The Apple Doesn't Fall Far….
The FERN ICLN Exercise - 2014

Jack Bennett, Section Chief
Dr. Katherine A. Kelley State Public Health Laboratory,
Rocky Hill CT

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EVERY CLOUD HAS A SILVER LINING
EXCEPT FOR THE MUSHROOM SHAPED ONES, WHICH HAVE A LINING OF IRIDIUM AND STRONTIUM 90

Radioanalytical Response at the CT Public Health Laboratory

*What is the ICLN???

(Next three slides courtesy of Dr. Marie Socha, DHS)

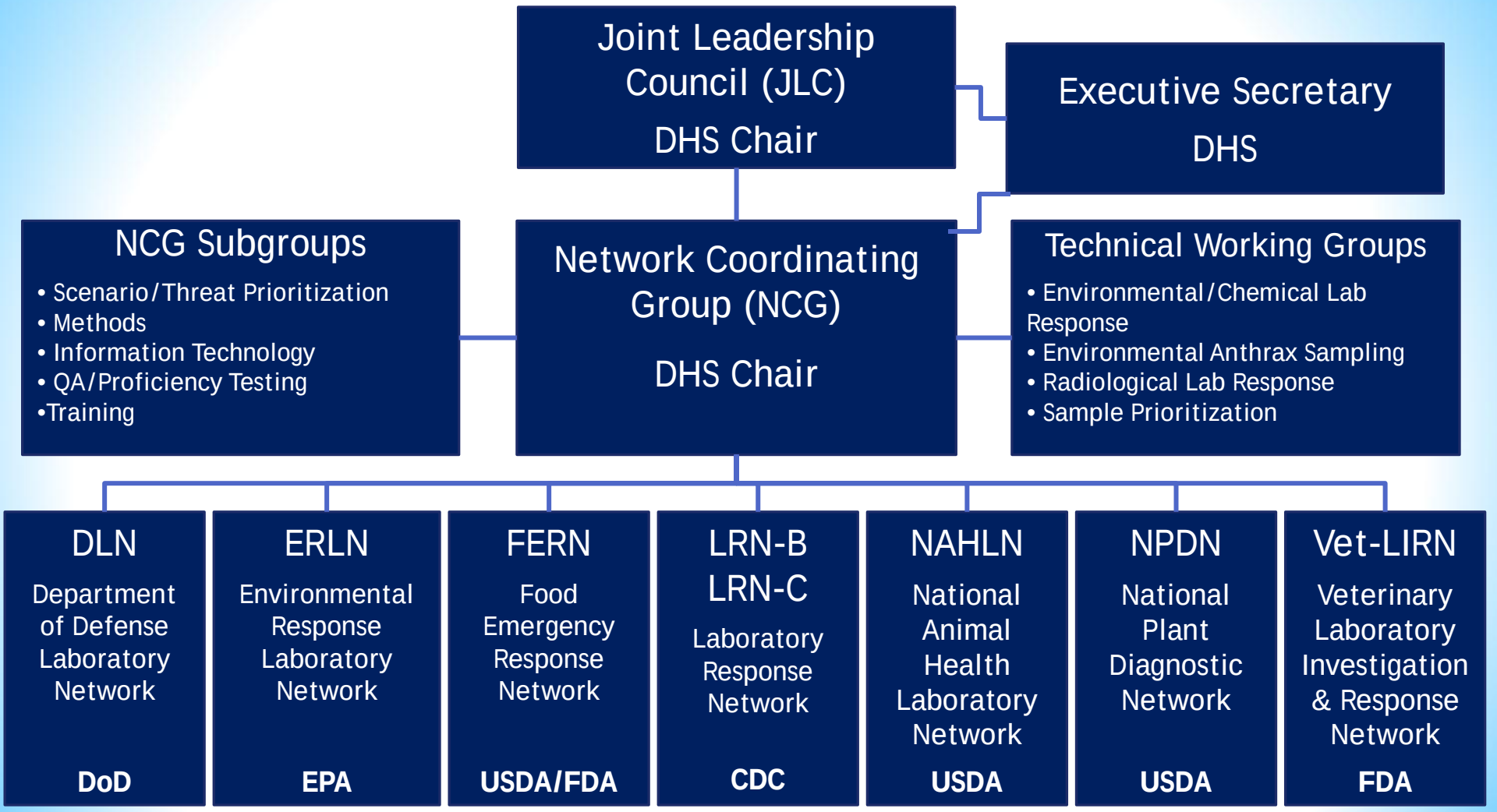
ICLN Vision as Cited in the MOA:

"To create a U.S. homeland security infrastructure with a coordinated and operational system of laboratory networks that provide **timely, high quality, and interpretable results for early detection and effective consequence management of acts of terrorism and other events requiring an integrated laboratory response** (ICLN MOA, June, 2005)."

Purpose of the ICLN:

- The purpose of the ICLN is to:
  - Work cooperatively to **optimize National public health laboratory preparedness** by improving coordination of laboratory response to incidents.
  - **Promote common standards of performance** across all lab response assets to ensure data supporting homeland security decisions is of best quality and defensible.
  - **Assess and fill gaps in coverage (capability and capacity)** across multiple sample types, potential victim groups (human, animal, plant, environment), all WMD weapons, and all response phases.
  - **Enhance laboratory interoperability.**

ICLN Organization



More than 450 distinct labs represented in member response networks.

FERN ICLN 2014 Exercise

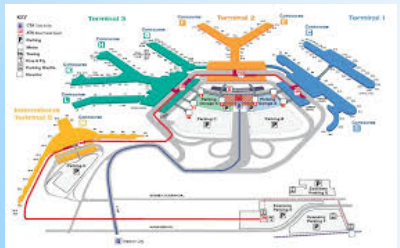
* This multi-agency exercise will cover analytical requirements for both the initial and the recovery phase of a radiological/nuclear event. The initial phase will test the network laboratories' screening capability and capacity within a 48 hour period, while the recovery phase will test confirmatory analytical capability and capacity over a 5 day period. Samples of apple juice will be spiked with known amounts of alpha or beta radioactivity in order to demonstrate positive or negative detection in early phase and quantitative detection in recovery phase. Each test sample of ~37 grams of apple juice will be individually spiked with either Sr-90 for analysis of beta radioactivity or Pu-239 for analysis of alpha radioactivity. Each participating laboratory will choose its own validated method and report test sample results as well as the results of blank and control samples

EXERCISE ** EXERCISE** EXERCISE
Denver, Colorado was impacted by an RDD containing Strontium-90

- An RDD was detonated at the State Capital building in downtown Denver. Extensive damage has occurred with some buildings and nearby automobiles being impacted. Many buildings in a 36 block area north/northeast of the blast are believed contaminated.
- Many fatalities and injuries are reported. Incident responders have observed definitive positive readings on Geiger counters.
- In the aftermath of the blast, attendees from Coor's field evacuated in a panic.
- The downtown arts festival and a Memorial Day fun run were taking place in the Central Business District. About 35,000 people were present at the time of the accident.

EXERCISE**EXERCISE**EXERCISE
Chicago O'Hare Airport was impacted by an RDD containing plutonium-239.

- An RDD was detonated just outside Terminal 1 at Chicago O'Hare. All incoming and outgoing air traffic for this terminal has been closed down.
- Terminal 1 is damaged and non-functional. The windows in the other airport terminals have been blown out by the blast. Several planes which were sitting at the gates in Terminal 1 are incapacitated.
- Emergency responders confirm radiation is present. All air handling systems within the airport have been shut down to minimize the spread of contamination.
- Fatalities include 240 passengers that were picking their baggage up in Terminal 1 when the device detonated and several people were hit with flying debris. An additional 100 airline employees were injured.
- At the time of the explosion, there were approximately 10,000 passengers in Terminal 1 waiting for their flights to leave. Many of these individuals evacuated the airport via the subway system.



How did the apple juice get contaminated???

* Timeline for the Exercise

1st RDD Explosion 05-12-14

Start early phase sample analysis 05-12-14

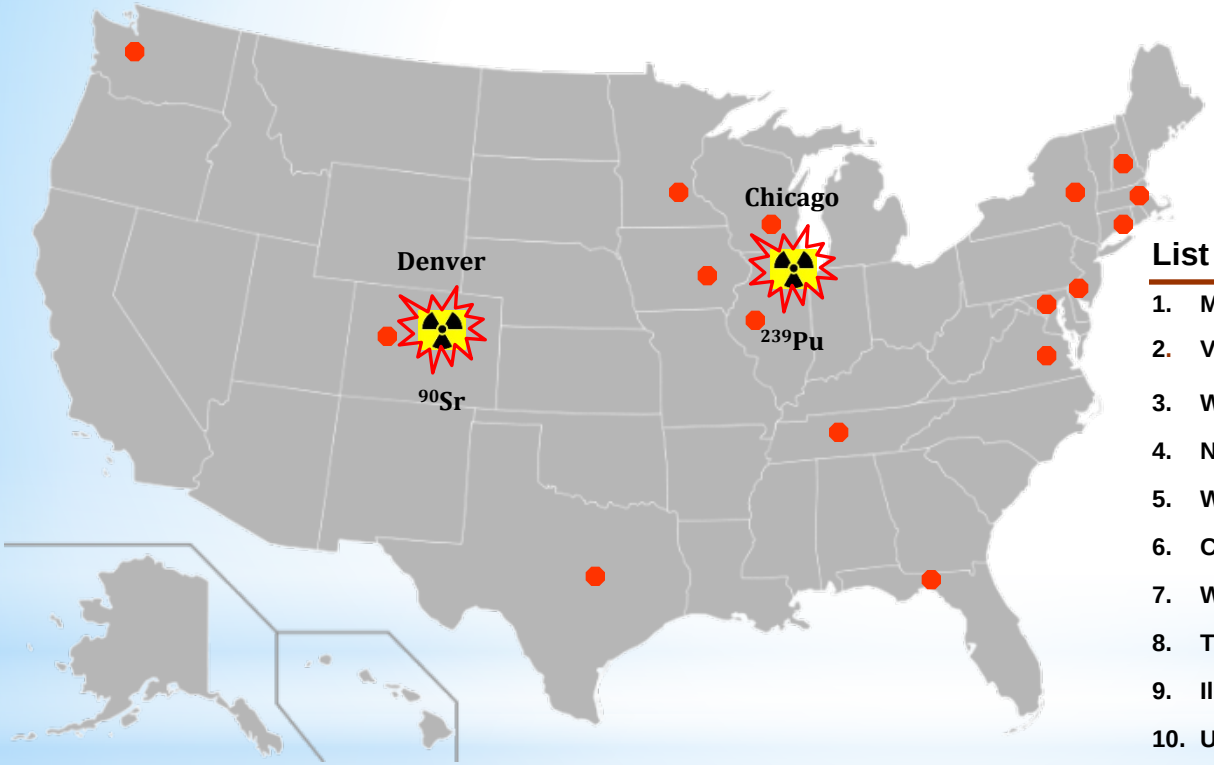
Start recovery phase sample analysis 05-14-14

End of Exercise 05-19-14

2nd RDD Explosion 05-12-14

Received 1st set of screening results 05-13-14

Received 1st set of isotopic results 05-15-14



List of Participants

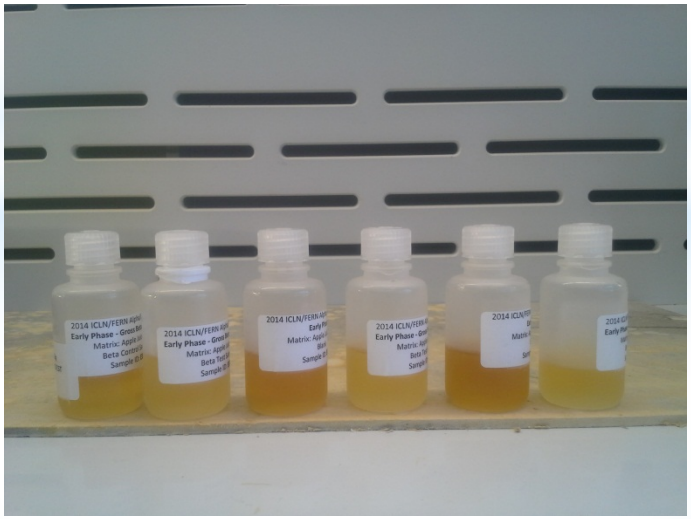
1. Maryland Dept. of Health and Mental Hygiene
2. Virginia Division of Consolidated Laboratory Services
3. Wisconsin State Laboratory of Hygiene
4. New York State Dept. of Health
5. Washington State Dept. of Public Health Laboratories
6. Colorado Dept. of Public Health and Environment
7. Winchester Engineering and Analytical Center
8. Texas Dept. of State Health Services Laboratory
9. Illinois Emergency Management Agency
10. University of Iowa Hygienic Laboratory
11. Tennessee Dept. of Health
12. Minnesota Dept. of Health
13. New Jersey Dept. of Health and Senior Services
14. New Hampshire Public Health Laboratories
15. Florida Dept. of Health Bureau of Radiation Control
16. State of Connecticut Public Health Laboratory

Sample Types

The test samples used in the exercise include:

- Unknowns Spiked with Single Radionuclide at two levels of Activity Concentrations
- Matrix-Matched Method Blanks
- Matrix-Matched Control Samples with Disclosed Known Values

The apple juice used for preparing test samples contain ~38 Bq/kg of naturally-occurring K-40, which implies that each test sample has ~1.4 Bq of K-40.



Radioanalytical Methods Applied in Exercise

Early Phase

- **LSC-Based Methods:**
  1. Direct Measurement
  2. Solid-Phase Extraction

- **GPC-Based Methods:**
  1. Wet Ashing/Counting
  2. Evaporation/Counting (EPA900)

Recovery Phase

- **GPC-Based Methods:**
  1. TBP Extraction/Counting
  2. Sr Resin/Counting
  3. Coprecipitation/Counting
  4. Extraction Resins/Counting (Eichrom ACW17 VBS)

- **LSC-Based Methods:**
  1. Sr Resin/Counting

- **Alpha Spec-Based Methods:**
  1. TRU and Anion Exchange Resins/Counting
  2. DGA Resin/Counting
  3. ASTM 3084-89
  4. Extraction Resins/Counting (Eichrom ACW17 VBS)

Sample Statistics

Early Phase:

Samples tested for alpha radioactivity = 76

Samples tested for beta radioactivity = 110

Recovery Phase:

Samples analyzed for Pu-239 = 51

Samples analyzed for Sr-90 = 85

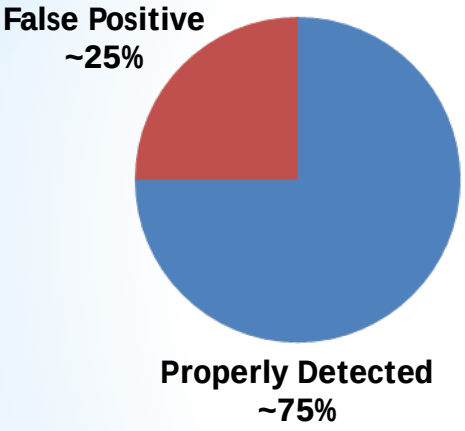
Total number of samples completed throughout the exercise

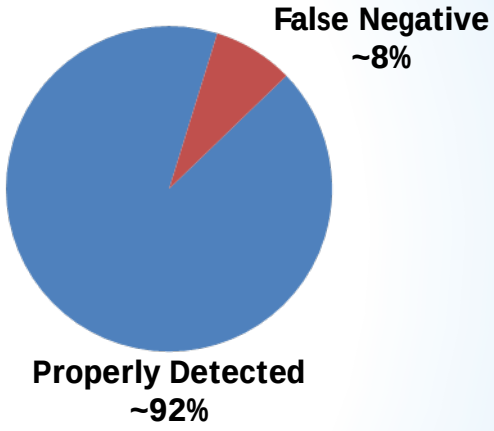
322

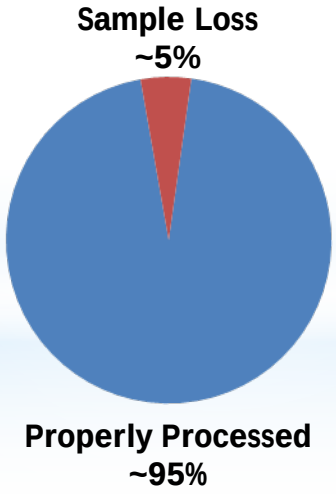
ICLN ‘s Expectation for FERN network

200 - 300

Overall Test Results





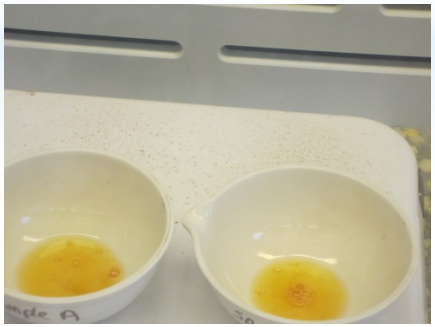


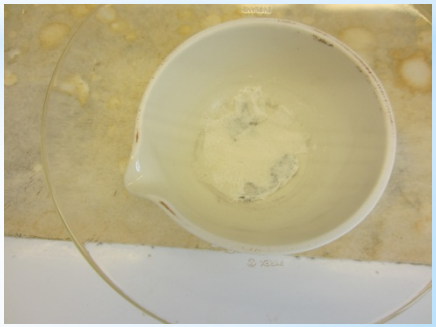
Stumbling Blocks

- K-40 Interference
- Instrument Failure
- Calculation Errors
- Problem with Uploading Results
- Sample Processing Errors
- Method Shortcoming
- Run out of Supplies

Connecticut's Screening Method

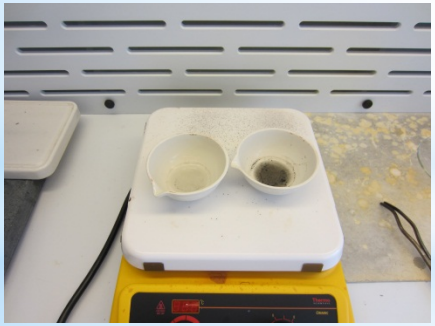
- 10 ml of apple juice was pipetted into ceramic dish with 10 ml of concentrated nitric acid.
- The hot plate was set to 170-185 degrees until sample evaporated to 1-2ml.
  - Higher temperatures caused the samples to char
  - Took several hours to evaporate
- The ceramic dish was then placed in the muffle furnace and ramped to 500 degrees and held for 2 hours.
  - At this point the sample remaining is a very powdery white ash and all the organic matrix has been removed.





Connecticut's Screening Method

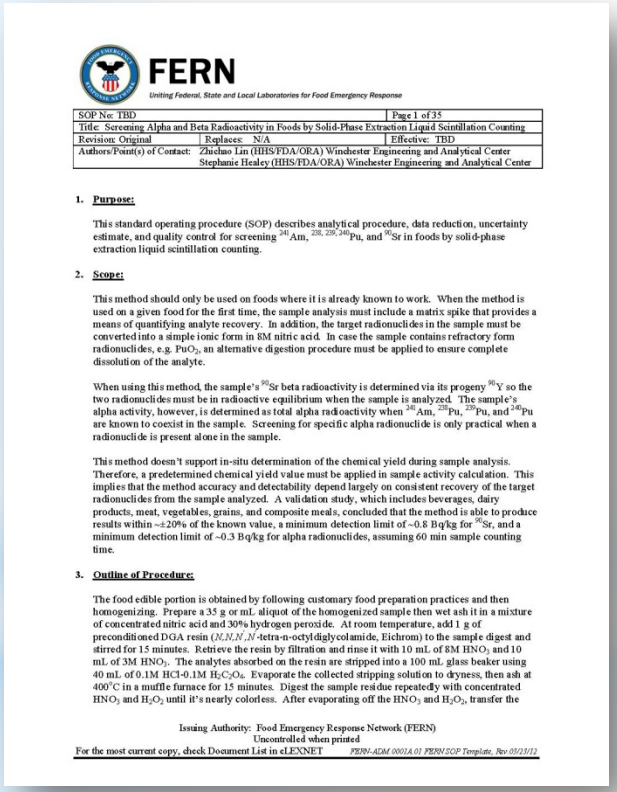
* 10 ml of concentrated nitric acid is added to the ashed sample and evaporated down to 1-2ml.
  * This step is repeated for a total of 2 acid rinses and evaporations.
* The sample is then planchetted and dried with a heat lamp before counting on a gas proportional counter for 100 minutes.



Connecticut's Screening Results Summary

Sample ID	Result (Bq/Kg)	True Value	% Recovery
Screen Unknown #1	400	193.14	207.10
Screen Unknown #2	81	77.28	104.81
Screen Unknown #3	363	196.8	184.45
Screen Known #1	362	315.1	114.88
Screen Blank #1	1.7	0	
Screen Blank #2	0	0	
Note: Blanks and Known were provided by the submitter			

Connecticut's Confirmatory Method



FERN

Uniting Federal, State and Local Laboratories for Food Emergency Response

SOP No: TBD		Page 1 of 35
Title: Screening Alpha and Beta Radioactivity in Foods by Solid-Phase Extraction Liquid Scintillation Counting		
Revision: Original	Replaces: N/A	Effective: TBD
Authors/Point(s) of Contact:	Zhichao Lin (HHS/FDA/ORA) Winchester Engineering and Analytical Center Stephanie Healey (HHS/FDA/ORA) Winchester Engineering and Analytical Center	

1. Purpose:

This standard operating procedure (SOP) describes analytical procedure, data reduction, uncertainty estimate, and quality control for screening ^{241}Am, $^{238, 239,240}$Pu, and ^{90}Sr in foods by solid-phase extraction liquid scintillation counting.

2. Scope:

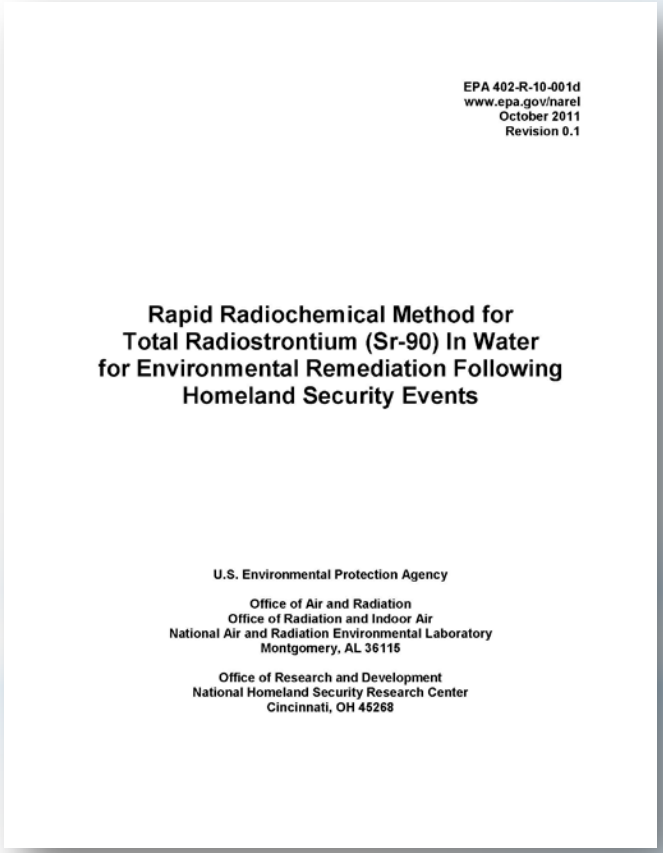
This method should only be used on foods where it is already known to work. When the method is used on a given food for the first time, the sample analysis must include a matrix spike that provides a means of quantifying analyte recovery. In addition, the target radionuclides in the sample must be converted into a simple ionic form in 8M nitric acid. In case the sample contains refractory form radionuclides, e.g. PuO_2, an alternative digestion procedure must be applied to ensure complete dissolution of the analyte.

When using this method, the sample's ^{90}Sr beta radioactivity is determined via its progeny ^{90}Y so the two radionuclides must be in radioactive equilibrium when the sample is analyzed. The sample's alpha activity, however, is determined as total alpha radioactivity when ^{241}Am, ^{238}Pu, ^{239}Pu, and ^{240}Pu are known to coexist in the sample. Screening for specific alpha radionuclide is only practical when a radionuclide is present alone in the sample.

This method doesn't support in-situ determination of the chemical yield during sample analysis. Therefore, a predetermined chemical yield value must be applied in sample activity calculation. This implies that the method accuracy and detectability depend largely on consistent recovery of the target radionuclides from the sample analyzed. A validation study, which includes beverages, dairy products, meat, vegetables, grains, and composite meals, concluded that the method is able to produce results within ~±20% of the known value, a minimum detection limit of ~0.8 Bq/kg for ^{90}Sr, and a minimum detection limit of ~0.3 Bq/kg for alpha radionuclides, assuming 60 min sample counting time.

3. Outline of Procedure:

The food edible portion is obtained by following customary food preparation practices and then homogenizing. Prepare a 35 g or mL aliquot of the homogenized sample then wet ash it in a mixture of concentrated nitric acid and 30% hydrogen peroxide. At room temperature, add 1 g of preconditioned DGA resin (*N,N,N',N'*-tetra-n-octyldiglycolamide, Eichrom) to the sample digest and stirred for 15 minutes. Retrieve the resin by filtration and rinse it with 10 mL of 8M HNO_3 and 10 mL of 3M HNO_3. The analytes absorbed on the resin are stripped into a 100 mL glass beaker using 40 mL of 0.1M HCl-0.1M $H_2C_2O_4$. Evaporate the collected stripping solution to dryness, then ash at 400°C in a muffle furnace for 15 minutes. Digest the sample residue repeatedly with concentrated HNO_3 and H_2O_2 until it's nearly colorless. After evaporating off the HNO_3 and H_2O_2, transfer the



EPA 402-R-10-001d
www.epa.gov/narel
October 2011
Revision 0.1

Rapid Radiochemical Method for Total Radiostrontium (Sr-90) In Water for Environmental Remediation Following Homeland Security Events

U.S. Environmental Protection Agency

Office of Air and Radiation
Office of Radiation and Indoor Air
National Air and Radiation Environmental Laboratory
Montgomery, AL 36115

Office of Research and Development
National Homeland Security Research Center
Cincinnati, OH 45268

Connecticut's Confirmatory Method

- Weigh 35 mL of juice into a beaker and add 100 mL conc HNO_3. Boil for 30 to 45 minutes.
- Carefully add 10 mLs of H_2O_2 and boil for 60 minutes.
- Cool, and bring up to 100 mLs with 8M HNO_3
- Remove a 5 mL aliquant (put in centrifuge tube), and add strontium and barium carriers

Connecticut's Confirmatory Method

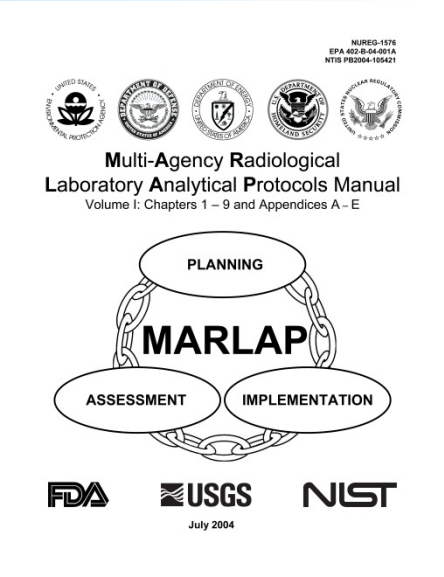
* Load solution onto a 2mL Sr-Resin column on a vacuum box
* Elute Strontium from column with 0.05M HNO_3, planchet and count for 100 minutes on a gas proportional counter.

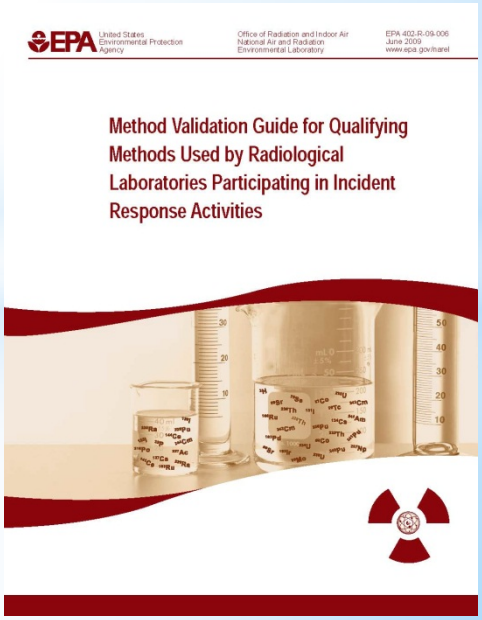
Connecticut's Confirmatory Samples Summary Data

Sample ID	Result (Bq/kg)	True Value	% Recovery
Confirm Unknown #1	177	156.52	113.08
Confirm Unknown #2	75	77	97.40
Confirm Unknown #3	168	157.39	106.74
Confirm Known #1	177	160.3	110.42
Confirm Blank #1	-0.9	0	
Confirm Blank #2	0	0	
Note: Confirm Blanks and Known were provided by the submitter			
Note: LFB Recovery = 107%			

What Next???

- Validate method according to MARLAP
  - EPA has developed a guidance document that helps understanding the MARLAP criteria
    - Also has additional items to consider
  - Will use Level D criteria





Tiered Approach to Method Validation

TABLE 3 – Method Validation Requirements and Applicable to Required Method Uncertainty

Validation Level [1]	Application	Sample Type	Acceptance Criterion [2]	Levels [4] (Concentration)	Replicates [3]	# of Analyses
B	Existing Method Radionuclide – Same, Similar or Slightly Different Matrix	Internal PT	Measured Value Within ±2.8 u_{MR} or ± 2.8 φ_{MR} of Validation Value	3	3	9
C	Similar Matrix: New Application	Internal or External PT	Measured Value Within ±2.9 u_{MR} or ± 2.9 φ_{MR} of Validation Value	3	5	15
D	Adapted, Newly Developed, Rapid Methods	Internal or External PT	Measured Value Within ±3.0 u_{MR} or ± 3.0 φ_{MR} of Validation Value	3	7	21
E	Adapted, Newly Developed, Rapid Methods	Method Validation Reference Materials	Measured Value Within ±3.0 u_{MR} or ± 3.0 φ_{MR} of Validation Value	3	7	21

Validation Criteria

- Method Uncertainty
- Detection Capability
  - Difference between blanks and samples spiked at the MDC
- Bias
  - Absolute and relative
  - No acceptance criterion, but important to know
- Specificity
  - Determined by spiking with non-target nuclides
- Ruggedness
  - Tracer yields, spectral quality

Connecticut Department of Public Health

Uncertainty Evaluation - Level 1 (0.5 AAL)

Isotope:	Sr-90
Required Method Uncertainty, Bq/kg (Note: if expressed as %, please convert to	10.3
Spike conc, Bq/	79
Spike Uncertainty Value, Bq/kg	1.77
Lower Recovery Limit, Bq/kg	48.1
Upper RecoveryLimit, Bq/kg	109.9

Sample ID	Activity, Bq/kg	Uncertainty, Bq/kg	Method Uncertainty Pass/Fail	
1	72	3.2	P	
2	76	3.4	P	
3	79	3.4	P	
4	73	3.2	P	
5	76	3.2	P	
6	84	3.5	P	
7	72	3.2	P	
Meets Required Method Uncertainty (Y/N)			Y	
Average	76	3.3		
Average Recov	96.20253			
Standard Devia	4.358899			
Effective Degrees of Freedom (for 7 Replicates)	27			
Critical Value (Value capped at 30 effective degrees of freedom))	2.052			
Test Statistic	1.241963			
Rel Bias (Y/N)	N		No Bias	

Uncertainty Evaluation - Level 2 (AAL)

Isotope:	Sr-90
Required Method Uncertainty, Bq/kg (Note: if expressed as %, please convert to Bq/kg)	20.06
Spike conc, Bq/kg	158
Spike Uncertainty Value, Bq/kg	3.53
Lower Recovery Limit, Bq/kg	97.82
Upper RecoveryLimit, Bq/kg	218.18

Sample ID	Activity, Bq/kg	Uncertainty, Bq/kg	Method Uncertainty Pass/Fail
AAL Test -1	152	3.5	P
AAL Test -2	124	2.9	P
AAL Test -3	121	2.9	P
AAL Test -4	164	3.5	P
AAL Test -5	121	1	P
AAL Test -6	117	2.9	P
AAL Test -7	148	3.4	P
Meets Required Method Uncertainty (Y/N)			Y
Average	135.2857	2.871429	
Average Recover	85.62387		
Standard Deviati	18.86544		
Effective Degrees of Freedom (for 7 Replicates)	9		
Critical Value (Value capped at 30 effective degrees of freedom))	2.262		
Test Statistic	2.854366		
Rel Bias (Y/N)	Y		Shows Bias

Uncertainty Evaluation - Level 3 (3x AAL)

Isotope:	Sr-90
Required Method Uncertainty, Bq/kg (Note: if expresse	61.56
Spike conc	473.6
Uncertainty Value,	10.59
Lower Recovery Limit,	288.92
Recovery Limit, Bq/kg	658.28

Sample ID	Activity, Bq/kg	Uncertainty, Bq/kg	Method Uncertainty Pass/Fail
3xAAL Tes	418	7.3	P
3xAAL Tes	447	7.6	P
3xAAL Tes	394	7	P
3xAAL Tes	407	7.6	P
3xAAL Tes	440	7.3	P
3xAAL Tes	398	6.7	P
3xAAL Tes	403	6.8	P
Meets Required Method Uncertainty (Y/N)			Y
Average	415.2857	7.185714	
Average R	87.68702		
Standard I	20.79835		
Effective Degrees of Freedom (for 7	47		
Critical Value (Value capped at 30	2.042		
Test Statis	4.421501		
Rel Bias (Y	Y		Shows Bias

In accordance with guidance provided in Appendix E of the Method Validation Guide the method is acceptable in spite of the apparent bias because all measured values are within the method uncertainty limits

MDC Test

Isotope	Sr-90
Required MDC, Bq/kg	44.4
MDC Spike Concentration, Bq/kg	44.4
Critical Net Concentration, Bq/kg	3.57

Sample ID	Activity, Bq/kg	Uncertainty, Bq/kg	Result </= CNC
1	43.3	2.6	N
2	41.5	2.5	N
3	49.9	2.5	N
4	43.7	2.6	N
5	41.1	2.5	N
6	40.1	2.4	N
7	33.5	2.4	N
8	39.8	2.6	N
9	33.1	2.4	N
10	31.9	2.3	N
Average	39.79	2.48	
Standard Deviation	5.588967	0.103279556	
Number of Results not meeting CNC Criterion	0		
Passes MDC Test (Y/N)	**YES**		

Blank Test

Isotope	Sr-90

Sample ID	Activity, Bq/kg	Uncertainty, Bq/kg	Date Prepared	Date Analyzed
1	4.8	1.6	6/25/2014	6/26/2014
2	4.7	1.5	7/2/2014	7/8/2014
3	7.2	2	7/28/2014	7/29/2014
4	4.7	1.8	7/30/2014	7/31/2014
5	2.3	1.5	8/12/2014	8/15/2014
6	1.6	1.6	8/5/2014	8/11/2014
7	4.5	1.4	6/16/2014	6/17/2014
Average	4.257143	1.628571429		
Standard Deviation	1.839255			
Effective Degrees of Fredom	6			
Critical Value	2.447			
Test Statistic	6.123862			
Absolute Bias (Y/N)	**Y**	**Shows Bias**		
Critical Net Concentration	3.568154			

The impact of this bias must be evaluated against the method data quality objectives

Acknowledgements

*Dr. Zhichao Lin, Winchester Engineering Analytical Center, Winchester MA

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Questions??

jack.bennett@ct.gov