Laboratory Proficiency Evaluation in Assessing Radioactive Contamination of Food

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All views and opinions expressed throughout the presentation are those of the presenter and do not necessarily represent views or official position of US Food and Drug Administration.

Significance

With growing risks posed by global aging nuclear facilities and widespread use of radioactive materials, FDA faces increasing challenges in safeguarding the nation's food supply from radioactive contaminations that may arise from nuclear accidents or acts of nuclear terrorism.

Nuclear Accident

Nuclear Terrorism

A major nuclear or radiological incident can prompt staggering demands on food monitoring given widespread public fear of radiation.

After the Fukushima nuclear disaster, almost **one million** food samples were analyzed in Japan alone over the next 4 years.[1]

1. Merz et al. *Environmental Science & Technology* **2015** *49* (5), 2875-2885

Food Emergency Response Network(FERN)

- To meet such demand for food monitoring, FDA has to work jointly with FERN radiological laboratory network to provide radioanalytical data needed for risk assessment and consequence management.
- The FERN integrates the nation’s food-testing laboratories at the local, state and federal levels into a network that is able to respond to emergencies involving biological, chemical or radiological contamination of food.

FERN **in Action**

https://www.fernlab.org/

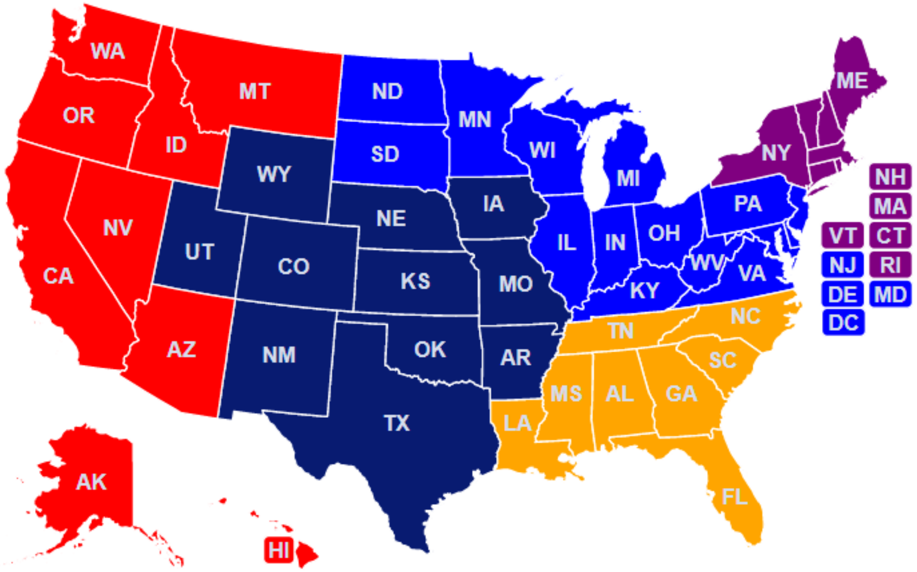
Assessing Laboratories

- FDA's decision-making will be based on large pools of data produced by different laboratories using diverse methods, which makes assessment of method acceptability and laboratory proficiency necessary.

- In analysis of food using radioanalytical techniques, difficulties and anomalies can arise from radionuclide characteristics, matrix disparity, or sample treatment.

- For instance, an existing gamma spectrometry method used for food analysis may require coincidence-summing and sample-attenuation corrections when analyte

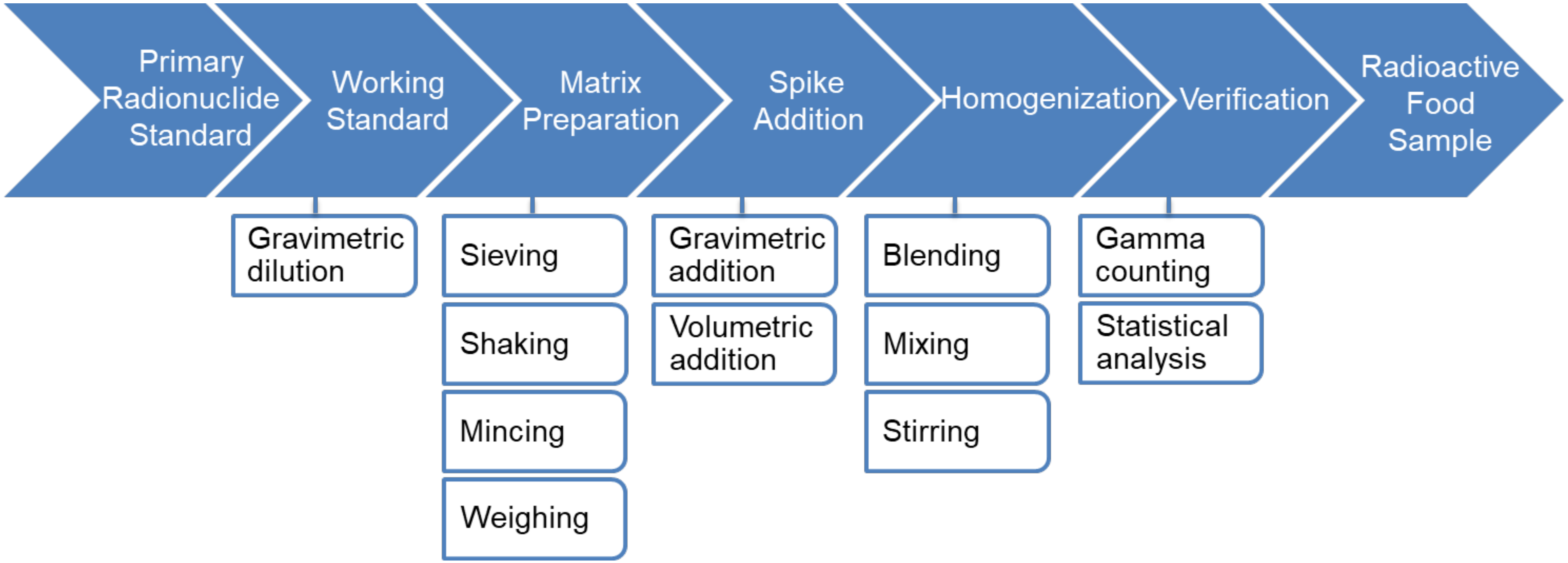
- **Evaluate the FERN labs capabilities by running proficiency testing program**



Preparing Radioactive Materials

Various preparation techniques were studied to provide fit-for-purpose radioactive food testing materials.

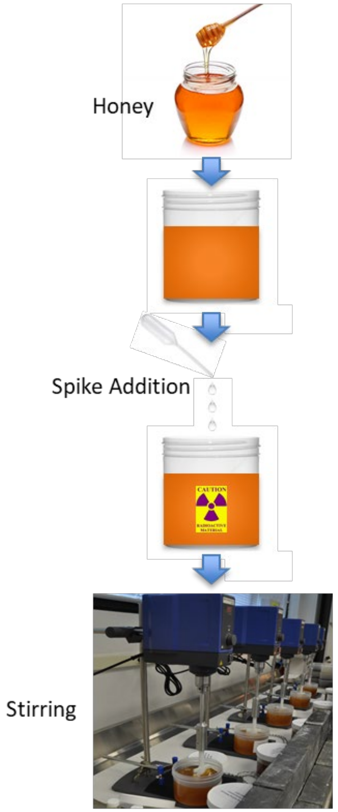
An effort was also made to ensure that the developed techniques are practical for producing sufficient test samples given broad network demand.



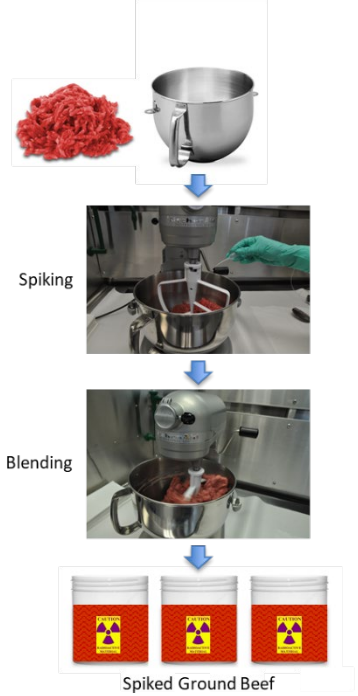
Preparing Radioactive Materials

Homogeneous Addition

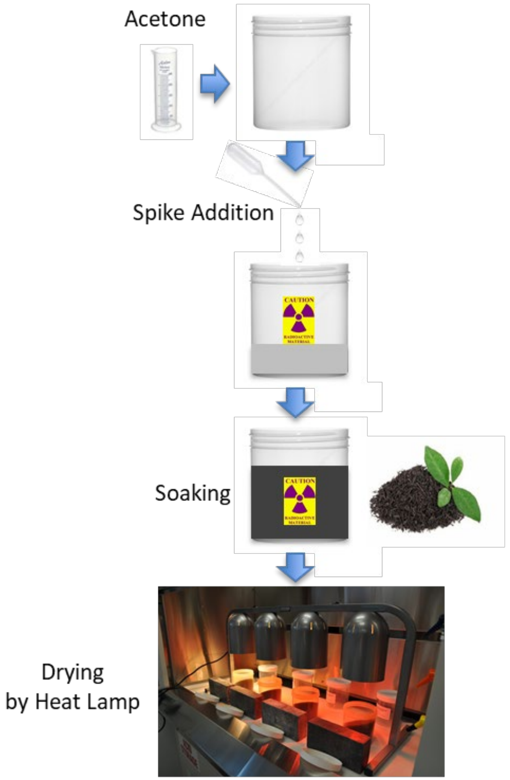
Mechanical Stirring



Mixing and Blending



Soaking



Heterogeneous Addition(Hot Spot)

Ground Beef

Tomato

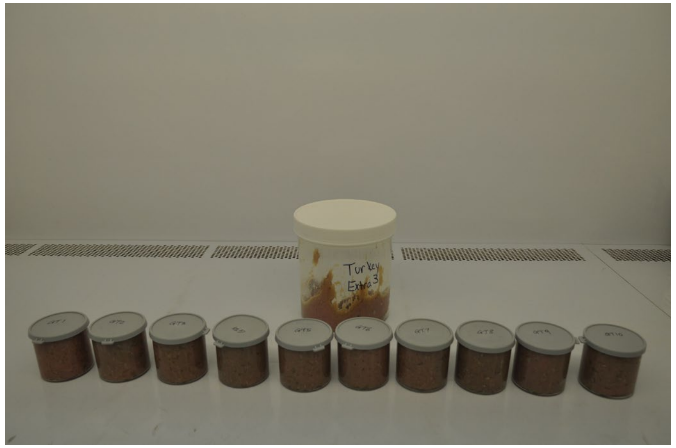
Preparing Radioactive Materials

Spiked Ground Turkey

A test sample was randomly selected and subdivided into 10 check samples. The measurement results showed a within-sample variability of <1.5%, indicating adequate sample homogeneity for the intended study.

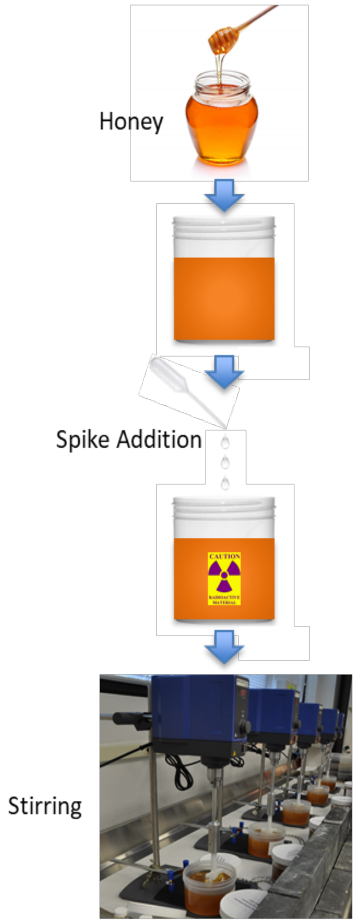
Sample Homogeneity Verification

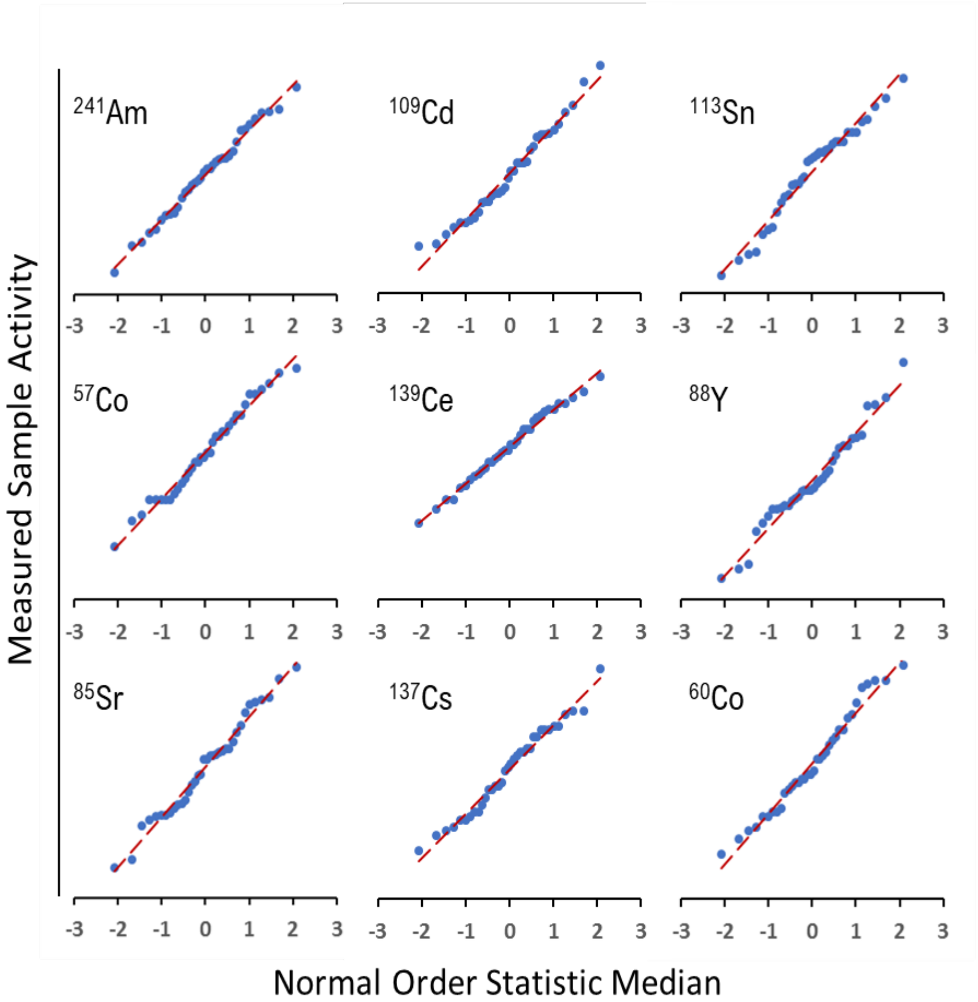
Check Samples



Sub ID	Geometry	Observed Activity	1s	1s, %
GT1	100-mL	0.443	0.011	2.37
GT2	100-mL	0.440	0.011	2.39
GT3	100-mL	0.434	0.010	2.37
GT4	100-mL	0.448	0.011	2.37
GT5	100-mL	0.428	0.010	2.38
GT6	100-mL	0.433	0.010	2.38
GT7	100-mL	0.446	0.011	2.35
GT8	100-mL	0.430	0.010	2.40
GT9	100-mL	0.441	0.011	2.38
GT10	100-mL	0.438	0.010	2.37
		Mean	0.438	
		1s	0.007	
		1s, %	1.5	

Preparing Radioactive Materials





A statistical analysis based on normal probability plot correlation coefficient (PPCC) plot was used to determine between-sample equivalence and within-sample homogeneity by comparing the calculated data linear correlation coefficient against normal PPCC critical values.

Method Performance Assessment and Laboratory Proficiency Study

Qualitive Scheme	Quantitative Scheme	Sequential Scheme	Time-Sensitive Scheme	Interpretive Scheme	Exercise Scheme
Evaluate proficiency in screening radioactivity in foods	Evaluate proficiency in quantifying radionuclides in foods	Evaluate accuracy in calibration of instrument efficiency	Evaluate laboratory ability to perform rapid emergency sample analysis	Evaluate proficiency in performing complex spectral analysis with reference spectra	Evaluate method performance characteristics

Based on FERN data quality objectives, a set of criteria were applied to evaluate method and laboratory performance:

For radionuclide identification:	Acceptable	if correctly identified
	Unacceptable	if incorrectly identified
For screening radioactivity:	Acceptable if	$D \leq 30\%$
	Unacceptable	if $D > 30\%$
For radionuclide quantification:	Acceptable	if $D \leq 10\%$
	Questionable	if $10\% < D \leq 15\%$
	Unacceptable	if $D > 15\%$

Study 1: Interlaboratory Comparison Study Using Qualitative Scheme

Assessing gamma-ray spectrometry methods for rapid screening of foods containing **heterogeneous** gamma radioactivity

Test sample sizes: 0.5 – 1.5 kg

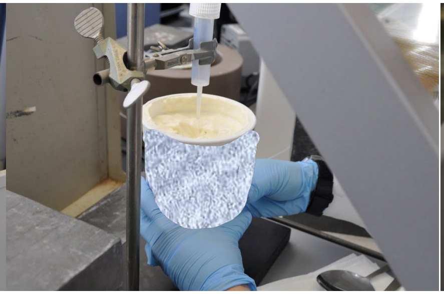
Radionuclides:

^{134}Cs	=	27.47±0.60	Bq/sample	12 – 40 times below regulatory limit
^{137}Cs	=	30.09±0.71	Bq/sample	16 – 53 times below regulatory limit
^{60}Co	=	99.88±1.81	Bq/sample	6 – 19 times below regulatory limit
^{133}Ba	=	17.25±0.47	Bq/sample	NA

Tomatoes

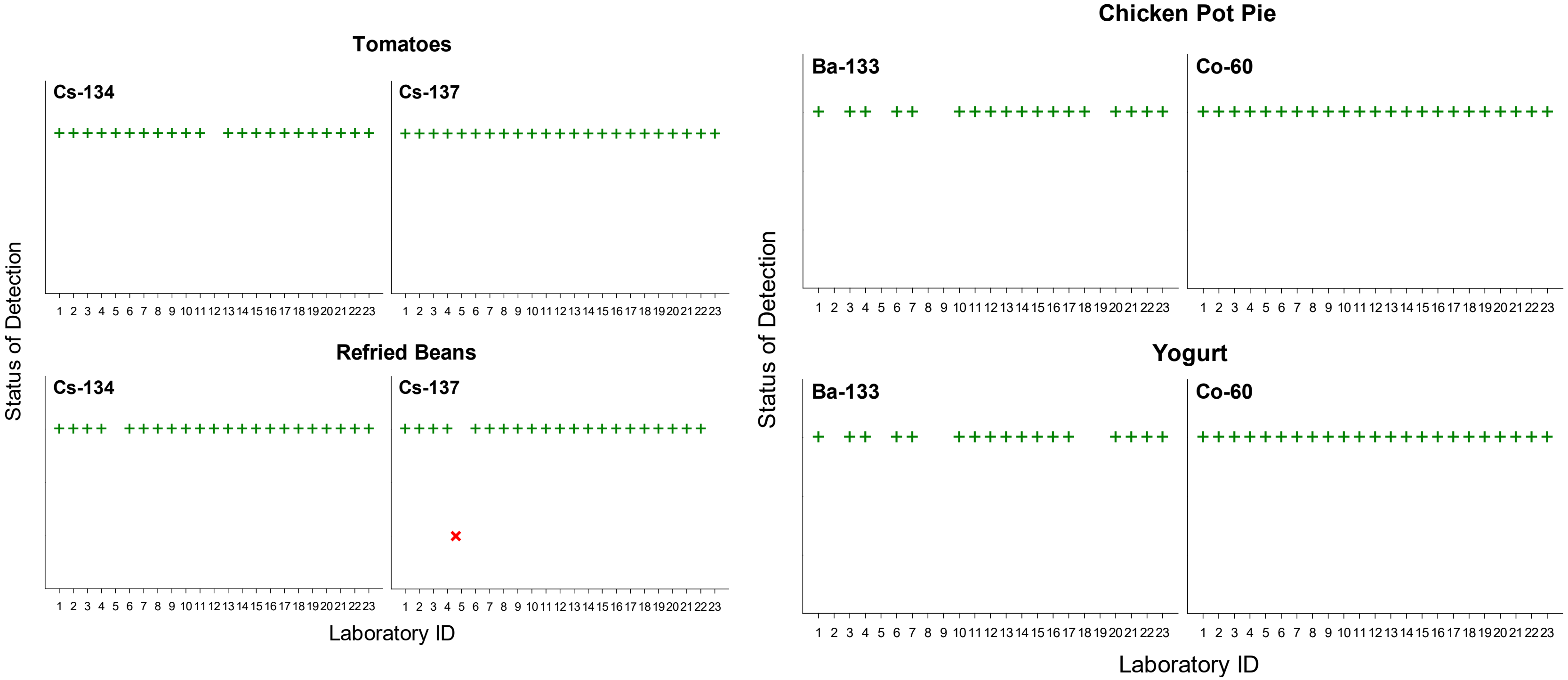
Chicken Pot Pie

Greek Yogurt

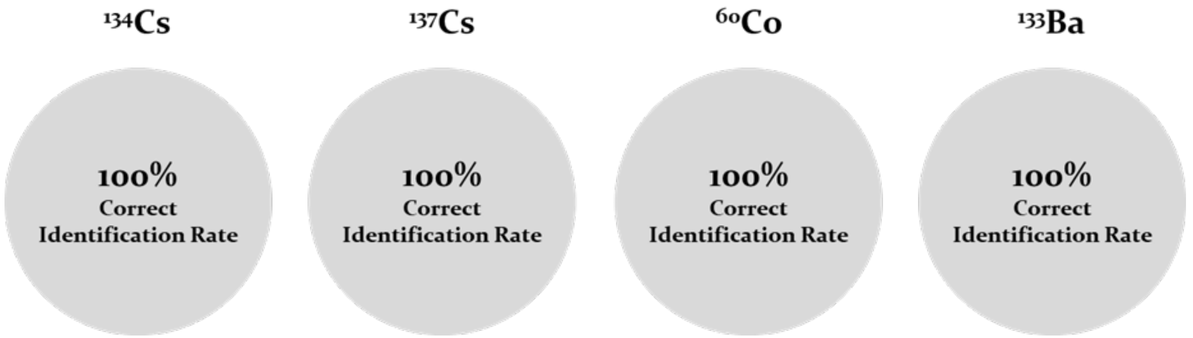


Refried Beans

Study 1: Interlaboratory Comparison Study Using Qualitative Scheme



Radionuclide **detection** observed in rapid screening of foods



Radionuclide **identification** observed in rapid screening of foods

Study 1: Interlaboratory Comparison Study Using Qualitive Scheme

Highlights of Study 1

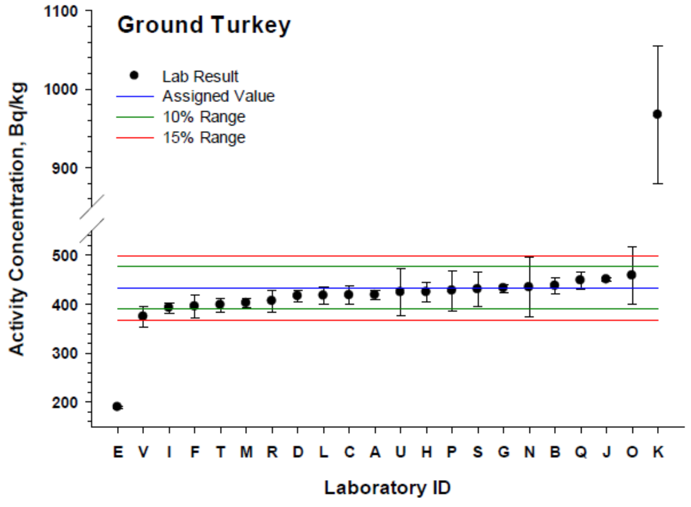
- ✓ When **detected,** all methods used by FERN radiological laboratories were able to rapidly identify unknown gamma-emitting radionuclides in foods
- ✓ Sample homogenization techniques practiced by FERN radiological laboratories were found to be adequate for screening **heterogenous radioactive contamination** in foods
- ✓ The **minimum detection limits** for all methods used were found to be much lower than 1/3 of regulatory limit for the radionuclides studied, except methods 5 and 12 due to partial sample loss and deficient peak search, respectively.
- ✓ While most methods were capable of detecting all radionuclides in the test samples, a few methods using smaller gamma detectors were having difficulty in detection of Ba-133

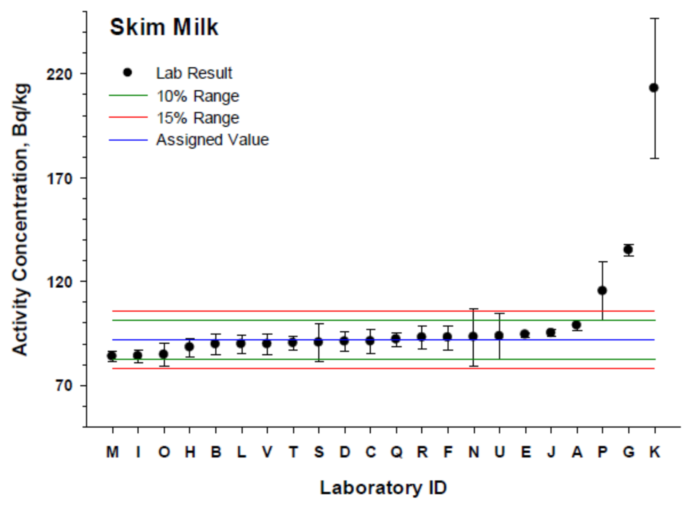
Study 2: Interlaboratory Comparison Study Using Quantitative Scheme

Assessing gamma-ray spectrometry methods for rapid screening of foods containing **homogeneous** gamma radioactivity

- Food Samples: Ground turkey, skim milk, Fruit Juice, green peas
- 22 FERN radiological laboratories for identification and quantification of ^{137}Cs by gamma spectrometry.

Study 2: Interlaboratory Comparison Study Using Quantitative Scheme

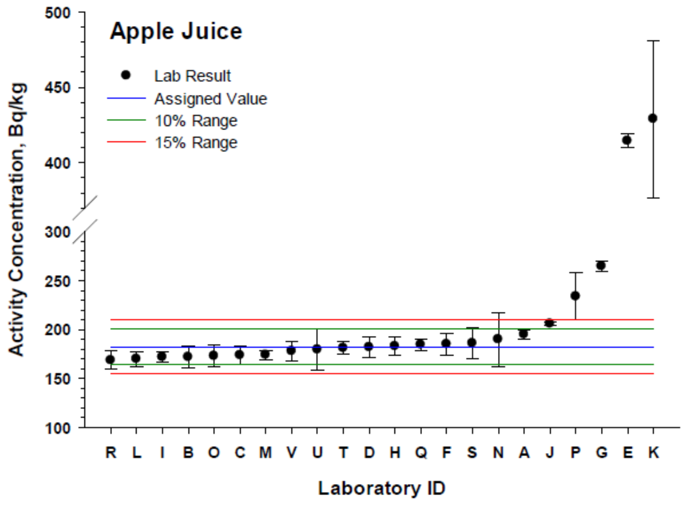


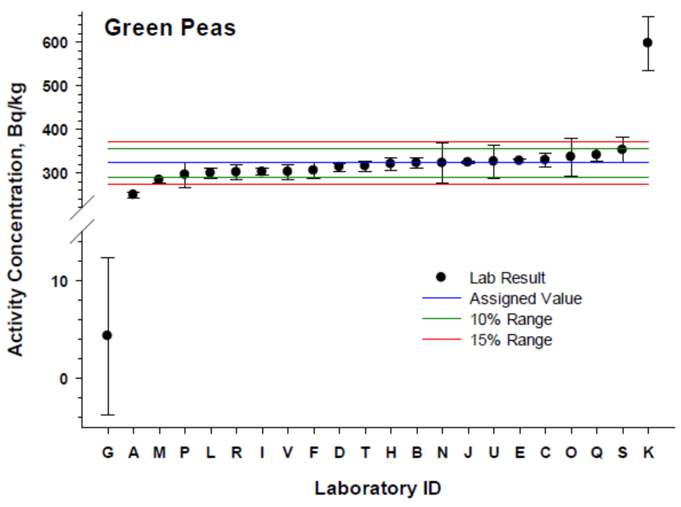


All laboratories were able to correctly identify the radionuclide Cs 137

> 82% of the laboratories were able to meet **±15%** acceptance criteria

The study also identified several laboratory performance issues that need to be further addressed, which include over- or under-estimated measurement uncertainties as shown by laboratories K and I, respectively, deficiencies in detector efficiency calibration as shown by laboratories K and G, and inadequate internal laboratory data review as shown by laboratories E and P.





Study 3: Interlaboratory Comparison Study Using Sequential Scheme

FDA

Assessing gamma-ray spectrometry methods for quantification of unknown gamma-emitting radionuclides in foods with **different compositions and densities**

Test Samples

Sample Size: 0.5 – 1 kg

Sample Matrix: Ground beef **Density = 1.17 g/mL**

Tea Leaf **Density = 0.56 g/mL**

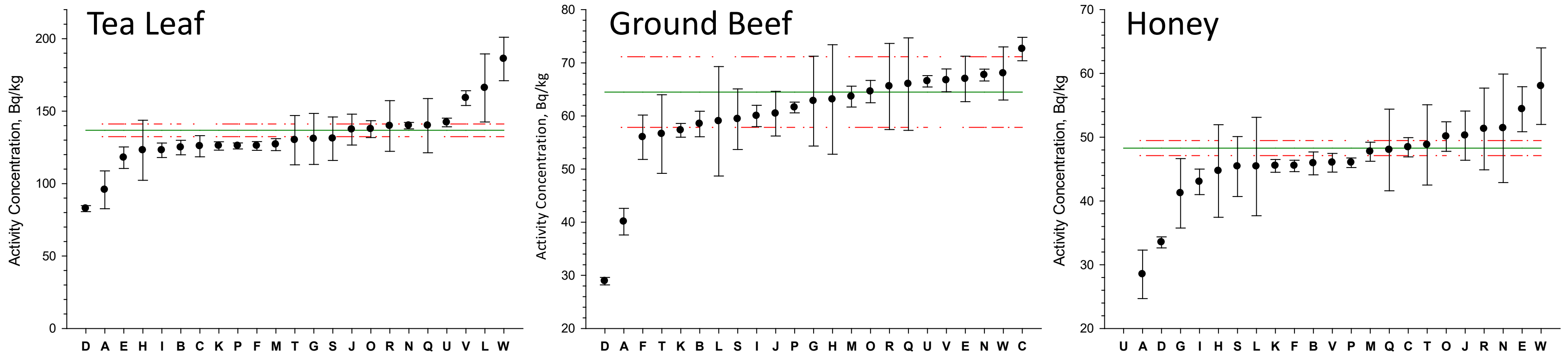
Honey **Density = 1.54 g/mL**

Radionuclide	Ground beef (BS)		Honey (HS)		Tea Leaf (TS)	
	Mean	±2s	Mean	±2s	Mean	±2s
^{241}Am	104.8	10.7	78.4	1.6	222.1	6.4
^{60}Co	64.5	6.6	48.3	1.2	136.8	4.4
^{137}Cs	40.7	4.2	30.5	0.9	86.3	3.0
^{88}Y	133.5	13.7	99.9	2.2	283.0	8.4
^{109}Cd	1340.6	139.4	1003.4	28.5	2831.9	99.1
^{57}Co	31.4	3.2	23.5	0.4	66.6	1.8
^{139}Ce	47.5	4.8	35.5	0.7	100.7	2.9
^{51}Cr	1349.4	137.3	1009.7	18.9	2860.0	79.1
^{113}Sn	81.1	8.4	60.7	1.5	171.9	5.6
^{85}Sr	107.5	10.9	80.4	1.5	227.8	6.3

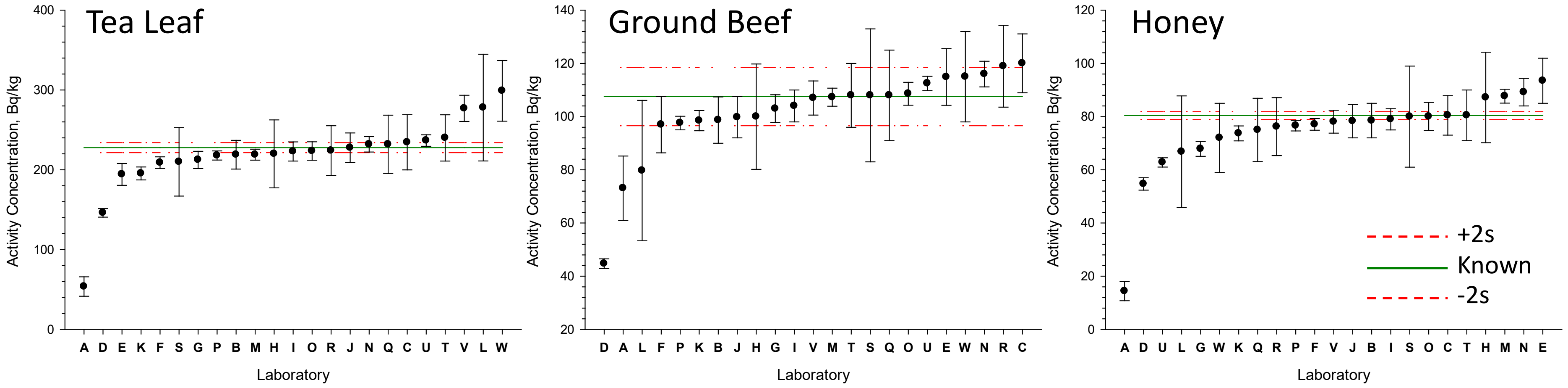
Note: The activity concentration is given at reference time of 4/1/2017 12:00 PM EST. The expanded uncertainty (2s) is estimated at 95% confidence level.

Study 3: Interlaboratory Comparison Study Using Sequential Scheme

Radionuclide = Co-60



Radionuclide = Sr-85



Density = 0.56 g/mL **Density = 1.17 g/mL** **Density = 1.54 g/mL**

Study 3: Interlaboratory Comparison Study Using Sequential Scheme

✓ From reviewing the plots, a few methodological issues were identified for method improvement and harmonization:

- ❖ Changes in sample density and coincidence summing of gamma rays notably biased the results of food analysis

- To improve accuracy, it requires implementation of counting efficiency corrections to compensate sample self-attenuation and coincidence-summing effects

- ❖ Efficiency calibrations for some methods were found to be problematic as indicated by consistently biased results for most radionuclides analyzed

- To obtain valid efficiency calibration, it requires use of traceable calibration standards and validation of all nuclide data files used for efficiency calibration and sample analysis

Summary

- Radioanalytical test capacity is enhanced by leveraging radioanalytical laboratory resources nationwide
- Various techniques for preparation of fit-for-purpose food-based radionuclide reference materials are developed, validated, and successfully used
- Most radioanalytical methods were sufficiently sensitive for detection of radioactive contaminants in foods much lower than the proposed regulatory limits
- Continuing study on improving current methods and developing new methods are needed to establish full radioanalytical capability for food monitoring and protection

Acknowledgements

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FERN Labs

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Thank You!

Questions or Comments?