Fundamentals of Analytical Chemistry

Chapter 16
Applications of Neutralization Titrations

Standard Acid Solutions

- No strong, primary standard acids
  - Constant boiling HCl solution (780 torr ≥ P ≥ 670 torr)
  - mass: molar boiling HCl(1) = 163.273 x 0.02039

- More commonly, standardization vs. a primary standard base
  - Sodium carbonate (Na₂CO₃)
  - Na₂CO₃ + 2HCl → 2NaCl + H₂CO₃
  - H₂CO₃ → H₂O + CO₂, CO₂ can be removed by boiling

- TRIS (or THAM)
  - Tri(hydroxymethyl)aminomethane
  - (HOCH₂)₃CNH₂ + HCl → (HOCH₂)₃CNH₂⁺ + Cl⁻

Standard Base Solutions

- Most common is NaOH
  - Must protect from atmospheric CO₂
  - CO₂ + 2OH⁻ → CO₃²⁻ + H₂O
  - Only affect endpoint when that endpoint is basic
    - Carbonate error
  - Solid hydroxides always have carbonate
    - High concentration of hydroxide will precipitate the carbonate
  - Standardization with primary standard potassium hydrogen phthalate

Elemental Analysis

- Sulfur
  - Burned in air, then bubbled through hydrogen peroxide
    1) S + O₂ → SO₂
    2) SO₂ + H₂O₂ → H₂SO₄

  Titrate with standard NaOH
  \[ \text{H}_₂\text{SO}_₄ + 2\text{NaOH} \rightarrow \text{Na}_₂\text{SO}_₄ + 2\text{H}_₂\text{O} \]

Kjeldahl Analysis

- Determination of Nitrogen
  - Nitrogen containing compound is digested, nitrogen is converted to ammonia which is then distilled and titrated with a standard acid or back-titrated with a standard acid and base.
  - If nitrogen content is known (or approximated) can determine amount of another analyte

Homework

- 19-31, 32, 33, 35, 39, 40, 48
Problem 16-38

- A 0.9092 g sample of wheat flour was analyzed by the Kjeldahl procedure. The ammonia formed was distilled into 50.00 mL of 0.05063 M HCl; a 7.46 mL back titration with 0.04917 M NaOH was required. Calculate the percent protein in the flour. (Gravimetric Factor for this protein wrt wheat is 5.70)

Carbonate/Hydroxide Mixtures

- Pure NaOH
  - $\text{Vol BG} = \text{Vol P}$

- Pure Na$_2$CO$_3$
  - $\text{Vol BG} = 2 \times \text{Vol P}$

Kjeldahl Analysis

- Can calculate moles of nitrogen originally present
  - moles N = moles of acid consumed

- Relate to mass of analyte
  - If formula of compound is known, compare moles of nitrogen to moles of compound
  - If %N is known, use %N to calculate mass of analyte
    - If “gravimetric factor” is known, multiply by the gravimetric factor to get mass of analyte

Carbonate/Hydroxide Mixtures

- Pure NaHCO$_3$
  - $\text{Vol P} = 0$
Carbonate/Hydroxide Mixtures

- Mixture NaOH and Na$_2$CO$_3$
  - Vol P < Vol BG < Vol 2P

Carbonate/Hydroxide Mixtures

- Mixture Na$_2$CO$_3$ + NaHCO$_3$
  - Vol BG > 2 x Vol P

Carbonate/Hydroxide Mixtures

- Quantitative Determinations
  - Determine what type of mixture (if any!)
  - Find the part of the curve due to the carbonate that is unaffected by the presence of the impurity
    - Calculate moles of carbonate
  - Find total moles from the part of the curve with the impurity
    - Total moles = moles carbonate + moles of impurity
  - We know moles of carbonate from previous!