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NICKEL-63 SEPARATION AND ANALYSIS IN STAINLESS STEEL

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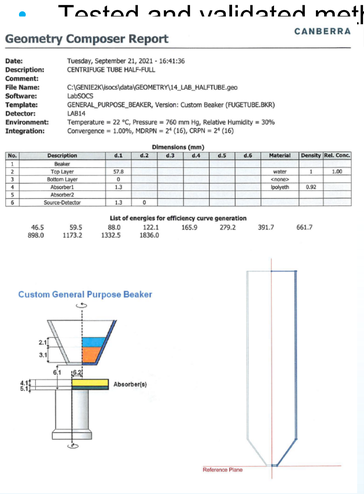
OBJECTIVES

- Quantification of ^{63}Ni activity required for waste characterization of a highly activated stainless steel component
- Modeled ^{63}Ni activity was suspected to be overestimated
- Interference from ^{60}Co necessitated separation of ^{63}Ni from matrix

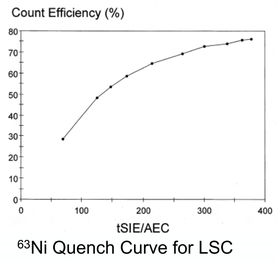
METHOD DEVELOPMENT

- Nickel chemically separated from ^{60}Co utilizing Eichrom TRU and Ni resins
- ^{63}Ni quantified in purified sample by Liquid Scintillation Counting
- ICP-OES used for measurement of Fe, Ni and Co concentrations
- Mirion/Canberra ISOCS calibration model developed to quantify ^{60}Co activity on HPGe gamma detector
- Tested and validated metho… …NIS… …vated Ma…

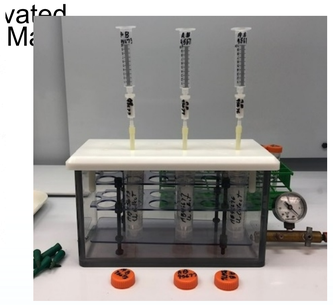
Stainless steel shavings



ISOCS model for centrifuge tube calibration



^{63}Ni Quench Curve for LSC



Vacuum box system with Eichrom cartridges

PROCESS

Sample Aliquot
- Weigh out subsample into beaker
- Prepare QC samples, spiking any lab control samples with ^{63}Ni

Digestion
- Add 3mL HNO_3 and 9mL HCl
- Digest on hotplate set at 150°C for 30 minutes
- Reduce volume to dryness, then bring up to 50mL in 8M HNO_3

ICP-OES Analysis
- Prepare 0.5:50 dilution
- Analyze for Ni, Fe and Co

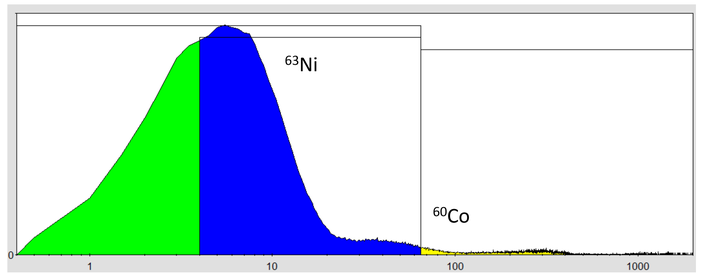
TRU Cartridge
- Use a 10mL portion of 50mL sample
- Process sample through TRU cartridge following Eichrom procedure *Iron-55 in Water*[2]

1st Nickel Cartridge
- Add 1mL Co (1000 ug/mL) to effluent collected from TRU
- Reduce volume to dryness on hotplate, then volume up to 5mL with 1M HCl
- Process sample through Ni cartridge following Eichrom procedure *Nickel-63/59 in Water*[3]

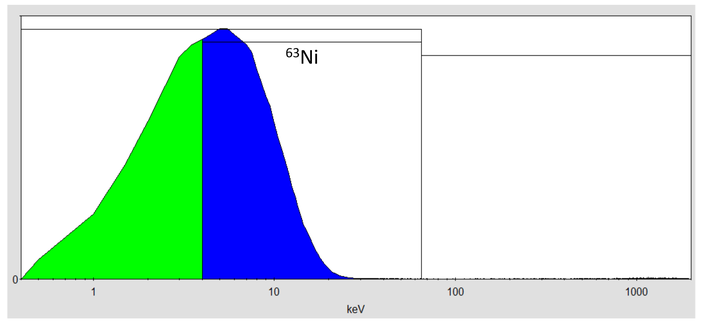
2nd Nickel Cartridge
- Add 1mL Co (1000 ug/mL) to Ni sample from 1st cartridge
- Reduce volume to NEAR dryness on hotplate, then volume up to 5mL with 1M HCl
- Run sample through Ni cartridge following Eichrom procedure *Nickel-63/59 in Water*[3]

Ni Analysis
- Volume Ni sample up to 25mL with 3M HNO_3
- Analyze by gamma spectroscopy for ^{60}Co
- 1:25 dilution for ICP-OES analysis for Ni, Fe, and Co
- Add 2mL to 18mL scintillation cocktail for LSC analysis

Data Reduction
- Use Ni yield to correct ^{63}Ni activity
- Propagate uncertainty from various data sources



LSC spectrum after first nickel separation



LSC spectrum after second nickel separation

RESULTS

- Iron successfully removed with TRU cartridge
- Addition of a second Ni cartridge effectively removed ^{60}Co interference and purified Ni
- Average ^{63}Ni recovery of 96.9 ± 0.03% on 21 validation samples per MARLAP criteria

CONCLUSIONS

- Method proved to be adequate for quantifying ^{63}Ni activity in a highly activated stainless steel matrix
- Measured ^{63}Ni activity was significantly lower than model predictions, effectively reducing radioactive waste disposal costs

REFERENCES

[2] Eichrom Technologies, LLC Analytical Procedure, Iron-55 in Water: May 1, 2014, FEW01, Rev. 1.1.

[3] Eichrom Technologies, LLC Analytical Procedure, Nickel-63/59 in Water: May 1, 2014, NIW01, Rev. 1.3.

[4] Ericksson, S., Vesterlund, A., Olsson, M., Rameback, H., Reducing Measurement Uncertainty in ^{63}Ni Measurements in Reactor Coolant Water with High ^{60}Co Activities (2013) Journal of Radioanalytical Nuclear Chemistry 296:775-779.

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