

Determination of Iron in Water

Introduction:

Any solution which is colored or can be made colored by adding a complexing agent can be analyzed using a visible spectrophotometer, such as the Spec 20. Solutions containing iron ions are colorless, but upon addition of ortho-phenanthroline, the iron (II) ions in the sample react immediately to produce a complex ion, which is orange in color. The more iron (II) ions in a sample will result in a deeper orange color while less iron (II) ions result in a fainter orange color. This follows the Beer-Lambert Law of spectroscopy. From data obtained from a series of iron (II) standards, it is possible to be able to determine the amount of iron in an unknown sample.

Safety:

Always wear safety glasses and an apron in lab.

Equipment and Materials:

Spectrophotometer Cuvettes and rack

Procedure:

Part I: Preparation of standards (steps 1 and 2 may have been done by the instructor)

1. Dissolve 0.7022 grams of ferrous ammonium sulfate, hexahydrate into deionized water. Dilute to 1.00 L. This solution is 100 mg/L of Fe^{2+} (same as 100 ppm).
2. Prepare standard solutions of the following concentrations: 0.0, 2.0, 4.0, 6.0, and 8.0 ppm. Use a pipet to transfer 0.0, 2.0, 4.0, 6.0, and 8.0 mLs of the 100 ppm stock solution into five separate 100 mL volumetric flasks. To each of the flasks, add 5 mL of a 0.25% ortho-phenanthroline solution with a graduated cylinder. Carefully dilute to the 100 mL mark by adding deionized water.
3. Clean a set of six cuvettes and dry the outside. Label the cuvettes: 0 ppm, 2.0 ppm, 4.0 ppm, 6.0 ppm, 8.0 ppm.
4. Rinse each cuvette with a **SMALL** amount of the appropriate solution and fill to about the half-way point.

Part II: Forming the standard curve.

5. Turn the spectrophotometer on and allow it to warm up for 20 minutes.
6. Adjust the wavelength to 510 nm.
7. With the sample compartment empty and the lid closed, set the instrument to 0%T using the Zero Control Knob. (left front)

8. Using a Kimwipe, wipe any finger prints from the cuvette containing the Blank (0ppm) and place this cuvette into the sample compartment being sure to align the index marks. Close the cover.
9. Set the instrument to 100% Transmittance using the 100% T/0A knob (right front).
10. Press the MODE button one time to change the mode from transmittance to absorbance.
11. Record the absorbance of the 0 ppm solution (it should be 0.000 A).
12. Insert each of the cuvettes containing standards into the sample holder, reading the absorbance values for each one. Record the values in your data table.
13. Construct a graph of solution concentration (x-axis) vs. absorbance (y-axis). Draw a “least squares” or best fit straight line through the data points.

Part III: Determination of an unknown.

14. Obtain an unknown sample from the instructor or prepare your own unknown sample using a collected water sample.
15. If using a collected water sample, add 5 mL of the 0.25% ortho-phenanthroline solution to a 100 mL volumetric flask. Dilute to the line with the collected water.
16. If using a collected water sample, use collected water without the 0.25% ortho-phenanthroline solution as the blank. Re-zero the spectrophotometer with this new blank.
17. Obtain an absorbance reading for the unknown sample.
18. From the standard curve, determine the concentration of iron in this unknown sample.

Name _____

Name _____

Period _____

Date _____

DETERMINATION OF IRON IN WATER

Data Table:

Concentration	Absorbance
0.0 ppm (blank)	
2.0 ppm	
4.0 ppm	
6.0 ppm	
8.0 ppm	
unk #	
unk #	

Questions

1. Explain why it was necessary to add ortho-phenanthroline to the solutions.
2. What is the purpose of preparing and analyzing standard iron solutions?
3. What other items could be analyzed using this method?

DETERMINATION OF IRON IN WATER TEACHER NOTES

Time: 45 minutes

Preparations:

Time: 10 minutes

- T:** Make available one set of materials for each group of students.

- T:** Prepare the 1,10-phenanthroline solution by dissolving 2.5 g ortho-phenanthroline in approximately 50 mL of 95% ethanol and diluting to 1.00 L with deionized water.

- T:** To save time, steps one and two may be done before the students arrive.

- T:** Turn on the spectrophotometers 20 minutes before using to warm up.

- V:** The van can supply spectrophotometers and any other material as requested.

Answers to Questions:

1. Explain why it is necessary to add ortho-phenanthroline to the solutions.

The ortho phenanthroline was added to complex with the iron. The complex is colored and can, therefore, be analyzed using a spectrophotometric method.

2. What is the purpose of preparing and analyzing standard iron solutions?

To prepare a standard curve and use it to determine the concentration of iron in the unknown water samples.

3. What other items could be analyzed using this method?

Any product containing iron which can be extracted and is in the +2 oxidation state or can be converted to this state (iron in vitamins).

Considerations:

While making a second blank for the unknown is not required, it will give better results if the unknown was not as clear as the standards before reacting. Prepared solutions can be kept for several weeks in a dark place. The blank will begin to turn slightly yellow but results do not seem to be drastically affected. Solutions can be poured

