Chapter W

Colorimetric determination of ferrous iron, Fe(II), in natural water, wastewater, and seawater.

By Peter M. Theodorakos

U.S. Geological Survey Open File Report 02-223-W

Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey

Contents

Table

Colorimetric determination of ferrous iron, Fe(II), in natural water, wastewater, and seawater.

By Peter M. Theodorakos

Code: T11 Accepted: 3/7/02

Principle

Dissolved iron occurs in two oxidation states, ferrous (Fe^{2+}) and ferric (Fe^{3+}). The determination of Fe^{2+} is accomplished using commercially available portable spectrophotometers that accept specially designed, sealed ampoules that, when the ampoule neck is broken, draw the water sample into a reagent mix. The 1,10-phenanthroline indicator in the ampoule reacts with the ferrous iron in the sample to form an orange color in proportion to its concentration.

Interferences

Errors can be caused by analysis of improperly collected or preserved samples, unfiltered samples, fingerprints, moisture or condensation on ampoules, and failure to zero the instrument prior to analysis. Above pH values of 4.5 Fe^{2+} is rapidly oxidized by the atmospheric or disolved oxygen to Fe^{3+}.

Scope

This is a rapid method for the determination of Fe^{2+} in water and water leachates of solid samples. A disadvantage of the method is the narrow concentration range provided, 0 to 3.0 mg/L. High concentration levels of iron in acid-mine drainage often require a dilution of the sample and the use of more than one ampoule (HACH Company Handbook,1996). This method is applicable to waters, wastewater, and seawaters.

Apparatus

HACH DR/2010 Spectrophotometer
Adapter, AccuVac Vial
Beaker, 50 ml
Vial, Zeroing

Reagents

Ferrous Iron (Fe^{2+}) AccuVac Ampuls (0 to 3.0 mg/L)
Water, demineralized

Procedure

1. Enter the stored program number for ferrous iron (Fe^{2+}) AccuVac ampuls.
   Press: **257 ENTER**
2. Rotate the wavelength dial until the small display shows: **510 nm**. When the correct wavelength is dialed in, the display will quickly show: **Zero Sample** then: **mg/L Fe^{2+} AV**
3. Fill a zeroing vial with at least 10 ml of sample (the blank). Collect at least 40 ml of sample in a 50-ml beaker.
4. Fill a Ferrous Iron AccuVac Ampul with sample.

5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.
6. Press: **SHIFT TIMER** a three-minute reaction period will begin.
7. Place the AccuVac Vial Adapter into cell holder.
8. When timer beeps, the display will show: **mg/L Fe^{2+} AV**.
9. Press: **ZERO** the display will show: **Zeroing…** then **0.00 mg/L Fe^{2+} AV**.
10. Place the AccuVac ampul into the cell holder. Close the light shield.
11. Press: **READ** the display will show: **Reading…** then the results in mg/L Fe^{2+} will be displayed.

Calibration

Prepare a ferrous iron stock solution (100 mg/L Fe^{2+}) by dissolving 0.7022 grams of ferrous ammonium sulfate-hexahydrate in demineralized water and bring to 1 liter. Dilute 1.00 ml of this solution to 100 ml with demineralized water to make a 1.00 mg/L standard solution. Prepare stock and working standards immediately before use.
Run the test using the 1.00 mg/L Fe^{2+} standard solution by the AccuVac procedure. Results should be between 0.90 mg/L to 1.10 mg/L Fe^{2+}.

Precision

Using an iron standard solution of 1.00 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the DR/2010, a standard deviation of + or – 0.009 mg/L Fe^{2+} was obtained.

Assignment of Uncertainty

Table 1.—Analytical performance summary for Ferrous Iron (Fe^{2+}) 0 to 3.00 mg/L for water, wastewater, and seawater utilizing the HACH DR/2010 spectrophotometer 1, 10 Phenanthroline Accu Vac Ampuls Method.

See page ix of the introduction to this Methods Manual for an explanation of the abbreviations used in the analytical performance summary tables.

Reference	*Description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Fe^{2+}	1.00 mg/L	20	0.989	0.013	1.00	1.308	101.1

Bibliography

Hach Company, 1996, Procedures manual, DR/2010 spectrophotometer handbook. *hm/dp 07-16-96 1 ed*