

## IRON-55 IN WATER

### 1. SCOPE

- 1.1. This is a method for the measurement of Iron-55 in water samples.
- 1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

### 2. SUMMARY OF METHOD

- 2.1. Iron-55 is preconcentrated from water samples using a ferric hydroxide precipitation and separated using Eichrom's TRU Resin column. Up to 2.5mg of stable iron carrier can be added to the sample, as a yield monitor. Once the iron fraction is separated, iron recovery can be determined from a batch yield sample, from chemical recovery of stable iron via atomic emission spectrometry or gravimetrically as an iron hydroxide precipitate. Fe-55 is measured by liquid scintillation counting.

### 3. SIGNIFICANCE OF USE

- 3.1. This is a rapid, reliable method for measurement of Fe-55 in water samples that is more cost-effective and efficient than traditional techniques.

### 4. INTERFERENCES

- 4.1. The maximum amount of iron to be loaded on a 2mL prepacked TRU column is 2.5mg. Samples with high iron content must be aliquotted appropriately, so that the 2.5mg of Fe/sample is not exceeded.

### 5. APPARATUS

- Analytical balance, 0.0001 g sensitivity
- Beakers, glass
- Centrifuge, with rotor and carriers for 50mL and 250mL tubes
- Centrifuge tubes, 50mL and 250mL

- Column rack, Eichrom Part: AC-103
- Vacuum filter apparatus
- Extension funnels, 25 mL, Eichrom Part: AC-120
- Filter, 0.45micron
- Fume hood
- Liquid scintillation counter
- Liquid scintillation vials
- Tweezers

## 6. REAGENTS

**Note: Analytical grade or ACS grade reagents are recommended. Evaluation of key reagents, such as aluminum nitrate and ammonium hydrogen phosphate, for contribution to method background levels from naturally occurring radioactive materials is recommended.**

<i>Sodium hydroxide, NaOH</i>
<i>Deionized water, All reagents are prepared using deionized water</i>
<i>Ferric nitrate, Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O</i>
<i>Isopropyl alcohol, C<sub>3</sub>H<sub>7</sub>OH</i>
<i>Nitric acid (70%), concentrated HNO<sub>3</sub></i>
<i>Phenolphthalein pH Indicator</i>
<i>TRU<sup>®</sup> resin, 2mL prepacked column, 100-150µm, Eichrom Part TR-C50-A</i>

- 6.1. *Iron carrier - (5mg/mL Fe) - Dissolve 3.6g of Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O in 80mL of water. Dilute to 100mL with water.*
- 6.2. *Phenolphthalein solution - dissolve 1g phenolphthalein in 100mL 95% isopropyl alcohol. Dilute with 100mL of water.*
- 6.3. *Nitric acid (2M) - Add 125mL of concentrated HNO<sub>3</sub> to 700mL of water. Dilute to 1L with water.*
- 6.4. *Nitric acid (8M) - Add 500mL of concentrated HNO<sub>3</sub> to 400mL of water. Dilute to 1L with water.*
- 6.5. *Nitric acid (0.5M) - Add 31mL of concentrated HNO<sub>3</sub> to 400mL of water. Dilute to 1L with water.*
- 6.6. *Sodium hydroxide (1M) - Add 20g of NaOH to 400mL of water. Mix to dissolve. Dilute to 500mL with water.*

- 6.7. *Sodium hydroxide (4M)* – Add 80g of NaOH to 400mL of water. Mix to dissolve. Dilute to 500mL with water.

## 7. PROCEDURE

- 7.1. Preconcentration of Fe-55 by ferric hydroxide precipitation
- 7.1.1. If necessary, filter water sample using 0.45µm filter to remove particulates.
  - 7.1.2. Acidify water samples of up to 1L to pH 2 using concentrated HNO<sub>3</sub>.
  - 7.1.3. To each water sample, add 0.75mL of phenolphthalein indicator, and 0.5 mL of 5mg/mL Fe carrier.

**Note:** *Fe is added as a yield monitor and to preconcentrate Fe-55 using a ferric hydroxide precipitation.*

- 7.1.4. Mix samples thoroughly and heat to 60-80°C for 15-20 minutes.
- 7.1.5. Allow samples to cool to room temperature.
- 7.1.6. While stirring, slowly add enough 1M NaOH to reach the pink phenolphthalein indicator endpoint.

**Note:** *NaOH should be used to adjust the pH. Adjusting the pH with NH<sub>4</sub>OH can lead to lower recoveries of iron during the ferric hydroxide precipitation.*

- 7.1.7. Allow precipitate to settle until supernate can be decanted or proceed to next step and centrifuge samples for quicker separation.
- 7.1.8. Transfer samples, including precipitate to 250mL centrifuge tubes. Centrifuge samples at 2000rpm for 10-20 minutes. Decant supernate. Repeat until entire sample has been transferred to centrifuge tube and centrifuged.
- 7.1.9. Rinse sample beaker with 10mL of deionized water. Transfer rinse to the appropriate centrifuge tube. Centrifuge samples for 10 minutes at 2000rpm. Decant supernate.
- 7.1.10. Dissolve precipitate in 5mL of concentrated HNO<sub>3</sub>.
- 7.1.11. Transfer dissolved samples to 15mL centrifuge tubes.
- 7.1.12. Rinse 250mL centrifuge tubes with 5mL deionized water.

- 7.1.13. Transfer rinses to the appropriate 15mL centrifuge tubes. Cap 15mL centrifuge tubes and mix.
- 7.2. Iron separation using Eichrom TRU Resin column
- 7.2.1. For each sample, place a TRU Resin column in the column rack. Attach a column reservoir to each column.
- 7.2.2. Place a beaker or a waste tray below each column. Remove the bottom plug from each column and allow to drain.
- 7.2.3. Add 5mL of 8M HNO<sub>3</sub> into each column reservoir. Allow solution to drain through columns.
- 7.2.4. Load sample in 8M HNO<sub>3</sub> on the column. Allow solution to pass through column.
- 7.2.5. Rinse sample centrifuge tubes with 5mL 8M HNO<sub>3</sub>. Add tube rinse to column reservoir. Allow solution to pass through column.
- 7.2.6. Rinse the column with 10mL of 8M HNO<sub>3</sub>. Allow solution to pass through column.
- 7.2.7. Place a clean, labeled 50mL centrifuge tube below each column. Elute Fe with 15mL of 2M HNO<sub>3</sub>.
- 7.2.8. Slowly add 4M NaOH until ferric hydroxide precipitate forms (pH 4-9).
- 7.2.9. Determine Fe yield using a batch recovery sample, atomic emission spectrometry (Steps in 7.3.) for stable iron or gravimetric recovery of stable iron (Steps in 7.4.).
- 7.3. Iron recovery by atomic emission spectrometry
- 7.3.1. Centrifuge at 2000rpm for 5-10 minutes.
- 7.3.2. Decant supernate.
- 7.3.3. Dissolve sample in 5mL of 0.5M HNO<sub>3</sub>.
- 7.3.4. Remove a 0.25mL aliquot of each sample from step to a 10mL volumetric flask for stable iron yield analysis by atomic emission spectrometry.
- 7.3.5. Dilute yield sample aliquot to 10mL with 0.5M HNO<sub>3</sub>.
- 7.3.6. Analyze sample for iron using atomic emission spectrometry.

- 7.3.7. Transfer remaining sample to a 20mL liquid scintillation vial.
- 7.3.8. Add 15mL of liquid scintillation cocktail.
- 7.3.9. Measure Fe-55 using liquid scintillation counting.
- 7.4. Iron recovery by gravimetric determination of stable iron
- 7.4.1. Filter the solution from 7.2.7 through a 25mm diameter glass fiber filter using a filter apparatus.
- 7.4.2. Dry the filter under a heat lamp completely.
- 7.4.3. Weigh the filter and record the weight.
- 7.4.4. Place the glass fiber filter in a 20mL liquid scintillation vial.
- 7.4.5. Add 15mL of liquid scintillation cocktail and count Fe-55 using a liquid scintillation counter.

## 8. CALCULATIONS

### *Atomic Emission Spectrometry Recovery of Fe Carrier*

$$\text{Yield} = \frac{C_{\text{meas}}}{7.5 \text{ ppm}}$$

where:

$C_{\text{meas}}$  = concentration in ppm measured by atomic emission spectrometry

7.5 ppm = Theoretical concentration in ppm for 3 mg stable Fe, 0.25mL aliquot of 5.0mL total, diluted to 10mL.

### *Gravimetric: Fe carrier*

$$\text{Yield} = \frac{(R_w - T_w - B_w)}{C_w}$$

where:

$R_w$  = residue+filter, mg

$T_w$  = tare weight of filter, mg

$B_w$  = blank weight, mg (extractant loss from column)

$C_w$  = Expected FeOH, mg (This is the wt. of Fe carrier added and then converted to FeOH equivalent wt.)

Fe-55 activity

$$A = \frac{(S-B)}{E \times V \times Y \times 60} \times \left( \frac{5}{4.75} \right)$$

where:

- A = Fe-55 sample activity (Bq/L)  
S = Fe-55 measured activity, cpm  
B = blank activity, cpm  
E = counting efficiency  
V = sample volume, liters  
Y = Fe-carrier yield  
60 = Conversion from cpm to cps

## 9. REFERENCES

- 1) E Philip Horwitz, Renato Chirizia, Mark L. Dietz, Herbert Diamond, and Donald M. Nelson. Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography. *Analytica Chimica Acta*, 281 (1993) 361.
- 2) Pacific Northwest Laboratory Method PNL-ALO-345, Iron-55 Analysis
- 3) ASTM Method D49922-09, "Standard Test Method for the determination of radioactive iron in water."

