Volumetric Analysis

Volumetric analysis is a widely-used quantitative analytical method. As the name implies, this method involves the

measurement of volume of a solution of known concentration which is used to determine the concentration of the analyte.

1. Prepare a solution from an accurately weighed sample to +/- 0.0001 g of the material to be analyzed.
2. Choose a substance that will react rapidly and completely with the analyte and prepare a standard solution of this substance. The concentration of the standard solution should be known to +/- 0.0001 M.
3. Place the standard solution in a buret and add it slowly to the unknown. This process is called titration and the solution in the buret is called the titrant. Continue the titration until the reaction is complete; that is, until the amount of reactant added is exactly the amount required to react with all the constituent being analyzed. This point is called the equivalence point, and can be detected by adding an indicator to the unknown solution before beginning the titration. An indicator is a substance that gives a color change at or near the equivalence point. The point at which the color change occurs is the end point of the titration.
4. Measure the exact volume of standard solution required from buret readings before and after the titration. Since the molarity of the standard solution is known, the number of

moles of titrant can be calculated. From a knowledge of the equation for the reaction, the number of moles of constituent present in the sample can also be calculated.

1. The most accurate and convenient way of preparing a standard solution is to weigh the reagent, dissolve it, and dilute the solution to a definite volume in a volumetric flask. This method can only be used if the reagent is a primary standard.
2. In order for a reagent to be a primary standard, it must be obtainable in pure form (generally at least 99.98% pure), stable both in pure form and in solution, easy to dry and keep dry, and soluble in a suitable solvent.
3. Many useful reagents do not meet those requirements, so the reagent is dissolved and diluted approximately to the concentration desired. The solution is then standardized by titrating it against a primary solution. This standardized soluton is caled a secondary standard.

1. Clean the buret before use and rinse wih water. If any drops of water collect on the walls, the buret is not clean. Once the buret is clean, rinse it with the titrant solution before filling it. Pour about 5 mL of the titrant into the buret and, holding the buret almost horizontally, rotate it slowly so that the titrant cleans the entire buret. Do this three times.

2. Place the buret in a buret clamp attached to a large ring stand. Using a funnel, fill the buret with titrant to a level above the zero mark. Place a beaker under the buret and open the stopcock for a few seconds to remove all air from the tip and fill it. The top of the solution should now be below the zero mark.
3. Read the buret to +/- 0.01 mL with the meniscus level with the eye to minimize parallax (see Fig. 1). Parallax is the varying of the apparent position of the meniscus due to eye level. If you are looking down on the meniscus, the reading will be low. If you are looking up at it, the reading will be high.

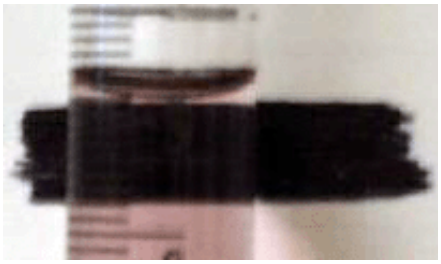


Figure 1. Place a black strip behind the buret to make the meniscus easier to see and the volume easier to read.

4. Place the solution that is to be titrated in an Erlenmeyer flask and add 3-5 drops of the appropriate indicator. Position the flask under he buret.
5. Add the titrant slowly from the buret while swirling the

contents of the flask to assure adequate mixing (see Figure 2). As the end point is approached, the titrant must be added very slowly - a drop at a time. Usually there is an indication as the end point is approached. If the end point is a color change, the change is produced momentarily where the reagent drops into the solution, but fades with stirring into the solution. This fading occurs more slowly as the endpoint is approached.

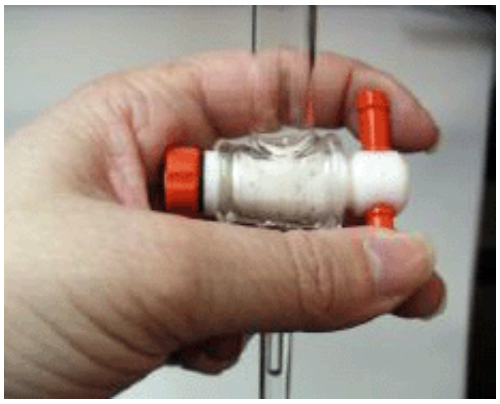


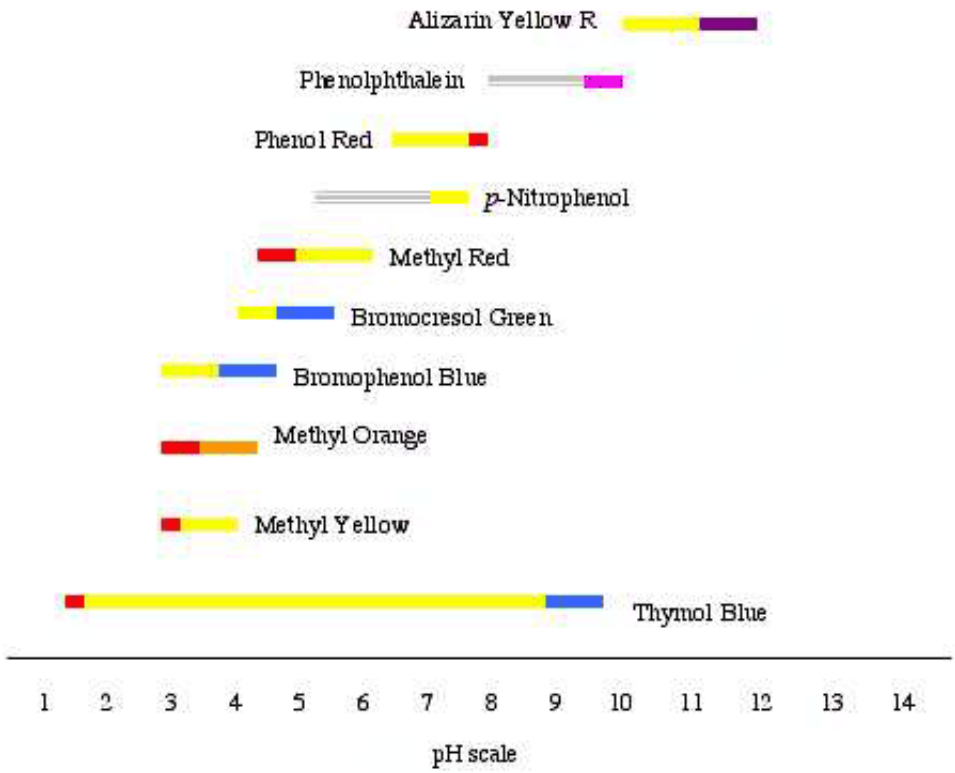
Figure 2. The proper handling of the stopcock of the buret. This allows for maximum control of the rate at which the titrant is added.

6. When the end point has been reached, allow the solution to sit for 10 seconds so the liquid in the buret can settle, then read the buret. Subtract the initial buret reading from the final reading to obtain the volume of titrant used.

Indicators are used to determine the end point of the titration. An indicator is used in acid-base and oxidation-

reduction titrations. The color change of the indicator should be near the equivalence point of the reaction. The following charts show commonly used indicators and their color changes.

Acid-Base Indicators



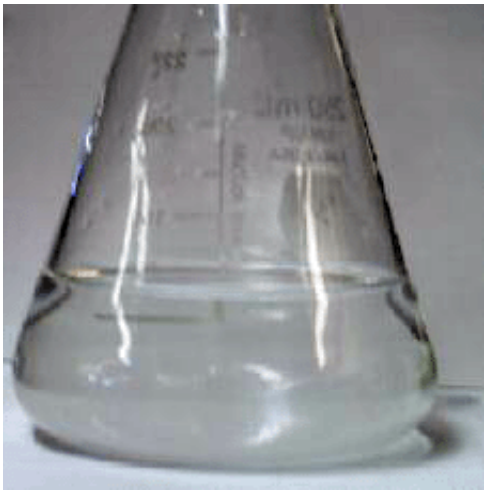
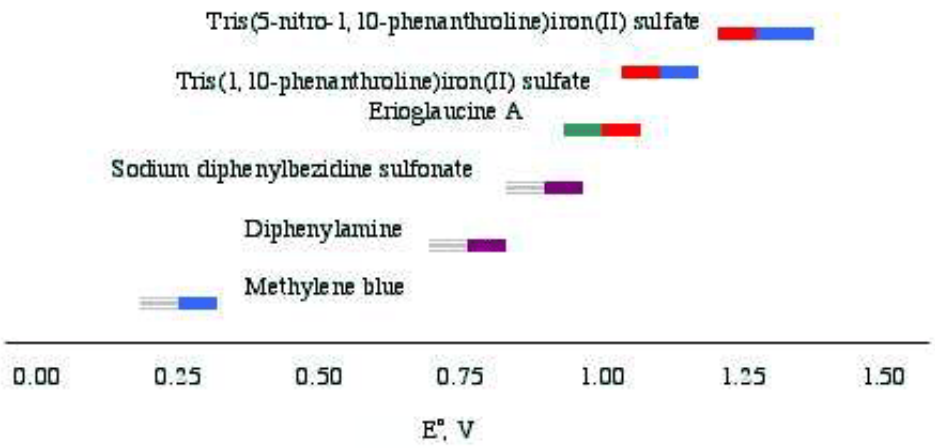


Figure 3. A solution containing the indicator phenolphthalein before titration.

Figure 4. A solution containing phenolphthalein after titration.

Oxidation-Reduction Indicators



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