

**COLORIMETRIC DETERMINATION OF IRON
IN MULTIVITAMINS & AN UNKNOWN**

TEST EXERCISE (105 pts)

Last Update: 3/14/2009 1:34 PM

Objective:

Determine **weight** of iron in a multivitamin pill
and **concentration** of iron in an unknown
solution - colorimetrically

Concepts:

Complexation Volumetric Dilution
Oxidation-Reduction Buffers
Beer's Law

Techniques:

Preparing precise dilutions and related calculations
Using Spectrophotometer

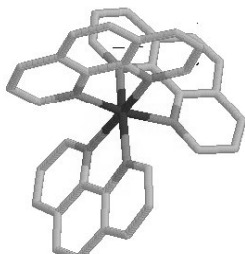
Apparatus:

Spectronic 20 Volumetric Flask Pipet

Stoichiometry of complex is 3 : 1
i.e., 3 mol of 1,10-Phenanthroline
react with 1 mol of **Ferrous Ion (Fe²⁺)**

The complex is
red-orange

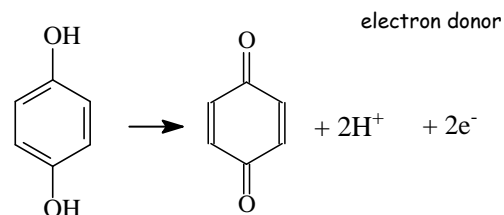
**Analytical
Wavelength**
(Absorbance
maximum) is
at 508 nm



1,10-Phenanthroline complex requires that
IRON be in the **+2 STATE - NOT +3**

OXIDATION - REDUCTION CHEMISTRY

Add **REDUCING AGENT - HYDROQUINONE**



Hydroquinone

Quinone

Procedure requires **MILDLY ACIDIC**
environment.

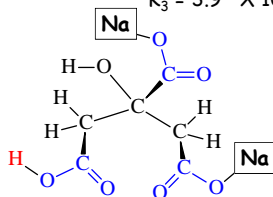
(pH 3.0 - 3.5)

BUFFERS

Use **Sodium Hydrogen Citrate Buffer.**

CITRIC ACID is a weak triprotic acid with
ionization constants (H₃Cit):

$$\begin{array}{ll} K_1 = 8.2 \times 10^{-4} & pK_a = 3.09 \\ K_2 = 1.77 \times 10^{-5} & pK_a = 4.75 \\ K_3 = 3.9 \times 10^{-6} & pK_a = 5.41 \end{array}$$



Its partially neutralized
salts (NaH₂Cit, Na₂HCit)
are natural buffers over
a range of acidic pH's

PROCEDURE

Work **IN PAIRS** for BEER'S LAW Determination
and **PILL - ALONE** for Unknown

Obtain ~50 mL of iron stock solution
in a **clean, DRY BEAKER**

BEER'S Law

Rinse buret with iron stock solution before
preparing dilutions

Prepare a **BLANK** solution

BLANK solution must contain
EVERYTHING other than the IRON

Keep BLANK in cuvette for entire exercise

Prepare **dilutions** of known concentration of Fe^{+2} - **1,10 Phenanthroline** complex using **BURET**

Deliver approximately
 10 mL, 7 mL, 5 mL, 3 mL, 1 mL
 of stock solution (all **MEASURED ACCURATELY!!**)
 into clean 100 mL volumetric flasks

To **each sample** of stock solution, add
Sodium Citrate solution
(to adjust pH to between 3.0 AND 3.5)
Hydroquinone solution using DeWick pipet
(to keep iron reduced)
Phenanthroline solution using DeWick pipet
(to form complex with iron)

Mix each solution thoroughly -
 then add water to bring to mark and
 mix again

Wait for complex to form!

Then, Measure absorbance of each solution at
 508 nm

Remember: Beer's Law Determination is critical to everything that follows. Make sure it is done correctly!

If you have doubts about your partner's data points, reproduce them yourself !!!!!

Measure **ABSORBANCE** of solutions in order of **INCREASING CONCENTRATION** -

(most dilute first)

Save dilutions in case you need to repeat absorption measurements after Beer's Law plot.

Use the same spectrophotometer for all measurements - Beer's Law, pill and unknown

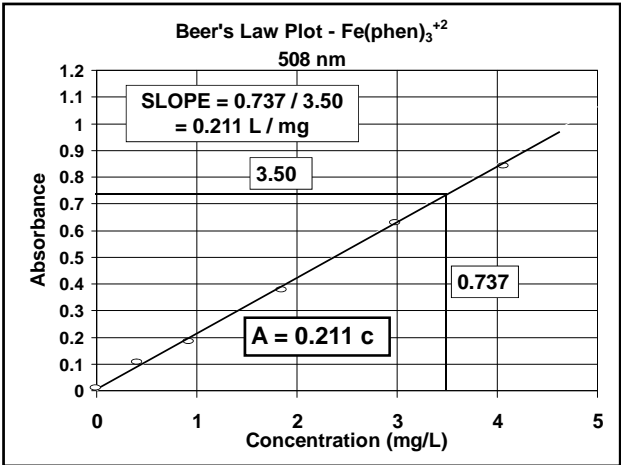
Concentration of Stock Solution = 40.0 mg / L

Vol of stock solution (mL)	Conc of Fe^{+2} (mg / L)	Absorbance
0	0	0
1.12	0.448	0.093
3.24		0.274
4.86		0.418
7.04		0.593
10.22		0.848

Conc (mg/L)	Abs
0	0.000
0.448	0.093
1.30	0.274
1.94	0.418
2.82	0.593
4.09	0.848

Slope should be 0.180 ± 0.020 L/mg

Spreadsheet requires **5 data points,**
 (in addition to the blank)
 in order of **increasing concentration**



From this graph, Beer's Law Constant, k , for Ferrous Phenanthroline complex at 508 nm is

$$k = \text{Slope of Beer's Law Plot} = 0.211 \text{ L / mg}$$

ABSORBANCE and CONCENTRATION are related by:

$$A = k c$$

$$= (0.211 \text{ L / mg}) c \text{ (mg/L)}$$

or, re-arranging

$$c = A / 0.211 = 4.74 A \quad (\text{mg/L})$$

WORK ALONE FROM THIS POINT ON

3. PREPARE SAMPLE SOLUTION FROM TABLET

Wish to determine **TOTAL IRON** in tablet

No need to weigh tablet -

HOWEVER, you must not lose any tablet material in preparing solution

DISSOLVE tablet in 6M HCl, **DILUTE** with 5 mL water, **COOL, FILTER, WASH,**

Bring solution up to 100.0 mL (SOLUTION A)

SOLUTION A contains the entire pill

DILUTE 5.00 mL aliquot of **SOLUTION A** to 100.0 mL - 20-fold dilution-call this **SOLUTION B**

4. BUFFER DETERMINATION

In Beer's Law part and unknown, procedure prescribes amount of buffer to use to adjust pH appropriately.

Why? Stock solution and unknowns are prepared with known pH

Not so with pill
varying composition -
varying amount of HCl, heating time, etc.

Must determine amount of buffer *empirically*

How will you do this?

To 10 mL aliquot of **SOLUTION B**
add **2.5% SODIUM H CITRATE**
solution To bring pH UP to 3.5
as measured by pH meter

Burets with Sodium H Citrate and pH meters
are already set up in lab

Record your REQUIRED AMOUNT OF BUFFER

5. CONVERT TABLET IRON TO COMPLEX

To a FRESH 10 mL ALIQUOT of **SOLUTION B**
in a 100 mL volumetric flask, add:

1. REQUIRED AMOUNT of Sodium H Citrate Buffer solution
2. HYDROQUINONE solution
3. PHENANTHROLINE solution
4. Mix thoroughly
5. Bring to 100 mL by adding distilled water.
6. Mix thoroughly again
Call this SOLUTION C

SOLUTION C contains 1/10th of the fraction of the pill that was in **SOLUTION B**

Wait for complex to form!

Then, measure the Absorbance of **Solution C**
Repeat determination with second sample of SAMPLE SOLUTION C

Suppose measured absorbance is $A = 0.530$
From Beer's Law Plot

$$c = 4.74 \times 0.530 = 2.51 \text{ mg/L}$$

100.0 mL (0.1000 L) of **SOLUTION C** contains

$$0.1000 \text{ L} \times 2.51 \text{ mg/L} = 0.251 \text{ mg of IRON}$$

and original solution (**SOLUTION A**, which contained the entire pill) contained

$$0.251 \times 200.0 = 50.2 \text{ mg OF IRON}$$

DETERMINE CONCENTRATION OF IRON IN UNKNOWN SOLUTION

Use 10.00 mL aliquot of unknown

Add sodium citrate, hydroquinone and phenanthroline AS IN PART 1

Dilute unknown aliquot to 100.0 mL

Wait for complex to form!

Measure absorbance of diluted solution and calculate concentration of diluted solution

Calculate concentration of original solution (X 10.00)

Repeat absorbance measurement a second time with a fresh sample of *the **SAME** diluted unknown solution*

Work in pairs for Beer's Law & Pill

TA's will assign half of groups to begin with the analysis of the **PILL**.

The other half will begin with the **BEER'S LAW** determination

If you begin with the **Pill part**, it is recommended that you analyze your unknown ***while your tablet is dissolving***.

However, watch that you don't let your tablet solution evaporate to dryness.

DATASHEET FOR UNKNOWN

Slope of Beer's Law plot = **0.211 L / mg**

Absorbance of diluted solution = **0.471**

Conc of diluted solution = **2.23 mg/L**

Conc of original solution = **22.3 mg/L**

On data sheets, "From Graph" means using the slope of the Beer's Law Plot

DATASHEET FOR PILL

Slope of Beer's Law plot = **0.211 L / mg**

Absorbance of solution C = **0.156**

Conc of solution C = **0.739 mg / L**

Conc of solution B = **7.39 mg / L**

Tot Vol of solution B = **100.0 mL**

Amt of Iron in solution B = **0.739 mg**

Amt of Iron in solution A = **14.8 mg**