Carolina Power & Light Company
New Hill, N.C.

Radiochemistry Laboratory

A Comparison of Standard and Extraction Chromatography Methods of Analysis For Nickel-59/63 and Tritium

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CP&L

Procedural Summary
for Nickel 59/63 Analysis

Standard Method

- Sample + Carrier
- Acidify/Evaporate
- PPT Fe $(OH)_3$
- Centrifuge/Filter
- Adjust pH = 8 - 9
- PPT Ni DMG
- Centrifuge/Wash PPT
- Dissolve PPT
- RE-PPT
- Centrifuge/Wash PPT
- Dissolve NI DMG
- Destroy DMG
- Convert to NIO
- Dissolve and LSC

Eichrom Column Method

- Sample + Carrier
- Evaporate
- Convert to Chloride
- Dissolve Residue
- Adjust pH = 8 - 9
- Load 10 ml Sample
- Rinse
- Elute with 3M HNO_3
- Measure NI-63 By LSC and NI-59 By X-Ray

Nuclear Power Plant
Sample Media Tested
<u>for Nickel 59/63 Analysis</u>

- **PWR Reactor Coolant**
- **BWR Reactor Water Cleanup Resin**
- **Dry Active Waste Smears**
- **Radwaste Filter Media**
- **Treated Laundry/Hot Shower Tank**
- **Waste Condensate Tank**

Typical Eichrom Nickel Column
Decontamination Factors

Nuclide	D.F.
Cr-51	350
Mn-54	8,000
Fe-55	400
Co-58	1,000
Co-60	1,100
Nb-95	130
Cs-134	2,800
Cs-137	3,000

COMPARATIVE Ni-63 ACTIVITY (µCi/unit) DETERMINATIONS

SAMPLE TYPE	STANDARD METHOD	NICKEL COLUMN	RATIO
TL/HS Tank	2.30 E-6	2.38 E-6	0.97
Lab Waste Tank	2.66 E-6	2.49 E-6	1.08
WECT Tank	4.31 E-6	4.17 E-6	1.03
NI-63 Spike	5.07 E-4	5.35 E-4	0.95
NI-59 Spike	1.00 E-2	1.07 E-2	0.93
DAW Smears	4.18 E-2	4.63 E-2	0.90
Radwaste Filter	7.40 E-1	7.79 E-1	0.95
RWCU Resin	1.83 E+0	2.05 E+0	0.89

COMPARATIVE Ni-63 ACTIVITY (µCi/unit) DETERMINATIONS (cont.)

SAMPLE TYPE	STANDARD METHOD	NICKEL COLUMN	RATIO
PWR Reactor Coolant	1.98 E-6	3.35 E-6	0.59
		4.53 E-6	0.44
PWR Reactor Coolant	1.93 E-6	1.60 E-6	1.21
DUPLICATE	1.84 E-6	1.71 E-6	1.08
PWR Reactor Coolant	6.89 E-6	5.86 E-6	1.18

MISCELLANEOUS OBSERVATIONS
ON EICHROM NICKEL COLUMN PERFORMANCE

PARAMETER	STANDARD METHOD	COLUMN METHOD
Chemical Yield	63 ± 9%	90 ± 14
LLD (50 ml/30 min)	1.2 E-7	6.5 E-8
Analysis Time:		
Overall	12 Hours	4 Hours
Hands On	8 Hours	2 Hours
LSC Cocktail	Mixed Waste	____

CAVEATS

- **Column has 3 Mg Ni Capacity**
- **Be Aware of Endogenous Nickel in Samples**
- **pH Adjustment is Critical**

Procedural Summary
for Tritium Analysis

Standard Method

- Distillation of an Alkaline Permanganate Solution
- LS Counting Aliquot of Distillate

Eichrom Column Method

- Pass Sample Through Conditioned Column
- Collect Effluent
- LS Counting of an Aliquot

Nuclear Power Plant
Sample Media Tested
<u>for Tritium Analysis</u>

- **Surface Water**
- **Groundwater**
- **PWR Reactor Coolant**
- **BWR Reactor Coolant**

COMPARATIVE H-3 ACTIVITY (µCi/l) DETERMINATIONS

SAMPLE TYPE	STANDARD METHOD	H-3 COLUMN	RATIO
Surface Water	4.48 E+3	5.18 E+3	0.86
Surface Water	7.74 E+3	8.07 E+3	0.96
Groundwater	3.37 E+4	3.41 E+4	0.99
Groundwater	4.67 E+4	4.48 E+4	1.04
BWR-RCS	2.77 E+6	2.74 E+6	1.01
PWR-RCS	4.37 E+8	4.10 E+8	1.06

MISCELLANEOUS OBSERVATIONS
ON EICHROM TRITIUM COLUMN PERFORMANCE

PARAMETER	STANDARD METHOD	COLUMN METHOD
Equipment	Distillation Apparatus	Column
Analysis Time:		
Overall	75 Minutes (including cleanup)	45 Minutes
Hands On	30 Minutes	15 Minutes