02
FE(III)-OXALATE COMPLEX

QUANTITATIVE ANALYSIS

CHEMISTRY 136L // FALL 2019

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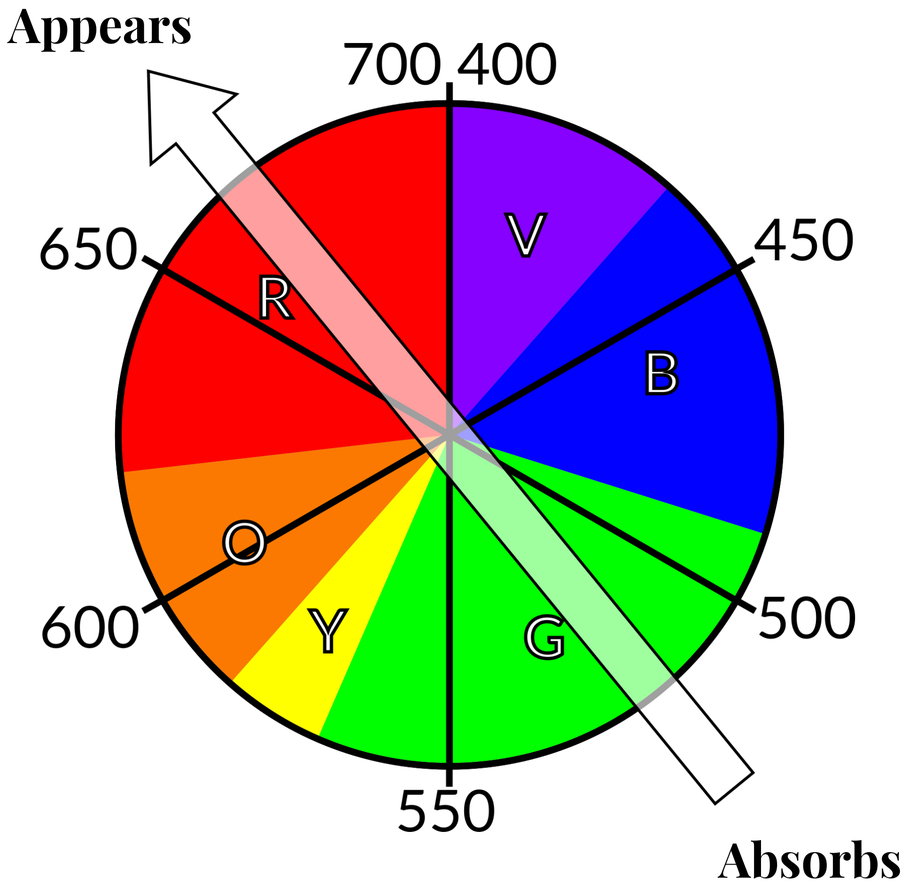
SPECTROPHOTOMETRY

A quantitative technique



Light absorption and the color of things

Complimentary Colors:

RED / **GREEN**

ORANGE / **BLUE**

YELLOW / **VIOLET**

If a chemical species appears **RED**,

it will strongly absorb **GREEN** light (wavelengths).

The species may absorb other colors as well.

ABSORBANCE (A)

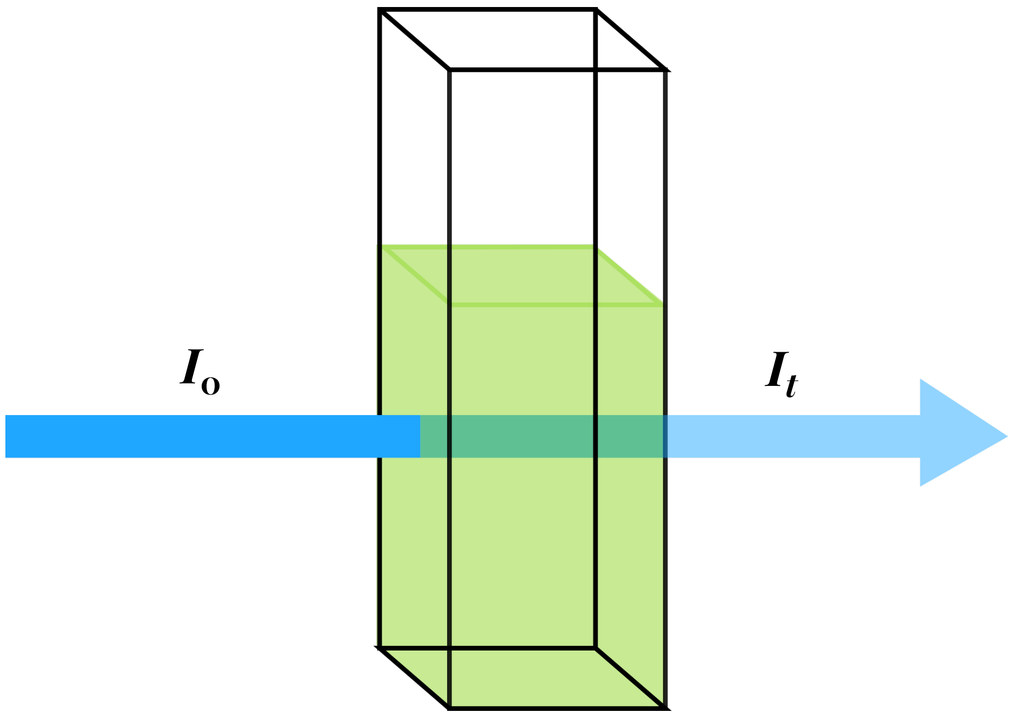
The experimental quantity of interest

We are interested in measuring absorbance (A):

$$A = -\log\left(\frac{I_t}{I_0}\right)$$

where I_0 is the amount of light going in and

I_t is the amount of light coming out after absorption.



Measure I_0 using a BLANK.

Measure I_t using your **SAMPLE**.

BEER-LAMBERT LAW

A linear relationship:

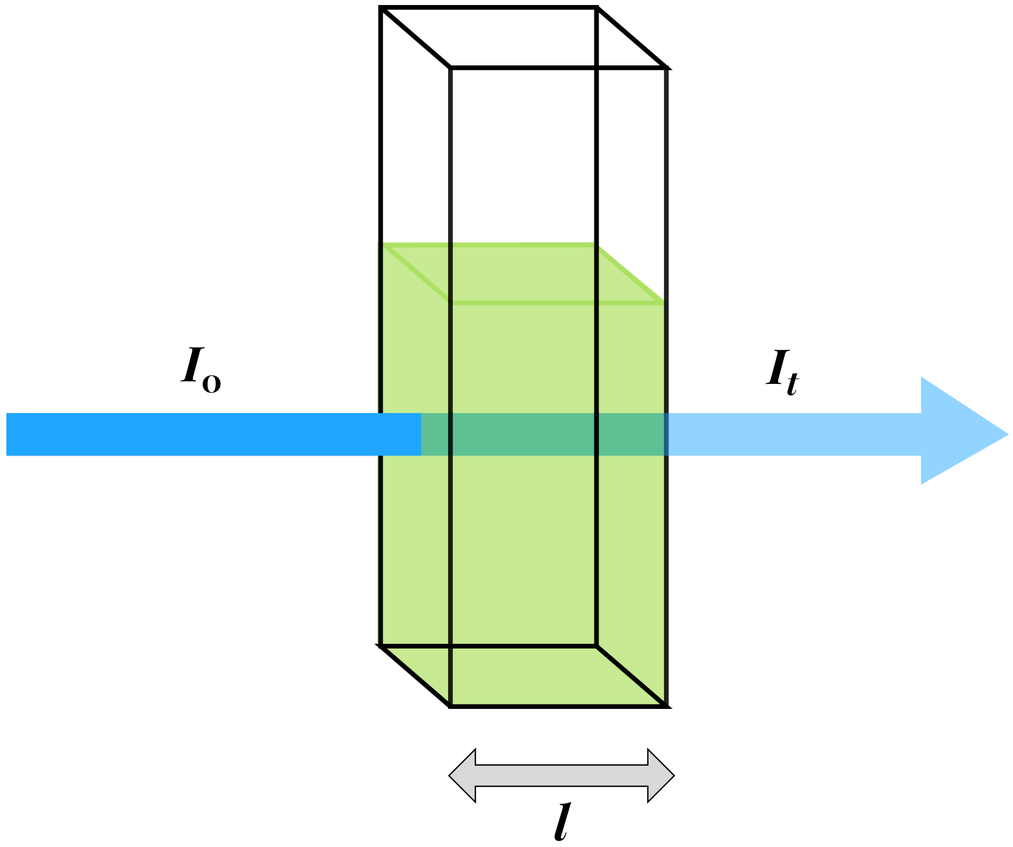
$$A = \varepsilon c l$$

where A is the absorbance (dimensionless),

c is the concentration (mol·L^{-1}),

l is the pathlength (cm), and

ε is the molar absorptivity (mol^{-1}·L·cm^{-1}),

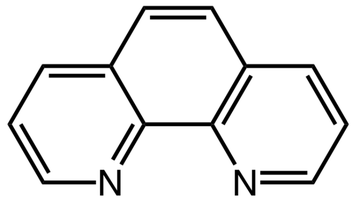


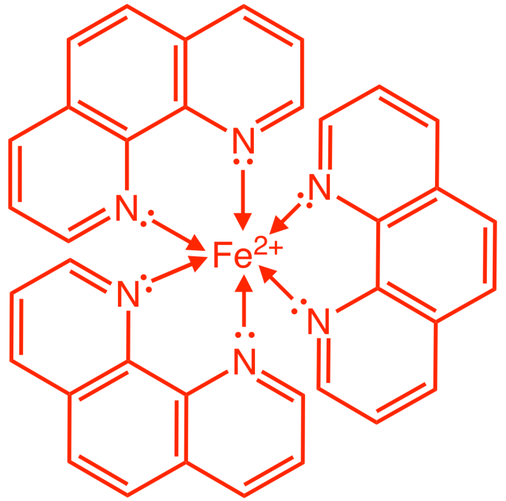
PART 1: SPECTROPHOTOMETRY OF FE(II)

Overview of procedure

The basic idea: Convert the species of interest into an *intensely colored* species.

Fe^{2+} (*aq*) + 3 *o*-phen (*aq*) → $[Fe(o\text{-phen})_3]^{2+}$ (*aq*)

COLORLESS Fe^{2+} + 3 

o-phen ⇌ 

RED

To ensure all Fe is in the 2+ state, we use hydroxylamine.

To optimize the pH, we use sodium acetate.

CALIBRATION

Overview of procedure

Prepare several calibrating solution of known concentrations of the colored complex

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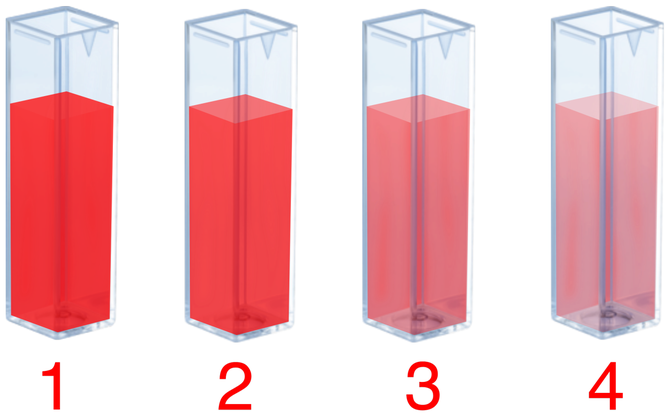
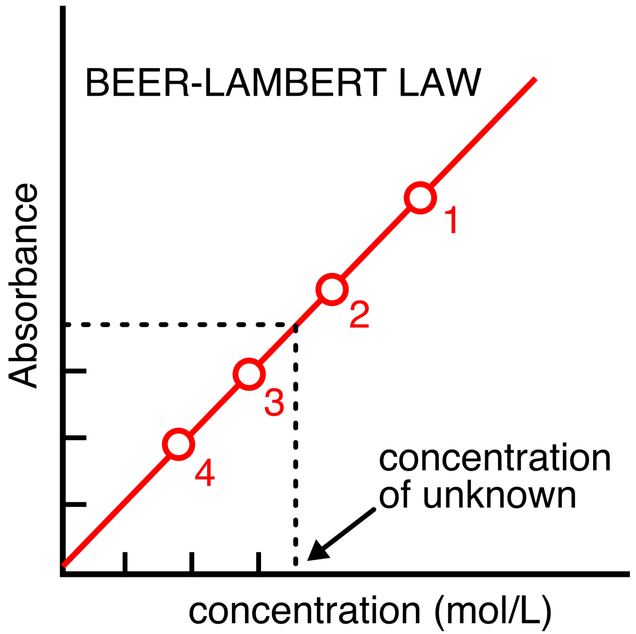
Measure absorbances with Vernier spectrometer

↓

Plot calibration graph to extract ε (the slope)

STANDARDS





PART 2: MASS % OF FE IN YOUR COMPLEX

Overview of procedure

Take a known mass of the complex synthesized from Expt. 1

↓

Reduce all the Fe(III) into Fe(II)

↓

Adjust the pH

↓

Complex Fe(II) with *o*-phen

↓

Measure absorbance

↓

Use calibration plot to determine Fe concentration and mass percentage.

PART 3: MASS % OF OXALATE IN YOUR COMPLEX

Overview of procedure

$$5\ C_2O_4^{2-}\ (aq) + 2\ MnO_4^-\ (aq) + 16\ H^+\ (aq) \rightarrow 2\ Mn^{2+}\ (aq) + 10\ CO_2\ (g) + 8\ H_2O\ (l)$$

Take a known mass of the complex synthesized from Expt. 1

↓

Dissolve in water

↓

Adjust H_2SO_4

↓

Titrate with MnO_4^- of known molarity