Staff Notes for Spectrophotometric Determination of Fe

Students will study the absorption of trace amounts of Fe complexed with 2,2-bipyridil. Students will use the LabQuests interfaced with an Ocean Optics spectrometer or a SpectroVis Plus to generate a working curve with which they can determine the concentration of Fe in an unknown. Since students will be using spectroscopy to relate concentration to %T, they should be familiar with Beer's Law and the fact that the relationship between concentration and A is linear at low concentrations. The chemistry of the reaction is quite interesting, and should be reviewed by you or the professor prior to lab. Note that there are some simple oxidation-reduction problems addressed in this lab. Students should have seen this before in previous courses, but a quick reminder wouldn't hurt. Some points to cover:

a. It's important that all Iron in solution exists as Fe^{2+} species. The HCl is added to minimize the air oxidation of the ferrous cation to the ferric cation. This oxidation happens readily in a pH neutral environment but is somewhat inhibited in an acidic environment.
b. Hydroxylamine solution is added to each sample to reduce all Fe^{3+} to Fe^{2+}. So hydroxylamine is referred to as a *reducing agent.*
c. The form of a complex as it exists in solution is dependent on the pH of solution. Thus to preserve a desired species, the solution must be strongly buffered. This means it has a large buffer capacity and is extremely resistant to a change of pH. We use sodium acetate as the buffer, in this case, to maintain a proper pH of around 4.5 as we dilute with the *complexing agent*—2,2 bipyridil, and water.

Prior to Lab:

Obtain a clean, labeled sample bottle from your students. Fill with an appropriate amount of unknown stock solution. Assuming the stock concentration of Fe solution is 40 ppm Fe, you should use a range of 30 - 40 mL to dispense to students. Accurately record the volumes you dispense. Students will perform a quantitative transfer and dilution.

Review how to use the LabQuests to collect data in units of %T.

Be familiar enough with Excel to help students perform a linear regression on their data.

During Lab:

Students will have to check out nine, 100 ml volumetric flasks and one 20 ml pipette at the beginning of the period. Review pipetting technique, how to transfer solutions, and the safe handling of strong acids. Be prepared to demonstrate how to use the LabQuests, too.

Ferrous ammonium sulfate is used to make a stock Fe solution. There is a vapor coming off the solid that can cause nausea and fainting spells. Tell your students to be extremely careful when handling this reagent.
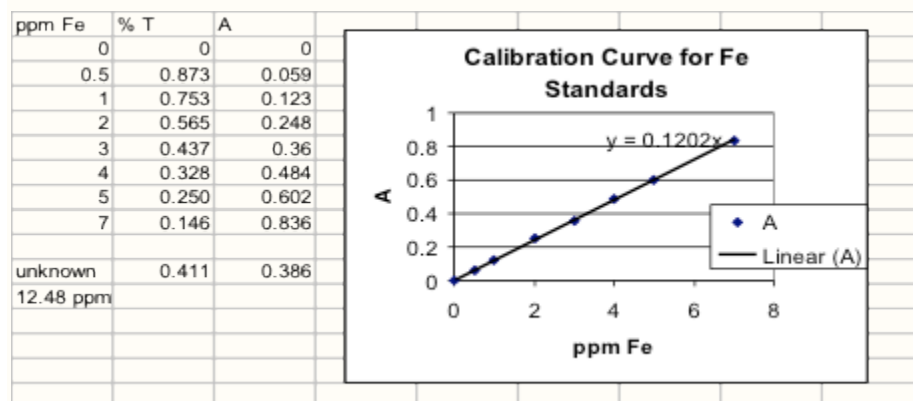
Things important to getting a good working curve:

a. Add reagents in the order presented in the table. Reasons are explained above.
b. Clean glassware and accurate pipetting techniques are VERY important to producing a good working curve. Any contamination or carelessness will skew the points of the working curve, or change the color of solution from red to blue.
c. Remind students they are performing a dilution of the Fe when they make their standards. They are also diluting the unknown sample in the same way. They must incorporate this dilution when trying to figure out the concentration of the unknown Fe sample. (The factor is 4—they should figure it out)
d. Use the blank they prepare to zero the instrument (or set 100% T).
e. Use the same instrument for all measurements. Each instrument has intrinsic offsets that cancel out, as long as you use the same one.

Students will use Excel to construct a working curve of their data, there its best the record the %T values vs. [Fe^{2+}] in their notebooks. Its true they can collect the data in units of Absorbance. They can even do the standard curve immediately using the LabQuests, and may remember how to do that vaguely from their experience in general chemistry. We want them to really think about the relationship between %T and A, manually do the conversions and work up

the data in stepwise fashion. They should use a 0,0 point—assuming the blank Transmittance is used to reset the instrument. Sometimes, a slight roll-off occurs at 7.0 ppm. They should not include this point in the linear regression. If their unknown concentration is greater than 5.0 ppm, they should use the slope of the line between 5.0 and 7.0 ppm to determine the unknown concentration.

Students that do not finish this lab by the end of the period can store their solutions in the plastic 125 ml sample bottles. The complex is very stable. The literature says it can sit for 2 years without noticing a change in color. Recently I uncovered a set of standards dated 1988 that looked good-as-new! Hopefully, students won't need them for that long!

Sample data. Stock solution prepared by dissolving 0.7022 g ferrous ammonium sulfate in 1.00 L flask.

ppm Fe	% T	A
0	0	0
0.5	0.873	0.059
1	0.753	0.123
2	0.565	0.248
3	0.437	0.36
4	0.328	0.484
5	0.250	0.602
7	0.146	0.836
unknown	0.411	0.386
12.48 ppm		

Calibration Curve for Fe Standards

$y = 0.1202x$

A
Linear (A)

ppm Fe

Preparation Notes: Spectrophotometric Determination of Iron

Overview
Students determine trace amounts of iron using spectrophotometric methods.

Chemicals
> 6 M HCl
>
> Ferrous Ammonium Sulfate ($Fe(NH_4)_2(SO_4)_2\ 6H_2O$) aka FAS
>
> 1 M HCl
>
> 1% Hydroxylamine
>
> 2 M Sodium Acetate
>
> 0.1% Bipyridyl
>
> Unknowns

Deployment

In each lab	TA prep room	At stockroom
Chemicals		
Fume Hood 1 L 6 M HCl		
Central Cart 500 g $Fe(NH_4)_2(SO_4)_2\ 6H_2O$ 2 L 1 M HCl 2 L 1% Hydroxylamine 2 L 2 M Sodium Acetate 2 L 0.1% Bipyridyl	Unknown Carboy – 40 ppm Ferrous Ammonium Sulfate	100 mL volumetric flasks Labquest chargers
Equipment		
Central Cart SpectroVis Plus – 8/section Cuvets		

Note: Lot # of unknown and bottles handed out MUST match.

Preparation
6 M HCl – 9677 mL / 20 L. See preparing acids and bases.
1 M HCl – 1613 mL / 20 L. See preparing acids and bases.

1% Hydroxylamine – 200 g / 20 L.

Sodium Acetate – 5443.2 g sodium acetate trihydrate / 20 L.

0.1% Bipyridyl – 20 g / 20 L.

Unknowns – 5.6176 g FAS + 8 mL 1 M HCl / 20 L. 1.4044 g FAS + 2 mL 1 M HCl / 5 L.

Safety Supplies at reagent shelf

Sodium Bicarbonate – for acid spills

Citric Acid – for base spills