7. Using a pH Electrode for an Acid-Base Titration

Green Profile
See Section 0

In this experiment you will use a pH electrode to follow the course of an acid-base titration. You will observe how pH changes slowly during most of the reaction and rapidly near the equivalence point. You will compute the first and second derivatives of the titration curve to locate the end point. From the masses of the unknown acid and the unknown base and the moles of titrant, you can calculate the molecular mass of each of the unknowns. Section 11-5 of the textbook provides background for this experiment.

Reagents

Standard 0.1 M NaOH and standard 0.1 M HCl: From storeroom

Bromocresol green indicator: Dissolve 0.100 g of the indicator in 14.3 mL 0.010 0 M NaOH and add 225 mL H_2O.

Phenolphthalein indicator: Dissolve 50 mg of the indicator in 50 mL of ethanol and add 50 mL H_2O.

pH calibration buffers: pH 7 and pH 4. Use commercial standards.

Unknowns: Unknowns should be stored in a desiccator by your instructor.

Suggested acid unknowns: potassium hydrogen phthalate (Table 10-4, FM 204.22), 2-(*N*-morpholino)ethanesulfonic acid (MES, Table 8-2, FM 195.24), imidazole hydrochloride (Table 8-2, FM 104.54, hygroscopic), potassium hydrogen iodate (Table 10-4, FM 389.91).

Suggested base unknowns: tris (Table 10-4, FM 121.14), imidazole (FM 68.08), disodium hydrogen phosphate (Na_2HPO_4, FM 141.96), sodium glycinate (may be found in chemical catalogs as glycine, sodium salt hydrate, $H_2NCH_2CO_2Na{\cdot}xH_2O$, FM 97.05 + x(18.015)). For sodium glycinate, one objective of the titration is to find the number of waters of hydration from the molecular mass.

Procedure

1. Your instructor will recommend a mass of each unknown (5–8 mmol) for you to

weigh accurately and dissolve in distilled water in a 250-mL volumetric flask. Dilute to the mark and mix well.

2. Following instructions for your particular pH meter, calibrate a meter and glass electrode, using buffers with pH values near 7 and 4. Rinse the electrode well with distilled water and blot it dry with a tissue before immersing in any new solution.

3. The first titration is intended to be rough, so that you will know the approximate end point in the next titration. For the rough titration, pipet 25.0 mL of unknown into a 125-mL flask. When you titrate the unknown acid, add 3 drops of phenolphthalein indicator and titrate with standard 0.1 M NaOH to the pink end point, using a 50-mL buret. When titrating the unknown base, add 3 drops of bromocresol green indicator and titrate with standard 0.1 M HCl to the green end point. Add 0.5 mL of titrant at a time so that you can estimate the equivalence volume to within 0.5 mL. Near the end point, the indicator temporarily changes color as titrant is added. If you recognize this, you can slow down the rate of addition and estimate the end point to within a few drops.

4. Now comes the careful titration. Pipet 100.0 mL of one of the unknown solutions into a 250-mL beaker containing a magnetic stirring bar. Position the electrode in the liquid so that the stirring bar will not strike the electrode. If a combination electrode is used, the small hole near the bottom on the side must be immersed in the solution. This hole is the salt bridge to the reference electrode. Allow the electrode to equilibrate for 1 min with stirring and record the pH.

5. Add 1 drop of indicator and begin the titration. The equivalence volume will be four times greater than it was in step 3. Add ~1.5-mL aliquots of titrant and record the exact volume, the pH, and the color 30 s after each addition. When you are within 2 mL of the equivalence point, add titrant in 2-drop increments. Since the pH changes rapidly near the equivalence point you need to be sure to have a sufficient number of volume, pH data points. When you are within 1 mL, add titrant in 1-drop increments. Continue with 1-drop increments until you are 0.5 mL past the equivalence point. The equivalence point has the most rapid change in pH. Add five more 1.5-mL aliquots of titrant and record the pH after each.

6. Repeat steps 4 and 5 for the other unknown.

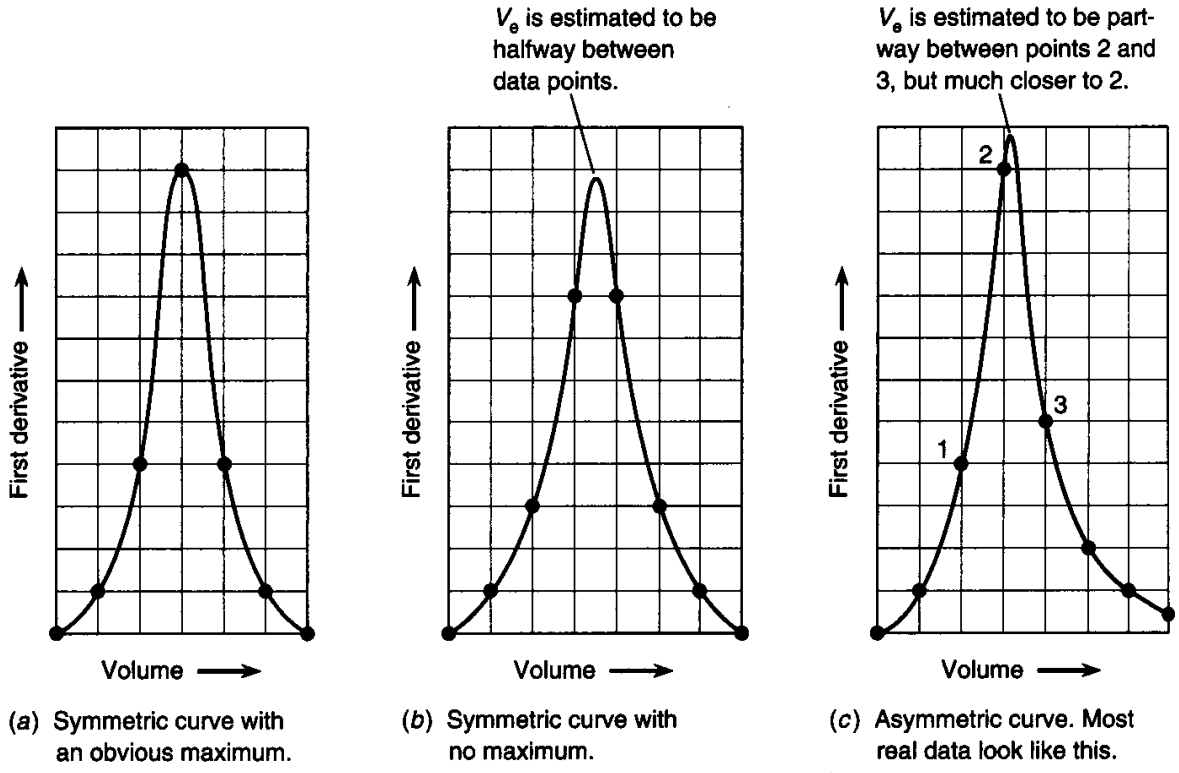

Figure 1. Locating the maximum position of the first derivative of a titration curve.

Data Analysis for Unknown Acid and Unknown Base

1. Construct a graph of pH versus titrant volume. Mark on your graph where the indicator color change(s) was observed.
2. Following the example in Figures 11-5 and 11-6 of the textbook, compute the first derivative (the slope, $\Delta pH/\Delta V$) for each data point within ±1 mL of the equivalence volume. From your graph, estimate the equivalence volume as accurately as you can, as shown in Figure 1.
3. Following the example in Figure 11-6, compute the second derivative (the slope of the slope, $\Delta(\text{slope})/\Delta V$). Prepare a graph like Figure 11-7 in the textbook and locate the equivalence volume as accurately as you can.
4. Go back to your graph from step 1 and mark where the indicator color changes were observed. Compare the indicator end point to the end point estimated from the first and second derivatives.
5. From the equivalence volume and the mass of unknown, calculate the molecular mass of each of the unknowns.