

FERTILIZER METHODS

Chapter

IRON CHELATES ANALYSIS

Subject

Iron Chelate – Pure Material ~ AA

SCOPE: This is an automated analytical procedure for the determination of iron chelate in fertilizer samples containing pure iron chelate, or iron chelate in combination with other iron compounds by flame AA analysis. **NOTE: Not applicable to mixed fertilizers or to samples containing non-chelated metals other than iron.**

PRINCIPLE: Applicable to Fe ethylenediamine tetraacetate (EDTA), Fe hydroxyethylenediamine triacetate (HEDTA), Fe diethylenetriamine pentaacetate (DTPA), Fe ethylenediaminedi-o-hydroxyphenylacetate (EDDHA), Fe citrate as well as liginosulfonates and polyflavonid extracts. The preparation for iron chelates determination is achieved by dissolving the sample in deionized water with a pH adjustment made to 8.5 with sodium hydroxide to precipitate out any non-chelated iron as $\text{Fe}(\text{OH})_3$. After appropriate filtration and dilution, samples are analyzed by atomic absorption spectroscopy (A.A.). By measuring the amount of light absorbed (at the specific wavelength), a quantitative determination of the amount of iron present can be made.

SAFETY: Each laboratory is responsible for maintaining a current file of the Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

APPARATUS & EQUIPMENT:

- Balance, accuracy to 0.001 g
- pH meter, scale graduated in 0.01 pH units
- Digital diluter (optional) or equivalent pipets
- Vortex shaker
- Acid fume hood
- Air compressor
- Auto sampler
- Atomic Absorption Spectrometer with associated hardware and software (or equivalent)
- Flask, 200 mL volumetric (class "A")
- Beaker, 300 mL

- Nalgene container, 20 liter
- Stopper, for 200 mL volumetric flask
- Disposable culture tube (16 x 125 mm)
- Disposable centrifuge tube (50 mL)
- Glass funnel
- Filter paper (Whatman 113V grade or better)

REAGENTS & CHEMICALS:

- Deionized (D.I.) water
- Hydrochloric acid (HCl) Certified A.C.S. grade or equivalent – **Caution: Strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment**
- **Sodium hydroxide (NaOH) NF/FCC grade or equivalent - Caution: Strong base. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment.**
- Buffer solution (pH 7)
- Buffer solution (pH 10)
- 0.2 N Sodium hydroxide (NaOH)
Weigh 8.0 g of sodium hydroxide to 800 mL of D.I. water. Bring to volume (1 liter) and mix well and allow to cool to room temperature.
Iron stock standard - 1000 ppm Fe in 2% Nitric or Hydrochloric acid
- Stock standards (Comply with UL ISO 9001 Quality Assurance System)
 - A. Copper stock standard - 1000 ppm Cu in 2% Nitric or Hydrochloric acid
 - B. Iron stock standard - 1000 ppm Fe in 2% Nitric or Hydrochloric acid
 - C. Manganese stock standard – 1000 ppm Mn in 2% Nitric or Hydrochloric acid
 - D. Zinc stock standard – 1000 ppm Zn in 2% Nitric or Hydrochloric acid
 - E. Custom Laboratory Internal Standard – 2000 ppm (Ca, Cu, Fe, Mg, Mn, Zn), 500 ppm Mo in 5% Hydrochloric acid

Calibration Standards:

- Calibration standard 4

Iron stock standard	20.0	mL
Hydrochloric acid	20.0	mL
Deionized water (D.I.)		

Pipet 20.0 mL of iron stock standard solution and 20 mL concentrated HCl into 1000 mL flask, bring to volume and mix well.
- Calibration standard 3

Iron stock standard	10.0	mL
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Hydrochloric acid 20.0 mL

Deionized water (D.I.)

Pipet 10.0 mL of iron stock standard solution and 20 mL concentrated HCl into 1000 mL flask, bring to volume and mix well.

- Calibration standard 2

Iron stock standard 5.0 mL

Hydrochloric acid 20.0 mL

Deionized water (D.I.)

Pipet 5.0 mL of iron stock standard solution and 20 mL concentrated HCl into 1000 mL flask, bring to volume and mix well.

- Calibration standard 1

Iron stock standard 0.5 mL

Hydrochloric acid 20.0 mL

Deionized water (D.I.)

Pipet 0.5 mL of iron stock standard solution and 20 mL concentrated HCl into 1000 mL flask, bring to volume and mix well.

SAMPLE HANDLING:

Chelated fertilizers should be analyzed as soon as possible upon receipt.

SAMPLE PREPARATION:

Weigh 1.0 g of sample directly into 200 mL volumetric flask.

1. Bring the sample to volume with D.I. water and shake vigorously for 30 seconds.
2. Allow sample to settle (Sample should be analyzed within one hour).
3. Filter through Whatman 113V grade or better, and collect filtrate (approximately 15 mL) in culture tube.
4. Make 1/50 dilution with D.I. water and transfer to 300 mL beaker.
5. Titrate the sample to pH 8.5 with 0.2 N NaOH. If the pH drifts above 8.8, discard solution and repeat the sample preparation.
6. Filter through Whatman 113V grade or better, and collect filtrate (approximately 10 mL) into culture test tubes.
7. Add 2 to 3 drops of concentrated HCl to culture tube to acidify the solution.
8. Analyze the sample within one hour using flame AA. (No dilution is needed.)

SYSTEM START-UP:

USE STANDARD OPERATING PROCEDURES FOR ALL ELEMENTS BY PERKIN-ELMER AAnalyst 100
