



Rapid Analysis of Emergency Water and Urine Samples



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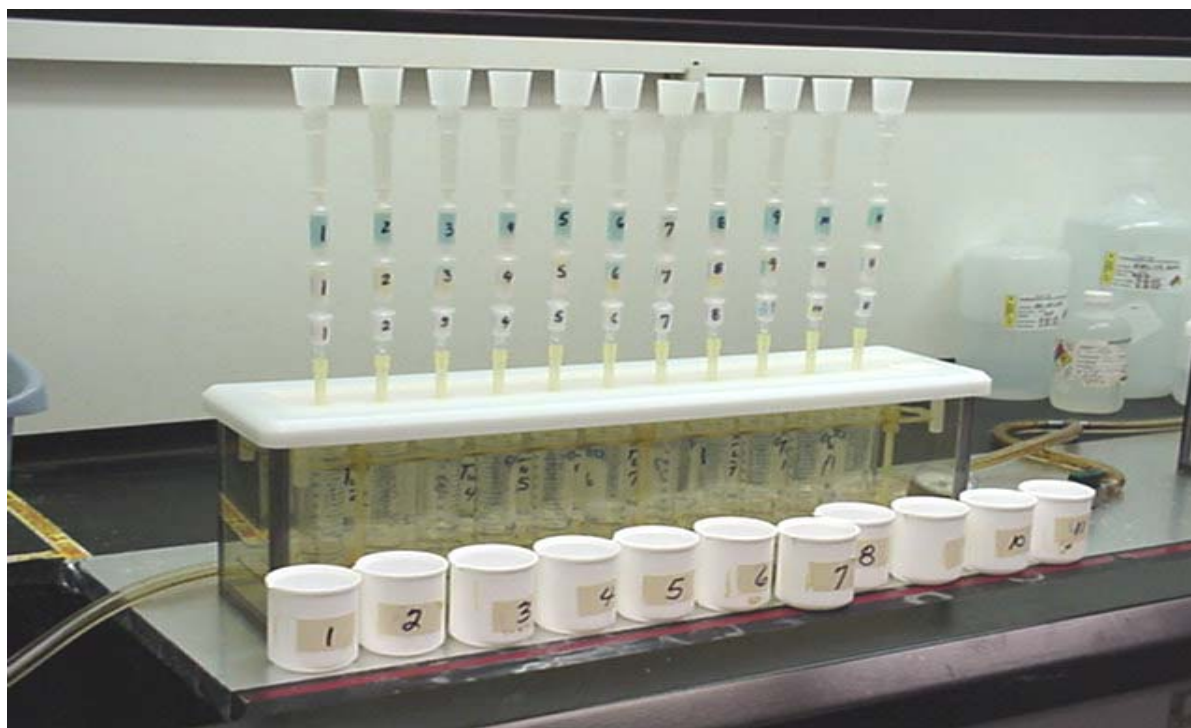
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SRS Lab Performance-NRIP-06

- Per NIST, SRS lab was only lab to report actinides and Sr-90 within 8 hrs.
- Quality of results was very good
- Demonstrated capability to perform rapid emergency analyses on water and urine
- Valuable lessons learned to improve emergency analysis capabilities at SRS lab

Emergency Method Technology

- Apply vacuum technology used for years at SRS
 - Rapid flow rates to save time/money
 - Combines actinide and Sr-89/90 separations



Actinides and Sr-89/90 in Water and Urine

- TEVA/TRU/Sr Resin –stacked cartridges
 - One sample preparation
 - Vacuum box flow rates
- Calcium phosphate ppt.
 - Sample aliquot directly in centrifuge tube
 - Ba added to remove carbonate/no heat



“Maxwell III, SL, “Rapid Column Separation for Actinides and Sr-89/90 in Water Samples”, Journal of Radioanalytical and Nuclear Chemistry, Vol. 267, No. 3, p 537”

Actinides and Sr-90 in Water/Urine

Calcium phosphate precipitation

- 1) Redissolve in 8 mL 6M HNO₃ and 8 mL 2M Al(NO₃)₃
- 2) Add 0.5 mL 1.5M Sulfamic Acid + 1.25 mL 1.5M Ascorbic Acid
- 3) Add 1 mL 3.5 M Sodium Nitrite

Vacuum box procedure

Beaker rinse: 3mL 3M HNO₃
5mL 3M HNO₃ to stacked cartridges

Separate cartridges:
TEVA Resin alone: 10 mL 3M HNO₃

Sr Resin alone:
10 mL 8M HNO₃
10 mL 0.05M strip Sr

Evaporate/ beta counting

Th Elution
20-25 mL
9M HCl

Pu (and/or Np) Elution
20mL
0.10M HCl - 0.05M HF - 0.03M TiCl₃

2 mL TEVA Resin
(50-100 um)

Add 0.5 mL 30 wt% H₂O₂

Cerium fluoride

Alpha spectrometry

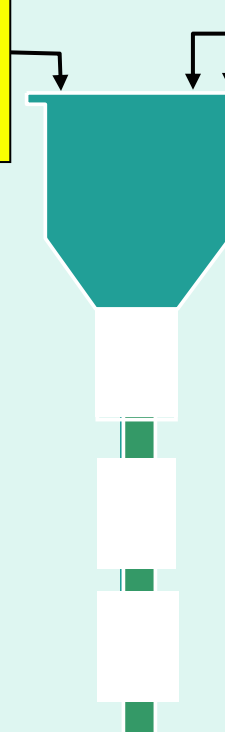
2 mL TRU-Resin
(50-100 um)

Cerium fluoride

2 mL Sr-Resin
(50-100 um)

TRU Resin alone:
Elute Am/Cm with 15 ml 4M HCl/add
15 ml H₂O+50 ug Ce+3 ml HF

Rinse 12 ml 4M HCl-0.2M HF
Elute U with 15 ml 0.1M NH₄HCO₄



Analysis Times-Water

Nuclide	Turnaround time
Gamma isotopes	6.2 hrs.
Gross alpha	6.4 hrs.
Gross beta	6.4 hrs.
Actinides	7.2 hrs. (>1 hr LIMS time)
Sr-90	4.6 hrs.

Analysis Times-Urine

Nuclide	Turnaround time
Gamma isotopes	5.3 hrs.
Actinides	7.4 hrs. (>1 hr LIMS time)
Sr-90	5.8 hrs.

Count Times

- Gamma isotopes 1-2 hours
- Alpha isotopes ~1 hour
- Gross alpha beta 10 minutes
- Strontium-90 20 minutes

Timeline: Actinides and Sr-90 Method

- 7:30 am -Pour aliquots / add tracers
- 7:40 am -Calcium phosphate precipitation
- 8:30 am -Sample loading to columns
- 9:45 am -Sr cartridge handed off to 2nd lab technician for final rinse / strip / mount
- 11-11:30am -Sr to count room (results by ~12 pm)
- 11:30-1:00 pm-actinide fractions to count room
- 12:30-2:00 pm-actinide counts complete

Analysis Results-Water

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Ba-133	910.1	854	-6.2 %
Co-60	671.2	647	-3.6 %
Cs-137	990.2	956	-3.5 %
Eu-152	830.9	785	-5.5 %
Gross alpha	15.00	19.1	27.3 %
Gross beta	2508	2233	-11.0 %

Analysis Results-Water

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Pu-238	1.712	2.040	19.2 %
Pu-239	1.632	2.090	28.1 %
Am-241	1.560	1.700	9.0 %
U-238	4.027	4.470	11.0 %
U-234	3.879	4.090	5.4 %
U-235	0.185	0.156	-15.7 %
Sr-90*	3.749	6.820	81.9 %
Sr-90**	3.749	4.330	15.5 %

* Counted on high background gas proportional counters-reported

**Counted on low background counters <8 hrs. but not reported

Analysis Results-Urine

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Ba-133	1067.35	1050.5	-1.6 %
Co-60	787.2	767.8	-2.5 %
Cs-137	1161.30	1146.8	-1.3 %
Eu-152	974.50	788.2	-19.1 %

Analysis Results-Urine

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Pu-238	2.008	2.138	6.5 %
Pu-239	1.914	1.888	-1.4 %
Am-241	1.830	1.458	-20.3 %
U-238	4.723	4.342	-8.1 %
U-234	4.549	4.632	1.8 %
U-235	0.217	0.164	-24.4 %
Sr-90*	4.397	8.128	84.8 %
Sr-90**	4.397	4.590	4.4 %

* Counted on high background gas proportional counters-reported

**Same mounts counted on low background counters later- not reported

Emergency Actinide Work

- 100 ml aliquot
- Used 10 pCi tracer levels (instead of 2 pCi)
- Initial alpha spectrometry counts
 - Water-60 minutes
 - Urine-80 minutes
- Recounted later for 4 hours for comparison

Actinides in Water Results: Longer Counts

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Pu-238	1.712	1.810	5.7 %
Pu-239	1.632	1.787	9.5 %
Am-241	1.560	1.572	0.77 %
U-238	4.027	4.386	8.9 %
U-234	3.879	4.207	8.5 %
U-235	0.185	0.237	28.1 %

4 hour counts instead of 1 hour

Actinides in Urine Results: Longer Counts

Nuclide	Reference (Bq/g)	Measured (Bq/g)	Difference
Pu-238	2.008	1.874	-6.7 %
Pu-239	1.914	1.852	-3.2 %
Am-241	1.830	1.614	-11.8 %
U-238	4.723	4.681	-0.89 %
U-234	4.549	4.789	5.3 %
U-235	0.217	0.288	32.7 %

4 hour counts instead of 1 hour

Lessons Learned

- Rapid methods are essential
- Requires planning of how to process
 - flow chart, dry run, etc.
- Coordination of chemists, lab technicians, count room
 - Roles and responsibilities

Lessons Learned

- May increase sample aliquot for actinides and Sr-90
- Need to use lower background counters for Sr-90 (instead of high background counters)
- Improve data transfer processing for actinides in LIMS (could have reduced by 1 -2 hours)

Summary

- SRS lab demonstrated rapid, efficient analysis of NRIP-06 emergency samples (< 8 hours)
 - Actinide methods can be adapted to ICP- MS
- Quality and speed met emergency response needs
 - Added to U.S. EPA Office of Radiation/ Indoor Air (ORIA) lab list
- Demonstrated technology
 - Eichrom procedure ACW17 based on SRS method
 - Same technology can be applied to air filters, vegetation, etc. after digestion
- Valuable exercise to improve SRS lab rapid response capabilities
 - Rapid feedback and certificates of traceability from NIST
 - Identified potential improvements at SRS