

Chem 141 Titration Lab Lecture Notes
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Q: What is the purpose of this experiment?

The purpose of this experiment is to explore two techniques by which we answer the question: How much? One technique we'll be using to answer this is weighing. The other is titration.

Q: What do we have to weigh?

You're going to weigh two different substances, NaOH, sodium hydroxide, on a top loading balance, and KHP, potassium hydrogen phthalate, on an analytical balance.

Q: Two different balances? What's the difference?

The analytical balance is a much more precise instrument, allowing mass determinations to ± 0.0001 g. The top loading balance typically allows masses to be determined to only ± 0.01 g. Thus the analytical balance is 100 times more precise than the top loading balance.

Q: Why do we use two balances with two different precisions?

You need precise determination of the mass of KHP, because weighing is the only method you'll use to quantify the amount of KHP in this experiment. Therefore you want to be able to measure and record the maximum number of significant figures.

It's ok to use the less precise top loading balances to find the approximate weight of the NaOH, since you'll be making a finer determination of the amount of NaOH by titration later in the lab.

Q: But why can't we just use the analytical balance to determine the exact mass of the NaOH?

NaOH is a hygroscopic compound. This means that it easily absorbs water from the air. This means that the mass of any solid "NaOH" sample that you measure will actually be a combination of NaOH and water. Since the fraction of NaOH that you're weighing is unknown, you might as well use the less precise measuring device.

Q: You say we'll use titration to determine the amount of NaOH. What's titration?

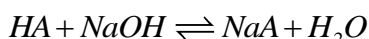
Titration is a way of determining how many moles of a chemical are in a solution phase sample by completely reacting it with another chemical.

Q: What reaction do we do to determine the number of moles of NaOH?

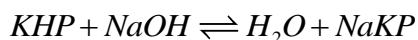
We're reacting KHP with NaOH. The products are water and a salt. This type of reaction is called a neutralization reaction because two caustic compounds, KHP, a mild acid, and NaOH, a strong base, are replaced by water and a weaker base.

Q: How much NaOH does it take to completely react with a sample of KHP?

The answer to this question is based on the balanced reaction for neutralization of an acid by NaOH:



Note that in this reaction there is a 1:1 mole ratio of acid to base. This equation is valid for all acids that contain only one acidic proton. All of the acids that you'll be titrating in today's experiment have just one acidic proton. This means we can write the reaction of KHP with NaOH as:



Q: Why is the 1:1 mole ratio significant?

It tells us that our reaction is complete when the number of moles of NaOH that we add is equal to the number of moles of KHP that are initially present.

Q: How can we tell how many moles of KHP are initially present?

Since you'll have weighed out a sample of KHP, you simply divide the measured mass of the KHP by the molar mass of the KHP:

$$\text{mol KHP} = \frac{\text{mass KHP}}{\text{molar mass KHP}}$$

Q: How do we determine the molar mass of the KHP?

The formula of the KHP is written in the lab manual. All you need to do is multiply the number of each type of atom by its atomic mass (which you can find on the back cover of the lab manual), and then add up the total masses for each element.

For example, perchloric acid, with formula HClO_4 , has a molar mass given by

$$\text{molar mass HClO}_4 = 1 \times m_H + 1 \times m_{Cl} + 4 \times m_O = 1.0079 + 35.453 + (4 \times 15.999) = 100.457 \text{ g / mol}$$

Q: Is there a special name for the point at which we've put equal moles of acid and base into our flask, so that the reaction is complete?

We call this the endpoint of the titration.

Q: How can you tell that you've reached the endpoint?

For some reactions there is a natural color change that can be used to tell when the reaction is complete. However, for our reactions today, no color changes occur. Therefore we have two options. One is to use a device called a pH meter to determine the change in acidity as NaOH is added, and then determine the endpoint graphically. We'll do this later in the semester.

The other option, the one we'll use today, is to add a compound called an indicator, which changes color when the endpoint is reached.

Q: What indicator will we use?

Our indicator of the day is called phenolphthalein. It is clear when a solution is acidic, and turns pink when the solution turns basic. When you reach your endpoint, the next drop of base you add will make your solution basic and the solution will turn pink.

Q: Will it turn bright pink?

Not immediately. When you reach the endpoint, the color of your solution will be the palest pink. A bold pink indicates that you've added too much base.

Q: Once I reach the endpoint, how can I calculate the concentration of the NaOH?

This is easy. You know the number of moles of NaOH, because it's the same as the number of moles of KHP. The concentration in units of molarity (moles/liter of solution) is just given by the number of moles of NaOH divided by the volume of liquid it was contained in:

$$[NaOH(M)] = \frac{n_{NaOH}}{V_{NaOH}} = \frac{n_{KHP}}{V_{NaOH}}$$

Q: How do we measure the volume of the NaOH that we've used?

We use a device called a buret. I'll demonstrate the use of the buret before you begin your experiment.

Q: Phew, that's a lot. So now we're done, right?

Well, no. There's no way to really know how good your answer is, so you'll need to repeat it at least twice. You'll often hear scientists talk of the reproducibility of their results. The term refers in part to the ability of others to reproduce your data, and in part to your ability to reproduce your own results. Redoing the experiment is the minimum confirmation that you have correct results.

Q: Do I report all three values?

No. You'll report the average (or mean) of the three values. You calculate the average by adding the three values together and then dividing by three.

Q: How can you tell how closely the three values agree if you're only reporting the average?

You can tell how close the values are by also reporting the standard deviation, a statistical measure of the precision of a group of measurements. The standard deviation is calculated with the formula:

$$s = \sqrt{\frac{\sum (\bar{M} - M_i)^2}{N - 1}}$$

Where \bar{M} is your average molarity, M_i represents the individual measurements, and N is the total number of molarities you measured.

What this really means is you take the absolute value of the difference between each of your three points and the average, square it, then add the three values up, divide by two, and take the square root.

The smaller s is, the more precise your measurement is.

What does it mean to be precise?

Precision is a measure of the reproducibility of your measurement. This means how close repeated values are to each other. It is also a measure of how finely ruled your measurement device is, since a finely ruled device tends to be more reproducible than a crudely ruled device.

Is precision the same as accuracy?

No! Accuracy is a measure of how close a value is to the correct value. Precision is a measure of how close repeated measurements are to each other.

How can you find out how accurate a measurement is then?

If a problem has been studied before, the values that are published in the literature (literature values) can be used as a measure of the true value. However, true values are usually hard to determine because new research or new techniques can result in an improvement on the published values.

Q: Ok. NOW we're done, right?

Not quite. At this point we've determined the concentration of the solution of NaOH we made, a process called standardization. Now we're going to use our standardized solution to determine the concentration of acid in a commercial bottle of vinegar.

Q: What kind of acid is in vinegar?

Acetic acid, C₂H₃O₂H. (Only the final H is acidic.)

Q: Why do we need to measure the concentration of the acid? It's right there on the bottle, 5%!

One of the common tasks given to chemists is to confirm the claims made by manufacturers, whether they make cigarettes and claim that they're low in tar, or orange juice to make sure that the vitamin C content is what is claimed. For solutions, titration is the method.

Q: Uh, by the way, what does 5% acid mean?

It means that 5% of the mass of the sample is acetic acid?

Q: How do we find the mass percent from the molarity we determine?

First you need to convert the moles of acetic acid to mass of acetic acid, but this is easy, since you just multiply the number of moles by the molar mass:

$$m_{\text{acetic acid}} = n_{\text{acetic acid}} M.M._{\text{acetic acid}}$$

Next you need to determine the total mass of your vinegar sample. To do this you need to know the density of your liquid, but this is given (on the final data sheet, page 12) as 1.005 g/mL. The total mass of liquid is given by the product of the volume and the density.

$$m_{\text{vinegar}} = V_{\text{vinegar}} d_{\text{vinegar}}$$

The mass percent is then given by the mass of the acetic acid over the total mass multiplied by 100:

$$\% \text{ acetic acid} = \frac{m_{\text{acetic acid}}}{m_{\text{total}}} \times 100\%$$

Q: You reported the density of the vinegar to three decimal places. That means our volume measurement has to be just as precise. How can we measure volume that precisely?

We use a device called a volumetric pipette, which I'll demonstrate for you at the end of the lab lecture.

Q: Is there anything different we're going to do in our second titration?

You'll have to do a 10-fold dilution of your NaOH solution before using it in the titration.

Q: Why?

Your NaOH solution is much more concentrated than the vinegar. You'd use so little NaOH that your results would be very imprecise. Decreasing the concentration of the NaOH increases the volume you'll use, and increases the precision of the measurement.

Q: How do we do the dilution?

You'll transfer about 30 mL of your NaOH stock solution into a clean beaker. Transfer 10 mL from the beaker using a volumetric pipette into a 100 mL volumetric flask. Then fill with distilled (RO) water until the meniscus touches the line in the neck.

Q: How do we calculate the new concentration?

There's a rule called the lever rule for dilution calculations:

$$M_{initial} V_{initial} = M_{final} V_{final}$$

If we solve this for the final molarity, it becomes

$$M_{final} = \frac{V_{initial}}{V_{final}} M_{initial}$$

Q: How many times to we have to repeat this measurement?

Just twice.

Q: In the lab manual it says to calculate the percent error. How is that defined?

$$\% \text{ error} = \left| \frac{\text{Experimental value} - \text{Literature value}}{\text{Literature value}} \right| \times 100\%$$

Q: But we don't know any literature value.

True. In this case, you're reporting the % difference between your measured value, and the value claimed on the label by the manufacturer. Use the same formula above, but substitute the value on the label for the literature value.

Two quick notes. First, be as careful about how many significant figures you use in this calculation as you would in any other calculation based on experimental numbers. Second, be aware that there are two possible reasons your value could deviate from the manufacturer's value – you could be wrong or they could be wrong. You should think about how to distinguish between the two possibilities.

Q: Do you have any hints?

Yes. Now I'll show you how to use a buret and read volumes from one, and how to use a volumetric pipette.

The analytical balance you'll be using is a very sensitive device. To ensure maximum precision, make sure that you don't lean on the counter while weighing, and make sure that after you place either your flask or the KHP container on the weighing pan, that you close the doors on the scale.

When you weigh, it isn't enough to record the mass of the KHP that you use. You also need to record the information that you use to determine this. So if you choose to weigh your flask, then add KHP and weigh again, you need to record both the mass of the empty flask and the mass of the flask with KHP in your notebooks as well as the difference.

Q: What safety concerns do we need to be aware of?

Make sure that you have safety glasses on at all times. NaOH is a very corrosive substance. Make sure that you handle both the solid and the solution carefully. KHP is caustic as well.

Q: What do we need to turn in?

Today you need to turn in the carbon copies of your lab notebook pages and your completed prelab assignment. The notebook pages should be a reasonably complete record of what you did, what you observed and the data you collected.

Next week you'll turn in the data report sheets on pages 11 and 12, sample calculations for molarity of the NaOH solution, for the concentration of the diluted NaOH solution and for the mass % of acetic acid (see page 7), and a brief discussion of the results including the precision, and accuracy of your results, and any sources of error in the procedure as you carried it out.

Honor Stuff: You may work together with your lab partner on all of the calculations that will be turned in on the two data report sheets. The brief discussion (part iv, on page 8) must be your individual effort. Don't forget to sign the pledge on what you turn in.

Next week: Qualitative Analysis of Reactions – Prelab and Notebook prep due at the beginning of lab.