

Performing a Titration

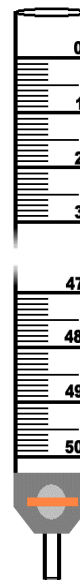
Supplemental Lab Handout
CHM2260

The term *titration* refers to a technique in which a solution of known concentration is carefully added to a measured amount of another solution of unknown concentration. The purpose of the titration is usually to determine the concentration of the unknown solution. The solution added is known as the *titrant*; the substance with which the titrant reacts is known as the *analyte*. The *end point* of a titration is reached when enough titrant has been added to react with all of the analyte.

Equipment for Titrations

When carrying out a titration the titrant is usually dispensed from a *buret* (see illustration at right). A buret is a long cylindrical tube with a stopcock at one end. Solutions are dispensed from the buret by opening and closing the stopcock. Stopcocks are typically open when vertical and closed when horizontal, so be sure the stopcock is closed before filling a buret with titrant. The volume of titrant dispensed from a buret is always determined as the difference between the starting and final volumes.

Most burets hold 50 mL of solution. Although you can refill a buret halfway through a titration, this is not a recommended practice. It is therefore a good idea to refill a buret at the beginning of each titration. Do not, under any circumstances, allow the volume of titrant to fall below the last mark on the buret. There is no way to measure or even estimate how much titrant you have added once this is allowed to happen.



All burets are graduated to nearest 0.1 mL. Since it is typical to estimate one more digit that you can read of a scale, always record buret volumes to two places past the decimal. Consider, for example, the buret shown to the left. The bottom of the meniscus lies below 7.2 but above 7.3; therefore the volume lies somewhere between 7.2 and 7.3 mL. To properly record the volume, estimate the last digit and record the volume as 7.23 or 7.24.

Types of Titrations

Titration falls into several categories. The most common titrations are acid-base titrations, in which an unknown acid is titrated with a base or vice-versa. Oxidation-reduction titrations are also rather common, in which a strong oxidizing agent such as $\text{S}_2\text{O}_8^{2-}$ (persulfate) or MnO_4^- (permanganate) is used as a titrant.

Indicators for Titrations

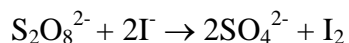
All titrations require some sort of indicator to determine the endpoint of the titrations. The purpose of an indicator is to provide some visual clue that the reaction is complete. Acid-base titrations typically employ an indicator that changes color with the acidity of the solution. Phenolphthalein, for example, is colorless when acidic and bright pink when basic and is commonly used when titrating an acid with a base. In a typical acid-base titration, base is added to an unknown acid solution until enough is added to react with all the acid. Once all the acid is neutralized, a slight excess of base changes the color from colorless to bright pink.

The MnO_4^- (permanganate) ion is commonly used as a titrant in oxidation-reduction titrations. This ion itself is intensely colored, but it is reduced to the nearly colorless Mn^{2+} ion during the course of the titration. It therefore serves as a built-in indicator; the titration is complete when there is nothing left to reduce the excess MnO_4^- and the solution takes on the purple color of this ion.

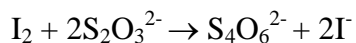
Soluble starch is often used as an indicator in titrations involving iodine because it forms an intensely colored blue-black complex with molecular iodine. As titrant is added, the iodine is reduced to the iodide ion. Therefore the endpoint of the titration is reached when the color of the solution turns from blue-black to colorless.

Titration Performed in this Lab

We will perform two titrations in this lab. The first of these is an oxidation-reduction titration. You will synthesize potassium persulfate (a strong oxidizing agent) by electrochemical methods. This is then reacted with an excess potassium iodide, which is oxidized to molecular iodine.

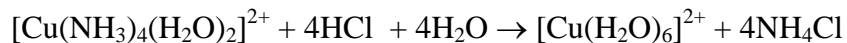


The molecular iodine is then titrated with sodium thiosulfate (a reducing agent) and reduced back to iodide.



This reaction employs starch as an indicator, which forms a blue-black complex ion with starch as previously discussed. Therefore, the sodium thiosulfate solution is added until the solution changes from blue-black to colorless. From the molarity and volume of sodium thiosulfate added, the number of moles of thiosulfate added can be determined. From the stoichiometry of the reactions given above, this is then related to the number of moles of iodine and present and ultimately to the number of moles of persulfate in the sample.

The second titration we will employ is an acid-base titration of a copper complex containing ammonia, $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4$, perhaps better written $[\text{Cu}(\text{NH}_3)_4(\text{H}_2\text{O})_2]\text{SO}_4$. This reaction employs 1.0 M hydrochloric acid as a titrant. This reacts with the ammonia in the complex ion, converting it into ammonium ion. The ammonium ion cannot coordinate, and the open coordination sites are replaced with water molecules.



The reaction that is really taking place here is the acid-base reaction between HCl and NH_3 .



Here the change in color of the complex ion serves as an indicator. While the $[\text{Cu}(\text{NH}_3)_4(\text{H}_2\text{O})_2]^{2+}$ ion deep blue-violet in color, the color of the $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$ ion is pale blue. The titration is complicated by the fact that in weakly basic solutions, copper hydroxide is formed, imparting cloudiness to the solution. Titration must therefore be continued until a clear, pale-blue solution is obtained.

Some Final Notes on Titrations (now pay attention)

1. Wash your buret with water and a few mL of titrant before beginning the titration.
2. Do not try to fill the buret to the zero line. Is that 0 mL, 0.0 mL, or 0.00 mL? No way to tell! It is better to start somewhere between 0 and 1.
3. Be sure to read your buret to the correct number of significant figures. When you record a buret volume you *read* the tenths place and *estimate* the hundredths place. That's *two* places past the decimal point! Once more, you should record your volumes to *two* places past the decimal point. (Are you getting the feeling you're going to have points taken off if you don't pay attention to this? Good.)
4. While adding the titrant, it is important to swirl the flask to keep the contents mixed. You can add the titrant rapidly during the initial stages of the titration, but you should slow down to a *dropwise addition* as you approach the endpoint. How can you tell you are approaching the endpoint? Look for a local color change around each drop of titrant that disappears as the flask is swirled. This tells you that you are getting close to the endpoint.
5. Never, ever, ever allow the volume in the buret to drop to below the 50-mL mark. Once that happens, your titration is ruined. You CANNOT estimate how much you have added once this happens. For this reason, it is recommended that you refill your buret at the start of each titration.

That's all!