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Rapid Radiochemical Analyses In Support of Fukushima

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Savannah River National Laboratory
Aiken, SC
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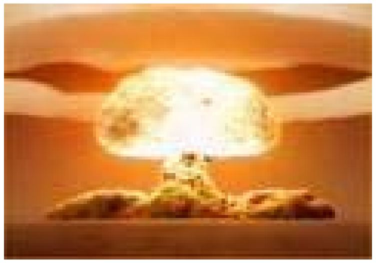
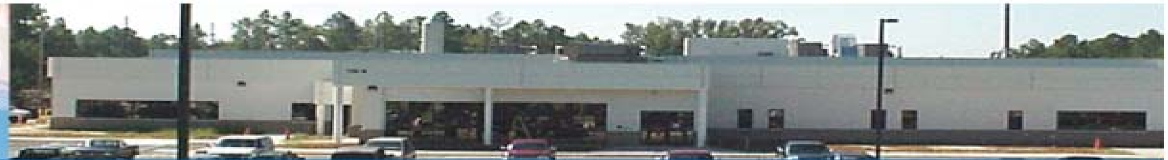
57th Radiobioassay and Radiochemical Measurements Conference
Destin, FL

Background

- Need for rapid radiochemical methods
  - Emergency response
    - IND, RDD, nuclear accident
    - Large numbers of samples
      - environmental and bioassay analyses
    - Rapid turnaround times
  - Routine sample analyses
    - Lowers costs
    - Allows more with less
- Rapid analysis support for Fukushima
  - Air filters and soil samples



SRS Environmental Bioassay Lab



Savannah River Nuclear Solutions, LLC

Analytical Laboratories: **Environmental Bioassay Laboratory**

Our Mission: To provide quality driven, cost competitive Environmental, Bioassay and Industrial Hygiene analytical services in a timely manner while meeting the needs of current and future Savannah River Site missions and for other customers. The Environmental Bioassay Laboratory (EBL) specializes in high volume sample loads (average of 42,000 samples/100,000 determinations per year) and rapid Turn Around Times (TAT) for analyses.

Accreditations and Certifications

Radiological Processing (Environmental Levels)	**Tritium**	**Gross AB**	**Gamma Spec**	**Sr-89/90** *(Sr-90)*	**Alpha Suites** *(Am, Np, Pu, U, Th, Cm Series)*	**Tc-99**	**I-129**	**C-14**	**Ni-63**
	Water, silica gel, vegetation, foodstuff	Water, air filter, rain ion column, vegetation, foodstuff, sediment, soil, concrete	Water, air filter, rain ion column, vegetation, foodstuff, sediment, soil	Water, air filter, rain ion column, vegetation, foodstuff, sediment, soil	Water, air filter, vegetation, foodstuff, sediment, soil	Water	Water, air filter, sedimen, soil	Water	Water
DOELAP Certified	**Tritium**	**Sr-90**	**Alpha Suites** *(Am, Np, Pu, U, Th, Cm Series)*	**Gamma Spec**					
	Urine	Urine, fecal	Urine, fecal	Urine					
SCDHEC Certified	**TSS**	**pH**	**Residual Chlorine Temperature**		**ICP-ES Metals**	**ICP-MS Metals**	**Mercury**		
	Water	Water	Water	Water	Water	Water	Water		
AIHA Certified	**Bulk Asbestos**	**Air Asbestos**	**Gravimetrics**	**Hexavalent Chromium by IC**	**Metals by ICP-ES**	**Beryllium by Optical Fluorescence**			
	Various	Filters	Filters	Filters	Filters, Wipes, Various	Filters, Wipes			

SCDHEC: South Carolina Department of Health and Environmental Control   AIHA: American Industrial Hygiene Association   Foodstuff includes fruit, greens, beef, fish, milk, deer, hog, and crops.

For all your EBL analytical laboratory needs, please contact:

80,000 sq ft facility (35,000 sq ft lab space)

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Approach

- Rapid Radiochemical Methods
  - Combine innovative sample preparation methods with rapid column extraction techniques
    - Soil, vegetation, air filters, water, food, milk, urine
  - Rugged, rapid preparation steps
  - Stacked cartridge technology
    - Sequential separation (5X faster than gravity flow)
    - Rapid flow rates
- Time is money

SRS – Rapid Extraction Chromatography

- Vacuum box technology
  - 1980's with ion exchange
- SRS Bioassay lab-switch to TEVA and TRU Resin -1998
  - higher chemical yields
  - better alpha peak resolution
  - lowers costs significantly

eichrom TECHNOLOGIES INC

New Technology For Faster Analysis!

The Vacuum Box System

Eichrom continues its tradition of being your "time saver" by introducing its Vacuum Box System and extraction cartridge line. By employing vacuum in our already advanced separation methods, analysis time can easily be cut in half over traditional gravity flow Eichrom columns. Customer comments and research in our Rad-lab indicate the same consistently high yields for actinides and strontium as with our conventional columns. Chromatography is improved with the new cartridges that use our smaller 'S' grade resin beads. Another advantage of the cartridge design is the ability to stack two together for tandem separations. Many of our actinide procedures use a common load solution that passes through two resins, for example UTEVA and TRU Resin in our ACW03 method. Using the Luer connections, the cartridges can easily be stacked during sample loading and initial rinse steps and then be separated for final analyte stripping. For sample loading to our cartridges, a standard syringe barrel is recommended, although many reservoirs with a male Luer connection can be used. The cartridges are connected to the vacuum box by two disposable tips. The white inner tip provides an excellent seal with the cartridge and the yellow outer tip provides support. As with our columns, each cartridge is labeled with an identifying lot number. In addition to Sr, Ln, TEVA, TRU and UTEVA cartridges currently in stock, our other resins are available by special order in the cartridge format. Though inventory is limited on the Vacuum Box at this introductory stage, we are now accepting orders. Please inquire for an up to date delivery schedule when placing your order.

The Vacuum Box
Clear polycarbonate construction
24 sample capacity
Sample collection via inner 50 mL C tube rack
Inexpensive cartridge to box connection

The Extraction Cartridge
Standard 2 ml resin volume
50-100µ beads
Available in Sr, Ln, TEVA, TRU and UTEVA
Stackable for multiple analyte separation

Eichrom and Westinghouse Savannah River Site Strive for Faster Bioassay Methods

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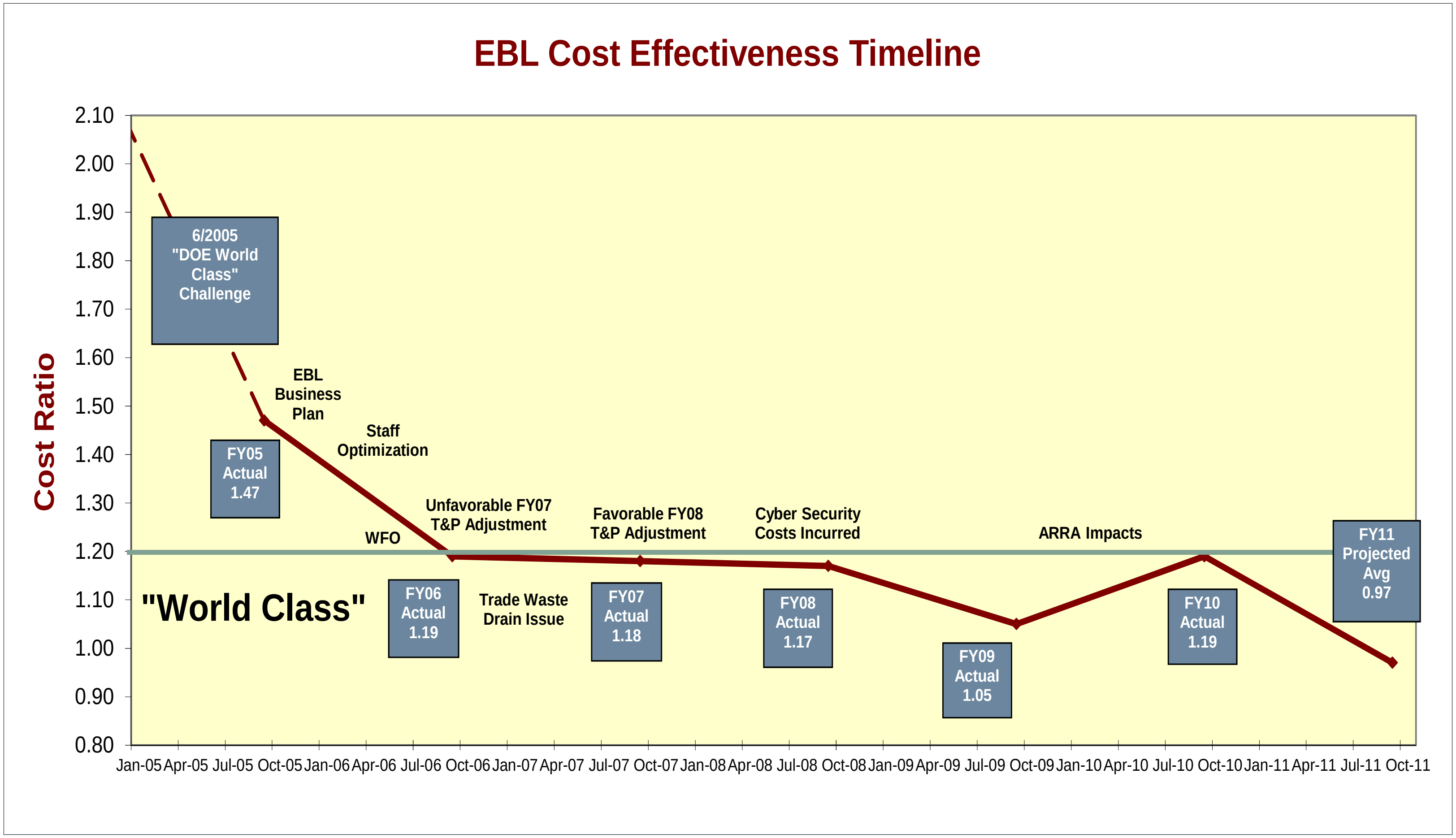
Gravity Flow vs. Vacuum

Cost Reduction vs Private Commercial Labs



Fukushima Air Filters

- Cellulose nitrate filters
  - HNO_3, H_2O_2, HF digestion
    - Repeat HNO_3/H_2O_2 to dryness several times, then with 3ml 3M HNO_3 -boric acid
  - Redissolve in 20 ml 8M HNO_3
  - Took 10 ml aliquot/held back 10 ml in reserve
  - Added 2 ml 2M $Al(NO_3)_3$
- Separate using 2 ml Sr Resin
  - twice for very high total beta samples (>1000 pCi/filter)
  - Important to ensure all beta interferences were removed
- High, consistent Sr gravimetric yields (85-95%)
- Gas flow proportional counting
  - Simultaneous drawer counting system
- Results within hours!

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Approach

- Actinides (Pu,Np, Am, Cm, U) and Sr-89/90
  - Air Filters
    - HNO_3+ HF digestion
    - Rapid and quantitative
    - TEVA Resin +TRU Resin + Sr Resin
    - CeF_3 microprecipitation-alpha spectrometry
    - Sr-89/90- gas proportional counting
      - Gravimetric recovery-Sr carrier (4mg)

Maxwell, S., Culligan, B. and Noyes, G. Applied Radiation and Isotopes Vol. 68, Issue 12, December 2010, Pages 2125-2131

Actinides and Sr-90 in Air Filters

Acid Digestion

1) Redissolve in 6 mL 6M HNO_3 and 6 mL 2M $Al(N0_3)_3$
2) Add 0.5 mL 1.5M Sulfamic Acid + 1.25 mL 1.5M Ascorbic Acid
3) Add 1.25 mL 3.5 M Sodium Nitrite

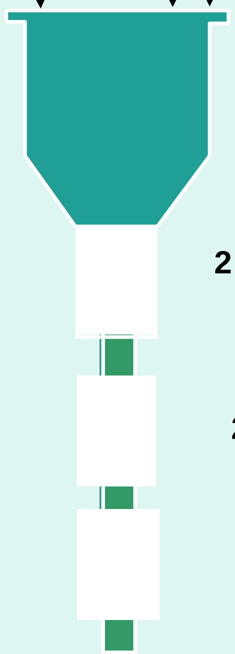
Vacuum box procedure

Beaker rinse: 3mL 3M $HN0_3$
10mL 3M $HN0_3$ to stacked cartridges

Separate cartridges:
TEVA Resin alone: 10 mL 3M $HN0_3$

Th Elution
20mL 9MHCl

Pu Elution
20mL
0.10M HCl - 0.05M HF - 0.01M $TiCl_3$



2 mL TEVA Resin (50-100 um)

2 mL TRU-Resin (50-100 um)

2 mL Sr-Resin (50-100 um)

Add 0.5 mL 30 wt% H2O2

Cerium fluoride

Alpha spectrometry

Cerium fluoride

Sr Resin alone:
15 mL 8M $HN0_3$
10 mL 0.05M $HN0_3$
Sr strip

Evaporate/ beta counting

TRU Resin alone:
Elute Am/Cm with 15 ml 4M HCL/add
15 ml H_20 + 50 ug Ce+ 3 ml HF
12 ml 4M HCl-0.2M *HF -0.002M $TiCl_3$*
Elute U with 15 ml 0.1M $NH_4HC_2O_4$

Optional enhanced Po-210 removal

NRIP-2009 Air Filters Turnaround Times

Nuclide	Turnaround Time (Hrs.)
^{238}Pu	3.9
^{240}Pu	3.9
^{241}Am	3.6
^{238}U	3.7
^{234}U	3.7
^{90}Sr	3.3

NRIP -2009 Air Filters Performance vs. NIST

Nuclide	Avg. Difference (%)
^{238} Pu	3.3
^{240} Pu	-7.3
^{241} Am	7.6
^{238} U	-3.1
^{234} U	-3.4
^{90} Sr	-9.9

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Routine Performance Test Results (air filters)

MAPEP 24

Analyte	Result	Ref Value	Flag	Notes	Bias (%)	Acceptance Range	Unc Value	Unc Flag
Radiological						Units: (Bq/sample)		
Americium-241	0.00036		A			False Positive Test	0.0002	
Cesium-134	3.60	3.49	A		3.2	2.44 - 4.54	0.17	
Cesium-137	2.32	2.28	A		1.8	1.60 - 2.96	0.15	
Cobalt-57	3.27	3.33	A		-1.8	2.33 - 4.33	0.14	
Cobalt-60	0.018		A			False Positive Test	0.057	
Manganese-54	2.71	2.64	A		2.7	1.85 - 3.43	0.25	
Plutonium-238	0.102	0.096	A		6.3	0.067 - 0.125	0.008	
Plutonium-239/240	0.075	0.0765	A		-2.0	0.0536 - 0.0995	0.006	
Strontium-90	1.43	1.36	A		5.1	0.95 - 1.77	0.084	
Uranium-234/233	0.170	0.178	A		-4.5	0.125 - 0.231	0.014	
Uranium-238	0.171	0.185	A		-7.6	0.130 - 0.241	0.014	
Zinc-65	3.35	3.18	A		5.3	2.23 - 4.13	0.34	

MAPEP = Mixed analyte Performance Evaluation Standards from DOE-RESL Lab Idaho, USA

± 20 acceptance limits

Sr-89/90 Fukushima Air Filter Work

AF Batch	N	Avg. Sr. Carrier % Recovery	+/- 1 sigma	% Recovery LCS	Approximate MDC (pCi/filter)
A	14	60.0	15.0	82.5	1 - 2
B	14	92.3	5.3	100.1	1 - 1.5
A`	16	91.1	7.3	88.6	1
B`	16	91.6	4.3	94.6	1
C`	16	92.7	7.3	104.0	1
ARF19	17	79.9	4.7	92.0	0.7
AF/Swipes A	7	93.3	4.0	94.1	0.5
AF/Swipes B	7	80.2	10.7	102.7	0.5
Avg.		85.1		94.8	

for the air filter batches A, B, A`, B` and C` - analyzed only 10 of the 20ml dissolved aliquot

ARF19 used 15 of 20 ml

AF/Swipe batches used the entire sample

Fukushima Soil Samples

- Received samples in early April, 2011
  - Rapid approval of USDA permit
- Via DOE FRMAC(Federal Monitoring and Assessment Center)
  - Gamma, Sr-89/90, actinides
  - Higher than normal activity samples
    - Rad Con and facility support
    - DOE RAP team

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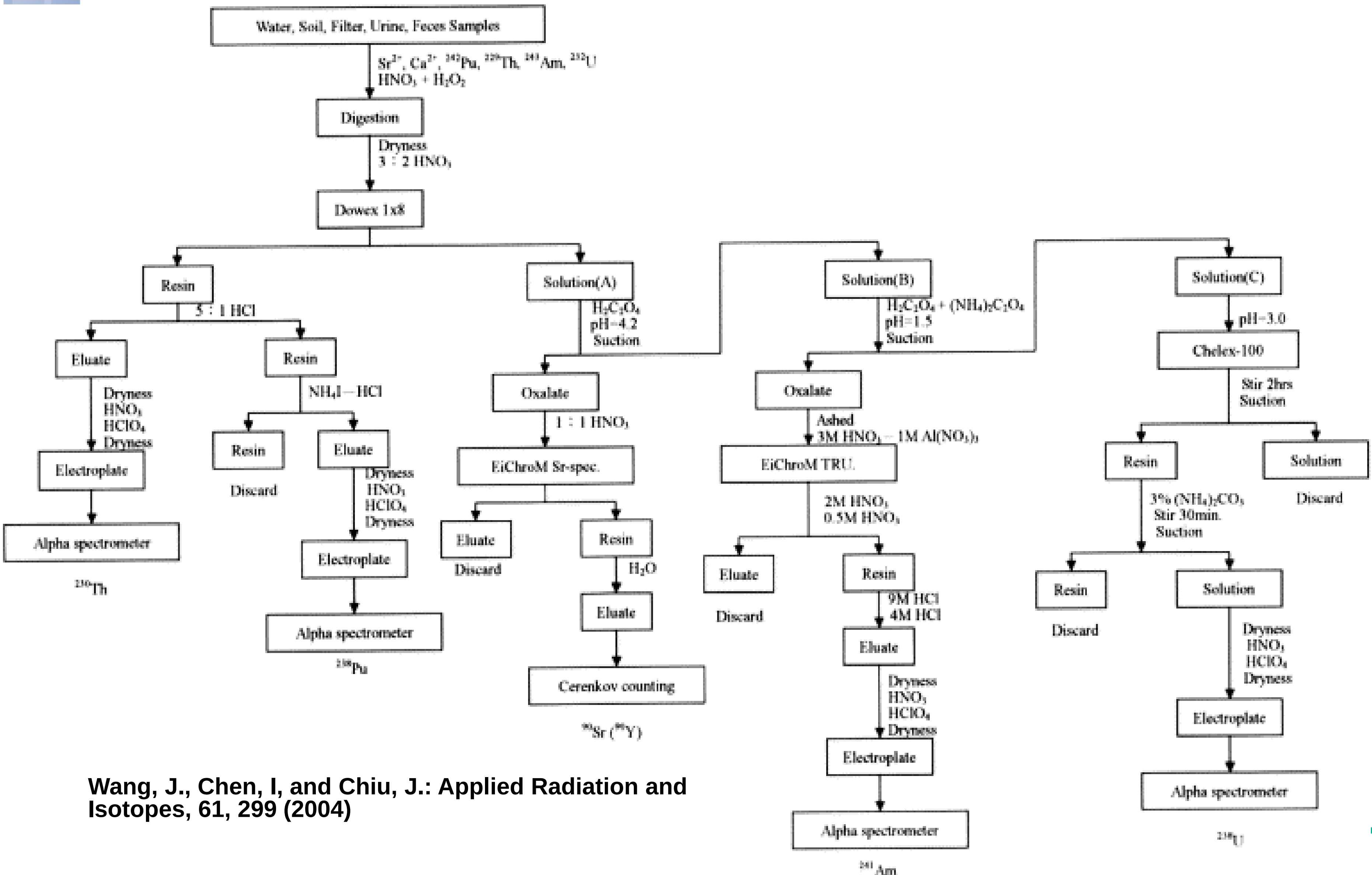
Wang, et al Flow Chart



Wang, J., Chen, I, and Chiu, J.: Applied Radiation and Isotopes, 61, 299 (2004)

Fukushima Soil Samples – Sr-89/90

- Was it ok to plant rice?
  - Required MDA 2 pCi/g (74 mBq/g)
  - Rapid turnaround needed
- Sr-89/90
  - 1.5g rapid fusion method
    - Fe and Ti OH ppt with calcium phosphate added
    - Remove silicates with La/Ca F ppt
    - 3ml Sr Resin (2ml+1ml cartridges/ 6.5 mg Sr carrier)
    - 0.8 pCi/g MDA (29.6 mBq/g)
  - Lower MDA needed later for Sr-89/90 MDA
    - used 10g leach, longer count times
    - Redissolve leachate in 1M HCl, then add NH_4OH, Fe /Ti OH ppt with calcium phosphate, plus La/Ca F ppt
    - 0.05 pCi/g MDA (1.85 mBq/g)

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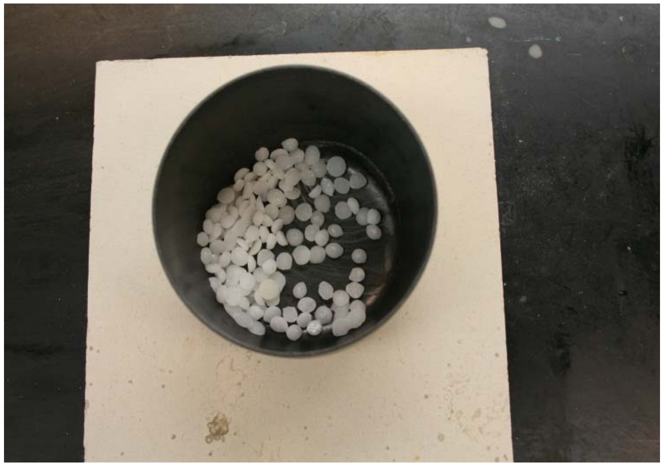
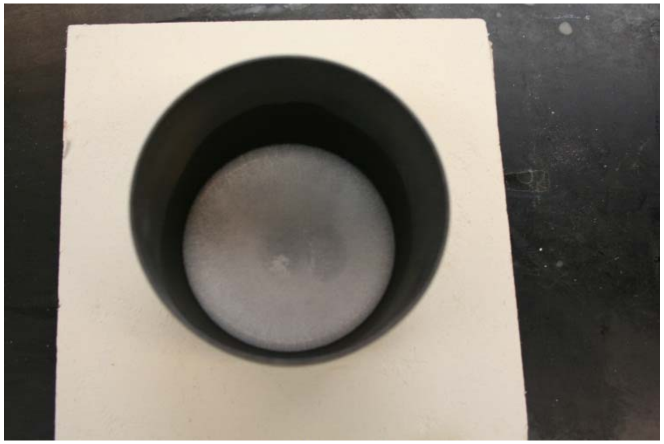
Rapid Sodium Hydroxide Fusion





Leach Preconcentration for Sr and Actinides

- Acid leaching with multiple rinses of solids
- Evaporate leachate and redissolve
- Iron/titanium hydroxide ppt. (+$Ca_3(PO4)_2$) preconcentration
- Lanthanum fluoride ppt. matrix removal

S. L. Maxwell, B. K. Culligan, and G. W. Noyes, Rapid Separation Method for 237Np and Pu isotopes in Large Soil Samples, Applied Radiation and Isotopes, 2010, July 2011, Pages 917-923

S. L. Maxwell, B. K. Culligan, V.D. Jones, S. T. Nichols, M. A. Bernard, G. W. Noyes, Determination of 237Np and Pu isotopes in Large Soil Samples by Inductively-Coupled Plasma Mass Spectrometry, Analytica Chimica Acta, 2010 Dec 3;682(1-2):130-136

Sr-89/90 Fukushima Soil Work

SOIL Batch	N	Avg. Sr Carrier % Recovery	+/- 1 sigma	% Sr-90 Recovery LCS	MS	Approximate MDC (pCi/g)
1	14	78.1	9.4	115.5	98.8	1
2	21	71.5	8.5	100.5	89.1	0.9
3	22	74.2	5.1	100.3	94.5	0.8
4	22	79.7	5.3	106.4	98.5	0.7
5	22	82.1	8.8	105.2	91.7	0.7
6	12	74.1	5.8	106.3	107.1	0.8
7	11	77.5	3.8	91.3	109.9	0.4
8	7	77.1	7.6	90.2	108.9	0.05
9	11	86.1	8.4	105.4	94.9	0.05
10	10	71.9	12.5	99.7	97.4	0.05
11	10	76.6	11.7	94.3	94.3	0.04
Avg.		77.2		101.4	98.6	

Fukushima Soil Samples - Actinides

- Actinides
  - Screening with rapid fusion method (2g)
  - Analysis of large sample aliquots to achieve lower MDAs
    - Volcanic island soil contains high levels of Fe
    - Limited sample aliquot size
  - Used multiple aliquots and loaded to TEVA+TRU+DGA
    - Recombined final purified solutions from multiple purified aliquots into a single CeF_3 micro-ppt
      - removes uranium with H_2O_2 present (U^{6+})
  - Needed to determine actinides isotopes by alpha/ICP-MS
    - Could not split purified aliquots between alpha and ICP-MS since we needed lowest MDA possible for Pu
    - so we counted 100% of aliquot by alpha spectrometry 1st
    - And then….

Fukushima Soil Samples - Actinides

- Further processing for ICP-MS
  - Redissolved actinides after alpha counting filters using HNO_3-boric acid
  - Loaded onto TEVA Resin, rinsed with 3M HNO3, and eluted Pu with ICP-MS friendly solution (0.25M HCL-0.005M HF-0.001M$TiCl_3$)
- Did not need to move Pu to DGA to remove U*
  - *since micro-CeF_3 ppt. with H_2O_2 present used to prepare counting sources removes 1000x uranium*
- Having a range different rapid separation 'tools' allowed us to adapt to specific sample needs

- * Health Physics: August 2011 - Volume 101 - Issue 2 - pp 180-186, Rapid Determination of 237Np and Plutonium Isotopes in Urine By Inductively-Coupled Plasma Mass Spectrometry and Alpha Spectrometry, Maxwell, Sherrod L.; Culligan, Brian K.; Jones, Vernon D.; Nichols, Sheldon T.; Noyes, Gary W.; Bernard, Maureen A.* [>10E6 U decontamination of Pu)

Actinides in Soil: Summary of SRS Approach

- 0.5 -2 grams direct fusion (NaOH)
- 2 -10 grams HNO_3-HF Si removal, then fusion
- 10 -100+ grams acid leach

- In all cases we use Fe/Ti OH precipitation followed by LaF_3 precipitation
  - to preconcentrate actinides and eliminate soil matrix
    - Silicates, Fe
  - Sr-89/90 can be collected also (Ca + PO4)

Fukushima Emergency Soil Samples

- Gamma
  - important to have different size calibrated geometries for soil
  - communication on gamma library (isotopes, parent-daughter, etc)
- Sr-89/90
  - Capability to increase to 10g aliquot was important to allow better determination of Cs/Sr isotopic ratios
  - Simultaneous gas proportional counters allowed longer count times without long delays in results
  - Samples with high total beta interferences may need 2nd column separation
- Actinides
  - Important to have large aliquot capability that can be used with alpha spectrometry and/or ICP-MS
  - May have to adapt methods to specific needs
  - High U-238 DF critical for Pu-239/ options
    - TEVA to DGA thru UTEVA (>10E6)
    - CeF_3+ H_2O_2

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Sr-89/90 in Seawater

- Received questions from Japanese scientists after Fukushima about Sr-89/90 in seawater
  - Japan still using fuming nitric method
- Asked about Eichrom Sr Resin methods
  - Seawater contains 8 mg/L Sr
  - Recommended
    - ICP-ES Sr assay, using stable Sr as yield monitor
    - Either calcium phosphate or calcium carbonate ppt
    - 8M HNO_3+$Al(NO_3)_3$ column load solution
    - 3ml of Sr resin (2ml resin if stable Sr 5mg or less)
    - Gas flow proportional counting

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Readiness for Events Requires Continual Progress

- Actinides in
  - Concrete/Brick
  - *Food*
- Ra-226 using Ra-225 tracer
  - Concrete, brick, vegetation, urine, filters

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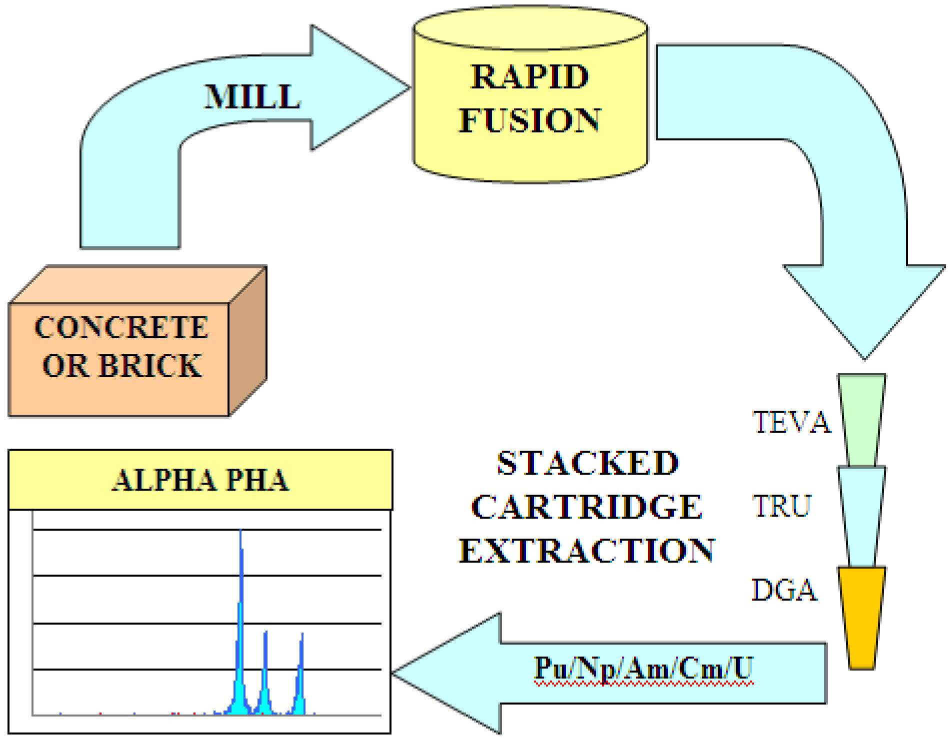
Rapid Fusion Application for Concrete and Brick



Anal Chim Acta. 2011 Sep 2;701(1):112-8. Epub 2011 Jun 15.
Rapid radiochemical method for determination of actinides in emergency concrete and brick samples.
Maxwell SL, Culligan BK, Kelsey-Wall A, Shaw PJ.

Fukushima Impact on Food Chain

- Radiation fallout from the wrecked Fukushima nuclear plant poses a growing threat to Japan’s food chain as unsafe levels of cesium found in beef on supermarket shelves were also detected in more vegetables and the ocean

Japan’s Food-Chain Threat Multiplies as Fukushima Radiation Spreads

By Aya Takada - **JUL 25, 2011 4:59 AM ET**

http://www.bloomberg.com/news/2011-07-24/threat-to-japanese-food-chain-multiplies-as-cesium-contamination-spreads.html

Rapid Actinide Method for Food

Rapid Determination of Actinides in Emergency Food Samples
S. L. Maxwell, B. K. Culligan, A. Kelsey-Wall and P. J. Shaw,
in press, Journal of Radioanalytical and Nuclear Chemistry

Ra-226 in Solid Samples

- Challenges
  - Difficulty separating Ca from Ra/Ba
  - Adequate tracer? (Ba-133)
  - Native barium interference on alpha spectrometry source preparation
    - Poor alpha resolution
    - Isobaric interferences using ICP-MS
    - MnO_2 resin can be used for waters but Ra precipitates with $Fe(OH)_3$ at high pH
- Different approach

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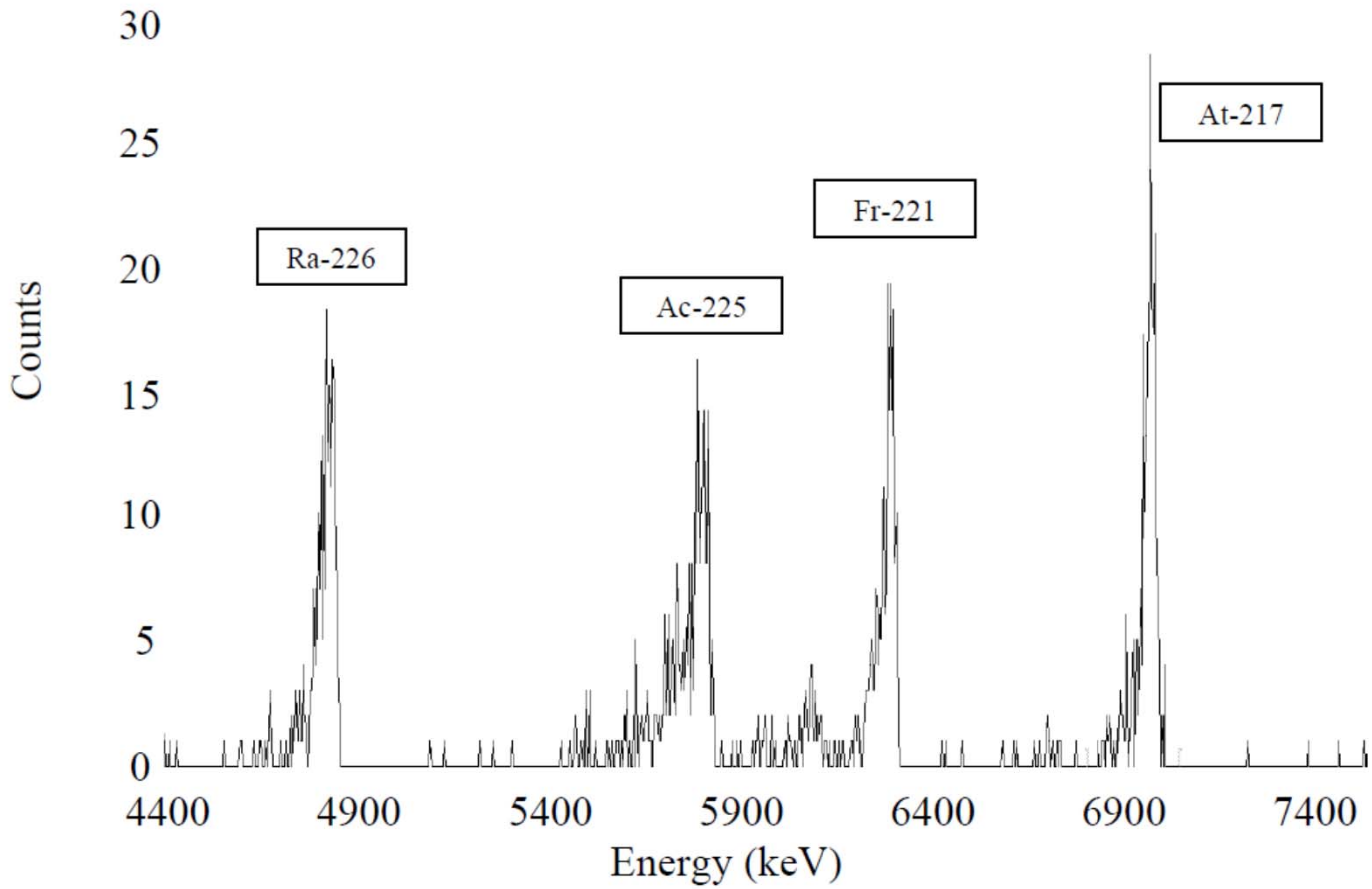
Ra-226 Spectra using At -217 tracer



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Summary

- SRS Environmental Bioassay Lab supported Japan and with fast, quality results
  - NRIP/EPA emergency preparedness testing helped prepare us
  - Rapid radiochemical methods are essential
  - Sr-89/90, gamma, actinides (alpha and ICP-MS)
- Ability to adapt / apply various analytical tools is important
  - Communication with customer
  - Data packaging/QC review was huge part of the effort
- Rapid methods for emergency response can result in cost savings for routine operations

Recent Publications

- Soil

Maxwell, S., Culligan, B. Noyes, G., Jones, V., Nichols, S.T. and Bernard, M. (2010), Rapid determination of 237Np and Pu isotopes in large soil samples by inductively-coupled plasma mass spectrometry, Anal Chim Acta. 2010 Dec 3;682(1-2):130-6. Epub 2010 Oct 8.

Maxwell, S. and Culligan, B. (2006), Rapid column extraction method for actinides in soil, J. Radioanal. Nucl. Chem, 270 (No.3), 699

Maxwell, S. (2008) Rapid method for determination of plutonium, americium and curium in large soil samples, J. Radioanal. Nucl. Chem, 275 (No.2), 395,– U.S. Patent 7,507,583

Maxwell, S., Culligan, B. and Noyes, G. (2010), Rapid method for actinides in emergency soil samples, Radiochimica Acta, Vol. 98, No. 12, pp. 793-800.

S. L. Maxwell, B. K. Culligan, and G. W. Noyes, Rapid Separation Method for 237Np and Pu isotopes in Large Soil Samples, Applied Radiation and Isotopes, 2010, July 2011, Pages 917-923

S. L. Maxwell, B. K. Culligan, V.D. Jones, S. T. Nichols, M. A. Bernard, G. W. Noyes, Determination of 237Np and Pu isotopes in Large Soil Samples by Inductively-Coupled Plasma Mass Spectrometry, Analytica Chimica Acta, 2010 Dec 3;682(1-2):130-136

- Concrete and Brick

Maxwell SL, Culligan BK, Kelsey-Wall A, Shaw PJ, Rapid radiochemical method for determination of actinides in emergency concrete and brick samples. Anal Chim Acta. 2011 Sep 2;701(1):112-8. Epub 2011 Jun 15. A, Shaw PJ.

- **Email me at sherrod.maxwell@srs.gov for a copy**

Recent Publications

- Urine and Water

Maxwell, S., Culligan, B. Noyes, G., Jones, V., Nichols, S.T. and Bernard, M. (2010), Rapid determination of 237Np and Pu isotopes in water by inductively-coupled plasma mass spectrometry and alpha spectrometry , J. Radioanal. Nucl. Chem, online first, DOI: 10.1007/s10967-010-0825-

Maxwell, S., Culligan, B. Noyes, G., Jones, V., Nichols, S.T. and Bernard, M. (2010), Rapid determination of 237Np and Pu isotopes in urine by inductively-coupled plasma mass spectrometry and alpha spectrometry , Health Physics Journal, in press

Maxwell, S. and Culligan, B., (2009), Rapid separation method for emergency water and urine samples, J. Radioanal. Nucl. Chem, 279 (No.3), 901

Maxwell, S. L and Jones, V. D., (2009), Rapid determination of actinides in urine by inductively coupled plasma mass spectrometry and alpha spectrometry: A hybrid approach, Talanta 80 (2009) 143–150

Maxwell, S. (2006), Rapid method for 226Ra and 228Ra analysis in water samples J. Radioanal. Nucl. Chem, 270 (No.3), 651

Maxwell, S. and Culligan, B., (2009), New column separation method for emergency urine samples, J. Radioanal. Nucl. Chem, Vol. 279, No.1, 105

Maxwell, S. (2006), Rapid column extraction method for actinides and 89/90Sr in water samples, J. Radioanal. Nucl. Chem, 267 (No.3), 537

Health Physics: August 2011 - Volume 101 - Issue 2 - pp 180-186, Rapid Determination of 237Np and Plutonium Isotopes in Urine By Inductively-Coupled Plasma Mass Spectrometry and Alpha Spectrometry, Maxwell, Sherrod L.; Culligan, Brian K.; Jones, Vernon D.; Nichols, Sheldon T.; Noyes, Gary W.; Bernard, Maureen A.*

Recent Publications

- Air Filters

  Maxwell, S., Culligan, B. and Noyes, G. (2010), Rapid separation method for actinides in emergency air filter samples, Appl. Radiation and isotopes, December 2010, Pages 2125-2131

- Vegetation

  Maxwell, S., Culligan, B. and Noyes, G. (2010), Rapid Separation of Actinides and Radiostrontium in Vegetation Samples, Journal of Radioanalytical and Nuclear Chemistry, Volume 286, Number 1, October 2010 , pp. 273-282(10)

- Food

  S. L. Maxwell, B. K. Culligan, A. Kelsey-Wall and P. J. Shaw, Rapid Determination of Actinides in Emergency Food Samples, in press, Journal of Radioanalytical and Nuclear Chemistry

- Animal Tissue

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- Milk

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