Experiment 9 – Volumetric Analyses (Titrations)

Pre-Lab Hints

1. The analyte is the substance or solution being analyzed.
   The indicator always goes in the flask, not the burette.
2. Read the first footnote at the beginning of the Introduction.
   Also, read the boldface print near the end of the Introduction.
3. See the paragraph in the Introduction with endpoint and stoichiometric point in boldface.
4. a. Review Technique 2 (Cleaning Glassware) and step 1 of Technique 16B (Pipetting a Liquid) in the Laboratory Techniques section near the front of your lab manual.
   b. Review boldface in step 1 of Technique 16C (Titrating a Liquid).
   Read step A5 of the Experimental Procedure also.
5. Moles acid = moles NaOH = Molarity (mol/L) times volume (L) of NaOH solution.
   Multiply moles of acid times its molar mass (g/mol) to get its mass in grams.
6. a. Show all of your calculations. Include all of the units and count the significant digits.
   Line A3 = mass ÷ molar mass = (Line A1) ÷ 204.22 g/mol
   Line A6 = final volume – initial volume = (Line A5) − (Line A4)
   Line A7 = moles ÷ volume = (Line A3) ÷ [(Line A6) × (0.001 L/ml)]
   Line B4 = final volume – initial volume = (Line B3) − (Line B2)
   Line B5 = Line A7 (same NaOH concentration)
   Line B6 = molarity (mol/L) × volume (L) = Line B5 × [(Line B4) × (0.001 L/ml)]
   Line B7 = moles ÷ volume = (Line B6) ÷ [(Line B1) × (0.001 L/ml)]
   b. Skip.

Procedure Notes

- At the top of your report sheet in part A, use (1.00 mol/L)(V) = (0.15 mol/L)(0.500 L) to find the volume of 1.00 M NaOH solution needed. Convert it to mL.
- In the second space at the top of your report sheet, find the moles of NaOH first, which are equal to (0.15 mol/L) times (0.015 L). The moles of NaOH and KHP are equal at the stoichiometric point. Convert those moles into the mass of KHP (204.44 g/mol).
- Skip step 1 of part A. Dry solid acid (KHP) is stored in the desiccator. Promptly return KHP container to desiccator after use. Do not ever leave cap off of container after use.
- Stir the solution to dissolve the solid KHP completely before beginning the titration.
- Read technique 16 (pages 28-32) thoroughly. Pay particular attention to the photos. You will need to accurately operate both a buret and a pipet in this experiment.
- Note that the buret reads **0 ml at the top**, and 50 ml at the bottom, so that the amount dispensed increases as the liquid's surface (meniscus) becomes lower.
- Read volume measurement to nearest 0.05 ml at the bottom of the meniscus.
- When cleaning the buret, be sure to allow both rinse water and titrant to flow through the buret tip, and that there are no air bubbles trapped in the tip.
- Titrant may initially be dispensed in 1-2 ml increments. When color change begins to persist, dispense titrant slowly. Ideally, dispense dropwise near endpoint.

Procedure Notes (continued)

- Do not overshoot the endpoint. If extra titrant was dispensed beyond the endpoint, there will be no way to determine the actual endpoint, and your calculations will be inaccurate. Also, there will be no indication that extra titrant was dispensed after the initial color change. Ideally, dispense dropwise near endpoint.
- For the acid sample in part B, initially draw liquid above the calibration line on the pipet, then use your index finger to drain excess liquid until the bottom of the meniscus is at the calibration line. Dispense by gravity flow only. Do not force remaining drop from tip, because the pipet is calibrated to include this drop.
- Moles OH^{-1} = (mol/L of NaOH) × (L of NaOH) for both parts of the experiment.
- Moles H^{+1} = moles OH^{-1} at the stoichiometric point for both parts of the experiment.
- Moles H^{+1} = (g of KHP) ÷ (g/mol of KHP) for part A of the experiment.
- Use (mol/L of H^{+1}) = Moles H^{+1} ÷ (0.0250 L of H^{+1}) for part B of the experiment.
- After completing the experiment, rinse the buret, and place it on the stand, upside-down with the stop-cock open.
- Also after the experiment, label the plastic bottle as NaOH, and include the concentration.
- Skip the standard deviation steps on the report sheet.
- Provide the balanced neutralization reaction where indicated at top of page in part B.
- **Provide all calculations on a separate sheet of paper.**

Lab Questions

1. Suppose you dispense titrant until a very, very faint pink color persists and does not disappear. Explain whether or not more titrant is needed, and how you would proceed.

2. An air bubble is inadvertently left in the NaOH buret tip prior to a titration, and it exits during the titration. Explain how this affects the volume measurement and concentration calculation for NaOH in part A.

3. Suppose some titrant liquid clings to the wall of the buret, and does not drain down during the titration. Explain how this affects the level of the meniscus and the final volume measurement.

4. Suppose one group left the cap off the container of slightly hygroscopic KHP before your group obtained it. Explain how and why your calculations of KHP, NaOH, and acid moles may be different from the actual moles of each.

5. Suppose you rinse the walls of the analyte flask with boiled, deionized water during the titration as directed in step A6, but you use a lot of water. Explain if, and how, this affects your moles of acid and base in the flask, and your concentration calculations.