Basic Techniques

Calorimetry

Calorimetry is used to measure the change in heat (released or absorbed) during a chemical reaction. In chemistry labs, the setup for calorimetry generally consists of a Styrofoam cup with a sealed lid and hole for thermometer to measure the change in temperature during the reaction.

Centrifugation

Centrifugation is used to separate solids and liquids in heterogeneous mixtures. Successful separation results in the solid portion (pellet) at the bottom of the centrifuge tube and the liquid portion (supernatant) above it. When using a centrifuge, it is important that it remains balanced at all times – any tube being spun should have one of equal volume placed in the opposite slot.

Quantitative Transfer

Proper quantitative transfer methods ensure that the entire quantity of a substance has been transferred (for example, if an experiment requires that 5g of a powder are transferred into a beaker, it is easy for small amounts of the powder to be lost during transfer or left behind on the transfer medium – this can affect accuracy of results).

When transferring powders, the mass should first be measured on creased weighing paper. Next, the weighing paper should carefully be folded and tipped into its destination (e.g. a beaker). A spatula should then be tapped against the paper to dislodge any remaining powder. Finally, the paper should be rinsed into the beaker to ensure full transfer of the powder.

When transferring a liquid, after it is poured into its destination, the sides of the vessel it was poured from should be rinsed with solvent and continue to be poured into the destination to ensure that all of the liquid has been transferred.

Titration

Titration allows for one to determine the endpoint of a reaction and the quantity of reactant used to achieve it. Titration generally involves a buret (to deliver precise amounts of the titrant) and a flask (generally an Erlenmeyer flask) containing the solution. An indicator that changes color is used to determine when the equivalency point has been crossed once sufficient quantity of the titrant has been added. Sometimes, a pH meter will be used to detect the endpoint rather than an indicator.

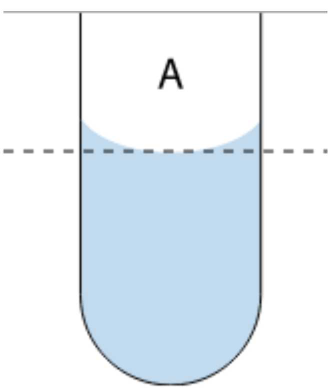
Basic Techniques – Continued

Filtration

Filtration is used to separate solids from liquids in a mixture. In some cases, pouring the solution through filter paper set in a Buchner funnel over a flask. To filter more effectively, a filter flask is used in combination with a vacuum outlet to effectively suck the liquid solution through the filter paper (vacuum filtration). After successful filtration, the solid is left behind on top of the filter paper while the liquid collects in the flask below.

Reading the meniscus:

When measuring volume in graduated glassware, one should always read a meniscus from the **bottom of the curve**. In the image below, one's eye should be level with the dotted line:



(Source: cropped from https://en.wikipedia.org/wiki/Meniscus#/media/File:Reading_the_meniscus.svg)

Drying glassware

Some glassware can be placed in an oven for drying, but volumetric glassware (e.g. pipets, burets, volumetric flasks, graduated cylinders, etc.) should **never** be placed in an oven - the heat can ruin the calibration.

Cleaning glassware

Once glassware is cleaned with soap or detergent in tap water, it should then be rinsed with deionized water to avoid potentially contaminating future solutions with small concentrations of residual ions.

Equipment

Beaker
Beakers can be used for the transfer of liquids, mixture of liquids, or for reactions – however, beakers are not accurate and should not be used for measurements.

Graduated cylinder
A graduated cylinder can be used for the transfer of liquids or for general measurements (with accuracy varying based on the extent of volume indications present – e.g., to the nearest mL). They are not the most accurate method of transferring liquids – a pipette or buret is better suited for transfer of liquids when very high accuracy is needed. A graduated cylinder (along with any other form of volumetric container holding a liquid) should always be read from the bottom of the meniscus (see Data Analysis for additional details).

Erlenmeyer Flask
Like a beaker, an Erlenmeyer flask can be used for the transfer of liquids, mixture of liquids, or for reactions – but is not accurate and should not be used for measurements. Since an Erlenmeyer flask has a narrower neck than a beaker, it helps prevent splashing outside of the container (this is particularly useful during titrations, where a liquid is being dropped into a solution).

Volumetric Flask
A volumetric flask is used as a container for a liquid of a known, precise amount (or the creation of a mixture to a known volume). These flasks have a very narrow neck along which there is a mark for the precise amount the flask holds (e.g. one can be confident a 250 mL volumetric flask with liquid up to the marking point contains almost exactly 250 mL).

Filter Flask
A filter flask is used during vacuum filtration. It is very similar in appearance to an Erlenmeyer flask, but the side of the neck contains a protruding “side arm” along which a vacuum hose can be fixed to create suction.

Test tube
Like beakers, test tubes can be used for the transfer of liquids, mixture of liquids, or for reactions – but they are not accurate for measurements of volume and should not be used for this purpose. They are generally useful for the transfer of smaller quantities of liquid.

Equipment – Continued

Pipet
A pipet is used for the measurement and transfer of very precise amounts of liquid. Pipettes may be graduated (with several markings for varying volume) or volumetric (a single marking for a set volume). It is important when transferring liquid with a pipette not to "blow out" or force any remaining liquid at the very end of the pipette after the initial transfer – a pipette is calibrated to account for this remaining liquid.

Buret
A buret is used for the transfer of very precise amounts of liquid. They are primarily used in titrations to deliver specific, precise amounts of the titrant. A buret contains a stopcock along its side that allows one to deliver solution gradually. Once the endpoint is reached, the stopcock is closed and the markings on the buret can be ready to determine the precise amount of liquid that was delivered to the solution.

Bunsen burner
A Bunsen burner produces a gas flame used for heating in the laboratory. Caution should always be taken when using an open flame.

Calorimeter
A calorimeter is used to measure the change in heat from a reaction while minimizing the transfer of heat to the outside environment. In general chemistry labs, a simple setup of two stacked Styrofoam cups, a lid with a hole, and a thermometer are used.

Balance (digital)
A digital balance is used to measure mass with a high degree of precision. A digital balance should be swept clean, then turned on and set to zero. Then, a creased piece of weighing paper should be placed on the balance and it should once again be set to zero (to exclude the weight of the weighing paper from the mass being measured). Finally, the mass being measured should carefully be transferred to weighing paper (gradually if necessary) and the measurement should be recorded.

pH Meter
A pH meter is used to measure the pH of a solution. Depending on the laboratory, this device may be analog or digital. A pH meter should be calibrated in solutions of known pH before use.

Equipment – Continued

Buchner Funnel
Used during vacuum filtration. It sits on top of the flask and holds the filter paper.

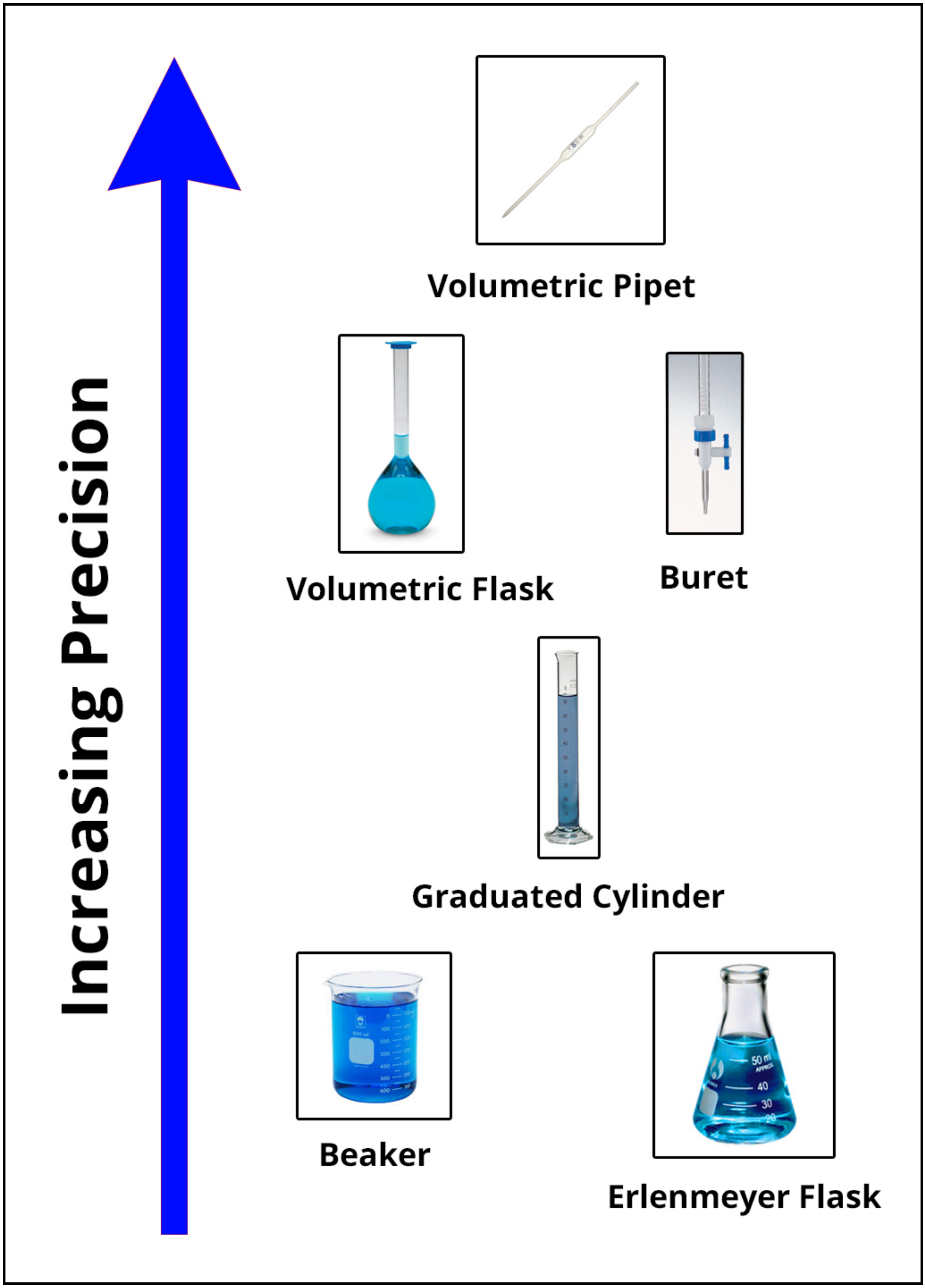
Gravity filter funnel
Used during gravity filtration (not vacuum filtration). Does not form the necessary seal for vacuum filtration and should not be used for this purpose.

Separatory funnel
Used to separate immiscible liquids

Dropping funnel
Used to add liquid reagents (one drop at a time) to a flask

Powder funnel
Used for adding solid reagents to a flask

Laboratory Equipment Listed in <u>Increasing Precision</u>



Error Analysis

To calculate the percentage error of a determined value against an actual value, the following formula should be used:

$$\frac{Determined - Actual}{Actual} \times 100 = \%\ error$$

Types of error:

Systematic Errors are errors that result from flawed equipment or experimental design. They can be detected and corrected for (for example, if your scale used to measure mass was not set to zero beforehand and thus every measurement is off by +5 mg, this error can be adjusted for). Systematic errors can still be precise but will not necessarily be accurate (for more on precision and accuracy, see the Data Analysis section below). All systematic errors produced from the same source will tend be in the same direction (e.g. in the incorrect calibrated scale example above, every measurement would be consistently heavier 5 mg).

Random Errors are errors that result from human interpretation of data. For example, when determining the quantity of liquid in a graduated cylinder by reading a meniscus, you may slightly misjudge the bottom of the curve, resulting in an error of measurement. Random errors can affect both precision (e.g., you may both overestimate and underestimate some readings) and accuracy.

Safety

Safety Shower
Used to wash off chemicals exposed to the skin. Clothing that has been exposed to chemical spills should also be removed while using the safety shower.

Eyewash
Used to rinse out eyes if exposed to chemicals. Eyelids should be held open during rinsing.

Safety Goggles
Safety goggles should be worn at all times during lab, and not removed until leaving the lab. They protect the eyes from exposure to chemicals from splashing or explosions.

Disposable gloves
Should be worn to protect the hands from accidental exposure to chemicals.

Spill neutralizers
Used to neutralize chemicals (particularly acids and bases) if accidentally spilled in the lab.

Fume Hood
Remove potentially harmful vapors that can be emitted during a reaction. All work performed under a fume hood should be done so at a reasonable distance away from the front of the hood so that proper ventilation is achieved.

Safety in the lab is a broad topic that can be covered by the DAT. It is suggested that you review your lab manual from general chemistry to refresh the basics, but in general, apply common sense when answering questions about safety. Know the fundamentals such as always wearing goggles, tying back hair, clearing the space around open flames, and the proper methods of dealing with incidents such as spilled chemicals, broken glass, and fires are. **The correct answer to dealing with an incident in the lab will usually be the one that effectively resolves the issue while exercising the most caution possible to minimize harm to oneself and others.**

Data Analysis

Significant digits

Significant digits are used to indicate the uncertainty of a measurement. For the DAT, you generally do not need to worry about significant digits when performing calculations, but you may be asked specifically about them during the exam. For this reason it is helpful to review how to determine the correct number of significant digits, and how to handle calculations so that the answer contains the appropriate amount of accuracy.

Shortcut to determine the number of significant digits present:

1. If there are decimals anywhere in the number, count all digits starting from the first non-zero number as significant. Examples: in the number 40.02, there are 4 significant digits. In the number 00.056700, there are 5 significant digits. The underlined numbers indicate the significant digits.

2. If there are no decimals anywhere in the number, start counting from the first non-zero number, and stop counting at the last non-zero number. Examples: in the number 56700 there are 3 significant digits. In the number 2002 there are 4 significant digits.

You should also know the correct number of significant digits to use in your answer when performing calculations:

1. When multiplying or dividing numbers with differing amounts of significant digits, the answer should be limited to the lesser number of significant digits. Example: say you were asked to multiply 45.00 * 5.0 First, determine the number of significant digits: 45.00 has 4 significant digits, while 5.0 has only 2 significant digits. Therefore, the answer should be rounded to 2 significant digits. 45.00 * 5.0 is 225, but our answer should only have 2 significant digits, so we round up to 230 as the correct answer.

2. When adding or subtracting numbers with differing amounts of significant digits, things get a bit more complicated. Here, the answer should be presented to the digit of least accuracy in the numbers being added or subtracted. Let us review two examples:

55.8 + 42.39

Data Analysis – Continued

Above, the least accurate place is underlined: it is the tenths place in 55.8. Therefore our answer should be rounded to the least accurate decimal place. 55.8 + 42.39 = 98.19, but since our answer must be rounded to the tenths place, the correct answer is 98.2. This applies to whole numbers as well, as we will see in our next example:
9876 + 25 + 36̲0

Data Analysis – Continued

Once again the least accurate place has been underlined: it is the tens place in the number 360. Therefore our answer can be no more accurate than that digit. 9876 + 25 + 360 = 10,261, but since our answer must be rounded to the tens place, the correct answer is 10,260.

Remember that zeros after a decimal (assuming they follow a non-zero number) do count as significant. So if you were to add 2.50 + 5.6691, the least accurate place is the hundredths place on 2.5<u>0</u> - the underlined zero counts as a digit of accuracy, so here our normal total would be 8.1691 but our adjusted total for accuracy is 8.1<u>7</u> – once again our answer is presented to the least accurate digit present.

Precision

Precision refers to how consistent and close together measured values are to each other. A set of values can be precise without necessarily being accurate.

Accuracy

Accuracy refers to how close a measured value is to the true (actual) value.