EXPERIMENT 3 Quantitative Analysis of Fe(III)-Oxalate Complex

DR. MIOY T. HUYNH CHEMISTRY 136L YALE UNIVERSITY FALL 2018

SPECTROPHOTOMETRY: A QUANTITATIVE TECHNIQUE

Light absorption and color of things



If a chemical species is **RED**, it will strongly absorb **GREEN** light.

It may absorb other colors too.



The experimental quantity of interest is Absorbance (A)

$$A = -\log\left[\frac{I_t}{I_0}\right]$$

where, I_0 = amount of light going in I_t = amount of light coming out after absorption

> Measure I_0 using a BLANK Measure I_t using the SAMPLE

BEER-LAMBERT LAW

 $A = \varepsilon c l$

A = absorbance (dimensionless) c = concentration (mol·L⁻¹)

l = pathlength (cm)

 ε = molar absorptivity (mol⁻¹·L·cm⁻¹)

SPECTROPHOTOMETRY: THE BASIC IDEAS Convert the species of interested to

another intensely colored species.

PART 1. Spectrophotometry of Fe(II)

Fe²⁺ (aq) + 3 o-phen (aq) \rightarrow [Fe(o-phen)₃]²⁺ (aq) COLORLESS COLORLESS

To make sure <u>all</u> Fe is in 2+ state, we use <u>hydroxylamine</u>. To adjust <u>pH</u> to the optimal value, we use sodium acetate.





Prepare several calibrating solutions containing known concentrations of the colored complex

Measure absorbances

Plot calibration graph $\rightarrow \varepsilon$ = slope



concentration (mol/L)

PART 2. Mass % of Fe in Fe(III)-oxalate complex

Take a known mass of the complex from Expt. 1 Reduce all the Fe(III) into Fe(II) Adjust the pH Complex with o-phen Measure absorbance Use the calibration curve to figure out [Fe] \rightarrow mass % PART 3. Mass % of oxalate in the same complex

 $5 C_2 O_4^{2-} (aq) + 2 MnO_4^{-} (aq) + 16 H^+ (aq) \rightarrow 2 Mn^{2+} (aq) + 10 CO_2 (g) + 8 H_2 O (l)$

Take another known mass. Dissolve in water. Add H_2SO_4 . Titrate with MnO_4^- of known molarity.