

Measurement of ^{55}Fe in Water (TEVA Separation)

Summary of Method ^{55}Fe is separated and measured from up to 1L aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation, dissolved in 4M HCl and loaded onto 2mL cartridges of TEVA Resin. Hold back carriers, 2mg each of Zn, Mn, Cs, Nb, Zr, and Co are added to improve separation from radionuclides of these elements. An iron phosphate precipitate at pH 2.8-3.2 is used to prepare samples for liquid scintillation counting and remove remaining traces of Zn, which can co-elute with iron from TEVA resin. Chemical recovery of iron is determined by ICP-AES measurement of 5mg of stable iron carrier. ^{55}Fe may also be determined using TRU resin, AN-1612 from nitrate media. AN-1612 allows ^{55}Fe incorporation into standard TEVA-TRU actinide separations methods, but is limited to 2mg Fe per sample for a 2mL TRU resin cartridge.

Reagents

TEVA Resin Cartridges (Eichrom TE-R50-S)
Deionized Water
Sodium Hydroxide
HCl
 HNO_3
 H_3PO_4
LSC Cocktail
Fe, Zn, Mn, Cs, Nb, Zr, Co carriers (10mg/mL)
Phenolphthalein pH indicator
 ^{55}Fe standard
Nitromethane or other LSC quench agent

Equipment

Vacuum Box (Eichrom AR-12-BOX or AR-24-BOX)
Vacuum Box Inner Liner (Eichrom AR-12-LINER or AR-24-LINER)
Yellow Outer Tips (Eichrom AR-1000-OT)
Inner Support Tube (Eichrom AR-1000-TUBE-PE)
Cartridge Reservoirs (Eichrom AR-200-RV20)
Centrifuge Tubes - 50mL and 250mL
20mL glass liquid scintillation tubes
Liquid scintillation counter
Calibrated pipets and disposable tips
Appropriately Sized Glass Beakers
ICP-AES system for Fe chemical yield measurement
Analytical balance
Vacuum Pump
Centrifuge
Hotplate

Sample Preparation

Up to 1L of water sample in glass beaker.

Add 5 mg Fe, 2mg Zn, Mn, Cs, Nb, Zr, Co

Evaporate to dryness or proceed to ferric hydroxide precipitation steps below.

Ferric Hydroxide Precipitation

Add pH indicator

Heat sample to 80°C

Adjust to pH 8-9 with NaOH.

Mix sample and allow to cool to room temperature.

Allow ppt to settle and decant supernate to <200mL.

Transfer to 250mL centrifuge tube. Rinse beaker with water to ensure complete transfer of ppt.

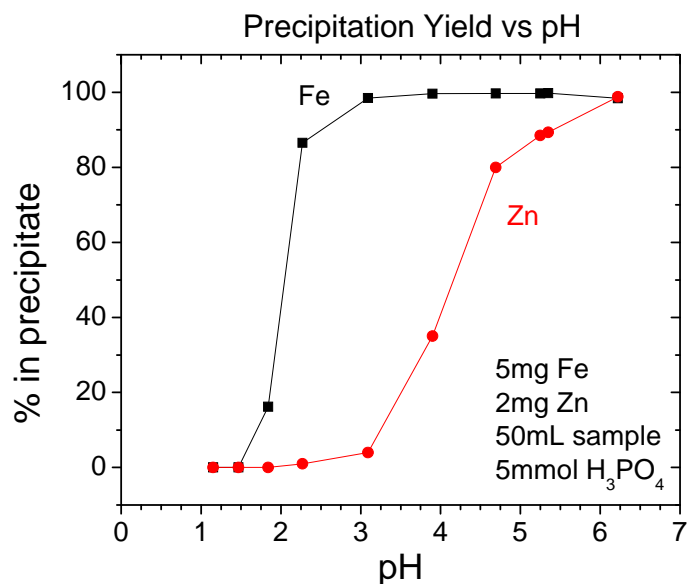
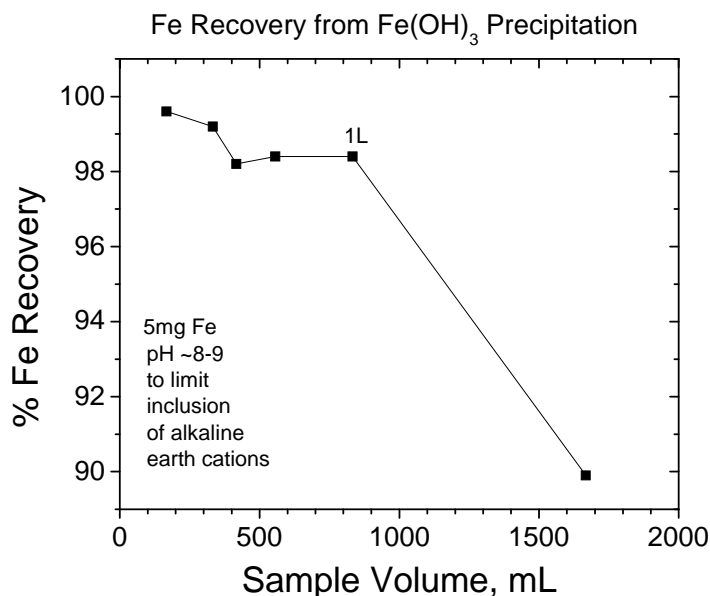
Centrifuge 10min. Decant supernate to waste.

Rinse ppt w/ 50mL Water. Centrifuge. Decant supernate.

Dissolve ppt/residue in 20mL 4M HCl.

Iron Separation

- | | |
|---|--|
| <ol style="list-style-type: none"> 1) Set up vacuum box with TEVA cartridges. 2) Precondition with 5mL 4M HCl. 3) Load samples on TEVA Resin. 4) Rinse tube with 5mL 4M HCl. Add to TEVA. 5) Rinse TEVA with 10mL 4M HCl. 6) Strip Fe from TEVA with 20mL 0.1M HNO₃. 7) Add 5mL 1M H₃PO₄. Mix. 8) Adjust to pH 2.8-3.2 with NaOH/H₃PO₄. Mix. | <ol style="list-style-type: none"> 9) Centrifuge. Decant Supernate. 10) Wash ppt with 50mL H₂O. Centrifuge. Decant Supernate. 11) Dissolve ppt with minimal 6M HCl. 12) Transfer to 10mL volumetric flask. Dilute to 10mL. 13) Take 0.1-0.2 mL, dilute to 10mL for ICP-AES Fe yield. 14) Transfer balance of sample to 20mL glass LSC vial. 15) Add 6 drops H₃PO₄. Evap. on hotplate to ~0.5mL. 16) Add 1mL H₂O. Cool. Add 15mL LSC cocktail. Mix. |
|---|--|



Method Performance

Method	Replicate	%Rec		Fe-55 Tracer corrected	Bias	Impurity*
		2mg Fe tracer	Fe-55 raw %rec			
TEVA	1	95.8	89.2	93.1	-6.9	<0.5%
	2	94.4	89.7	95.0	5.0	
	3	97.6	87.2	89.4	10.6	
	4	95.3	88.2	92.6	7.4	
	5	83.9	79.8	95.1	4.9	
	6	89.1	89.6	100.5	-0.5	
	7	80.6	86.4	107.2	-7.2	
	AVG	91.0	87.2	96.1		
	SD	6.6	3.5	5.9		

References

- 1) ASTM Method D4922. "Standard Test Method for Determination of Radioactive Iron in Water."