

Summary of Method Nickel-59/63 is separated and measured from up to 500mL aliquots of water. Samples are preconcentrated by evaporation or ferric hydroxide precipitation, dissolved in 1M HCl, buffered with ammonium citrate and adjusted to pH 8-9 with ammonium hydroxide. Citrate complexes Fe(III), preventing precipitation at pH 8-9. Nickel is loaded onto 2mL cartridges of Nickel Resin. Yields can be improved by adding a 2mL cartridge of prefilter resin below the Nickel cartridge to minimize losses of the Ni-DMG complex. Nickel is recovered in 3M HNO₃ and measured by liquid scintillation counting. Chemical recovery of nickel is determined by ICP-AES measurement of 1-2mg of stable nickel carrier.

Reagents

Nickel Resin Cartridges (Eichrom NI-R50-S)
 Prefilter Resin Cartridges (Eichrom PF-R50-L)
 Deionized Water
 Ammonium Citrate
 Ammonium Hydroxide
 Sodium Hydroxide
 HCl
 HNO₃
 Iron(III) Carrier (10mg/mL)
 Nickel Carrier (10mg/mL)
 Phenolphthalein pH indicator
 Liquid Scintillation Cocktail

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 Ni chemical yield measurement
 Analytical balance
 Vacuum Pump
 Centrifuge
 Hotplate

Sample Preparation

Up to 500mL of water sample in glass beaker.

Add 1-2 mg of Ni carrier

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

Ferric Hydroxide Precipitation

Add 2mg of Fe(III) carrier and 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.

Load Solution Preparation

- 1) Dissolve ppt/residue in 5-10mL 1M HCl.
- 2) Add 1-2mL of 1M ammonium citrate.
- 3) Add pH indicator.
- 4) Adjust to pH 8-9 with ammonium hydroxide.
- 5) If ppt. forms, add additional ammonium citrate.



Nickel Separation

- 1) Set up vacuum box with Nickel cartridges.***
- 2) Precondition with 5mL 0.2M ammonium citrate.
- 3) Load samples on Nickel/Prefilter Resin.
- 4) Rinse Nickel/Prefilter Resin with 20mL 0.2M ammonium citrate.
- 5) Strip Ni with 10-15mL of 3M HNO₃.
- 6) Take aliquots for ICP-AES and Liquid Scintillation.

***optional: prefilter cartridges below Ni to improve yield.

References

- 1) Eichrom Method NIW01VBS. "Nickel-59/63 in water," <http://www.eichrom.com/eichrom/radiochem/methods/eichrom/>