Here is the basic procedure for determining a volume using density:

1. Record the mass of a dry, empty container. Beakers or Erlenmeyer flasks work well.

2. Measure an amount of liquid using a volume measuring device and pour the liquid into the container of known mass from step 1. For example, measure 25 mL of water with a graduated cylinder and pour the water into a beaker.

3. Record the mass of the container holding the water.

4. Determine the mass of the water that was poured into the container with the massing-by-difference approach you learned about previously.

5. Use the known density of the water to calculate the actual volume of water that was delivered.

Volumes and densities change as the temperature changes, but mass does NOT change with temperature. You will need to record the temperature in the room and look up the true density of water at that temperature using the US Department of the Interior water density table.

Here is an example of what a record of this could look like in your notebook and the accompanying calculations.

Mass of 150 mL beaker: 240.345 g

Recorded Volume on graduated cylinder: 25.0 mL

Mass of 150 mL beaker containing water: 265.234 g

Mass of water delivered: $265.234\ g - 240.345\ g = 24.889\ g$

Temperature: 20.9 degrees Celsius

Water Density: 0.9980 g/mL

Actual Volume Delivered: $24.889\ g \times \frac{1\ \text{mL}}{0.9980\ g} = 24.94\ \text{mL}$

In this case I measured 25.0 mL with the graduated cylinder, but the mass tells me that I actually poured 24.94 mL of water into the container.

For each of the four tools, complete four trials of measuring 25 mL. There will be sixteen total trials. The container does not need to be emptied between each trial for a tool.

Waste Disposal

Only distilled water is used in this lab. All waste generated can be disposed of in the sink.

Safety Considerations

Different types of glassware are utilized in this laboratory. Take special care to handle volumetric pipets gently as the neck of the pipet can easily break if a large force is applied.

Pre-Lab Questions

1. What described tools should not be used to measure volumes?

2. Which tools are continuous (can measure any volume) and which tools are discrete (can only measure certain volumes)?

3. What is a meniscus in the chemistry lab and how is it important in volume measurement tools?

4. You have a 15.3 gram mass of liquid water. What is the volume of the water at 21 degrees Celsius? What is the volume of water at 93.3 degrees Celsius?

5. Explain, using a particulate level diagram, what is happening with the total volume and the water molecules in the solution as you change the temperature from 21 to 93.3 degrees Celsius.

Post-Lab Calculations and Questions

1. Calculate the average volume delivered for each tool. You should have four averages.

2. Calculate the relative average deviation (RAD) for each tool. You should have four RAD values.

3. Calculate the percent error for each tool. You should have four percent errors.

4. Rank the tools in order of their accuracy.

5. Rank the tools in order of their precision.

6. Rank the tools in order of their ease of use.

7. Are any tools unacceptable to use? Explain why.

8. For future labs, we will typically use either a graduated cylinder or a volumetric pipet to deliver a specific volume. What are the advantages and disadvantages of a graduated cylinder compared with a volumetric pipet?

9. When do you think it would be appropriate to use graduated cylinders compared with volumetric pipets?

Reaction Types and Qualitative Analysis - Technique Laboratory

Introduction

A wide variety of chemical reactions can be classified into a few types of chemical reactions with different driving forces for what causes the reaction to occur. In this laboratory we will look at a variety of different types of chemical reactions and see how we can use some of those chemical reaction properties to identify different compounds. Our previous labs have all been quantitative, meaning we took measurements to determine precise values. This lab is a qualitative lab, where we don't need to worry about precise values and amounts. In a qualitative lab, keen observations are key to being successful. Practicing scientists use combinations of qualitative and quantitative observations to solve problems. Building both quantitative and qualitative laboratory skills and knowing when to utilize each skill set will help you grow as a scientist.

Observing Chemical Reactions

We can often see chemical reactions occurring by making specific observations that utilize our everyday senses.

Observation 1: Color Changes

The formation of new chemical during a reaction can often produce new colors. Changes in color associated with chemical reactions are most often observed when a transition metal is involved in the reaction.

Observation 2: Formation of Gases and Smell

Gas evolution is often the driving force for a chemical reaction to occur. We can see gas evolution reactions by seeing the bubbling of a gas. Even if we cannot see a gas being formed, we can still check for properties of a gas being formed.

Testing a Gas With a Lit Wooden Splint

A wooden splint (popsicle stick) can be lit with a match. If we place the lit splint over a reaction that is occurring, we can see if a produced gas has any impact on the lit splint. If the gas supports combustion (oxygen gas is a good example of this), then the flame will glow brighter. If the gas is combustible (methane and hydrogen are good example of this), then a quick "pop" is often heard. If the gas

is neither combustible nor supporting of combustion (carbon dioxide is a good example of this), then the flame will extinguish on the splint.

Wafting To Smell a Gas

Many gases have characteristic smells, so even if we don't see the bubbling for the formation of a gas, we can still detect its production via smell.

We never want to inhale a larger amount of a new compound produced in a reaction. To avoid inhaling large amounts, we use a technique called "wafting." To waft you gently wave your hand over the top of a container and lift your hand to your nose. This keeps the concentration that reaches your nose low to avoid harmful impacts.

Observation 3: Formation of a Solid

Another common driving force for chemical reactions in solution is the formation of a solid precipitate. We can detect the formation of a solid by seeing if a solution gets cloudy (hard to see through) when we mix two liquids. A cloudy solution means that solid particles are suspended in the liquid, signifying that a solid was formed.

Clear, Cloudy, and Colorless

In the chemistry lab we use terminology that you use in your everyday life, but sometimes it has a slightly different meaning in the chemistry lab.

In your everyday life you probably use clear and colorless as interchangeable terms. But in chemistry they refer to two completely different ideas. In the chemistry laboratory clear is the opposite of cloudy. When we say a solution is clear, that means that there are not any suspended solids or gases in the solution. Cloudy means there are suspended particles in the solution. A colorless solution means that the solution has no color, and the opposite is when a solution does have color.

In the chemistry lab we can have a clear red solution. Clear means it is free of suspended particles and red gives the color. A clear and colorless solution both has no particles and no color. We can also have a cloudy colorless solution or a cloudy blue solution.

Observation 4: Heat Change Associated With a Reaction

Many chemical reactions occur with large amounts of heat being consumed or produced. We can sense these changes by determining if the temperatures of the materials change when they are mixed together. Simply holding a beaker in your hands will allow for the detection of these changes.

Oxidation-Reduction Reactions: Movement of Electrons

Oxidation-reduction reactions involve the transfer of electrons from one atom to a different atom. Many types of chemical reactions are oxidation-reduction reactions; combination reactions, many decomposition reactions, combustion reactions, and single-displacement reactions are all classified as oxidation-reduction reactions. The key feature to identify is the changing oxidation numbers of different atoms when these reactions occur. When transition metals are involved, we will typically see color changes occurring when the transition metal undergoes a change in oxidation state.

Red-Ox Reaction 1: Oxidation States of Manganese

Here we will look at the reaction of potassium permanganate ($KMnO_4$) with iron(II) chloride ($FeCl_2$) under acidic conditions.

In a medium test-tube, mix about 3 mL of the potassium permanganate solution with 2 mL of HCl solution and 1 mL of iron(II) chloride solution. Record your observations. Manganese compounds in aqueous solutions tend to have different colors based on the oxidation state of the manganese. When manganese is in the +2 oxidation state, aqueous solutions are a pale pink color. When manganese is in the +4 oxidation state, it tends to form insoluble brown compounds. When manganese is in the +7 oxidation state, aqueous solutions have a rich purple color.

Oxidation-reduction reactions involve the transfer of electrons. Is manganese losing or gaining electrons in this process? Some other atom in the reaction must have the opposite behavior with its oxidation state. What other atom do you think is changing its oxidation state? Is it being oxidized or reduced?

Write the reduction half-reaction, oxidation half-reaction, and the balanced Red-Ox reaction for this process.

Red-Ox Reaction 2: Zinc With Hydrochloric Acid

In a small test tube and a small amount of zinc metal to 3 M hydrochloric acid. Zinc is typically found in the +2 oxidation state in ionic compounds.

Record your observations.

Write the reduction half-reaction, oxidation half-reaction, overall balanced chemical reaction, and net ionic equation that occurs and identify all the oxidation states of the atoms.

Red-Ox Reaction 3: Zinc With Copper(II) Chloride Solution

In a small test tube add a small amount of zinc metal to the copper(II) chloride solution.

Record your observations.

Write the reduction half-reaction, oxidation half-reaction, overall balanced chemical reaction, and net ionic equation that occurs and identify all of the oxidation states of the atoms.

Acid-Base Reactions: Movement of H^+

Bronstead-Lowry Acid-Base reactions involve the transfer of hydrogen ions (H^+) from one chemical species to another. We can often determine if a liquid is acidic or basic by seeing if it performs an acid-base reaction with an indicator.

Testing With Litmus Papers and Phenolphthalein

Litmus and phenolphthalein are two different compounds that change color depending on whether hydrogen ions (H^+) are added or removed from them. Litmus turns red when an H^+ is added to it and turns blue when an H^+ is removed from it. We can classify solutions as basic if they remove a hydrogen ion from litmus and acidic if they add a hydrogen ion to litmus.

Most litmus paper is sold in two types (Red and Blue). Red litmus paper will turn blue if a solution is placed on it that is basic. Blue litmus paper will turn red if a solution that is acidic is placed on it.When testing with litmus paper, it is important to dip a stir rod into the solution and then touch the stir rod to the litmus paper. This keeps the liquid free from contamination and allows for multiple tests with a single piece of litmus paper.

Phenolphthalein is often used as an indicator in acid-base reactions. Phenolphthalein is colorless when the solution is acidic, but when the solution is

basic, phenolphthalein loses an H^+ and then changes to a pink color. To test solutions with phenolphthalein, only a few drops (2 to 3) of the indicator are added to a solution.

We will test each of the following 1M solutions with red and blue litmus and with phenolphthalein.

NaCl, HCl, NaOH, Ammonium Chloride, and Sodium Carbonate

Classify all these solutions as acidic, basic, or neutral based on your observations.

Acid-Base Reaction 1: HCl With Sodium Carbonate

Mix equal volumes of the HCl and sodium carbonate solutions you tested above. Make observations based on your senses to determine if a reaction occurred.

Record all observations and write the balanced chemical reaction along with the full and net ionic equations. Was the reaction exothermic or endothermic?

Acid-Base Reaction 2: HCl With NaOH

Mix equal volumes of the HCl and sodium hydroxide solutions you tested above. Make observations based on your senses to determine if a reaction occurred.

Record all observations and write the balanced chemical reaction along with the full and net ionic equations. Was the reaction exothermic or endothermic?

Acid-Base Reaction 3: Ammonium Chloride With NaOH

Mix equal volumes of the ammonium chloride and sodium hydroxide solutions you tested above. Make observations based on your senses to determine if a reaction occurred.

Record all observations and write the balanced chemical reaction along with the full and net ionic equations. Was the reaction exothermic or endothermic?

Precipitation Reactions: Solid Formation

Precipitation reactions occur when two soluble ionic compounds react to form an insoluble solid (precipitate). These reactions can be predicted by knowing solubility rules. The driving force for these reactions is the formation of an insoluble solid.

We will test the following solutions with both silver nitrate and barium nitrate. The silver and barium tests are a good way to determine what anions are present in a solution of an ionic compound.

Sodium Chloride, Sodium Acetate, Sodium Sulfate, Sodium Carbonate, Sodium Nitrate, and Sodium Chromate.

Figure 4.1: Lead nitrate and sodium iodide are mixed to give a lead iodide precipitate. This Wikimedia image is shared under a CC BY-3.0 license.

Precipitation Reactions 1: Silver Nitrate

Record your observations when adding three drops of silver nitrate solution to a small amount of each of these solutions in a small test tube.

Write balanced full and net ionic equations for each ch emical reaction that occurs.

Precipitation Reactions 3: Barium Chloride

Record your observations when adding three drops of silver nitrate solution to a small amount of each of these solutions in a small test tube.

Write balanced full and net ionic equations for each chemical reaction that occurs.

Flame Tests

Flame tests are often used to identify cations in soluble ionic compounds. Think about the vibrant colors you see in fireworks. The same process occurring to produce color in fireworks is what we will see with flame tests. Many cations give off characteristic colors when placed in the outer cone of a flame.