DETERMINATION OF IRON IN BEER

(USE AN APPROPRIATE TITLE)

(THIS IS NOT A SAMPLE JOURNAL FORMAT REPORT

REQUIRED:

Single-sided, white paper only
1" margins

Graphs: EXCEL (see examples)
(If approved by instructor: graphs can be done on good quality graph paper (1" by 20; or 1 cm by 10))

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Chemistry 464L
Spring 2002
M 2-5
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Lab Partner: Thomas R. Quilleran
(lab partner's name required)

The information in italics is for instructional purposes only

1. Prepare a table indicating how you prepared the standard solutions for the analysis of iron in beer. Tabulate the absorbances measured at 510 nm. Include in your table the relevant information for the beer sample

(Note: question is repeated and bolded to set it apart from the answer below)

Table 1. Data for the Beer's Law Plot for Determination of Iron in Beer.
(Final volume = 1000 µL)

(note all column headings and numbers are centered)

Standard Curve	µL 1.00 x10^{-3} M [1] Fe^{2+}	µL H_2O	µL 0.0100 M [2] PHE	µL 20 % [3] ASC	µL 20 % [4] NaOAc	Fe^{2+1} 10^{-5} M (in cuvet)	Absorbance 510 nm
1	0	100	20	100	780	0.00	0.025
2	20	80	"	"	"	2.00	0.225
3	40	60	"	"	"	4.00	0.495
4	60	40	"	"	"	6.00	0.665
5	80	20	"	"	"	8.00	0.945
6	100	0	"	"	"	10.00	1.075
[5] Beer							
7	--	25	"	"	"	--	0.095
9	--	25	"	"	"	--	0.092
9	--	50	"	"	"	--	0.167
10	--	50	"	"	"	--	0.163

(Note: some explanatory information is provided in footnotes)

1 $Fe_2(NH_4)_2(SO_4)_2 \cdot 6\ H_2O$ was used as the primary standard for a 0.1987 M stock solution in 0.010 M HCl. A portion of this stock solution was diluted with 0.010 M HCl to achieve a 1.00 x 10^{-3} M solution used to prepare the standard solutions.

2 PHE is 1,10-phenanthroline dissolved in 0.010 M HCl water.

3 ASC is ascorbic acid, w/v %.

4 NaOAc is sodium acetate, w/v %.

5 Coors Lite beer, undiluted

Beer's Law has nothing to do with beer!

2. Using your data for the standard iron solutions, construct two Beer's law plots. For Figure 1 plot absorbance versus mg/mL iron(II); for Figure 2, plot absorbance versus M of iron(II). Attach these plots to the end of your report and any spreadsheet(s) used to construct the plots. Show calculations for the extinction coefficient below for Figure 2.

Extinction coefficient:

The extinction coefficient is found using Beer's Law equation, $A = \varepsilon\ c\ l$, where ε is the extinction coefficient, c the concentration, and l the path length of the cuvet (1.00 cm):

ε = slope of plot of A vs concentration in mol/L from standard plot = $0.108 \times 10^5\ M^{-1}\ cm^{-1}$
= $1.08 \times 10^4\ M^{-1}\ cm^{-1}$

See regression analysis for complete determination of extinction coefficient.

3. Determine the unknown iron in your beer sample by two methods. For Method I, use Figure 1 and read the iron concencentration in mg/mL in the cuvet from the plot directly. Convert it to mg/mL iron in your sample. For Method II, use the extinction coefficient determined from the slope of Figure 2 to find the unknown iron concentration in M in the cuvet. Convert it to the molarity of iron in your sample.

Method I:

Sample Calculation for Sample #7: *(Note 2 sig. figs. are used because method is less accurate)*

ABS at 510 nm = 0.095 - 0.025 (*blank correction*) = 0.070

Iron concentration in cuvet = 0.33×10^{-3} mg/mL *(Read directly from Figure 1)*

Iron concentration in sample = 0.33×10^{-3} mg/mL x $\frac{1000\ \mu L}{25\ \mu L}$ (*volume correction for dilution into cuvet*) = 13×10^{-3} mg/mL

Method II:

The concentration of iron in an unknown sample is determined using Beer's Law equation, $A = \varepsilon\ c\ l$, where the individual absorbance values are divided by the known extinction coefficient multipled by the path length

Sample Calculation for Sample #7:

ABS at 510 nm = 0.095

$$\text{Iron in cuvet} = \frac{0.095 - 0.025\ \text{(blank correction)}}{1.08 \times 10^{4}\ M^{-1}\ cm^{-1}\ (1.00\ cm)} = 0.0648 \times 10^{-4}\ M = 6.48 \times 10^{-6}\ M$$

(Carry extra digit - this is not final "answer")

$$\text{Iron in Beer} = 6.48 \times 10^{-6}\ M \times \frac{1000\ \mu L}{25\ \mu L} = 2.6 \times 10^{-4}\ M$$

(*volume correction for dilution into cuvet*)

(Correct sig. figs. for final "answer")

(Note: where multiple calculations are tabulated in a spreadsheet, it is not necessary to show all calculations. If all calculations are shown, they must be summarized in a table with proper headings. The summary table is placed BEFORE the calculations)

4. Compare the iron concentration in your beer sample determined by Method I and Method II. Note that you will have to convert mg/mL to M or visa versa. To be consistent, convert mg/mL to M.

(carry extra digit here)

Method I: $\frac{13.4 \times 10^{-3}\ \text{mg}}{\text{mL}} \times \frac{1\ \text{g}}{10^{3}\ \text{mg}} \times \frac{10^{3}\ \text{mL}}{1\ \text{L}} \times \frac{1\ \text{mole}}{55.85\ \text{g}} = 2.4 \times 10^{-4}\ \text{M}$

Method II: 2.56×10^{-4} M

Average Iron in sample: $2.5 \pm 0.10 \times 10^{-4}$ M
(sig figs for comparison limited by least accurate determination)

Sample	Iron value	Deviation from average
Method I:	2.4×10^{-4} M	0.10×10^{-4} M
Method II:	2.6×10^{-4} M	0.10×10^{-4} M
Average:	2.5×10^{-4} M	0.10×10^{-4} M

$$\frac{\text{Deviation}}{\text{Average}} = \frac{0.10 \times 10^{-4}\ \text{M}}{2.5 \times 10^{-4}\ \text{M}} \times 100\ \% = 4.0\ \%$$

The two methods give the same iron in the beer sample to within 4.0%

5. What are the two forms of iron that may be found in beer? Why is it likely to find one form and not the other?

Iron has two oxidation states, Fe^{3+} and Fe^{2+}. The Fe^{2+} ion is not likely to be present because it is readily oxidized by dissolved oxygen. Therefore the Fe^{3+} ion is likely to be the one present in beer. It is important to note that beer contains dissolved acids (lactic, citric, etc.) from the manufacturing process using yeast; these acids will chelate to the Fe^{3+} thereby preventing its precipitation from the beer as Fe_2O_3 (rust!). Since it taints the flavor of beer, Fe^{3+} levels are keep at a low level in beer.

6. Write the chemical reaction that is occurring between iron and 1,10-phenanthroline in this system? What is the function of the ascorbic acid and the sodium acetate?

Iron (II) and 1,10-phenanthroline form a complex where three phenanthroline molecules surround the iron(II) ion forming a colored complex:

$$Fe^{2+}_{(aq)} + \quad 3\ PHE \quad \circ \quad Fe(PHE)_{3(aq)}$$

The ascorbic acid acts as a reducing agent to reduce any Fe^{3+} that could be present to Fe^{2+} so that it can bind to the phenanthroline.

The sodium acetate acts to maintain the pH around 4 to 5 which is optimal for the formation of the colored complex.

(Note: the question has 3 parts that are clearly answered!)

7. How do you know that the absorbing species in the beer sample is iron(II)-phenanthroline complex and not some other metal ion-phenanthroline complex?

The species giving the color with the beer sample was shown to be the iron(II)-phenanthroline complex after comparison of the visible spectra of the reagent mixtures of the beer sample and one of the standards (see Figure 3). Both spectra both had the same wavelength maximum and the same overall shape. They were not identical because the iron concentration in the beer sample was not the same as in the standard sample.

8. Discuss three sources of errors in your experiment.

The major source of error is in pipetting technique. The pipetors are not calibrated and so that the volumes delivered may not be what is dialed in the window. Secondly, poor pipeting technique (pipeting from source at a distance, delivery from the pipet tip that is not at eye level, careless uptake and delivery in other ways, etc.). Finally, another source of error is inadequate mixing; solutions pipetted together do not necessarily mix thoroughly.

(Note: provide a thoughtful analysis of errors in your experiment. Do not copy this paragraph into any of your reports!)

(DO NOT SEPARATE ANSWERS FROM QUESTIONS ON DIFFERENT PAGES)

Standard Plot for the Determination of Iron

Note: all calculations are performed in the spreadsheet

Note: other reagents are left off of this spreadsheet

Stock Solution of Iron Standard **0.0558 mg/mL**

1.00 10^{-3} M

Final volume in cuvet **1000 mL**

MW Fe **55.85 g/mole**

(Can also use trendline information)

Sample	mL Std	Fe^{2+} 10^{-3} mg/mL in cuvet	Fe^{2+} 10^{-5} M in cuvet	ABS 510 nm	CORR ABS 510 nm
1	0	0.00	0.0	0.025	0.000
2	20	1.12	2.0	0.225	0.200
3	40	2.23	4.0	0.495	0.470
4	60	3.35	6.0	0.665	0.640
5	80	4.47	8.0	0.945	0.920
6	100	5.58	10.0	1.075	1.050

SUMMARY OUTPUT

ABS vs M

Regression Statistics	
Mult R	0.9966
R^2	0.9933
Adj R^2	0.9916
Standard E	0.0372
Obs	6

	Coef	*Std Err*
Intercept	0.0052	0.0269
X Var 1	**0.1083**	0.0044

X Variable 1 = Extinction Coefficient

0.108 x 10^{5} M^{-1} cm^{-1}

(Beer's Law: A = ε c l)

ε is the slope of A vs c with l = 1.00 cm

(cannot ignore magnitude of x axis)

or

1.08 x 10^{4} M^{-1} cm^{-1}

Sample	Beer Undiluted[1] mL	ABS 510 nm	CORR ABS 510 nm	Calc Iron in cuvet[2] 10^{-3} mg/mL	Iron in Beer 10^{-3} mg/mL	Iron in Beer 10^{-4} M
				Using Figure 1: Absorbance versus Fe^{3+} mg/mL		
7	25	0.095	0.070	0.33	13	2.4
8	25	0.092	0.067	0.32	13	2.3
9	50	0.167	0.142	0.71	14	2.5
10	50	0.163	0.138	0.68	14	2.5
				AVE =	**13**	**2.4**
					10^{-3} mg/mL	**10^{-4} M**

(cannot ignore magnitude)

[1] *- The beer required no predilution*

[2] *- Obtained from plot of Absorbance versus mg/mL Fe^{2+} by reading directly from graph; fewer sig figs*

Using Figure 2: Absorbance versus Fe^{2+} M

Calc Iron in cuvet[3] 10^{-4} M	Iron in Beer 10^{-4} M
0.0648	2.59
0.0620	2.48
0.1315	2.63
0.1278	2.56
AVE =	**2.56**
	10^{-4} M

(cannot ignore magnitude)

[3] - Obtained from unknown absorbance and extinction coefficient, ε, and A = εcl

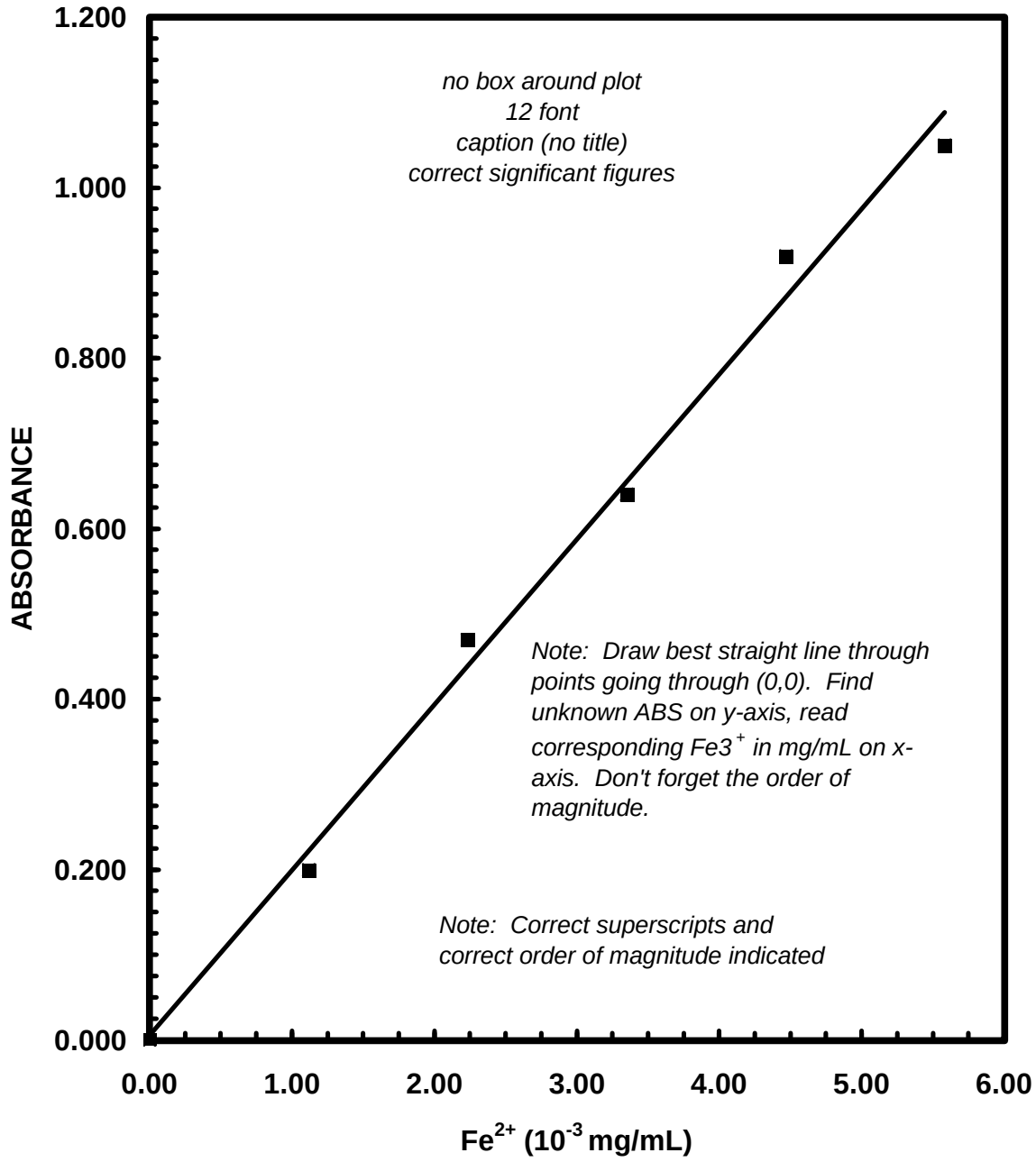

Figure 1. Standard plot for the determination of iron using 1,10-phenanthroline and ascorbic acid in sodium acetate solution. The absorbance is measured at 510 nm.

Use "Print Preview" mode to check that caption wraps correctly

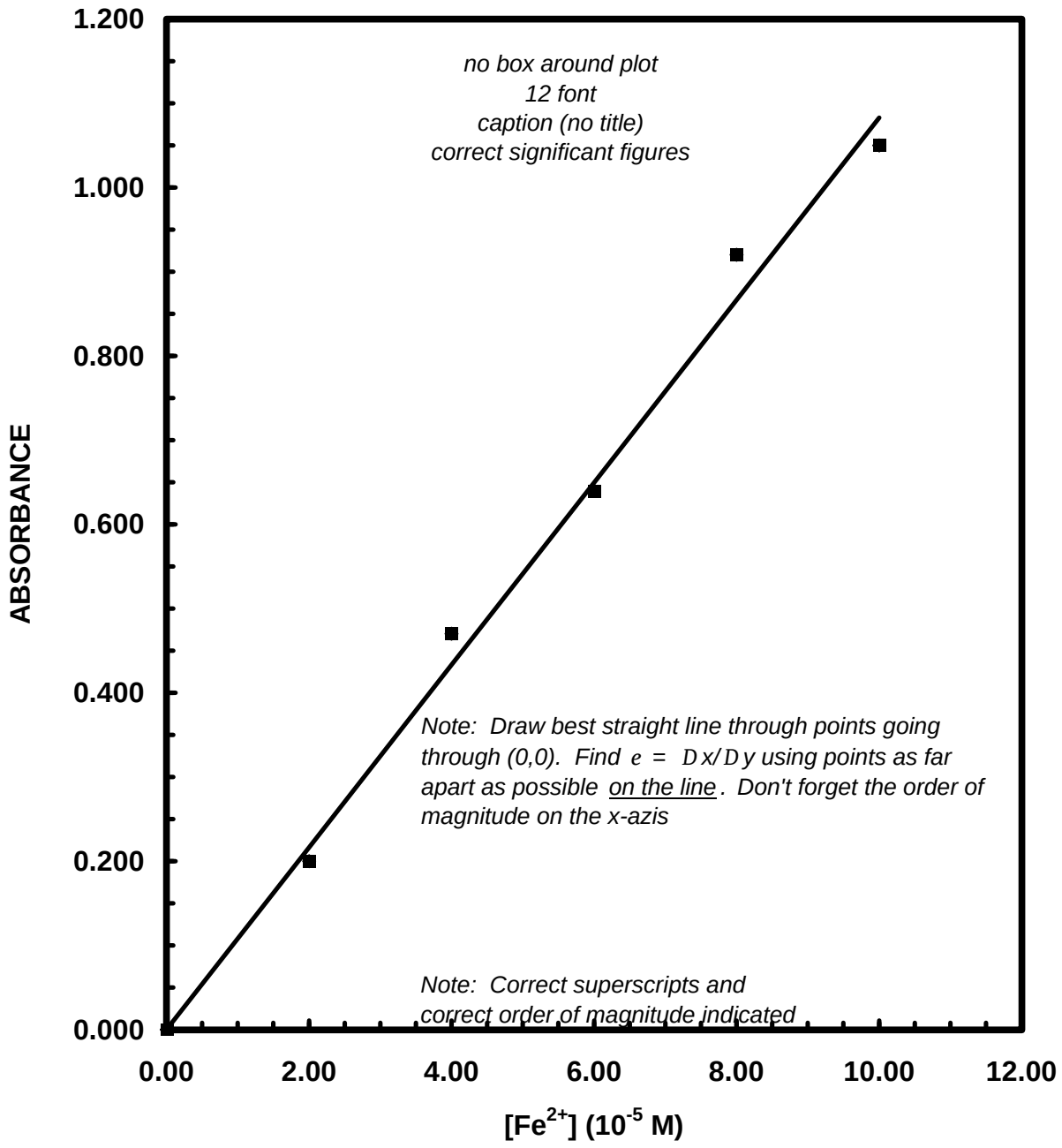

Figure 2. Standard plot for the determination of iron using 1,10-phenanthroline and ascorbic acid in sodium acetate solution. The absorbance is measured at 510 nm. The extinction coefficient from these data is 1.08×10^{-4} M^{-1} cm^{-1}.

Use "Print Preview" mode to check that caption wraps correctly

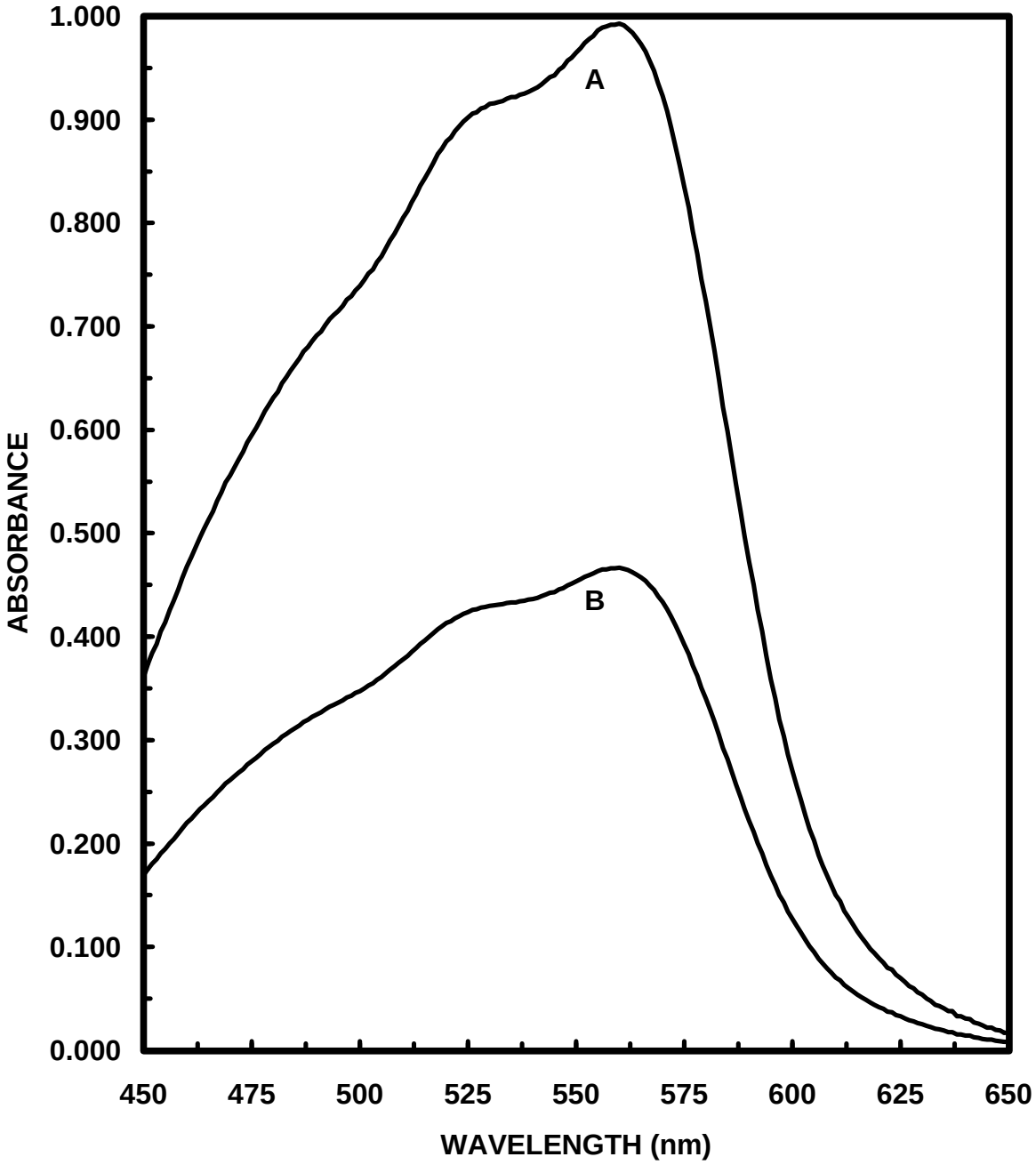

Figure 3. **(A) Spectrum of iron(II)-phenanthroline complex prepared from a standard iron sample. (B) Spectrum obtained from the analysis of a Lite Coors beer sample.**