AP13

DETERMINATION OF IRON-55 IN WATER

PART A

PRINCIPLE

Iron-55 (Fe-55) is separated from other isotopes by using a selective chromatographic resin (TRU® Resin from Eichrom Technologies, Darien, IL). Iron and the actinides are loaded on the column in 8 M HNO_3. Iron is eluted from the column with 2 M HNO_3. Under these conditions, the actinides are retained on the column. The Fe-55 activity is determined via liquid scintillation counting. A batch yield is used to determine chemical recovery. The maximum amount of iron that can be loaded on the column is 3 mg.

REFERENCE

1. Eichrom Technologies, Inc., Analytical Procedures, "Iron-55 in Water", April 30, 2001.

2. Backstrom, Guy, personal communication, 2006.

Certification Record for

AP13
DETERMINATION OF IRON-55 IN WATER

		CHECKPOINTS
1.	JOB HAZARD ANALYSIS (JHA)	_____
2.	MSDS/HAZARDS DISCUSSED	_____
3.	SAMPLE DIGESTION	_____
4.	COLUMN SEPARATION	_____
5.	COUNTING PREPARATION	_____
6.	FINAL CALCULATIONS	_____

ANALYST SIGNATURE: _____

CERTIFIED BY: _____

DATE: _____

ANALYSIS VALUE: _____

KNOWN VALUE: _____

MEASURED/KNOWN RATIO: _____

See Task _____, Batch_____ for the original data.

COMMENTS:_____
_____
_____
_____

PART B

1.0 PURPOSE AND SCOPE

This procedure provides the analytical method for determination of Fe-55 in water samples.

2.0 REAGENTS

All chemicals are hazardous. See MSDS for specific precautions. **See step 2.0 of AP12 JHA.** Unless otherwise indicated, all references to water should be understood to mean reagent grade water.

Ammonium hydroxide, NH_4OH, concentrated.

Fe-55, NIST traceable standardized solution.

Hydrochloric acid, HCl, concentrated, 12 M.

Hydrochloric acid, 1 M: Add 83 mL concentrated HCl to 900 mL water and dilute to 1 L with water.

Hydrochloric acid, 0.5 M: Add 250 mL 1 M HCl to 250 mL water and mix.

Iron carrier, 1 mg/mL, AA quality.

Liquid scintillation cocktail - Packard Instrument Ultima Gold-LLT, or equivalent.

Nitric acid, HNO_3, concentrated, 16 M.

Nitric acid, HNO_3, 8 M: Add 500 mL of concentrated HNO_3 to 300 mL water and dilute to 1 L with water.

Nitric acid, HNO_3, 2 M: Add 127 mL of concentrated HNO_3 to 700 mL water and dilute to 1 L with water.

TRU® resin, 2 mL pre-packed cartridge, 50-100 μm particle size resin.

3.0 APPARATUS

Analytical balance
Beakers, various sizes
Centrifuge tubes
Filters, 0.45 μm
Fume hood
Hot plate

Liquid scintillation analyzer (LSA)
Liquid scintillation vials
Syringe, 10 mL
Transfer pipette
Vacuum box assembly
Vacuum pump

4.0 PROCEDURE

4.1 General Requirements

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program (QP) Manual. See page two for a copy of the certification record.

A batch yield sample must be run with each batch to determine chemical recovery for the batch (see below for calculations). This is not a QC sample; two QC samples must be run with each batch.

4.2 Iron Separation Using TRU® resin

4.2.1 Filter the sample through a 0.45 μm filter unless otherwise stated in the Laboratory Work Request (LWR). Measure 0.025 to 0.25 L of the sample into an appropriate size beaker. Add 1 mL Fe carrier to each sample, blank, Laboratory Control Standard (LCS), and batch yield sample. Add a known amount of Fe-55 to the LCS and batch yield sample. **See step 4.2.1 of AP13 JHA**.

4.2.2 Add 20 mL concentrated HNO_3 to each sample and evaporate to near dryness (~1-2 mL). Remove from hot plate and allow to cool. **See step 4.2.2 of AP13 JHA**.

4.2.3 For each sample, place a TRU® resin cartridge in the vacuum box assembly.

4.2.4 Place a 10 mL syringe on each cartridge.

4.2.5 Condition each cartridge by adding 5 mL 8 M HNO_3 and allow the solution to drain into a waste container. **See step 4.2.5 of AP13 JHA**.

4.2.6 Add 20 mL 8 M HNO_3 to each sample from step 4.2.2. **See step 4.2.6 of AP13 JHA**.

4.2.7 Load the samples from step 4.2.6 onto the TRU® cartridge. Allow solution to drain into a waste container at 1 to 2 mL per second. A yellow color will appear on the cartridge. **See step 4.2.7 of AP13 JHA**.

4.2.8 Rinse each beaker with 5 mL 8 M HNO_3 and pour onto cartridge. Allow rinse to drain into a waste container. **See step 4.2.8 of AP13 JHA**.

4.2.9 Rinse the column with 10 mL 8 M HNO_3 and allow the solution to drain into a waste container. Pour the waste liquid into the dilute acid waste container. **See step 4.2.9 of AP13 JHA**.

4.2.10 Place a labeled centrifuge tube under each cartridge.

4.2.11 Strip the Fe with 15 mL of 2 M HNO_3. **See step 4.2.11 of AP13 JHA**.

4.2.12 Add 10 mL of concentrated NH_4OH to precipitate the iron as iron hydroxide and digest in a hot water bath for 15 minutes. **See step 4.2.12 of AP13 JHA**.

4.2.13 Centrifuge the sample(s) at 2000 rpm for 10 minutes and then decant the supernate. **See step 4.2.13 of AP13 JHA**.

4.2.14 Add 3 mL of 0.5 M HCl to dissolve the precipitate and heat in a water bath for 10 minutes. **See step 4.2.15 of AP14 JHA**.

4.2.15 Transfer the solution to a glass scintillation vial and add 17 mL of scintillation cocktail. Mix the solution with vigorous shaking. **See step 4.2.15 of AP13 JHA**.

4.2.16 Submit samples for scintillation counting.

5.0 CALIBRATIONS

5.1 Instrument Background Preparation

5.1.1 Add 1 mL Fe carrier and 15 mL 2 M HNO_3 to a centrifuge tube.

5.1.2 Add 10 mL concentrated NH_4OH to precipitate iron hydroxide. Heat in a hot water bath for 10 minutes. **See step 4.2.12 of AP13 JHA**.

5.1.3 Centrifuge the sample(s) at 2000 rpm for 10 minutes and then decant the supernate. **See step 4.2.13 of AP13 JHA**.

5.1.4 Add 3 mL of 0.5 M HCl to dissolve the precipitate and heat in a water bath for 10 minutes. **See step 4.2.15 of AP14 JHA**.

5.1.5 Transfer the solution to a glass scintillation vial and add 17 mL of scintillation cocktail. Mix the solution with vigorous shaking. **See step 4.2.15 of AP13 JHA**.

5.2 Instrument Efficiency Standard Preparation

5.2.1 Add 1 mL Fe carrier and 15 mL 2 M HNO_3 to a centrifuge tube.

5.2.2 Add Fe-55 standard to the vial. **See step 4.2.1 of AP13 JHA**.

5.2.3 Add 10 mL concentrated NH_4OH to precipitate iron hydroxide. Heat in a hot water bath for 10 minutes. **See step 4.2.12 of AP13 JHA**.

5.2.4 Centrifuge the sample(s) at 2000 rpm for 10 minutes and then decant the supernate. **See step 4.2.13 of AP13 JHA**.

5.2.5 Add 3 mL of 0.5 M HCl to dissolve the precipitate and heat in a water bath for 10 minutes. **See step 4.2.15 of AP14 JHA**.

5.2.6 Transfer the solution to a glass scintillation vial and add 17 mL of scintillation cocktail. Mix the solution with vigorous shaking. **See step 4.2.15 of AP13 JHA**.

5.2.7 The Laboratory Manager must review and approve the counting efficiency.

6.0 CALCULATIONS

Critical data values will be documented on standard forms maintained as critical records. The following equations define the critical data values. All data will be recorded and reduced according to these calculations.

$$Concentration = \frac{G - B}{E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma\ Error = \frac{1.96\sqrt{(G + B) \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

$$2\sigma\ TPU = C \cdot 1.96\sqrt{\frac{(G + B) \cdot T}{((G - B) \cdot T)^2} + RE^2 + RY^2 + RQ^2} = pCi / unit$$

$$MDC = \frac{3 + 4.65\sqrt{B \cdot T}}{T \cdot E \cdot Y \cdot Q} = pCi / unit$$

To calculate efficiency:

$$E = \frac{G_E - B}{A} = cpm / pCi$$

To calculate batch yield:

$$Y = \frac{G_{BY} - G}{E \cdot AY} = no\ units$$

where:

A	=	activity in pCi for efficiency determination
AY	=	activity in pCi for yield determination
B	=	background cpm beta
C	=	concentration in pCi/unit
E	=	counting efficiency (cpm/pCi)
G	=	sample gross cpm beta
G_{BY}	=	batch yield gross cpm beta
G_E	=	efficiency gross cpm beta
MDC	=	minimum detectable concentration
Q	=	quantity
RE	=	1σ relative uncertainty of the efficiency
RQ	=	1σ relative uncertainty of the quantity
RY	=	1σ relative uncertainty of the yield
T	=	time minutes
TPU	=	total propagated uncertainty
Y	=	chemical yield

7.0 RECORDS

7.1 Reference QP Manual for general record requirements.

7.2 The raw count data are saved during the weekly backup of the LSA to the ORISE network disks.

7.3 Hard copies of assignment and calculation sheets are maintained in the archived site file. Electronic copies of assignment and calculation sheets are saved during the daily incremental backup of the network system. The following data sheets show the required data and information. These forms or the equivalent should be completed and retained:

- Fe Analysis Assignment Form
- Fe Lab Data Sheet
- Fe Concentration and Uncertainty Report (This report may be generated using approved Excel spreadsheets or from the database, if available.)

AP13 (Rev 5) - Fe-55 ANALYSIS ASSIGNMENT FORM

Assigned To: _____ Date: _____ Batch: _____

Task #: _____ LWR #: _____ Activity Level*: _____

Sample #s: _____

_____

_____

Analysis Required:

Batch Yield ☐ Sample # _____
Initial below sample

Fe-55 STD # _____ Quantity: _____
Units: _____

Eff. Spike ☐ Fe-55 STD # _____ Quantity: _____
(see Special Instructions, if any) Units: _____

QC Required:

Blank ☐

LCS ☐ Fe-55 STD # _____ Quantity: _____ Initials ☐
Units: _____

Pipette # _____ Volume (mL) _____ Weight (g) _____

Replicate ☐ Sample # _____ # Replicates: _____

Matrix Spike ☐ Sample # _____

Fe-55 STD # _____ Quantity: _____ Initials ☐
Units: _____

1 mg Fe CARRIER ADDED? Initials ☐

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

* If Activity Level is indicated as Moderate or High, perform area survey.

COMMENTS: _____

_____

_____

AP13 (Rev 5) - Fe-55 LAB DATA SHEET

			BATCH YIELD	SAMPLE			
Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #							
Quantity							
Units							

Sample #			
Quantity			
Units			

AP13(Rev 5) - Iron-55 (by batch yield) Concentration and Uncertainty Report

INPUT BY:

DATE:

TASK#

BATCH#

Batch Yield (BY) Calculation	
BY sample ID	
BY sample cpm	
BY Sample Quantity (SQ)	
BY SQ error	
Sample cpm	
SQ	
SQ error	
BY pCi added	
BY pCi added error	
BY	
BY Error	
BY Relative Error	

Efficiency (Eff) Calculation	
Eff spike cpm	
Background cpm	
pCi added	
pCi added error	
Eff (cpm/pCi)	
Eff Error (cpm/pCi)	
Eff Relative Error	

Counting time for Eff and BYcalculations (min)	

Position #	SAMPLE ID	GROSS cpm	SQ	SQ ERROR	UNITS	TIME (min)	CONC.	TPU	4.65 sigma MDC
1									
2									
3									
4									
BY									
BY Sample									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									

Fe-55 Known Activity	Unc.	Meas./ Known	Unc.

BLANK CORRECT? YES[ ] NO[ ] INIT_____

LCS CORRECT? YES[ ] NO[ ] INIT_____

BATCH YIELD CORRECT? YES[ ] NO[ ] INIT_____

IF NO, SPECIFY REASON:

ANALYST REVIEW: _____ DATE: _____

REVIEWED BY: _____ DATE: _____

GIVEN TO: _____ DATE: _____

QC ENTERED BY: _____ DATE: _____