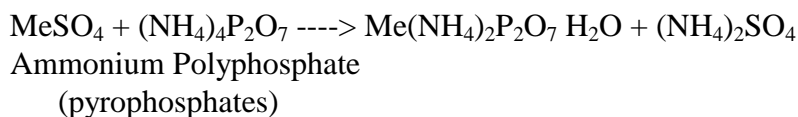
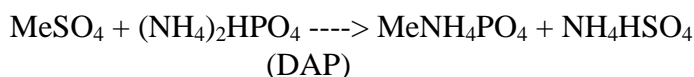


	FM-832	
	FERTILIZER METHODS	Chapter SECONDARY/MICRONUTRIENT ANALYSIS
		Subject Iron – Soluble ~ AA

SCOPE: This is an automated analytical procedure for the determination of soluble iron in mixed or pure material fertilizers. Some examples of soluble iron sources used in fertilizers are: ferrous sulfates, chlorides, nitrates and sucates.

PRINCIPLE: Samples are extracted in a 1% sulfuric acid (H₂SO₄) solution and shaken. After appropriate dilution, samples are analyzed by atomic absorption (A.A.) spectroscopy. By measuring the amount of light absorbed (at the specific wavelength), a quantitative determination of the amount of iron present can be made. Ferric oxides are slightly soluble (1-2%) at this low pH. This low solubility of ferric oxides is not enough to give a positive bias. This extraction solution insures that all of the ferrous sulfate sources are fully recoverable. Because of the extraction solution's low pH value there is no significant loss of soluble iron in samples containing soluble phosphates. The most probable reaction between the soluble micronutrient and the phosphate salt is a recombination reaction which forms a stable (reciprocal) salt pair. This reaction proceeds in an irreversible manner to near completion in which the salt pair is of much lower solubility than the original compounds.



This particular reaction has been documented in Lehr (1972), where he listed a number of such reactions that may take place during the manufacture or storage of fertilizers. Many reactions require only water which is attracted to the hygroscopic fertilizer ingredients. Logic tells us that if a reaction can happen in a bulk product, it can happen when the materials are put into solution for analysis. This must be avoided during analytical procedures.

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:

- Atomic Absorption Spectrometer with associated hardware and software (or equivalent)
- Balance (accuracy to 0.0001 g)
- Digital Diluter or equivalent pipets (Class A)
- Acid fume hood
- Volumetric flasks (Class A 200 mL, 500 mL and 1 L)
- Plastic funnel
- Culture tubes (16 x 125 mm or equivalent)
- Vortex shaker
- Filter column, 40 to 50 micro meter or equivalent
- pH meter (accuracy to 0.01 pH units)

REAGENTS & CHEMICALS:

- Deionized (D.I.) Water
- Sulfuric Acid (H₂SO₄) 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**
1% (v/v) H₂SO₄ / D.I. water Extraction Solution: Add 10 mL of concentrated sulfuric acid to a 1 L volumetric flask containing 100 mL D.I. water, bring to volume with D.I. water and mix solution thoroughly.
- The volume of extraction solution may be adjusted to meet the number of samples.
- 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.**
- Stock standards
 - A. Copper stock standard - 1000 ppm Cu in 2% Nitric acid
 - B. Iron stock standard - 1000 ppm Fe in 2% Nitric acid
 - C. Manganese stock standard – 1000 ppm Mn in 2% Nitric acid

D. Zinc stock standard – 1000 ppm Zn in 5% Nitric acid

E. Custom Laboratory Internal Standard – 2000 ppm (Ca, Cu, Fe, Mg, Mn, Zn), 500 ppm Mo in 5% Hydrochloric acid

- Calibration standard 4

Copper stock standard	20.0	mL
Iron stock standard	20.0	mL
Manganese stock standard	20.0	mL
Zinc stock standard	5.0	mL
Hydrochloric acid, conc.	20.0	mL

D.I. water

To a Liter flask containing 500 mL of D.I. water pipet 20 mL of HCl, pipet 20 mL each of copper, iron, and manganese and pipet 5 mL zinc stock standard solutions into the 1000 mL flask, bring to volume with D.I. water and mix well.

- Calibration standard 3

Copper stock standard	10.0	mL
Iron stock standard	10.0	mL
Manganese stock standard	10.0	mL
Zinc stock standard	3.0	mL
Hydrochloric acid, conc.	20.0	mL

D.I. water

To a Liter flask containing 500 mL of D.I. water pipet 20 mL of HCl, pipet 10 mL each of copper, iron, and manganese and pipet 3 mL zinc stock standard solutions into the 1000 mL flask, bring to volume with D.I. water and mix well.

- Calibration standard 2

Copper stock standard	5.0	mL
Iron stock standard	5.0	mL
Manganese stock standard	5.0	mL
Zinc stock standard	1.0	mL
Hydrochloric acid, conc.	20.0	mL

D.I. water

To a Liter flask containing 500 mL of D.I. water pipet 20 mL of HCl, pipet 5 mL each of copper, iron, and manganese and pipet 1 mL zinc stock standard solutions into the 1000 mL flask, bring to volume with D.I. water and mix well.

- Calibration standard 1

Copper stock standard	0.5	mL
Iron stock standard	0.5	mL
Manganese stock standard	0.5	mL
Zinc stock standard	0.5	mL
Hydrochloric acid, conc.	20.0	mL

D.I. water

To a Liter flask containing 500 mL of D.I. water pipet 20 mL of HCl, pipet 0.5 mL each of copper, iron, manganese and zinc stock standard solutions into the 1000 mL flask, bring to volume with D.I. water and mix well.

SAMPLE**HANDLING:**

For samples containing "free-floating" particles filter a portion of the sample into a culture tube using filter column or better.

SAMPLE**PREPARATION:**

1. Weigh 0.5 g sample into 200 mL volumetric flask containing 100 mL of the 1% (v/v) H₂SO₄ / D.I. water extraction solution when the guarantee is less than 5%. Weigh 0.3 g when the guarantee is greater than 5%. **For pure materials**, weigh 0.3- 0.5 g of sample into 1 L volumetric flask containing 500 mL 1% (v/v) H₂SO₄ / D.I. water extraction solution.
2. Bring the sample to volume with D.I. water and shake vigorously for 30 seconds.
3. **If sample is deficient**, verify total iron by FDACS Fertilizer Method FM-822.

SAMPLE**ANALYSIS:**

1. Make appropriate dilution using dilution chart, analyze on AA.

Dilution Chart

% G	DILUTION
0.01 – 0.30	No dilution
0.31 – 1.50	1/5
1.51 – 2.50	1/10
2.51 – 3.00	1/25
3.01 – 6.00	1/50
> 6.00	1/100

2. A custom internal standard is weighed and analyzed with each set (Fe = .20%) to check the precision and accuracy of the method and the AA.

Weigh 0.5 g of the custom internal standard into a 200 mL volumetric flask containing 100 mL of 1% (v/v) H₂SO₄ / D.I. water extraction solution, bring to volume with D.I. water and analyze with each soluble iron set.

3. AA spectrometer parameters used in Fe analysis:

ELEMENT	BURNER HEAD ALIGNMENT	WAVELENGTH (nm)	SLIT WIDTH (nm)	ACETYLENE FLOW (L/min)	OXIDANT FLOW (L/min)
Fe	Straight	284.3	0.2	1.7	4.0

4. Use D.I. water for blanks in the standardization of the instrument.

SYSTEM START-UP:

See **FSFL-SOP-508: Operation of PE Flame AAanlyst 100.**

QA/QC:

The correlation coefficient (calibration standard) should be 0.999 or higher.

CALCULATION:

% Soluble Fe found = [ppm (AA)] (flask volume) (dilution factor) * 100 / (sample weight) (10⁶ mcg/g)

APPROVAL:

Approved by:



Date: 12/28/09

Signature

Bureau Chief

Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	6/15/98	Replaces M-113	J. Corry
Revised	9/23/09		Jack Andreu

REFERENCE:

*QC Corporation - Baltimore, M.D. (Method# AWWA B402-90)
FSFL-SOP-508*