Fundamentals of Lab Analysis

Many labs you'll do in chemistry classes will have you trying to determine something about an unknown chemical or sample: identifying an unknown solution, finding its concentration, or assessing a sample's purity. In all of these cases, you're trying to determine something that cannot be measured directly. We can't use a concentration-ometer to find out how dilute your permanganate solution is. Instead, we need to measure indirectly, through a series of reactions and procedures. This worksheet is intended to help you understand some of the philosophy behind this type of lab analysis. Ideas to keep in mind will be in **bold**.

How do we measure something indirectly? We put a sample through a series of one or more reactions until we get to a position where we *can* measure directly. Let's say that we want to determine the concentration of an $NaC\ell$ solution, and we do so by treating it with $AgNO_3$. Why? Because the Ag^+ ions and the $C\ell^-$ ions will form a precipitate. That precipitate can be captured, filtered and weighed — a direct measurement.

The lab manual for such an experiment might give these instructions:

1. Pipette 25 mL of $NaC\ell$ into a volumetric flask. Fill to 100 mL.
2. Pipette 10 mL from the flask to an Erlenmeyer flask. Add 40 mL of distilled water.
3. Add 50 mL of the 0.5 M $AgNO_3$ solution and stir well.
4. Use a scale to find the mass of a piece of filter paper and weighing boat.
5. Slowly pour the resulting solution through a funnel with the paper filter in it. Allow the precipitate to dry and weigh it in the weighing boat.

That's a lot of steps, but they're all there for a reason. Most importantly, **we want to know about the original sample, not the sample after we've done something to it**. That's usually the purpose of the experiment. Imagine a mining operation handing you a sample of something they'd like to work with. They have more of it than the sample they gave you; they want to know what they have. The first step of the $NaC\ell$ lab instructions has us diluting the solution. There's no point reporting the molarity of the solution after *we* diluted it because it's not what *they* have. So why dilute it at all?

The best way to analyze a sample is to make the active chemical in the sample the limiting reagent in some reaction. We dilute the original $NaC\ell$ solution to make its concentration smaller so the amount of $NaC\ell$ we use in the reaction with $AgNO_3$ is smaller. The reaction between $NaC\ell$ and $AgNO_3$ is one-to-one, so as long as we've diluted the $NaC\ell$'s concentration to lower than the 0.5 M for the $AgNO_3$, the amount of precipitate we get at the end is determined by the quantity of $NaC\ell$, not the quantity of $AgNO_3$. We can then use the mass of precipitate to find the concentration of $NaC\ell$.

Many experiments similar to this one use indicators for pH and a process called titration. We achieve titration when we have proportional molar quantities of the reactants according to stoichiometry. In a case like this, all the chemicals are limiting reagents.

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In our calculations, we'll work backwards from the last step (where we know a lot about the chemicals we have) to the beginning (where we don't know enough about the chemicals we're attempting to analyze). The calculations section of the lab manual will have you working through these steps, which have been labelled with the step in the method from the previous page in which you found the information you'll use:

a. Determine the mass of the dry precipitate by subtracting the two masses you measured. [Instruction 4 & 5]
b. Determine the number of moles of AgCℓ in your precipitate using the molecular weight of AgCℓ.
c. Determine the number of moles of NaCℓ in the reaction vessel using the stoichiometry of the reaction.
d. Find the concentration of the 10 mL of solution you pipetted. [Instruction 2]
e. Use the dilution factor from the volumetric flask to find the concentration of the original solution. [Instruction 1]

Steps b and c required the molecular weight of a compound and the reaction equation, and you didn't need to perform the experiment to know either of those things. Other than those steps, we're walking backwards through the experiment, finish to start. This also means that if you're ever unsure which solution to use for some purpose (there were five different volumes/concentrations of solutions containing NaCl during this experiment alone), you can use where you are in the method to help you.

What about preparing 25 mL into 100 mL, then only using 10 mL of it? **We take samples** for two reasons. First, it keeps the quantity low so it's more likely to be the limiting reagent, and also, it's **so that we can do multiple runs of an experiment.** It should always be possible to replicate results. That means we should get results that agree with each other if we do it over, but we need to start from the same place. Using 10 mL out of 100 mL means we can do the same experiment up to 10 times if necessary.

It is sometimes necessary to standardize a solution (find out its actual current concentration) **before it can be used on your sample.** Permanganate cannot be prepared in advance and stored because the solution breaks down over time. It must be standardized so you know what its concentration is on the day of the lab. Often this means creating the same reaction as your main lab, but with the unknown solution swapped for a stock solution of known concentration.

Percent error is allowed to be negative. That just means your experimental value is less than the book/literature/true value.

PRELAB / POSTLAB

Finally, your teacher may have you doing a prelab or postlab along with the lab itself. This is worksheet that is intended to help you with the calculations in the lab. You'll be given data to work with similar to what you might get during the lab, but because it's a worksheet, they can print the correct answer on the sheet. This way, you can check your technique in doing the calculations. After all, you can't check your work in an experiment that easily; there's no printed answer!