

# TestAmerica



THE LEADER IN ENVIRONMENTAL TESTING

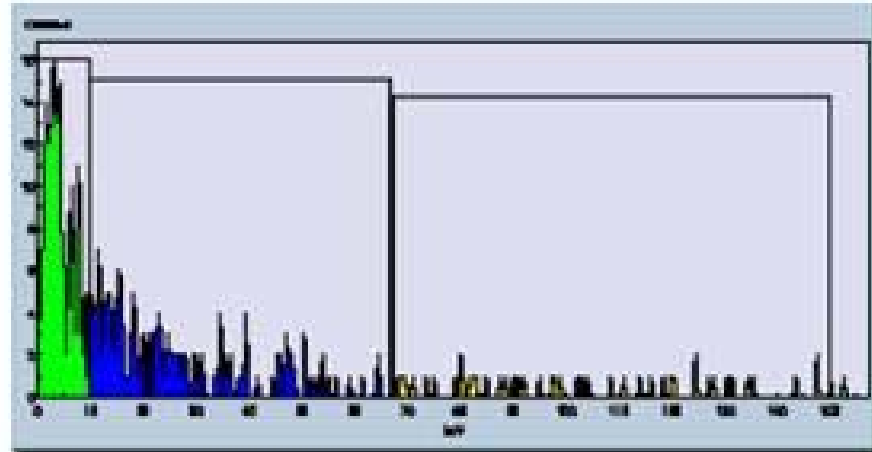
## Removal of Tc-99 Interference from Ni-63 Analysis of Water Sample

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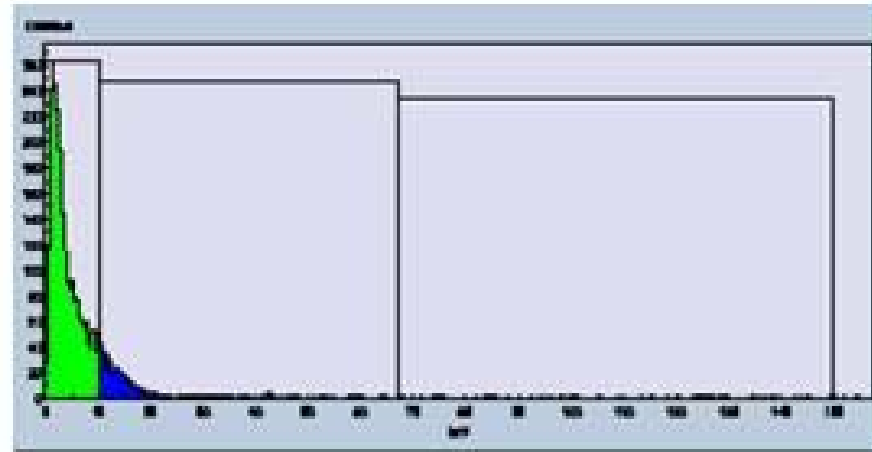
- **Client water sample submitted for analysis of Ni-63 exhibited unusual peak/shape in LSC spectrum**
- **Desire to report without interference**
- **Ni-63**
  - ~ 100 year half-life
  - ~ Beta emitter
  - ~ 65.87 keV Max; 17.13 keV Avg

# Initial Spectra

The MB:

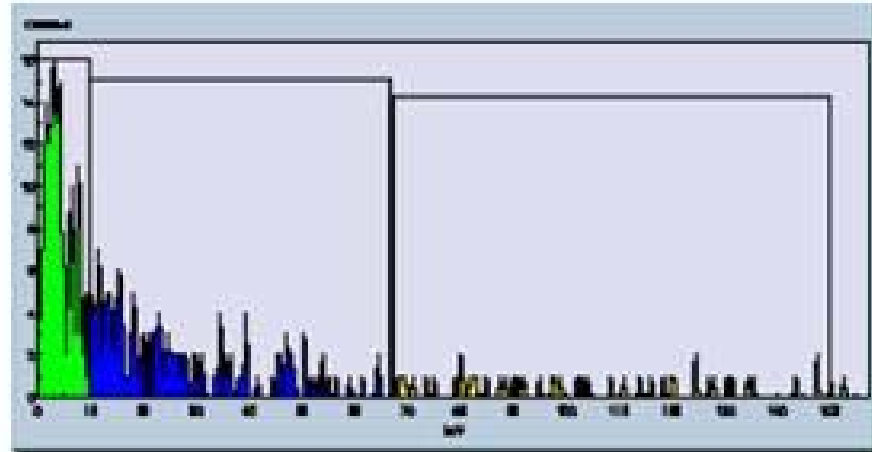


The LCS:

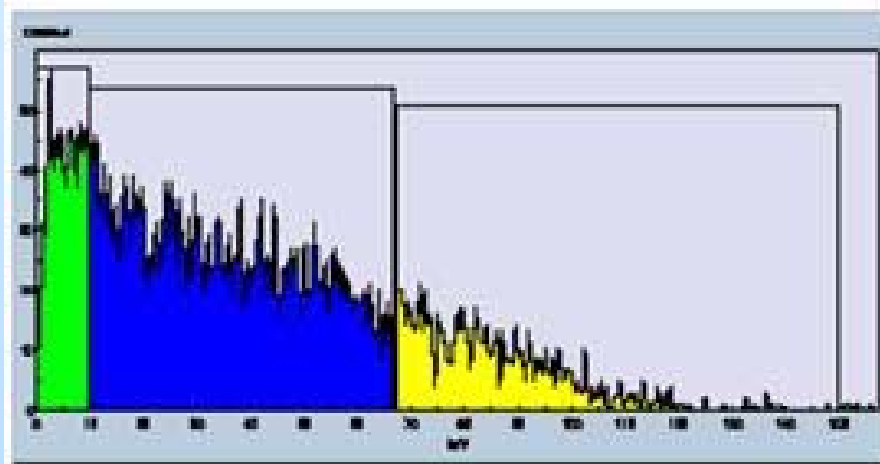


# Initial Spectra

The MB:



The SMP:



## What do we know?

- **Client unable to provide much information regarding chemical composition of this well water**
- **No time to perform characterization**
- **Spectrum similar to Tc-99 (different cocktail mix)**
- **Well has shown elevated gross beta and Tc-99 in past. No Sr-90 of note.**
- **Nitrate < 7 mg/L; VOA “ND”**

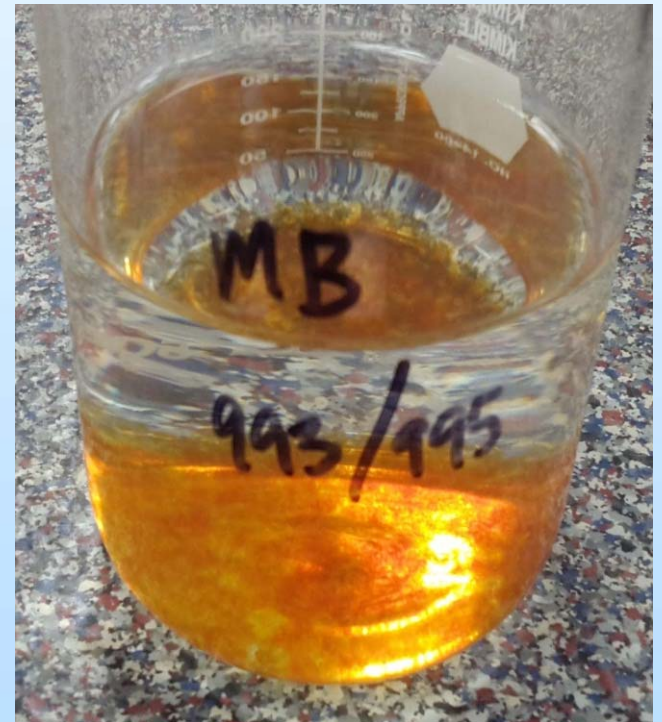
- **We will proceed with (fairly good) assumption that interference is caused by Tc-99**
- **Assume that typical chemistry is used.**

## Ni-63 Water Prep

- 500 mL sample (preserved)
- Ni and Fe carriers
- Spike LCS (Ni-63)



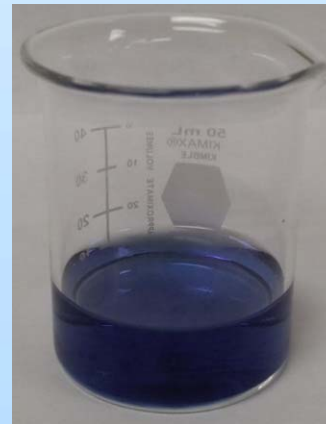
- Hydroxide precipitation
  - ~ Heat near boiling
  - ~ Slowly add 10M NaOH to pH ~8-9
- Centrifuge/discard supernate





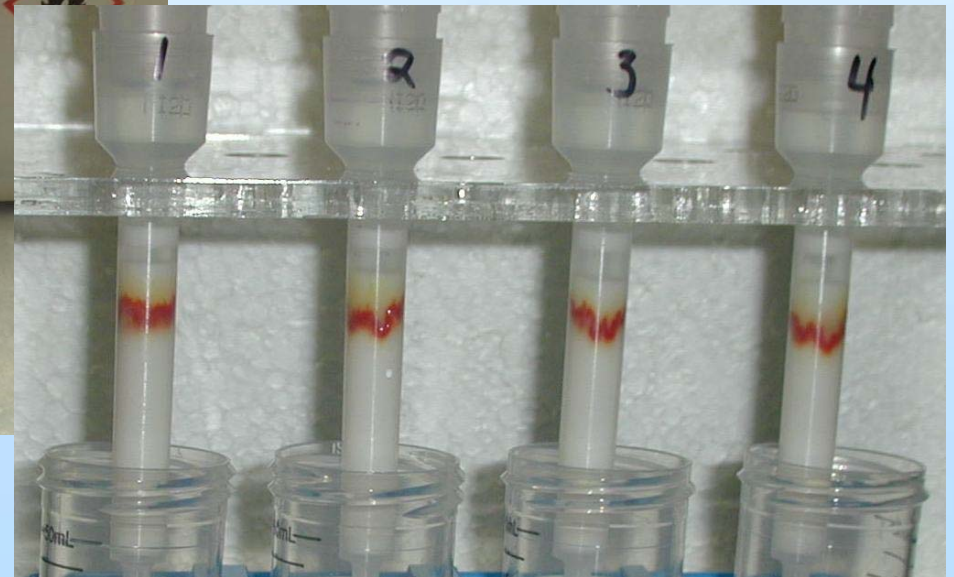
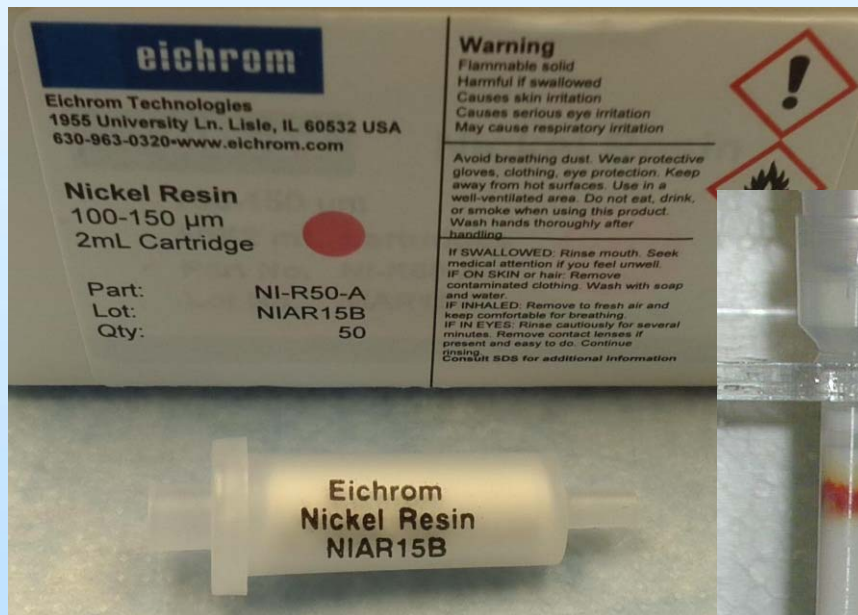
## Ni-63 Water Prep (cont'd)

- Dissolve sample residue in 10 mL 1M HCl
- Add 1 mL 1M ammonium citrate (dibasic)
  - ~ Gently heat, then allow to cool
- Add drop(s) 4% thymol blue
  - ~ Adjust pH to ~8-9 w/  $\text{NH}_4\text{OH}$  (blue color)
  - ~ +1mL 1M  $\text{C}_6\text{H}_{14}\text{N}_2\text{O}_7$  if still cloudy



## Ni-63 Water Prep (cont'd)

- Load onto conditioned (pH 8-9 H<sub>2</sub>O) Ni cartridge
- Rinse w/ 10 mL 0.2M C<sub>6</sub>H<sub>14</sub>N<sub>2</sub>O<sub>7</sub>

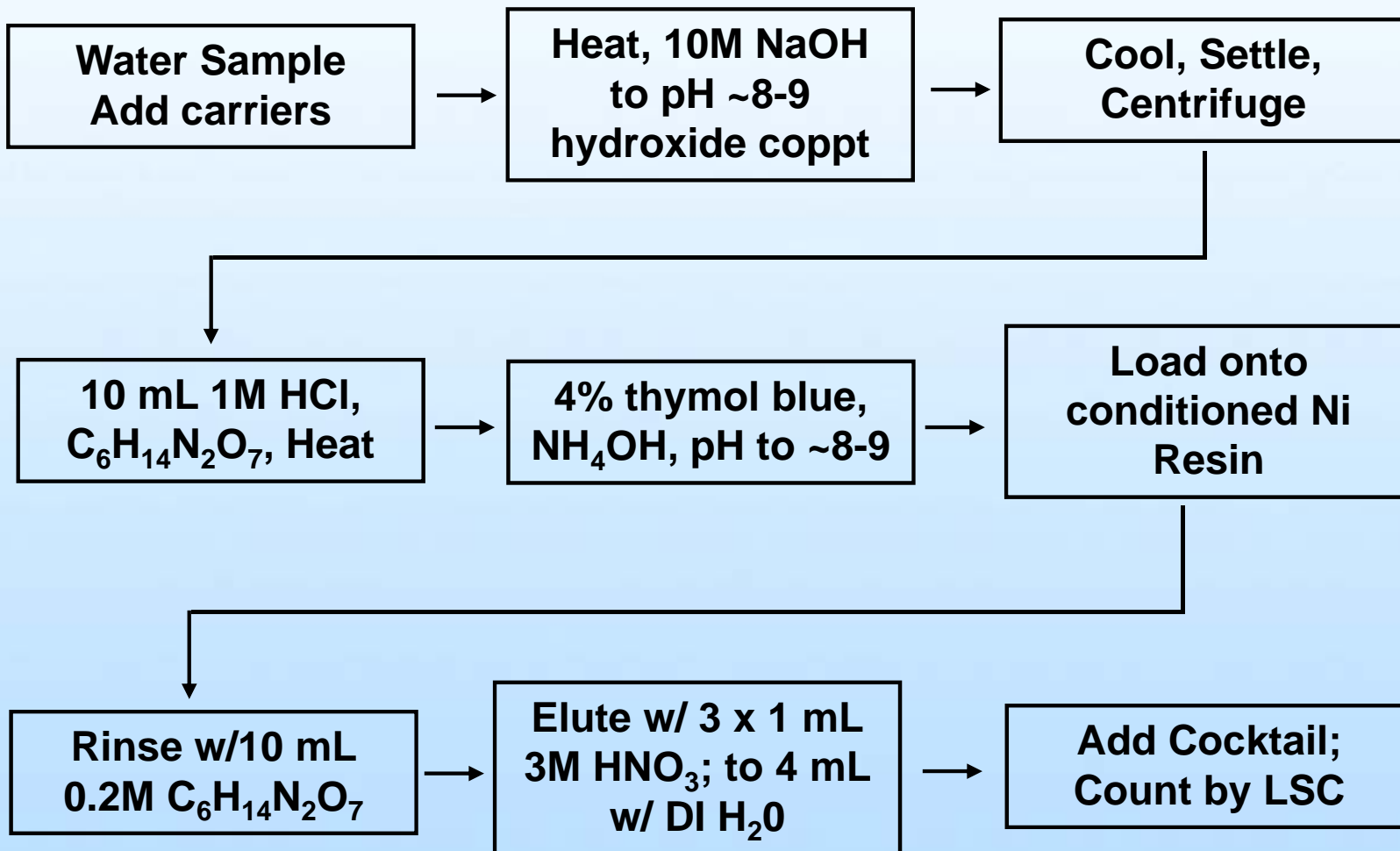


## Ni-63 Water Prep (cont'd)

- New collection tube, elute with 3 x 1 mL portions of 3M HNO<sub>3</sub>;
- Bring to 4 mL w/ DI
- 0.1 mL to metals for chemical recovery
- Load into 15 mL Ultima Gold AB



# Initial Water Prep



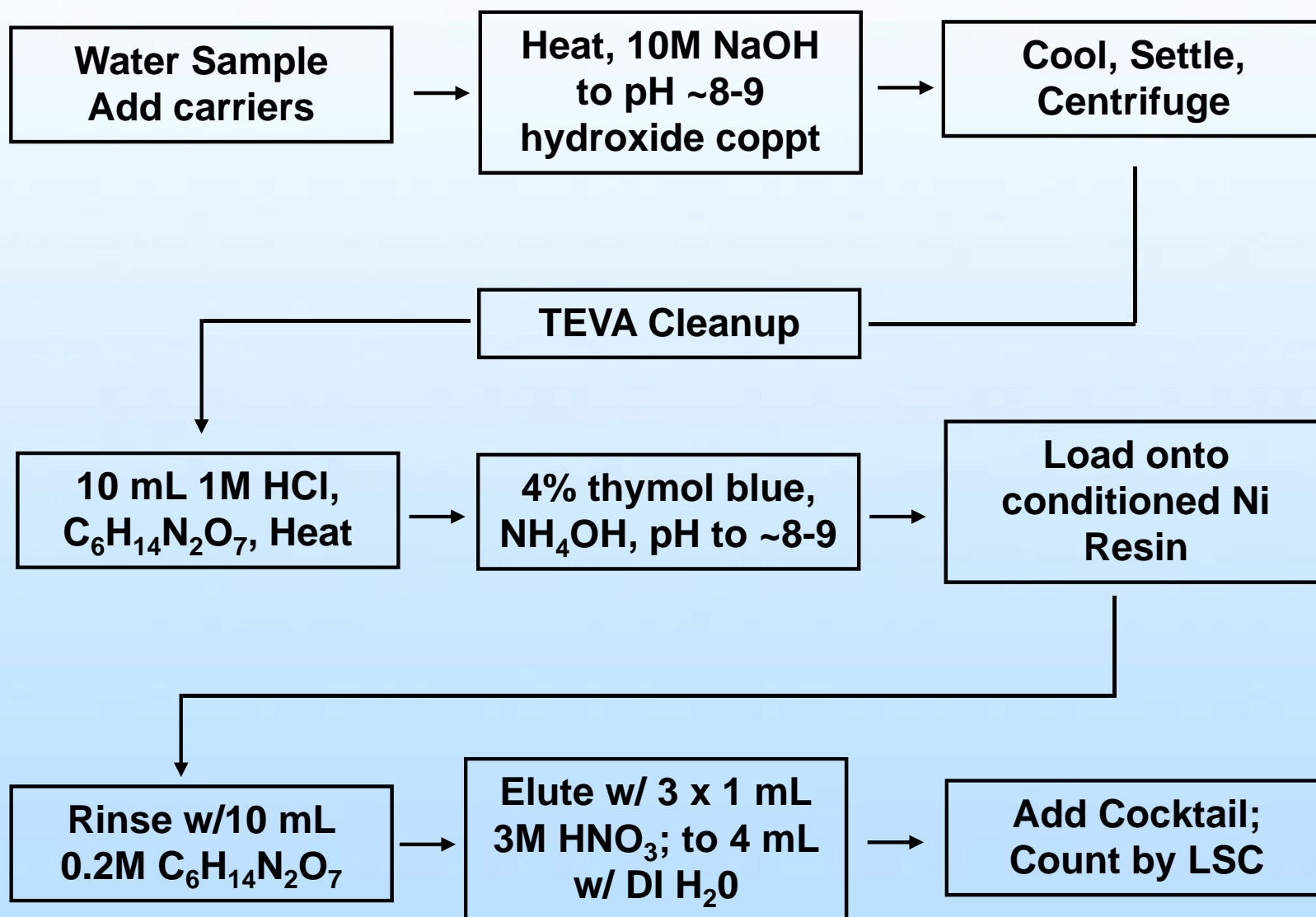


## Additional Step

- Additional Step added for removal of the Tc-99
  - ~ After initial pre-concentration, before loading on Ni cartridge
  - ~ Dissolve solids/residue with 1M HNO<sub>3</sub>
  - ~ Load on TEVA resin, collect eluant
  - ~ Evap. to dryness

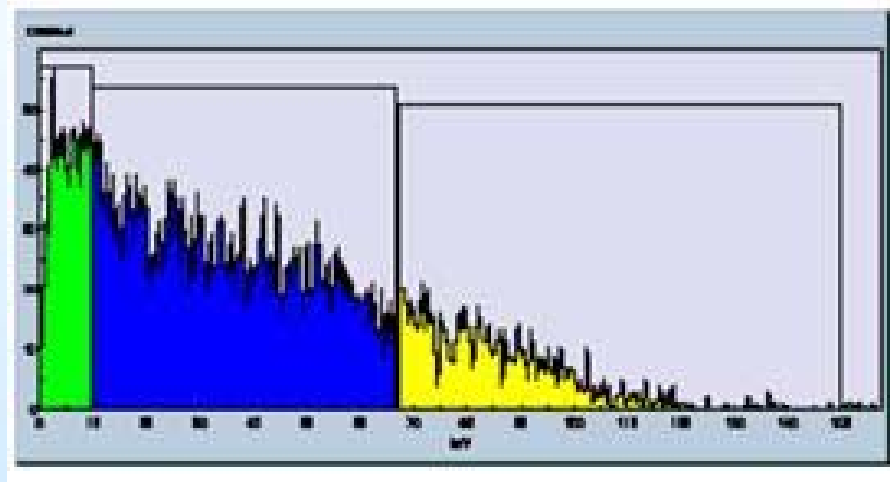


## Additional Step

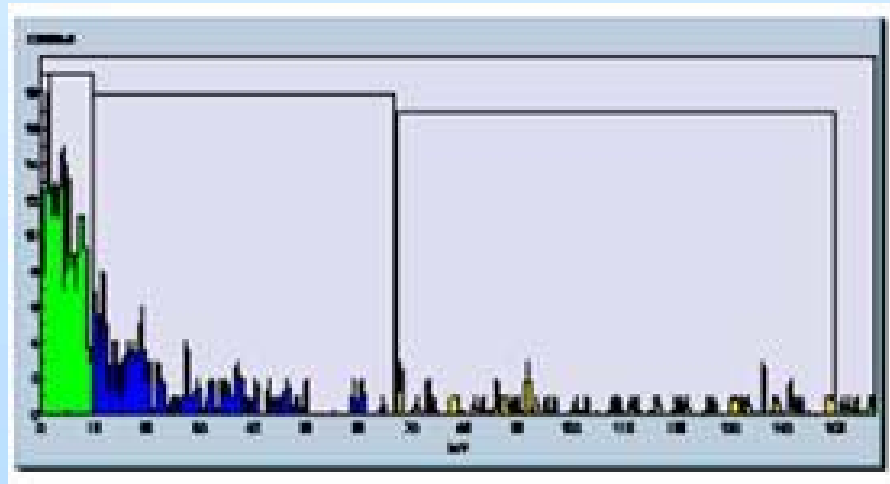


# Spectral Comparison

Initial:



w/ Cleanup:





## Hindsight, Thoughts

- The lab originally prepped the sample without hydroxide co-precipitation; used evaporative concentration option of SOP instead.
- Ferric hydroxide co-precipitation should not carry pertechnetate. Thus, it is possible if this had been the pre-concentration used the TEVA cleanup may not have been necessary.
- Tc(IV) does follow ferrous hydroxide if effective reducing agent present.
- The sample may have reducing effects (unknown), so without some step to ensure the sample is oxidized, TEVA may be necessary.

- Spectral review is an important part of the analysis process
- TEVA is an effective means to remove Tc-99 from well water samples
- Obtaining “process knowledge” from the client can often help decision-making process for handling apparent interferences or matrix issues
- Thorough interview of analysts is prudent when solving analysis issues

## Special thanks to:

- Dan McAlister, Eichrom Technologies
- Terry O'Brien, Eichrom Technologies

Both have been more than generous with their resources!!

# Questions?

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