

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

Summary of Method ^{55}Fe is separated and measured from up to 500mL aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation and purified on 2mL cartridges of TRU Resin. Holdback carriers, 0.1-1mg each of Zn, Mn, Cs, Nb, Zr, and Co are added to improve separation from these nuclides of these elements. An iron phosphate precipitate is used to prepare samples for liquid scintillation counting. Chemical recovery of iron is determined by ICP-AES measurement of 2mg of stable iron carrier. ^{55}Fe may also be determined from chloride media using TEVA resin (Eichrom AN-1611). AN-1612 provides higher Zn decontamination and can be incorporated into TEVA-TRU actinide separations, but is limited to 2mg total Fe per 2mL cartridge. AN-1611 can process 5-6mg of Fe, but is less rugged for Zn decontamination.

Reagents

TRU 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 2mg Fe. 1mg Zn, Mn, Cs, Co. 0.1mg Nb, Zr .

Evaporate to dryness or proceed to $\text{Fe}(\text{OH})_3$ ppt 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.

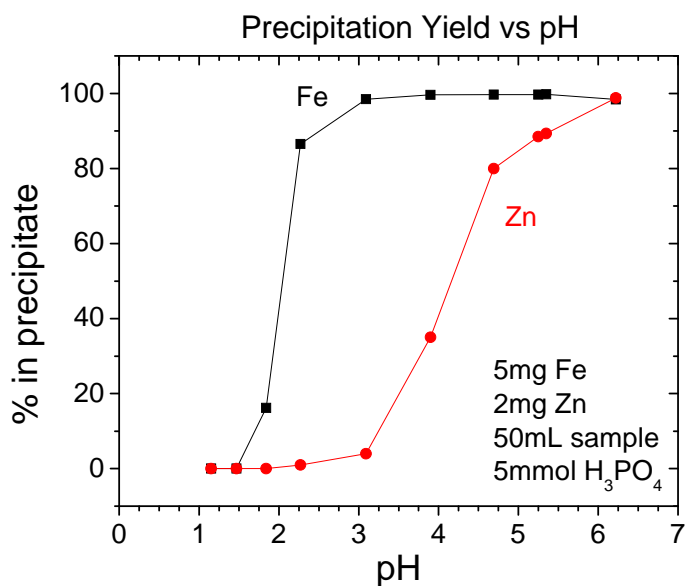
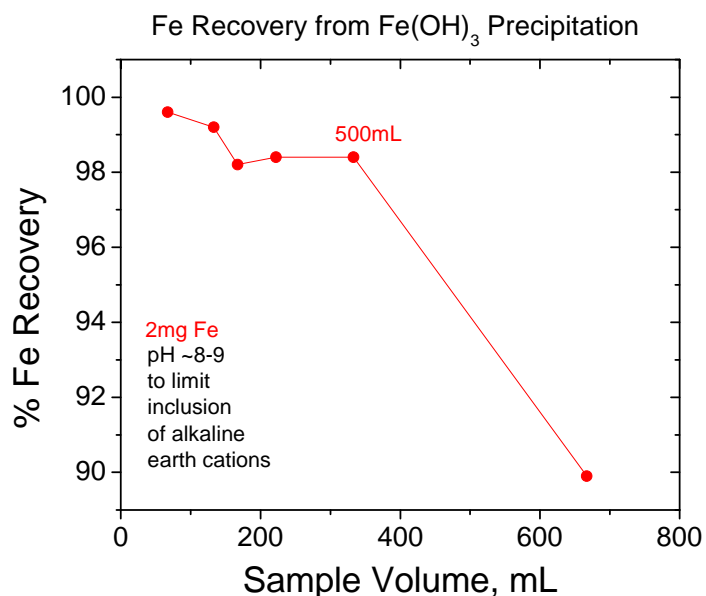
Centrifuge 10min. Decant supernate to waste.

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

Dissolve ppt/residue in 10mL 8M HNO_3 .

Iron Separation

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| <ol style="list-style-type: none"> 1) Set up vacuum box with TRU cartridges. 2) Precondition with 5mL 8M HNO₃. 3) Load samples on TRU Resin. 4) Rinse tube with 5mL 8M HNO₃. Add to TRU. 5) Rinse TRU with 10mL 8M HNO₃. 6) Strip Fe from TRU with 15mL 2M HNO₃. 7) Add 5mL 1M H₃PO₄. Mix. 8) Adjust to pH 2.8-3.2 with NaOH. 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. |
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Method Performance

Method	Replicate	%Rec		Fe-55 Tracer corrected	Bias	Impurity*
		2mg Fe tracer	Fe-55 raw %rec			
TRU	1	90.6	93.1	102.8	2.8	<0.5%
	2	90.0	92.3	102.5	2.5	
	3	94.8	92.4	97.5	-2.5	
	4	89.5	94.0	105.0	5.0	
	5	95.8	94.3	98.5	-1.5	
	6	95.8	92.8	96.9	-3.1	
AVG		92.8	93.2	100.5		
SD		3.0	0.8	3.3		

References

- 1) Eichrom Method FEW01VBS. "Iron-55 in water," <http://www.eichrom.com/eichrom/radiochem/methods/eichrom/>