General Chemistry Lab Techniques

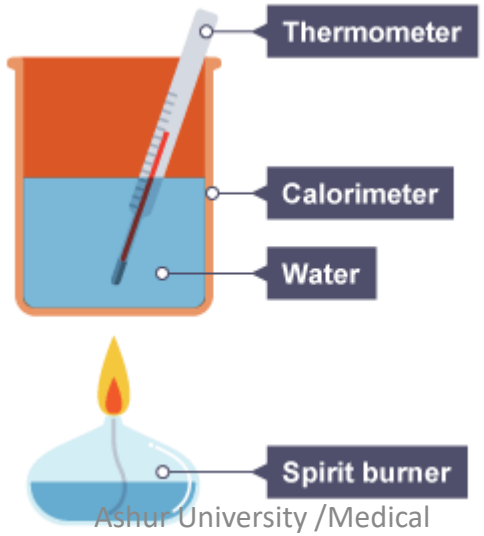


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Basic Techniques

- **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.

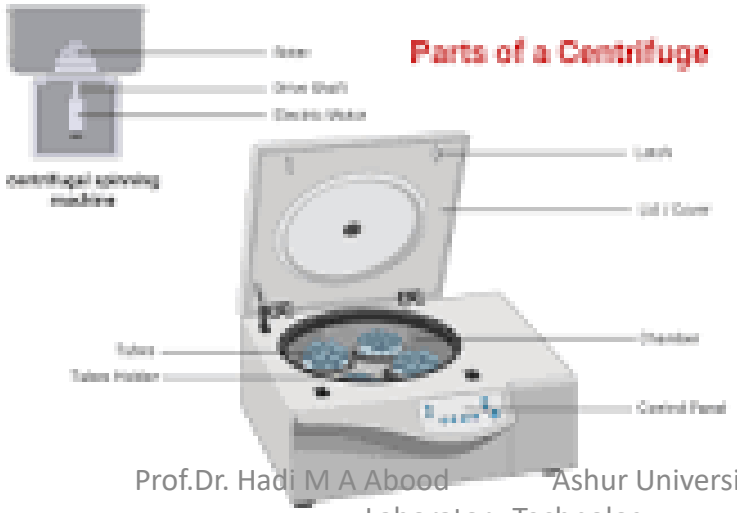


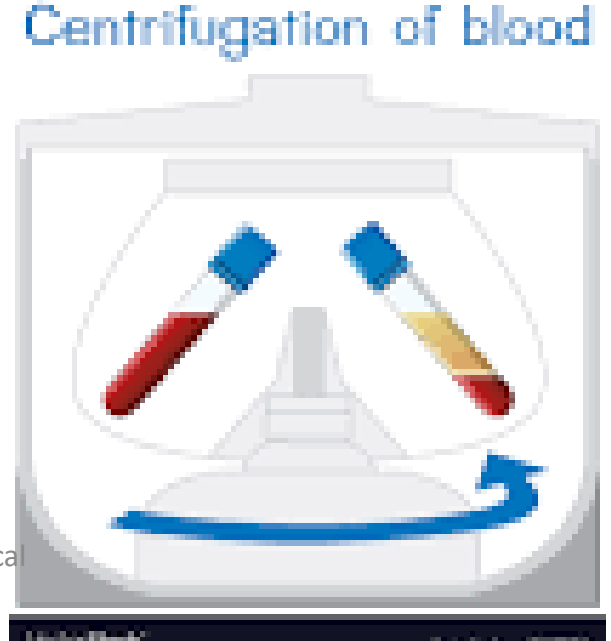
Worked example – calculating energy per gram of fuel

- 3.5 g of a fuel is burned to heat 50 cm^3 of water. The temperature of the water increased from 22°C to 71°C. Calculate the energy released per gram of fuel using the following equation.
- Energy given out = mass of water × 4.2 × temperature change
- Energy measured in joules, J
- Mass of water measured in grams (1 cm^3 = 1 g)
- 4.2 is the specific heat capacity of water, J/g°C
- Temperature change = temperature of water after heating – temperature of water before heating
- Energy given out per gram = energy given out ÷ mass of fuel
- Temperature change = 71 – 22 = 49°C
- Energy given out = 50 × 4.2 × 49 = 10,290 J
- Energy per gram = 10,290 ÷ 3.5 = 2,940 J/g

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.





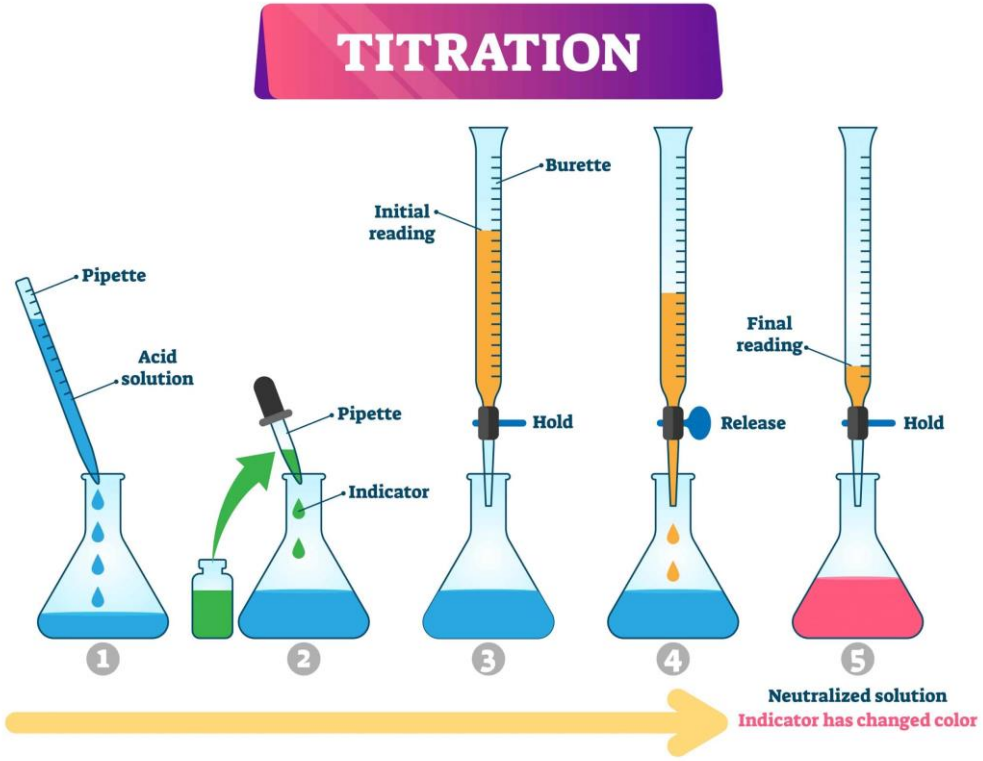
Quantitative transfer



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- 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.

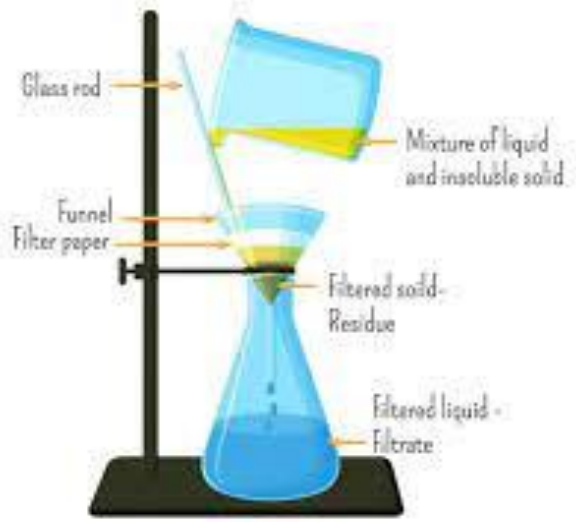
Titration

- Titration allows 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.



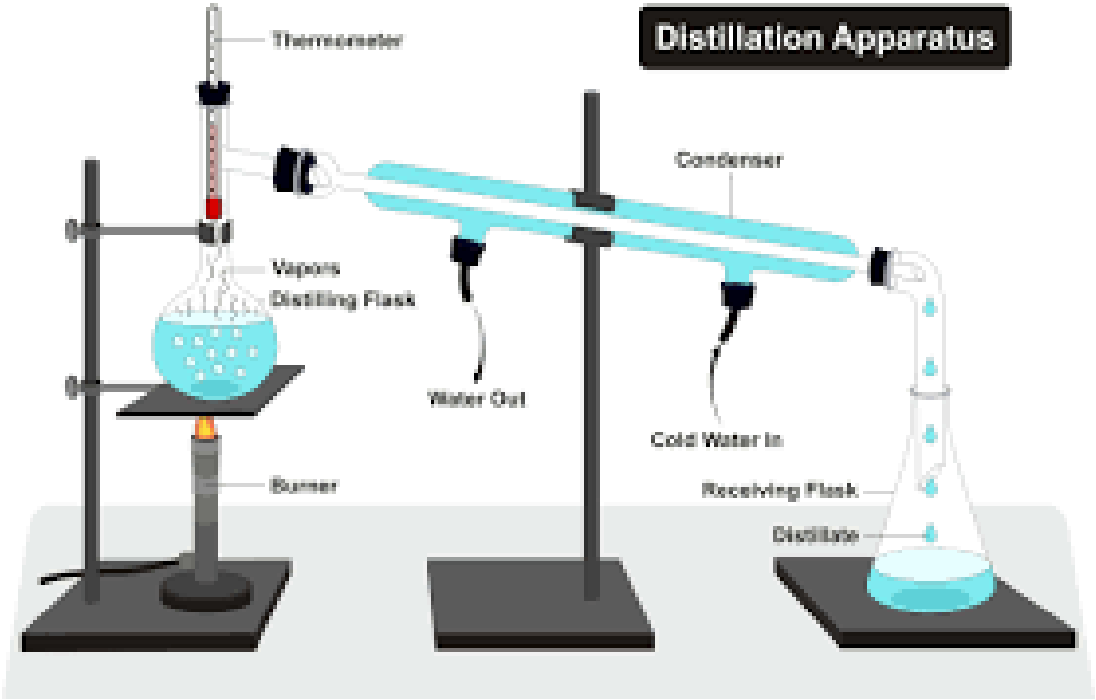
Filtration

- 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.



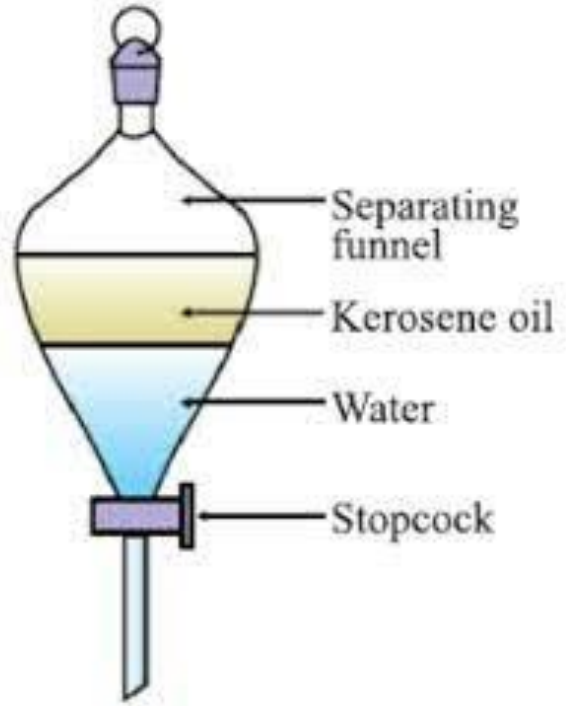
distillation

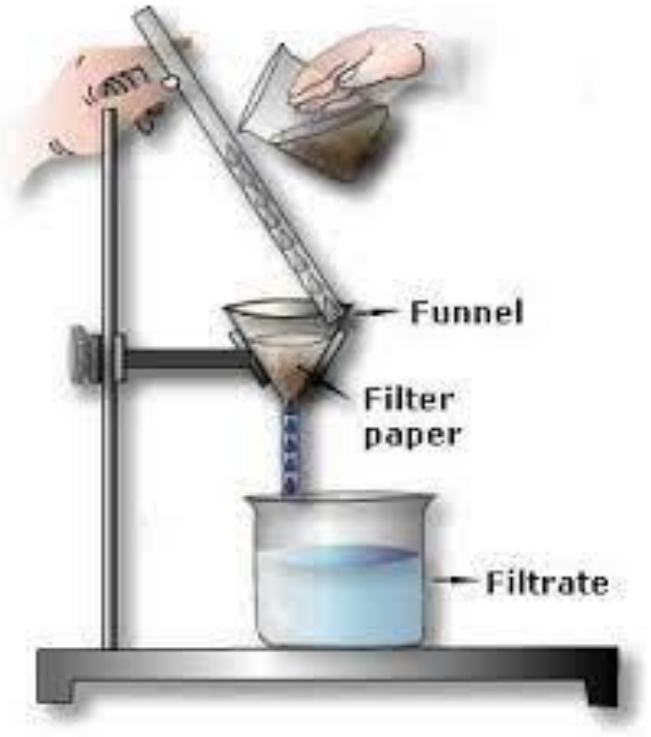
Separates miscible liquids (mix together well) according to differences in boiling points. Often uses a piece of equipment known as a condenser.

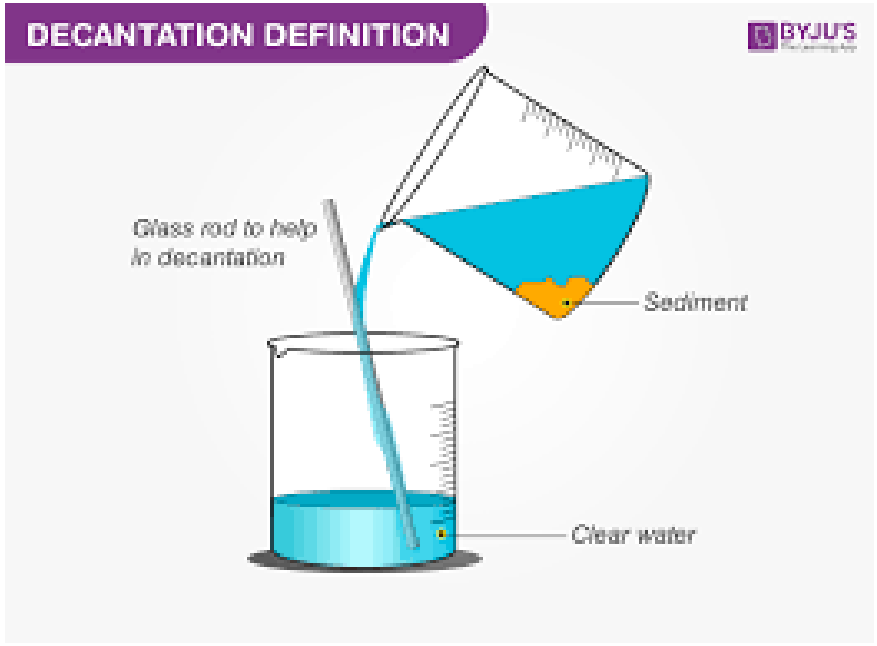


Decantation

- Separates liquids from solids by pouring the liquid carefully off the top of a container.

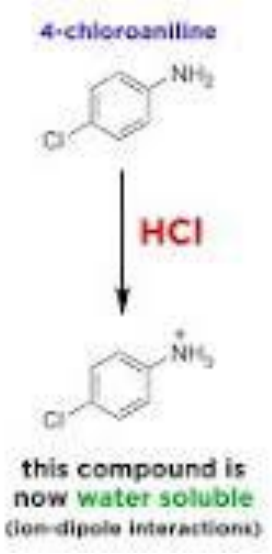


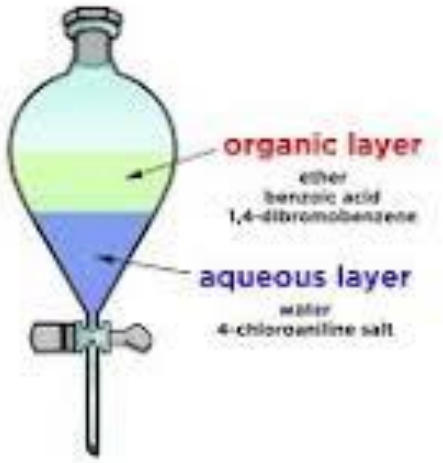


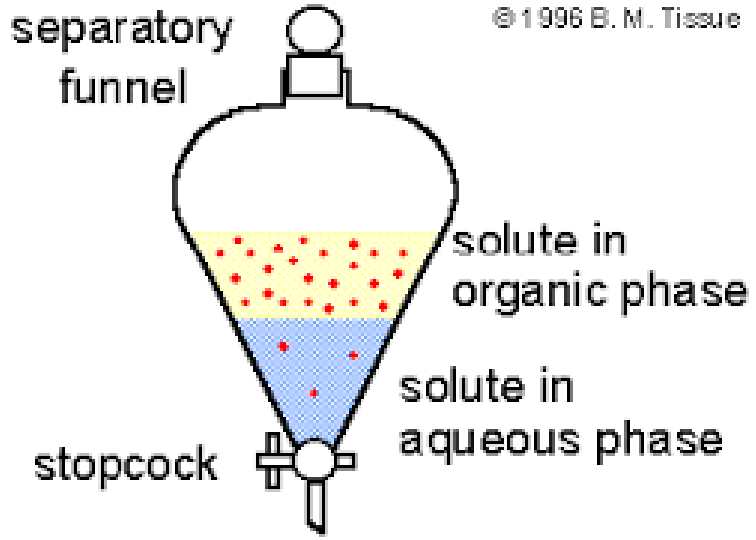


Extraction

- Separates two liquids that don't mix very well due to a difference in polarity, i.e. separating water-soluble (polar) liquids from nonpolar ones. This procedure uses an instrument known as a separatory funnel.





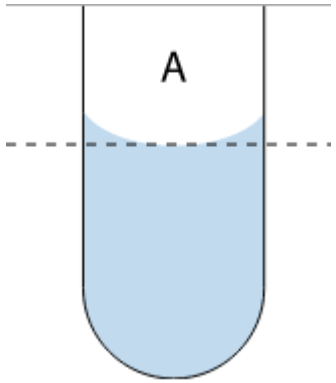


Reading the meniscus:

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 lir

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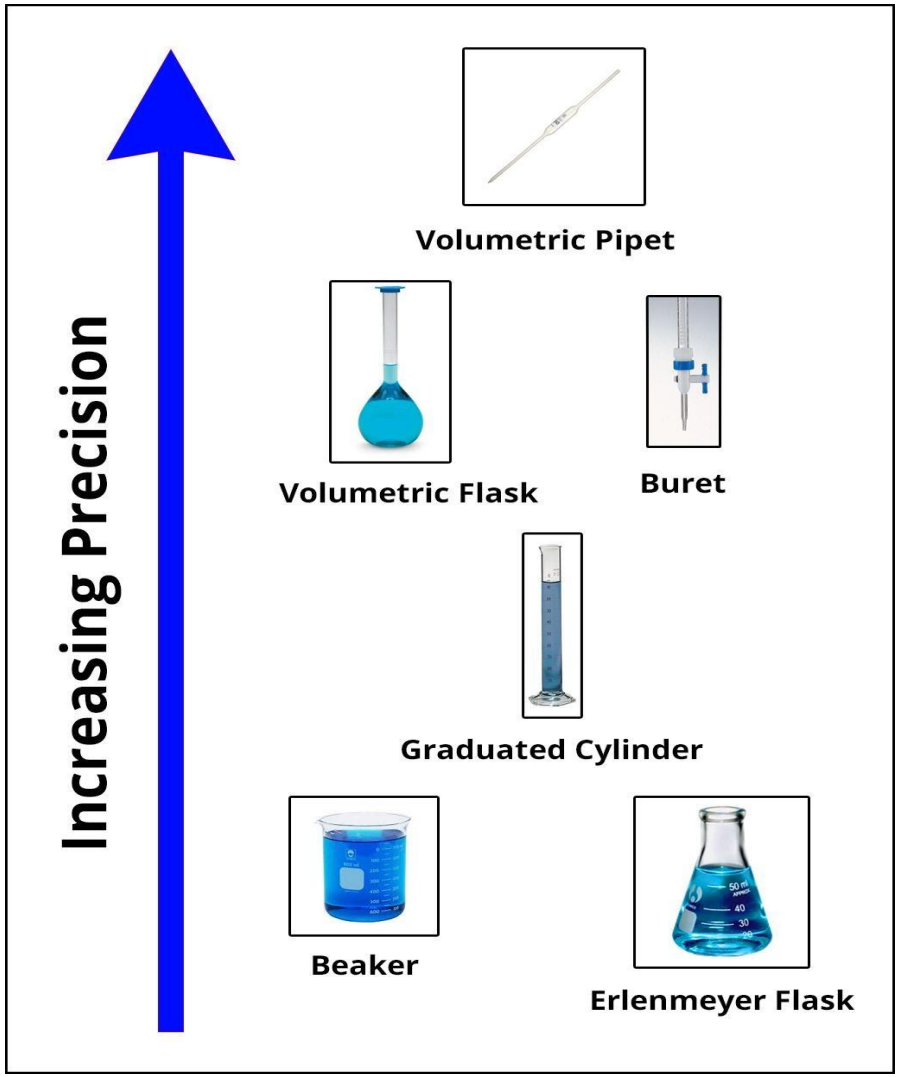
Drying glassware

- 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.





Laboratory Equipment Listed in Increasing Precision



Error Analysis

- To calculate the percentage error of a determined value against an actual value, the following formula should be used:
- (Actual - Theoretical) / Theoretical ×100 = % error

Types of error:

1. **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 to be in the same direction.
2. **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.

Data Analysis

- **Significant digits**
- Significant digits are used to indicate the uncertainty of a measurement. 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.

cont Data Analysis

- 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

  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 + 360

cont Data Analysis

- 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 2.50 - 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 8.17 – once again our answer is presented to the least accurate digit present.