Advice & Counsel

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Titrating, Part II

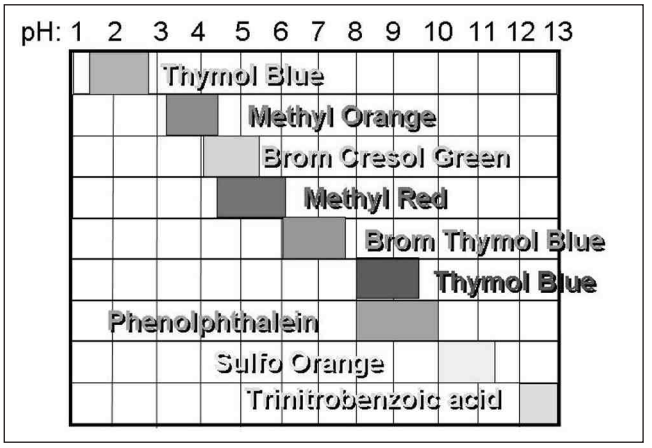

Fig. 1—Common indicators and pH range.

Common Indicator Color Changes:

Indicator	Lower pH	Higher pH
Thymol Blue:	Red	Yellow
Methyl Orange:	Red	Orange
Brom Cresol Green:	Yellow	Blue/Green
Methyl Red:	Red	Yellow
Brom Thymol Blue:	Yellow	Blue
Thymol Blue:	Yellow	Blue
Phenolphthalein:	Clear	Pink
Sulfo Orange:	Yellow	Orange
Trin-nitro benzoic acid:	Clear	Orange

Fig. 2—Color change indicator.

Following up on the topic we began last month:

Our sample was prepared for titration last month, but we did not discuss the types of titrations conducted. They basically fall into:

A. Acid-base
B. Oxidation-Reduction
C. Chelation

This month, let's stick to acid-base titrations. These are the easiest to conduct in most cases (a boric acid determination is a big exception and is very difficult to do). In an acid-base titration, we will react a known volume of either an acid or a base with the opposite reagent at a known concentration. For example, what if we wanted to know the caustic content of our alkaline cleaner? We would take a small known volume (let's say 2 mL) and transfer it into a flask. De-ionized or distilled water is then typically added to make for a more manageable volume. By reacting this known

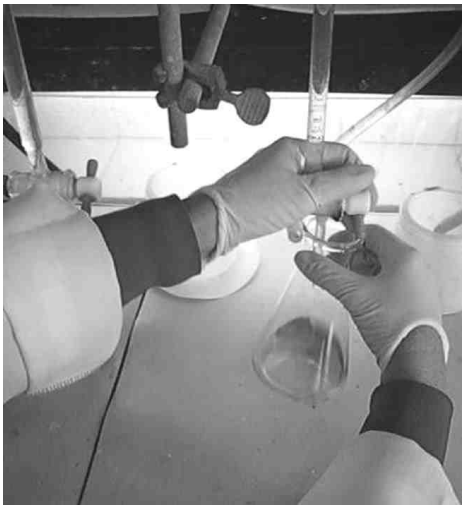

Fig. 3—Conducting the titration.

Titration Procedure:

- Ideally Endpoint should be achieved within one drop or less than 1 drop of reagent from buret
- Partial drop can be added by (gently) touching side of flask to drop hanging from tip of buret, then mixing into liquid in flask

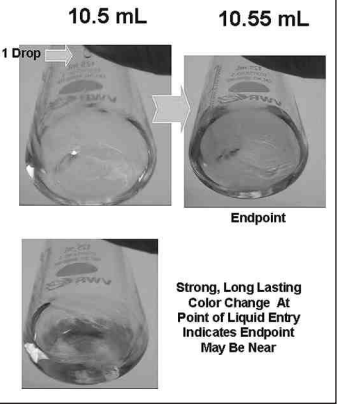

Fig. 4—Titration procedure.

volume of cleaner with an acid of known strength, we will be able to use a simple math equation to determine the caustic content of the cleaner:

Va x Na = Vb x Nb

This equation states that the volume of an acid times its normality is equal to the volume of a base times its normality. We took a 2 mL sample of our "base" which was the cleaner, so Vb = 2 mL. We don't know the strength of the cleaner, so Nb is unknown. By titrating with 0.1 normal sulfuric acid and measuring the volume necessary for neutralization, we will have Na = 0.1 and Va = to the titration volume. If, for example, the titration is 15 mL, the equation becomes:

15 x 0.1 = 2 Nb

Nb = 1.5/2 = 0.75

By definition, a 1 normal sodium hydroxide solution contains 40 g/L of NaOH. Our titration determined that our cleaner contains 0.75 normal or 40 x 0.75 = 30 g/L of sodium hydroxide.

So how do we know when the acid has neutralized the caustic in the flask? The most common way to do this is to use a chemical "indicator." Indicators are weak acid or weak base compounds that change color depending upon the pH level of the liquid in which they are injected. There are dozens of indicators and each responds to a certain pH range. The analytical procedure you will be following will specify which indicator to use. It is important not to substitute an alternate unless you know your chemistry. Figure 1 shows some common indicators and the pH range they respond to. Figure 2 shows the color change when the pH changes from a higher value to a lower value.

Let's go back to the titration. We add just a few drops of the indicator specified in the procedure. Don't add too much, as your sample may turn cloudy and color changes will be hard to see. Typically 3–4 drops is enough.

Fill the buret and "zero" the volume. We could begin our titration at any level of volume in the buret by writing down the initial reading, but this is not considered good practice (unless you are an "old pro").

Once the buret is zeroed, we can begin the titration. The titration is conducted by swirling the flask with your right hand and grasping the stopcock properly (fig. 3) and slowly adding a few drops of reagent from the buret into the flask. Note any color change at the point of entry. This will give you an idea of what the color change will be at the end of the titration. If there is no color change, you can add the reagent at a faster rate until you see a color change at the point of entry (fig. 4). As you continue to add reagent, the area of the color change spot in the flask will broaden. This is an indication that you are near "the endpoint." At the endpoint the color of the liquid in the flask will very quickly (in most cases) change, and you need to immediately stop adding reagent. With a little practice, a good chemist can make the entire volume in the flask change color by the addition of a single drop (sometimes less than a drop) of reagent. A partial drop can be added by letting the drop hang from the buret tip and touching the inside of the flask to the drop.

A good way to practice is to make or purchase a 0.1 N solution of sodium hydroxide and 0.1N solution of sulfuric acid and some phenolphthalein indicator (they are readily available from most any chemical/plating supply house). Pipet 10 mL of the acid into a flask, add about 2 mL of DI water and a few drops of phenolphthalein. Then titrate the contents with the 0.1 normal sodium hydroxide until a pink color flashes on. If you are good, the titration will be exactly 10.0 mL. ***P&SF***