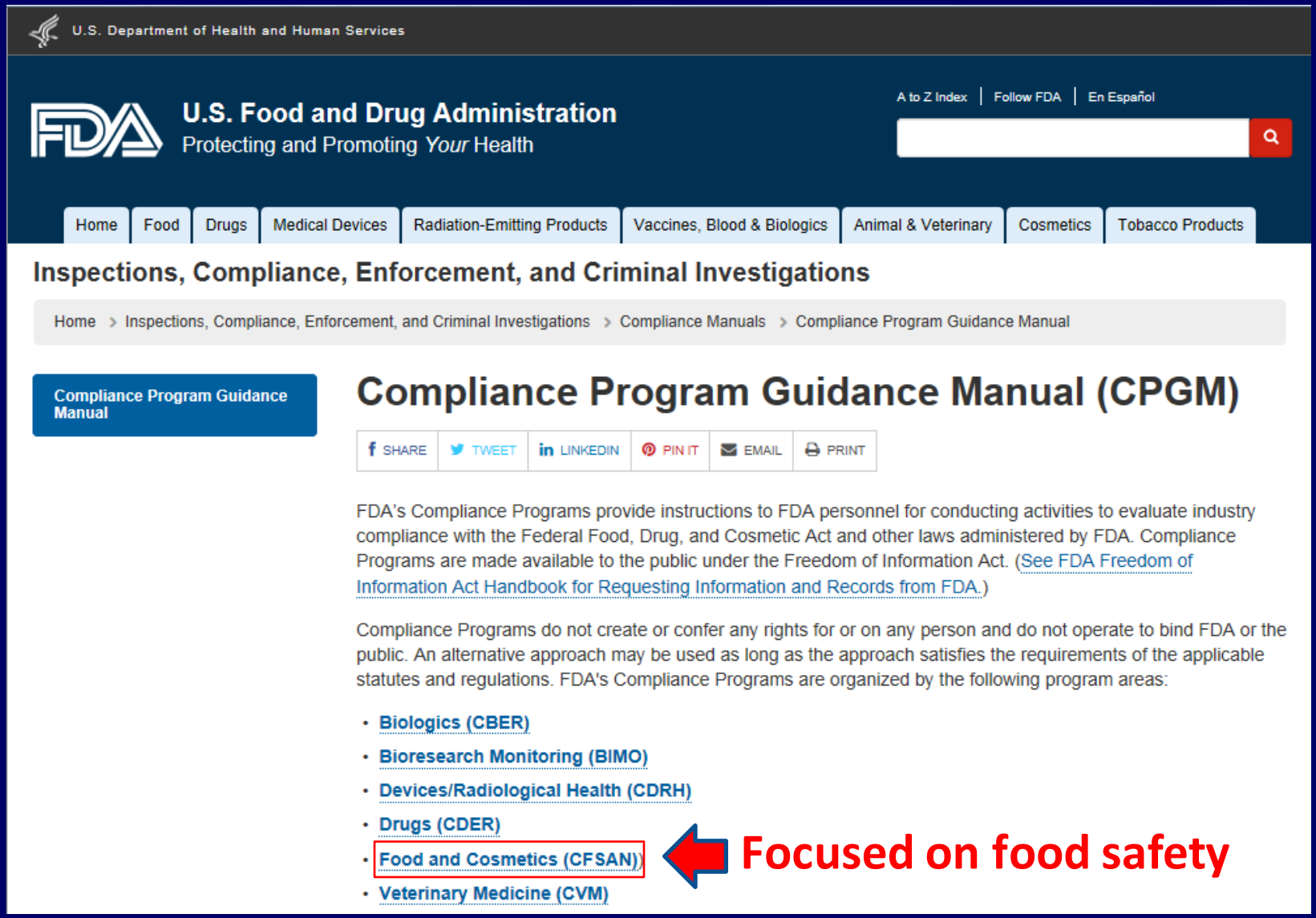
FDA Compliance and Emergency Response Programs for Safeguarding the Nation's Food Supply from Radioactive Contamination

Zhichao Lin, Stephanie Healey, Patrick Regan

Analytical Branch
Winchester Engineering and Analytical Center
Food and Drug Administration

Presentation to 24th Annual CIRMS Conference
National Institute of Standards and Technology
Gaithersburg, Maryland 2016

FDA's Compliance Program Areas



U.S. Department of Health and Human Services

U.S. Food and Drug Administration
Protecting and Promoting *Your* Health

A to Z Index | Follow FDA | En Español

Home | Food | Drugs | Medical Devices | Radiation-Emitting Products | Vaccines, Blood & Biologics | Animal & Veterinary | Cosmetics | Tobacco Products

Inspections, Compliance, Enforcement, and Criminal Investigations

Home > Inspections, Compliance, Enforcement, and Criminal Investigations > Compliance Manuals > Compliance Program Guidance Manual

Compliance Program Guidance Manual

Compliance Program Guidance Manual (CPGM)

SHARE | TWEET | LINKEDIN | PIN IT | EMAIL | PRINT

FDA's Compliance Programs provide instructions to FDA personnel for conducting activities to evaluate industry compliance with the Federal Food, Drug, and Cosmetic Act and other laws administered by FDA. Compliance Programs are made available to the public under the Freedom of Information Act. (See FDA Freedom of Information Act Handbook for Requesting Information and Records from FDA.)

Compliance Programs do not create or confer any rights for or on any person and do not operate to bind FDA or the public. An alternative approach may be used as long as the approach satisfies the requirements of the applicable statutes and regulations. FDA's Compliance Programs are organized by the following program areas:

- Biologics (CBER)
- Bioresearch Monitoring (BIMO)
- Devices/Radiological Health (CDRH)
- Drugs (CDER)
- Food and Cosmetics (CFSAN)
- Veterinary Medicine (CVM)

Focused on food safety

FDA's Food Compliance Programs

Program No.	Compliance Program Title
7303.003	Import Acidified and Low-Acid Canned Foods Program
7303.037	Domestic and Imported Cheese and Cheese Products
7303.039	National Drug Residue Milk Monitoring Program
7303.803	Domestic Food Safety Program
7303.803a	Domestic Acidified and Low-Acid Canned Foods
7303.819	Import Foods - General
7303.842	Seafood Processor Inspection Program - Domestic and Foreign Facilities
7303.844	Import Seafood Products Compliance Program
7303.847	Juice HACCP Inspection Program
7304.004	Pesticides and Industrial Chemicals in Domestic and Imported Foods
7304.018	Chemotherapeutics in Seafood Compliance Program
7304.019	Toxic Elements in Food & Foodware - Import and Domestic
7304.839	Total Diet Study
7307.001	Mycotoxins in Domestic and Imported Foods
7309.006	Imported Foods - Food and Color Additives
7318.002	Retail Food Protection - State
7318.003	Milk Safety Program
7318.004	Molluscan Shellfish Compliance Program
7318.029	Interstate Travel Program - Conveyances and Support Facilities
7321.002	Medical Foods - Import and Domestic
7321.005	Domestic and Import NLEA, Nutrient Sample Analysis General Food Labeling Program
7321.006	Infant Formula Program - Import and Domestic
7321.008	Dietary Supplements - Import and Domestic

ORA's Mission:

Protects consumers and enhances public health by maximizing compliance of FDA regulated products and minimizing risk associated with those products

ORA's staff are dispersed throughout the country

ORA is comprised of

- 5 Regional offices
- 20 District offices
- 11 Laboratories
- 1 Forensic Laboratory
- **1 Radiological Laboratory**
- 150 Resident posts & border stations

FDA uses the following programs to monitor radioactive contaminants in foods:

- Import Food Program
- Domestic Food Program
- Total Diet Study Program
- FERN Emergency Response Program

Import Food Program

Interest: Concern on excessive dietary exposure to radiation
Typical level of radionuclides in foods from different regions

Objectives: Monitor level of radionuclides in foods imported from countries most likely to have foods products with radionuclide contamination

Approach: Collect import food products from the affected areas/regions evenly throughout fiscal year

Deliverable: Activity concentrations of ^{134}Cs, ^{137}Cs, and ^{90}Sr (if necessary)

Foreign Site Uploading

Imports Arrival

CBP Screening

FDA Inspection

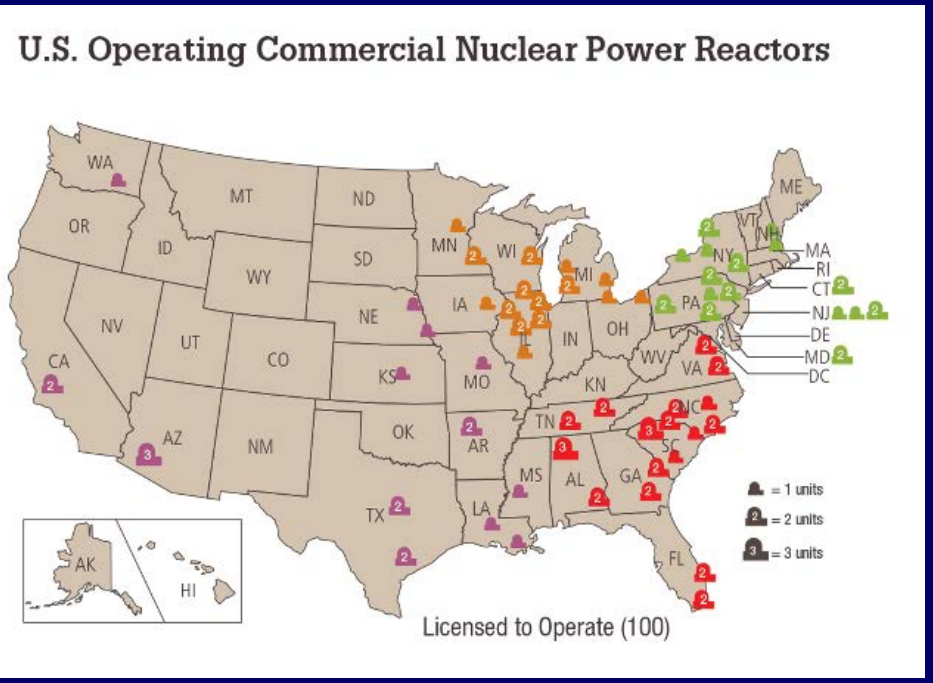
Domestic Food Program

Interest: Concern on dietary exposure to radiation
Current level of radionuclides in foods and trends

Objectives: Monitor radionuclides in foods produced in the vicinity of nuclear power plants in the US

Approach: Collect local foods within 10 miles from the power plants such as fish, milk, raw vegetables, and food crops (4 sites/year)

Deliverable: Activity concentrations of ^{3}H, ^{90}Sr, and γ-emitters



Total Diet Study Program

Interest: Impact on nation's food supply from environmental contamination, changes in agricultural practices, and food processing technology

Objectives: Determine prevailing level of radionuclides in foods
Identify trends in radioactivity concentration in representative American diet
Estimate daily dietary exposures for the total U.S. population

Approach: Collect market basket samples over 5-6 week period from different US cities 2 times a year for radionuclide analysis

Deliverable: Activity concentrations of ^{226}Ra, ^{232}Th, ^{241}Am, ^{140}Ba, ^{134}Cs, ^{137}Cs, ^{60}Co, ^{131}I, ^{140}La, ^{103}Ru, and ^{106}Ru in typical american diet

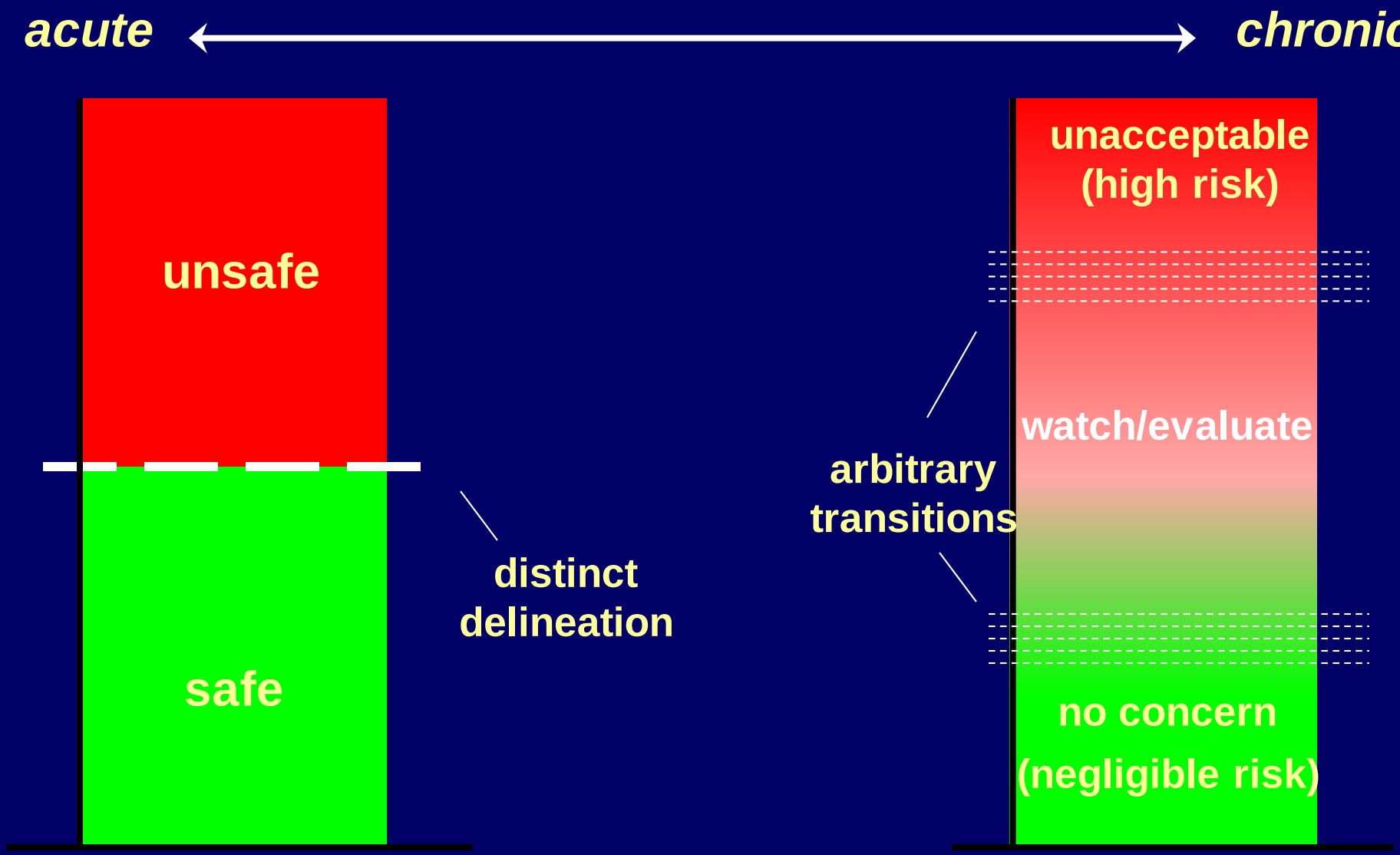
FERN Emergency Response Program

Interest :	Quick and coherent decision making in the event of radiological emergency
Objectives:	Develop rapid and high-throughput methods to detect radionuclides in foods for nuclear or radiological emergency Obtain greater geographic coverage and operation flexibility
Approach:	Communicate and collaborate with state laboratories Provide resources, trainings, and proficiency tests
Deliverable:	Full rang of radioanalytical capability for detecting all radionuclides concerning food safety Sufficient FERN laboratory network sample surge capacity Ability to provide analytical results within 48 hours after sample receiving

FDA's DILs were developed for the radionuclides that contribute predominant radiation dose through ingestion

Accident/Source Types	Principal Radionuclides
Nuclear reactors	I-131, Cs-134, Cs-137, Ru-103, Ru-106
Nuclear fuel reprocessing plants	Sr-90, Cs-137, Pu-239, Am-241
Nuclear waste storage facilities	Sr-90, Cs-137, Pu-239, Am-241
Nuclear weapons (dispersal wo/detonation)	Pu-239
Radioisotope thermoelectric generators and heater units for space vehicles	Pu-238
Others (such as RDD and IND events)	?

COMPARISON OF RISK TYPES



FDA derived intervention level (DIL)

The Basic Formula:

$$\text{Derived Intervention Level (Bq/kg)} = \frac{\text{PAG}}{f \times \text{food intake (kg)} \times \text{dc (mSv/Bq)}}$$

Where:

PAG = Protection action guide for a given radionuclide (mSv)

f = Fraction of the food intake assumed to be contaminated

dc = Dose coefficient; the radiation dose received per unit of radioactivity ingested (mSv/Bq)

FDA DILs for Principal Radionuclides

Radionuclide	DIL, Bq/kg	Age Group
Sr-90	160	15 years
I-131	170	1 year
Cs-134	958	15 years
Cs-137	1370	15 years
Cs-(134+137)	1200	15 years
Ru-103	6800	3 months
Ru-106	450	3 months
Ru-(103+106)	(C3/6800+C6/450)< 1	3 months
Pu-238	2.5	3 months
Pu-239	2.2	3 months
Am-241	2.0	3 months
Pu-(238+239)+Am-241	2.2	3 months

FDA DILs for Other Radionuclides

Radionuclide	DIL, Bq/kg	Age Group
Sr-89	1400	3 months
Y-91	1200	3 months
Zr-95	4000	3 months
Nb-95	12000	3 months
Te-132	4400	3 months
I-129	56	10 years
I-133	7000	1 year
Ba-140	6900	3 months
Ce-141	7200	3 months
Ce-144	500	3 months
Np-237	4	3 months
Np-239	28000	3 months
Pu-241	120	3 months
Cm-242	19	3 months
Cm-244	2	3 months

Guidance Levels for Radionuclides in Foods, Bq/kg

FDA		Health Canada			IAEA		WHO FAO	
Domestic & Import			Milk	Others	General Consumption		General Consumption	
^{238}Pu ^{239}Pu ^{241}Am	2	^{238}Pu ^{239}Pu ^{240}Pu ^{242}Pu ^{241}Am	1	10	^{239}Pu ^{241}Am	10	^{239}Pu ^{241}Am	10
^{90}Sr	160	^{90}Sr	30	100	^{90}Sr	100	^{90}Sr	100
^{131}I	170	^{131}I	100	1000	^{131}I ^{134}Cs ^{137}Cs	1000	^{131}I ^{134}Cs ^{137}Cs	1000
^{134}Cs ^{137}Cs	1200	^{134}Cs ^{137}Cs	300	1000				
^{103}Ru ^{106}Ru	(^{103}Ru/6800+ ^{106}Ru/450)<1	^{103}Ru	1000	1000	Milk & Infant Foods		Milk & Infant Foods	
		^{106}Ru	100	300	^{239}Pu ^{241}Am	1	^{239}Pu ^{241}Am	1
		^{89}Sr	300	1000	^{131}I ^{90}Sr	100	^{131}I ^{90}Sr	100
					^{134}Cs ^{137}Cs	1000	^{134}Cs ^{137}Cs	1000

Issue:	International community uses consensus PAG dose value to drive intervention level. However, the values of other components in the basic formula for computing DILs are selected differently by countries or organizations.
Result:	Different DILs are recommended.
Status:	Lack of harmonized DILs
Practice:	Exported foods subject to importing country's DILs Commission of European Communities' DILs govern trade within Europe CODEX (FAO/WHO) DILs for use in international trade Foods imported to USA subject to FDA's DILs
FDA's View:	DILs should be referred as regulatory guideline rather than hard/fixed control limits

Historic Nuclear Events and FDA Responses

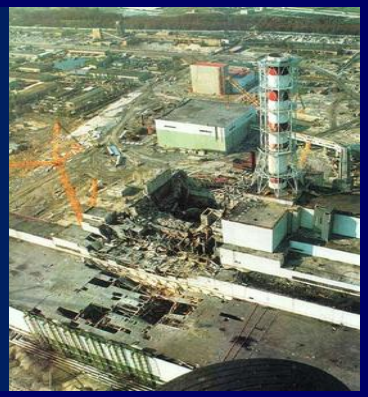
Three Mile Island, USA

1979

Milk, fish and water within a 20-mile radius of the facility were sampled and analyzed. No food or other products were contaminated.

Chernobyl, Ukraine

1986



2600 imported food shipments including cheeses, mushrooms, pasta, reindeer/elk meat, spices were sampled. 23 of the shipments were detained based on the positive detections in 705 test portions from 1987-1992

Fukushima, Japan

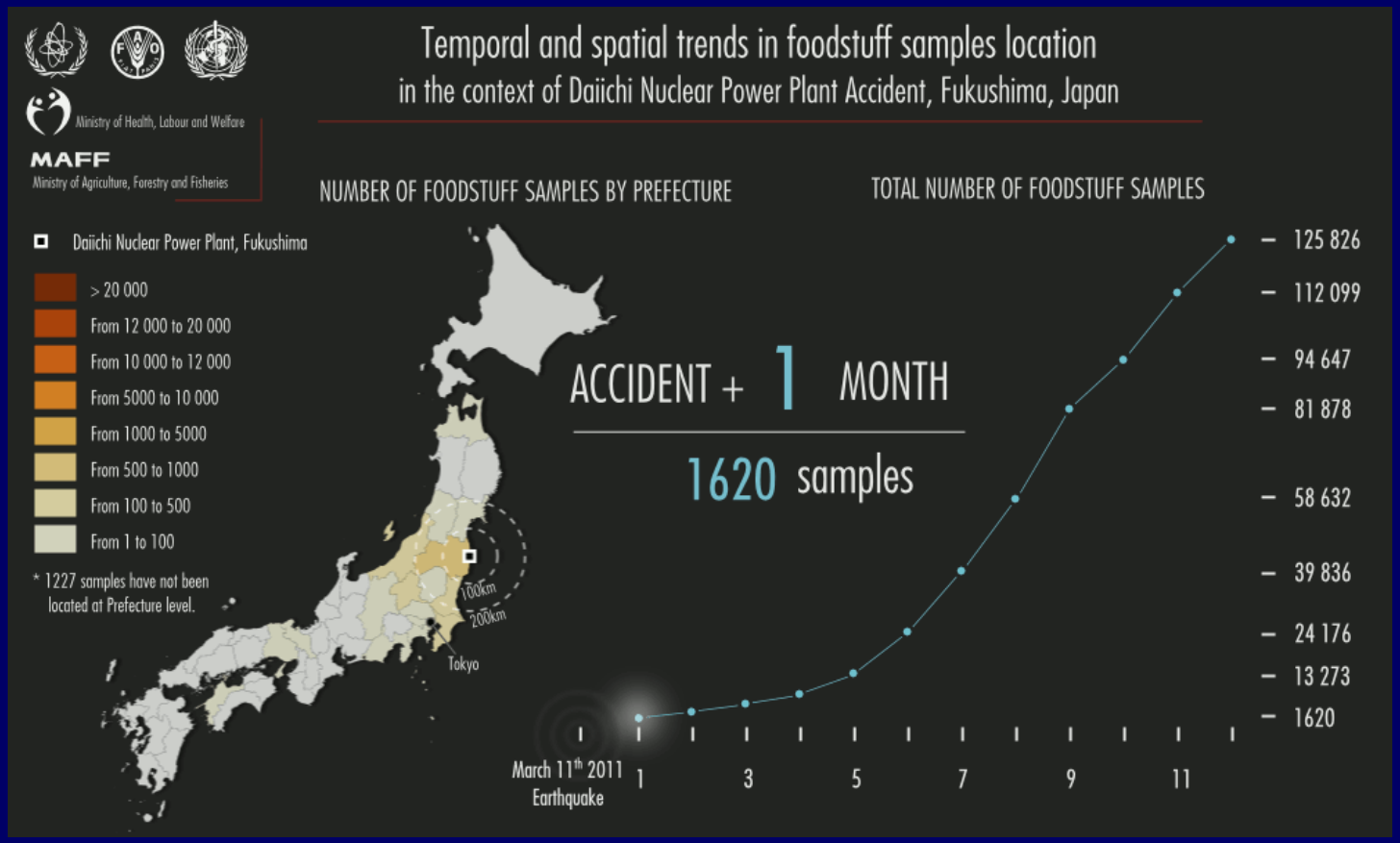
2011

~5000 food samples were collected from the food shipments imported from Japan. Only two food samples, ginger and green tea, showed low levels of ^{131}I, ^{134}Cs, and ^{137}Cs, that are far below the FDA's DILs

Food monitoring after Fukushima nuclear power accident

Number of Foods Sampled and Analyzed by Japan

Huge Demand on Radioanalytical Resources and Surge-Capacity



FDA Food Radiological Emergency Response Network

Location of Participating Laboratories

Air Shipping of FDA-Regulated Products for Radioactivity Analysis

Concerns:

Cost
Time
Safety
Availability
Efficiency
Contamination

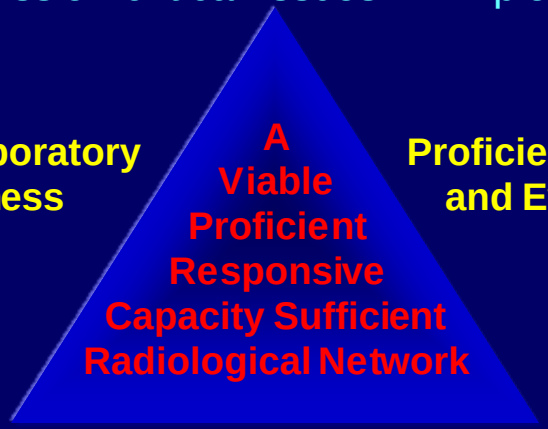
Key Components for Building and Maintaining FERN Radiological Measurement System

- Instigate effective methodology transfer
- Promote common standards of performance
- Improve skill & extend knowledge base
- Communicate on emerging & mission-critical issues

Training on Laboratory Preparedness

- Improve & streamline data reporting
- Provide a basis for data comparability
- Ensure laboratory competence
- Implement quality control & assurance

Proficiency Testing and Evaluation



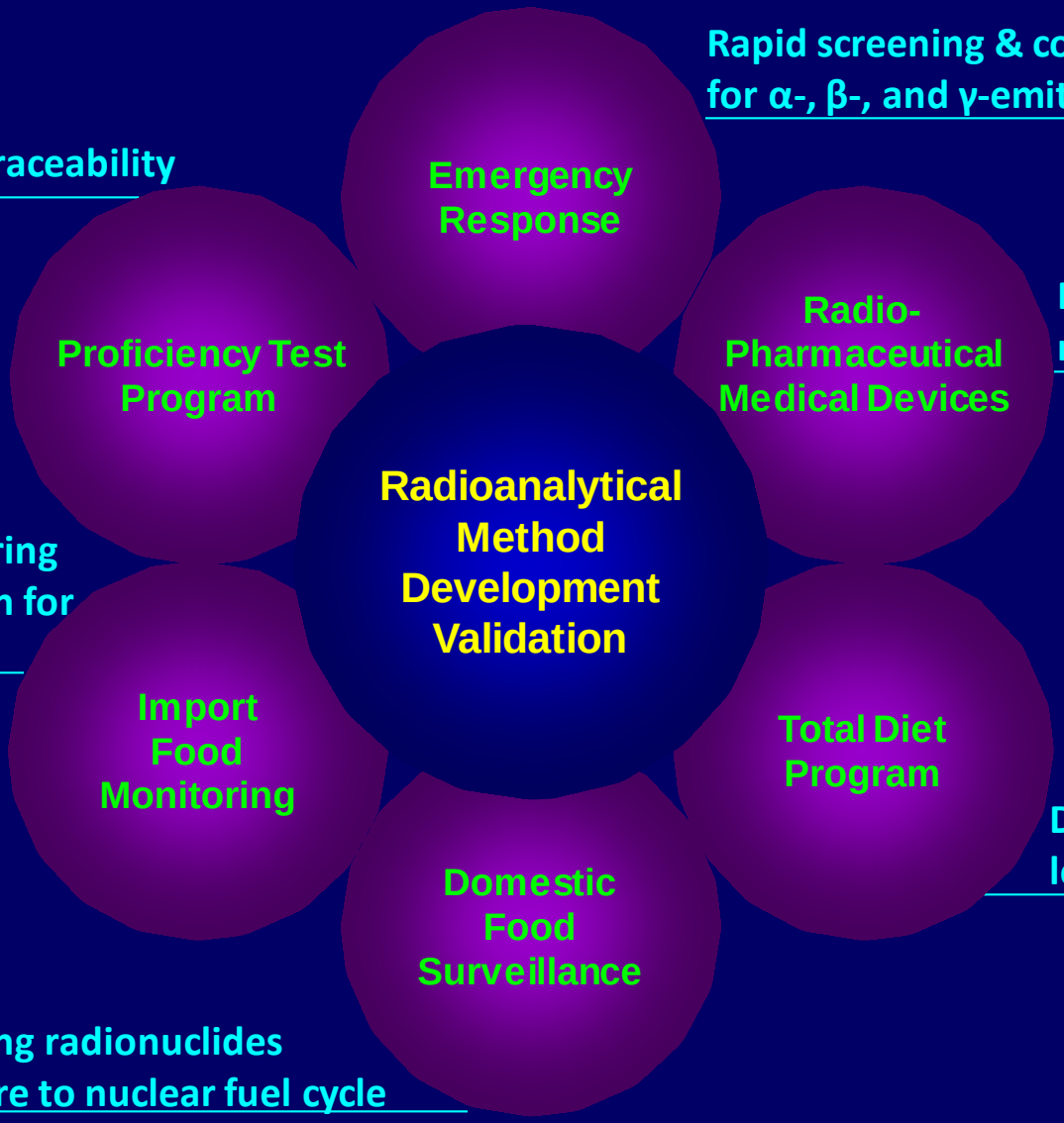
Collaborative Method Development and Validation

- Develop & validate standardized methods
- Conduct matrix extension study
- Identify & eliminate method deficiency
- Share expertise & ideas

Needs and Objectives for Radioanalytical Method Development

Emergency Response	To achieve rapid and high-throughput sample analysis for screening α-, β-, and γ-radioactive contaminants in foods
Proficiency Test Program	To obtain accurate reference values and confirmatory analysis for ensuring PT samples with verifiable traceability
Import Food Monitoring	To provide official analysis assuring analytical justification for regulatory enforcement and action
Total Diet Program	To determine background levels of α-, β-, & γ-emitting radionuclides in foods for customary consumptions and maintain laboratory readiness
Radio-Pharmaceuticals	To verify drug potency & detect radioactive impurities
Domestic Food Surveillance	To analyze and monitor the radionuclides that are signature to nuclear power production and nuclear fuel-waste cycle

Needs and Objectives for Radioanalytical Metrology Development



Rapid screening & confirmation analysis for α-, β-, and γ-emitters

Accuracy & precision for PT verification & traceability

Detecting radioactive impurities

Official analysis assuring analytical justification for regulatory action

Detecting ultra-low level α-, β-, & γ-emitters

Analyzing radionuclides signature to nuclear fuel cycle

Considerations in Development of FERN Radioanalytical Methods

➢ **Objective of Analysis**	Screening/High throughput/Qualitative/Positive Presence Confirmatory/Specific/Quantitative/Necessary action
➢ **Prospective Threats**	Radionuclides of greatest concern
➢ **Detection Capability**	Workable at level of regulatory significance
➢ **Robustness & Reliability**	High tolerance to a wide range of food matrices Consistent, Accurate, & Confident
➢ **Versatility**	Adaptable to different analytical procedures and detection technologies for a broad range of applications
➢ **General Availability**	Ability for implementation in common radiological laboratories and use of mostly accessible instruments

Elevated ionizing radiation encountered during imported product inspections

After 911, with increased security and safety measures, FDA inspectors started to carry radiation pagers while performing imported product inspections. Over the years, certain imported products had alarmed by the pagers as containing radioactivity and were sent to WEAC for confirmatory analysis. Here are some interesting examples:

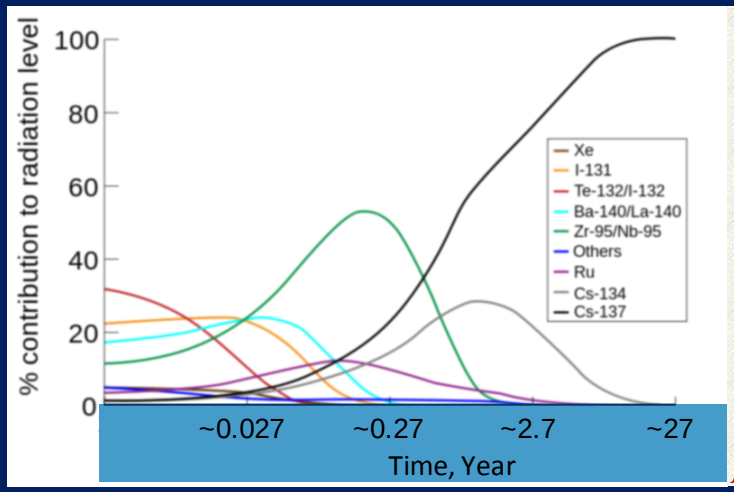
- Radioactive mushrooms and blueberry concentrate - Food
- Radioactive fragrance - Cosmetic
- Radioactive foot patch - Health Product
- Radioactive "Oil Karat" - Cooking Ware
- Radioactive pendant - Health Product/Cosmetic
- Radioactive ginger powder & green tea - Food

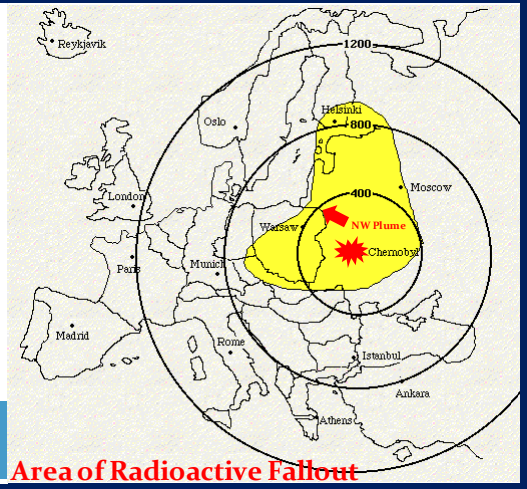


Imported foods carrying signature of Chernobyl nuclear fallout

Today, the FDA investigators still occasionally encounter radioactive mushrooms and blueberry concentrate imported from certain eastern european countries during import food products inspection.

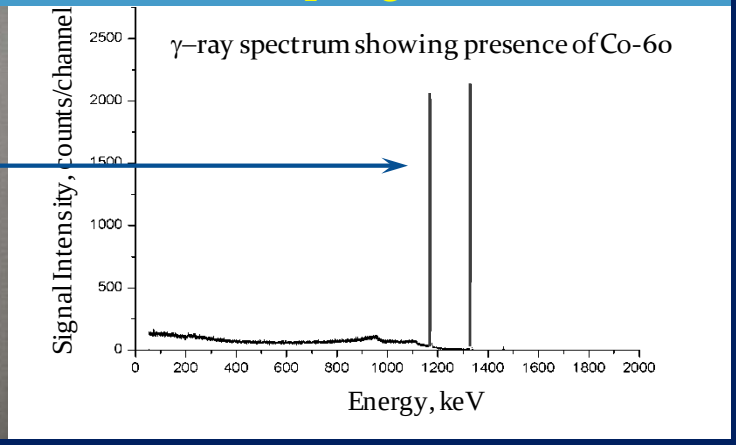
It is known mushrooms grow in lichen and mosses which accumulate Cs-137, and the blueberries used for making juice concentrate was likely contaminated due to specific behavior of Cs-137 in forest ecosystem





Imported Fragrance Carrying Signature of Radioactive Contaminated Steel

Radioactivity found in stainless steel metal spring



Trail of Radioactive Contamination

Mishandled/lost Co-60 source

↓

Mixed/melted down with scrap metal

↓

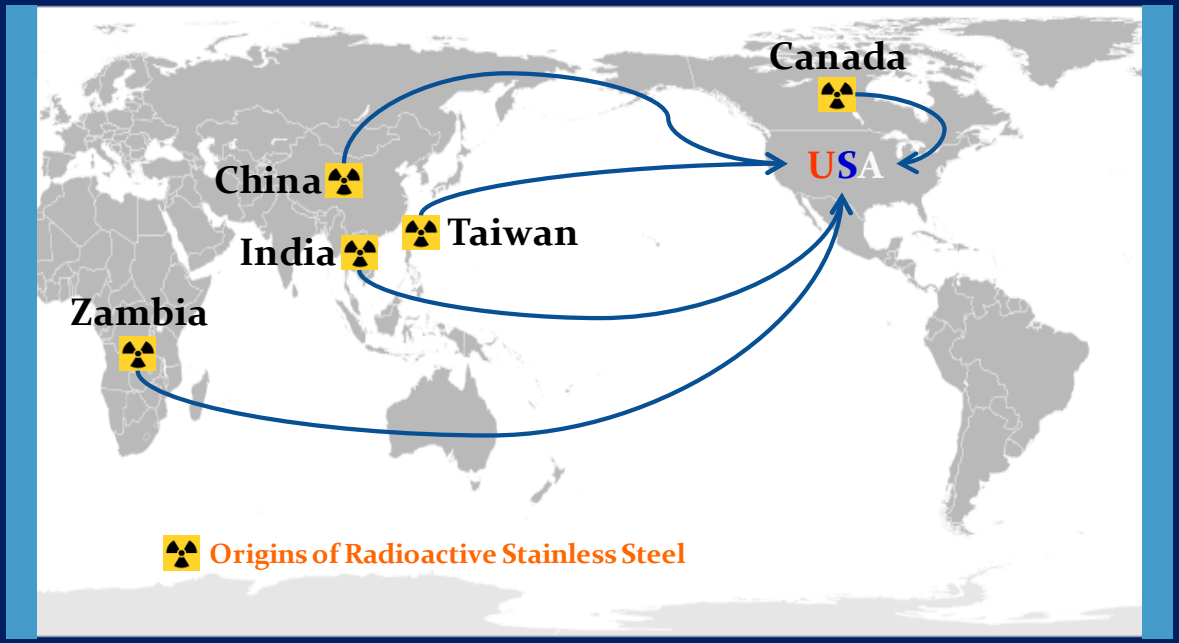
Tainted steel entering global markets

↓

Used for making various commercial products

↓

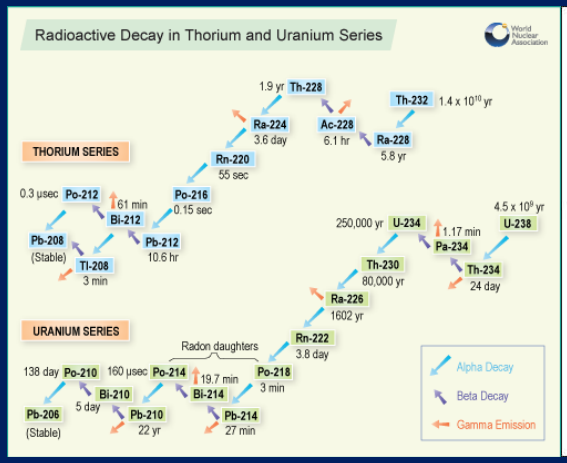
Tainted commercial products imported into US

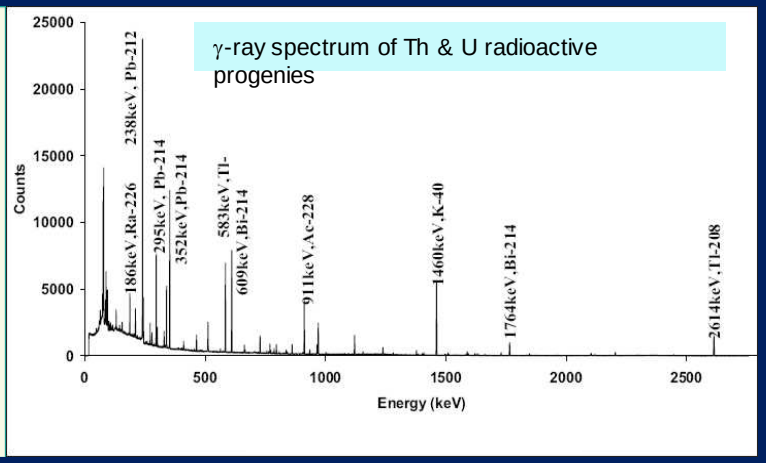


Imported Foot Patch Containing Naturally-Occurring Radioactive Thorium and Its Progenies

Foot patch are dubiously advertised for detoxing body and improving blood circulation. It is mostly made of plant extracts, **mineral powders**, binding agents, and crustacean shells. It is well-known fact that high content of naturally-occurring radioactive thorium presents in certain types of minerals.

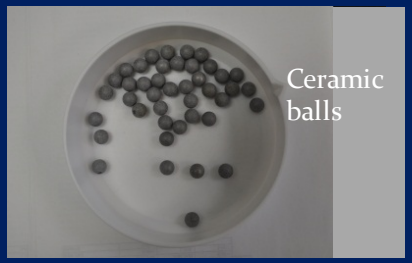
Gamma ray spectrometry analysis confirmed that the high radiation reading for the foot patch is coming from radioactive progenies of naturally-occurring thorium.





Imported “Oil Karat” containing naturally-occurring radioactive thorium and its progenies

A perforated stainless steel cage containing small ceramic balls was found by FDA inspector to emit high level of radiation at the time of inspection. The imported product was marketed to prolong useful life of cooking oil for frying. Per the user’s instruction, the product needs to be submerged in the oil so that ceramic balls can suppress oil molecules to bond together.



Each ceramic ball is comprised of a layer of clay rich in thorium, which through natural radiation emits far infrared and activates the three other layers, composed of titanium dioxide, activated carbon, and a mixture of titanium dioxide and platinum.

The ceramic balls were taken out of the cage and analyzed by gamma ray spectrometry. High level gamma radiation was detected and radioactive thorium progenies were positively identified.

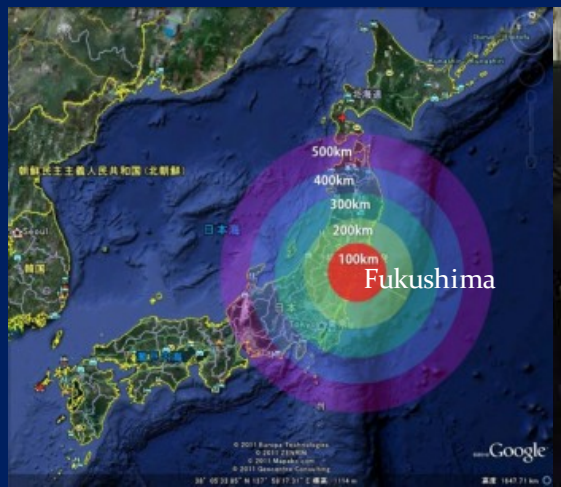
Imported “Quantum Pendant” Found Radioactive

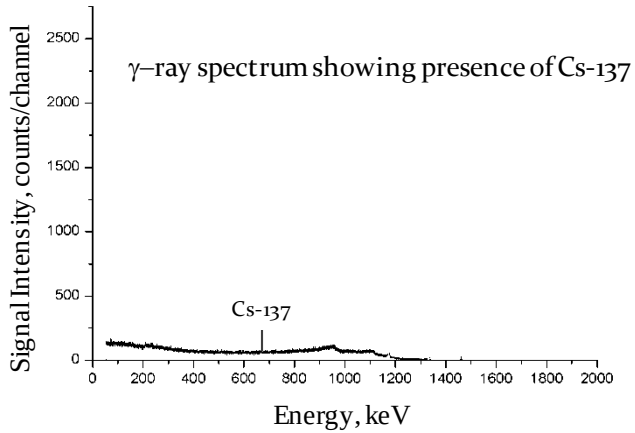
Imported “Quantum Pendant” been falsely claimed by the maker to provide various health benefits was found quite radioactive. While wearing, it is kept in direct contact with the skin and its long-term wearing arises radiation safety concerns.

The artifact is made from natural minerals that are rich in natural thorium and uranium. It is their radioactive progenies giving off noticeable ionizing radiation . Despite its widely known radiation hazard, this radioactive pendant is still being sold in US today.

Imported Japanese Ginger Powder & Green Tea Found Tainted with Radioactive Cs-137

In response to Fukushima nuclear power plant accident , >3000 food products imported from Japan to US were tested by FDA/WEAC for possible radioactive contaminants. Among all foods been tested, only two, i.e., dried ginger powder and green tea, were found to contain detectable amount of Cs-137. However, the levels of radioactivity detected were well blow the FDA regulatory limit.





FERN Method Development Guideline and Review Process

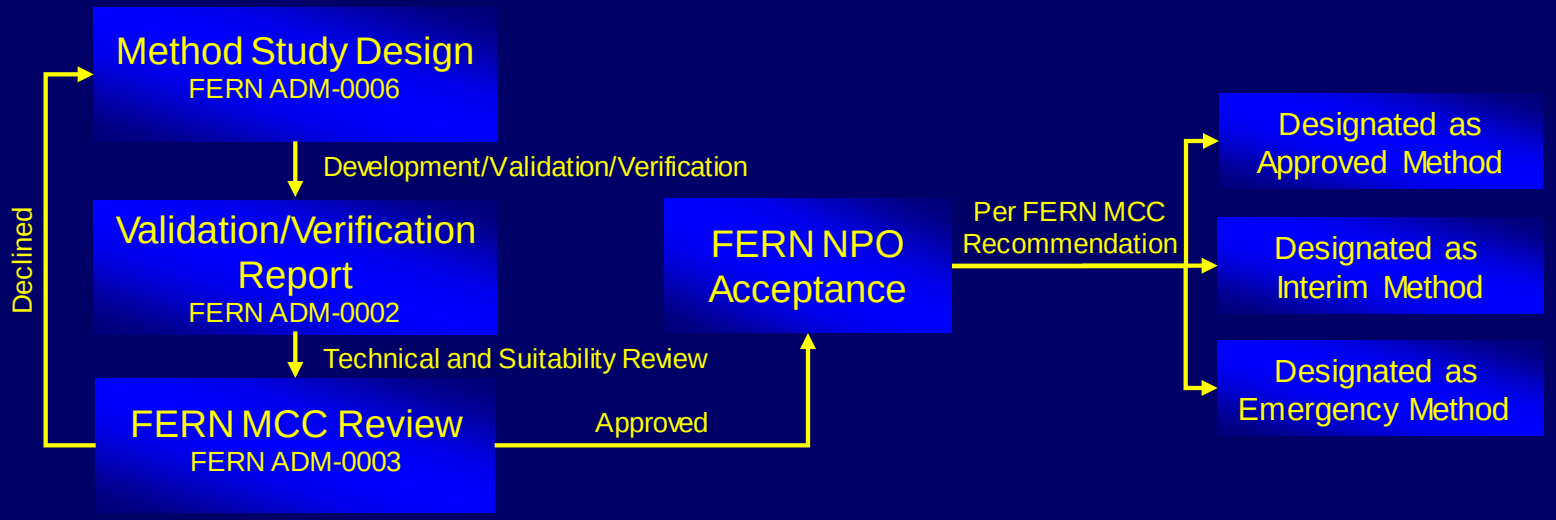
Three Method Categories

- **Emergency Method**
- **Interim Method**
- **Approved Method**

Note:
A-Quantitative
B-Qualitative

Four Validation Levels

	# of Matrices	# of Source	# of Lab	# of Spike Levels	# of Replicates
Level-1	≥1	≥1	1	≥1	≥3
Level-2	≥1	≥1	1	3	5
Level-3	≥1	≥1	2-7	3	5
Level-4	≥5	3-5	$8^{A}/10^{B}$	2	$2^{A}/6^{B}$



Radionuclides of Greatest Concern

Radionuclide	Principal Emission
^{241}Am	α
^{238}Pu	α
^{239}Pu	α
^{240}Pu	α
^{210}Po	α
^{242}Cm	α
^{243}Cm	α
^{244}Cm	α
^{252}Cf	α
^{226}Ra	α
^{237}Np	α
^{228}Th	α
^{230}Th	α
^{232}Th	α
^{234}U	α
^{235}U	α
^{238}U	α

Radionuclide	Principal Emission
^{137}Cs	γ
^{60}Co	γ
^{153}Gd	γ
^{192}Ir	γ
^{75}Se	γ
^{170}Tm	γ
^{169}Yb	γ
^{141}Ce	γ
^{144}Ce	γ
^{57}Co	γ
^{134}Cs	γ
^{125}I	γ
^{129}I	γ
^{131}I	γ
^{99}Mo	γ
^{103}Pd	γ
^{103}Ru	γ
^{106}Ru	γ
^{198}Au	γ
^{109}Cd	γ
^{99m}Tc	γ
^{140}Ba	γ
^{140}La	γ
^{65}Zn	γ
^{85}Sr	γ
^{7}Be	γ

Radionuclide	Principal Emission
^{89}Sr	β
$^{90}Sr/^{90}Y$	β
^{147}Pm	β
^{227}Ac	β
^{3}H	β
^{32}P	β
^{241}Pu	β
^{228}Ra	β
^{63}Ni	β
^{99}Tc	β
^{204}Tl	β
^{14}C	β
^{85}Kr	β
^{68}Ge	*EC*
^{55}Fe	*EC*

Sources of Radionuclides:

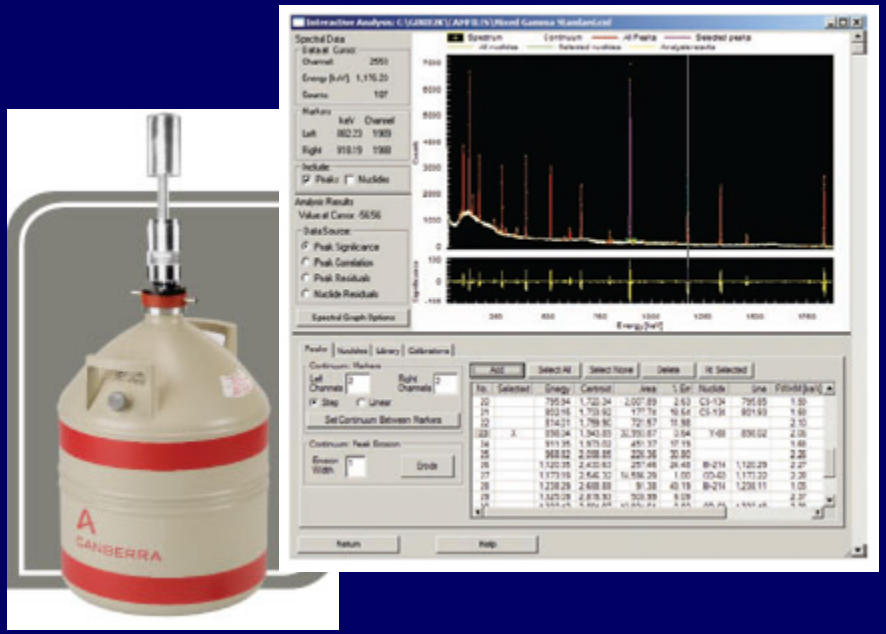
- Natural Origin
- Weapon Tests
- Nuclear Power
- Mining Industries
- Space Explorations
- Nuclear Medicine
- Waste Disposals
- Terrorist Activities

Radioanalytical Methods Developed & to be Developed for FERN Applications

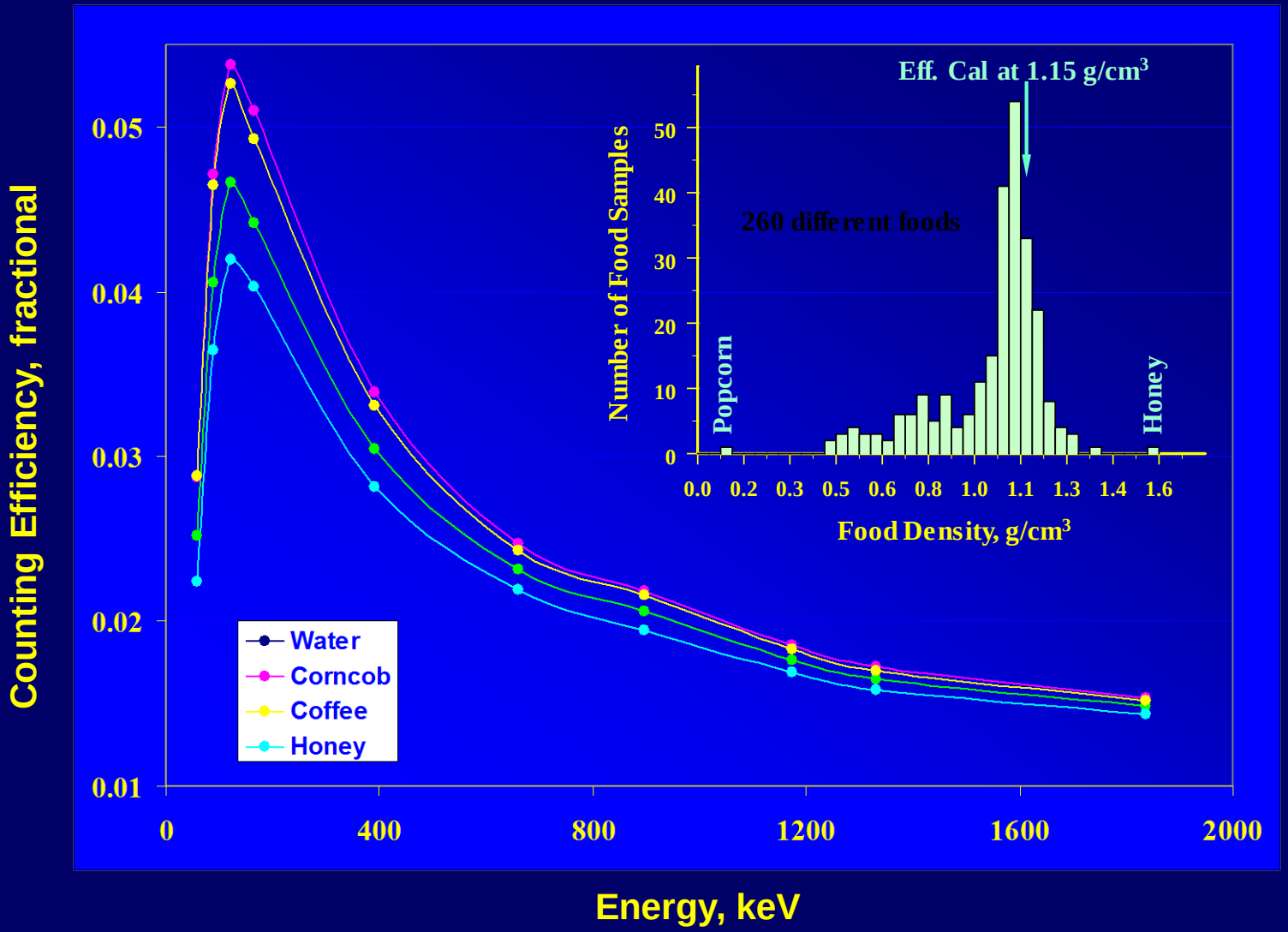
- Determination of γ-Emitting Radionuclides in Foods
- Analysis of ^{210}Po in Foods by Isotope-Dilution Alpha Spectrometry
- Analysis of $^{238,\ 239,\ 240}$ Pu in Foods by Alpha Spectrometry
- Analysis of ^{241}Am in Foods by Alpha Spectrometry
- Determination of ^{90}Sr in Foods by Internal Gas-Flow Proportional Counting
- Screening ^{241}Am, ^{238,239,240}Pu, and ^{90}Sr in Foods by Solid-Phase Extraction LSC
- Screening ^{243}Cm and ^{244}Cm in Foods by DGA/LSC Counting
- Rapid Detection of $^{239+240}$Pu and Pu Isotopic Ratio in Foods by ICPMS
- Rapid Detection of ^{89}Sr and ^{90}Sr in Foods by Cerenkov LSC
- Isotopic Analysis of Processed U in Foods by ICPMS
- Analysis of γ-Emitting Radionuclides in Foods Portable BEGe Gamma Spectrometry
- Analysis of γ-Emitting Radionuclides in Foods by Computational Gamma Spectrometry

Summary of Method

A homogenized food sample, prepared in 400-mL cylindrical geometry, is counted using a calibrated high purity Ge gamma-ray spectrometer. The radionuclides in the sample are identified and quantified using Canberra's Apex software in conjunction with a user developed Excel spreadsheet. The method employs additional calibrations for correcting measurement inaccuracy rising from sample density variations and coincidence summing effects.

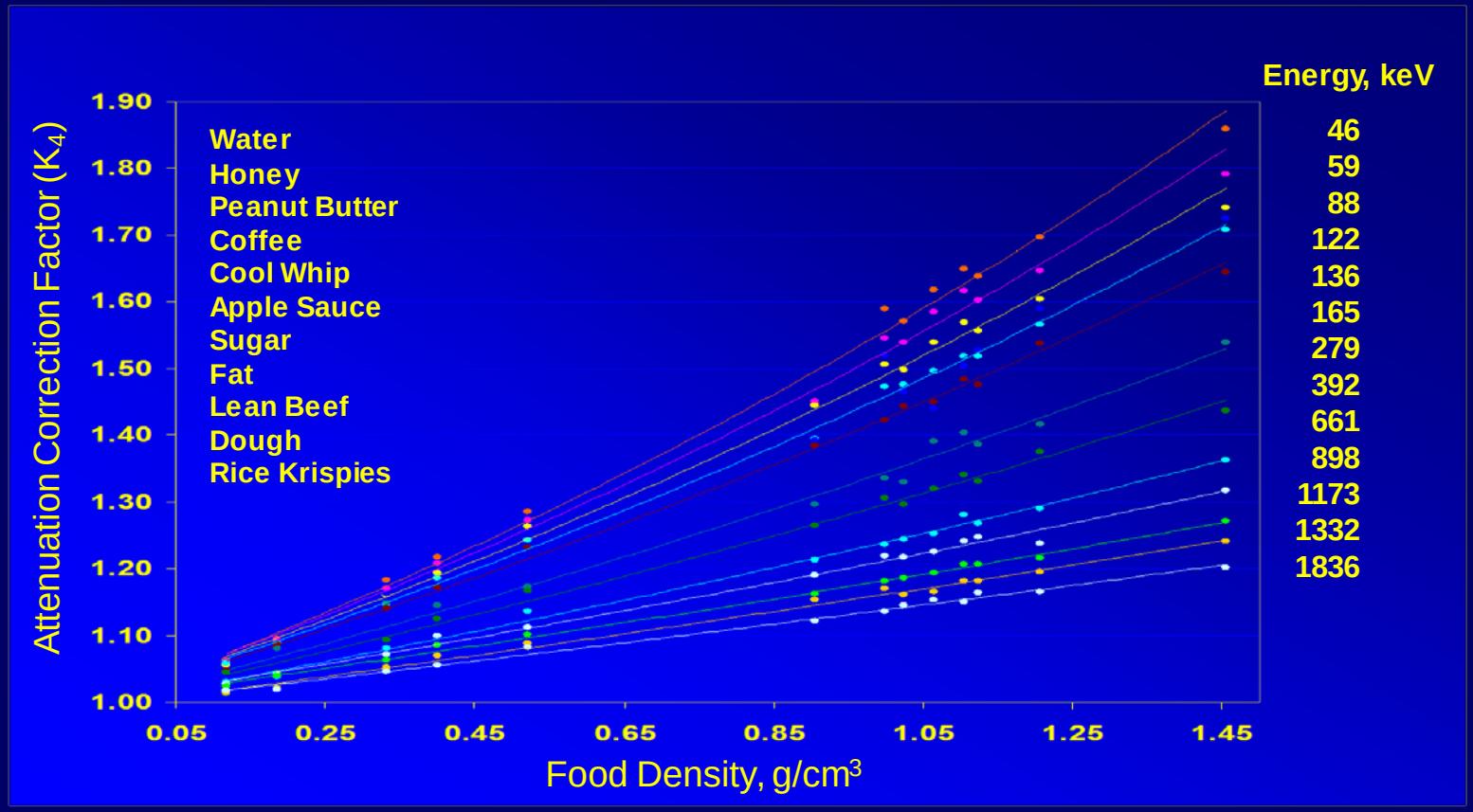


Counting Efficiencies Measured with Different Mixed-Gamma Food Standards



Sample Attenuations vs Food Densities & Compositions

Major Food Components	Empirical Formula	Weight Percent
Carbohydrates	$C_6H_{12}O_6$	C:40.0%, H:7%, O:53%
Fats	$C_{55}H_{98}O_6$	C:77.3%, H:11.5%, O:11.2%
Proteins	$C_{400}H_{620}O_{100}P_1S_1$	C:54.5%, H:7%, O:21.8%, N:15.9%, P:0.4%, S:0.4%
Water	H_2O	H:11%, O:89%



Coincidence-Summing Correction Effects

Techniques:
- Peak-to-Total Calibration
- Monte-Carlo Simulation

Dependency:
- Detector Size
- Counting Geometry
- Decay Scheme
- Sample Composition
- Shielding

Radionuclide	Energy keV	Coincidence-Summing Correction Factor	Error %	Radionuclide	Energy keV	Coincidence-Summing Correction Factor	Error %
BA-140	162.7	0.928	0.038	CO-60	1173.2	0.905	0.100
BA-133	356.0	0.965	0.065	CO-60	1332.5	0.902	0.084
BA-140	537.3	1.000	0.093	SE-75	136.0	0.855	0.052
BA-133	81.0	0.859	0.050	SE-75	264.7	0.863	0.062
CE-144	80.1	0.999	0.034	SE-75	279.5	0.905	0.069
CS-134	569.3	0.789	0.067	SE-75	400.7	1.590	0.088
CS-134	604.7	0.865	0.092	YB-169	109.8	0.840	0.057
CS-134	795.8	0.867	0.070	YB-169	130.5	0.848	0.037
IR-192	296.0	0.764	0.069	YB-169	177.2	0.881	0.043
IR-192	308.5	0.781	0.058	YB-169	198.0	0.892	0.040
IR-192	316.5	0.836	0.040	YB-169	307.7	1.090	0.049
IR-192	468.1	0.872	0.034	YB-169	63.1	0.846	0.038
LA-140	1596.2	0.866	0.096	MO-99	140.5	0.994	0.032
LA-140	328.8	0.795	0.051	MO-99	739.5	0.878	0.140
LA-140	487.0	0.844	0.074	Y-88	1836.1	0.902	0.110
LA-140	815.8	0.957	0.120	Y-88	898.0	0.911	0.110

Major Sources of Uncertainty and Their Relative Contributions ---- Analysis of ^{137}Cs in Rice by Gamma Ray Spectrometry ----

Variable	Symbol	Value	Rel. Standard Uncertainty, %	Sensitivity Factor	Contribution to Combined Uncertainty, %
Sample weight, kg	w	0.1379	0.0725	1	0.06
Counting efficiency	ε	3.708E-2	0.639	1	4.3
Sample dry content, fractional	K_1	0.9075	0.055	1	0.03
Decay correction	K_2	0.9640	3.53E-4	1	0
Decay during counting correction	K_3	0.9999	9.47E-7	1	0
Attenuation correction	K_4	1.0	0.5	1	2.7
Random summing correction	K_5	1.0	1.22E-5	1	0
Coincidence summing correction	K_6	1.0	0	1	0
Net peak area , counts	N_{net}	3057.7	2.949	1	92.3
Sample counting time, sec	t_s	259200	0	1	0
Emission probability	γ	0.851	0.235	1	0.6

Activity concentration of ^{137}Cs, Bq/kg	3.10
Combined standard uncertainty, Bq/kg	0.10
Expanded uncertainty, Bq/kg	0.20

Summary of Method

A food sample is weighed, ashed, spiked with Sr and Y carries, and digested in nitric acid. The digest is then mixed with tributylphosphate (TBP) in a separatory funnel where ^{90}Y is separated from ^{90}Sr and the sample matrix. After removal of Fe and REEs by F^- and OH^- precipitations, the purified ^{90}Y is deposited onto a glass fiber filter as YOX and counted using a low-background internal gas-flow proportional counter. The ^{90}Y concentration is calculated from the observed ^{90}Y count rate, attenuation-corrected counting efficiency, chemical yield, decay correction factor, and sample weight, which is equal to ^{90}Sr concentration in the sample.

Analysis of ^{90}Sr in Foods by Gas-Flow Proportional Counting

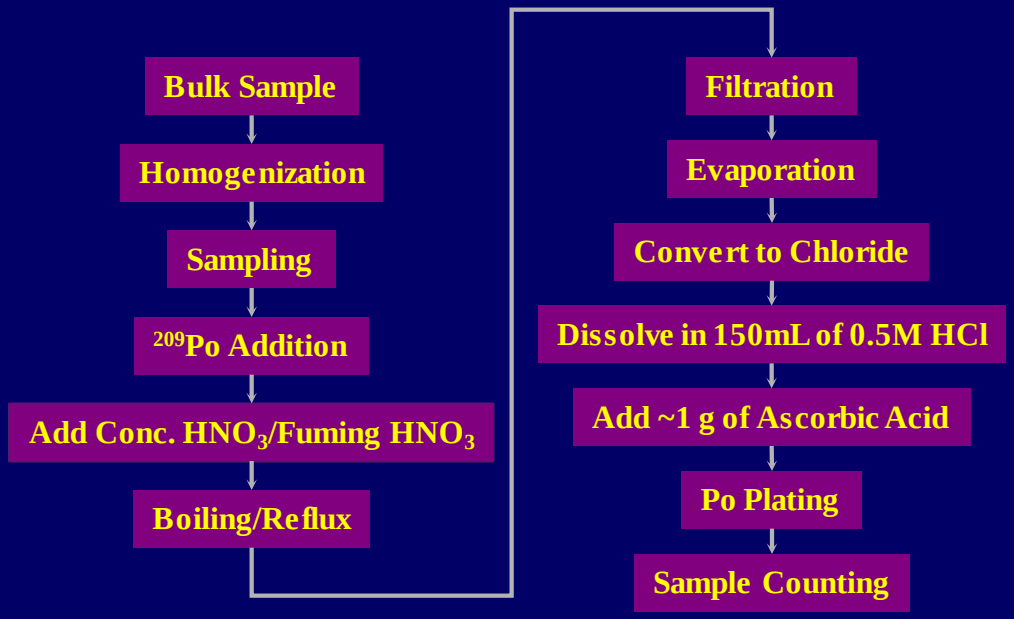
Calculation Model: $C_{Sr90} = \dfrac{A_{Sr90} - A_{MB}}{W_S}$ where, $A_{Sr90} = \dfrac{R_{Y90} - B_{Y90}}{E_Y \times Y_Y \times D_Y \times D_{Sr90} \times 60}$

Description of Source of Standard Uncertainty x_i	Evaluation Method for $u(x_i)$, The Standard Uncertainty of x_i (A) Statistical Method (B) Other Method	Relative Standard Uncertainty Component $u_i(y)/y$, %
Sample weight, W_S	u_{Ws} estimated (B)	0.09
Gross count rate of sample, R_{Y90}	$u_{Rs,}$ estimated (A)	6.76
Background count rate, B_{Y90}	u_{RB} estimated (A)	3.10
Counting efficiency of Y-90, E_Y	$u_{EY,}$ estimated (A)	4.53
Chemical yield of Y-90, Y_Y	$u_{YY,}$ estimated (A)	0.77
Net weight of yttrium oxalate on filter, W_{YOX}	$u_{WYOX,}$ estimated (B)	0.77
Weight of yttrium oxalate loaded filter, W_L	$u_{WL,}$ estimated (B)	0.14
Weight of unloaded filter, W_{UL}	$u_{WUL,}$ estimated (B)	0.10
Concentration of yttrium carrier solution, C_{YOX}	$u_{CYOX,}$ estimated (A)	0.28
Volume of yttrium carrier solution, V_{YOX}	$u_{VYOX,}$ estimated (A)	0.70
Relative total combined standard uncertainty of C_{Sr90}, %		≤ 9
Coverage factor, $k = 2$		x 2
Relative expanded uncertainty of C_{Sr90}, %		≤ 18

Summary of Method

A homogenized food sample is weighed, spiked with ^{209}Po standard, and digested in nitric acid. After converting to chloride form, the dissolved Po is either readily plated on a silver disc by spontaneous deposition or chemically purified upon levels of interfering elements or radionuclides in the sample. If necessary, Po is purified using a chromatography column packed with Sr resin. The Po on the sliver disc is counted using an alpha spectrometer. The observed peak energies and areas of ^{209}Po and ^{210}Po are used for ^{210}Po identification and quantification.

Procedure Flow Chart



Analysis of ^{210}Po in Foods by Isotope-Dilution Alpha Spectrometry

Calculation Model:

$$C_{210} = \frac{1}{W_s} \times \left[\left(\frac{R_s^g - R_s^i - R_s^t - R_s^p}{R_{st}^g - R_{st}^i - R_{st}^p} \times A_s \right) \times e^{\lambda T_s} - \left(\frac{R_{mb}^g - R_{mb}^i - R_{mb}^t - R_{mb}^p}{R_{mbt}^g - R_{mbt}^i - R_{mbt}^p} \times A_{mb} \right) \times e^{\lambda T_{mb}} \right]$$

- **Analyte/Tracer Equilibrium**
  - **Incomplete Dissolution**
  - **Oxidation States**
- **Interferences**
  - **Isotopic**
  - **Peak Overlap**
- **Blank corrections**
  - **Tracer Impurity**
  - **Reagent Blank**
- **Counting Statistics**
  - **Low Recovery**
  - **Source Geometry**

Summary of Method

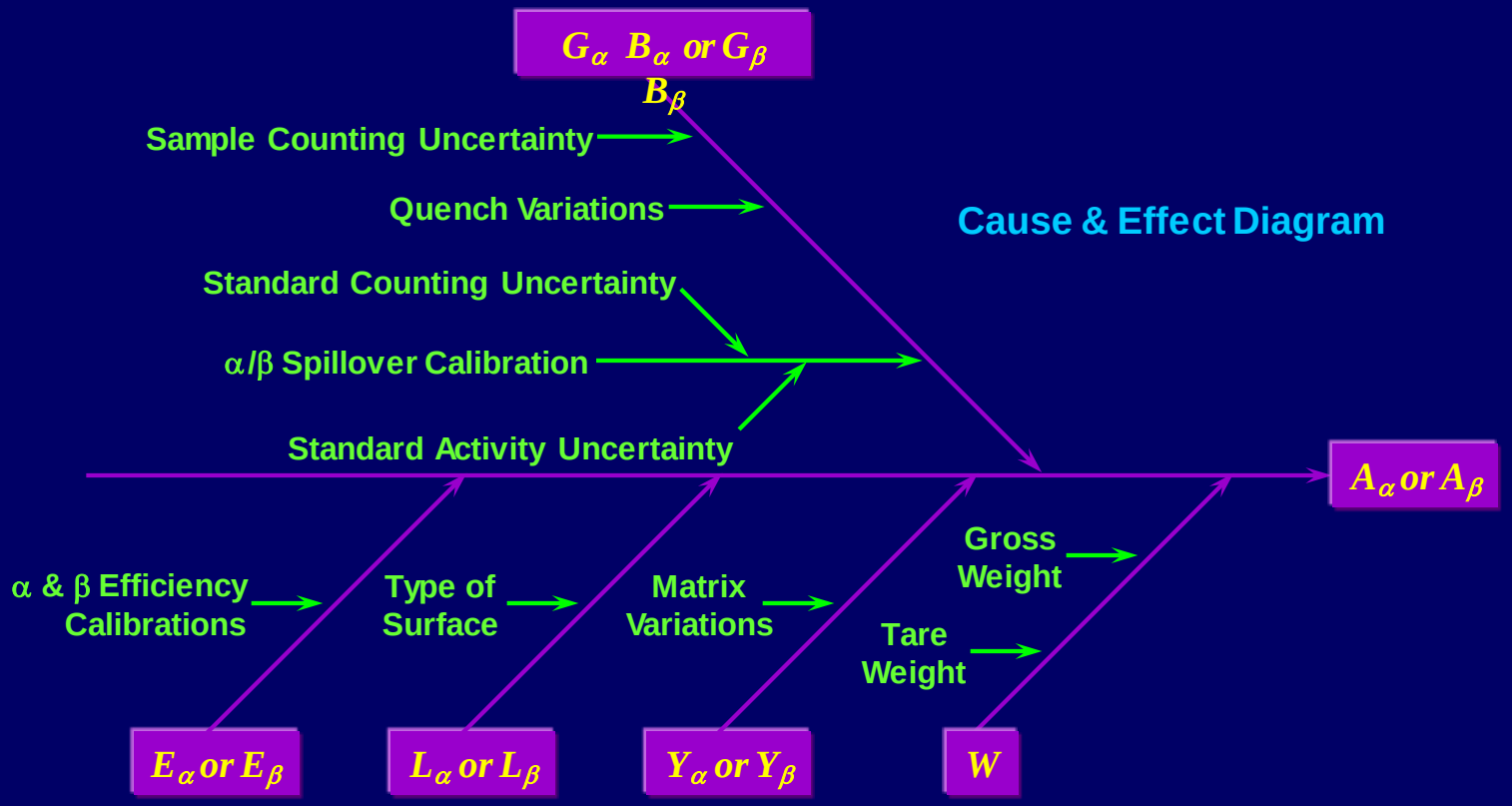
Foods are digested in concentrated nitric acid followed by addition of 1 gram of DGA extraction resin at room temperature. After stirring for 15 minutes, the resin is separated from the digest and the analytes are stripped from the resin using 0.1M HCl-0.1M $H_2C_2O_4$. The stripping solution is evaporated to dryness and the remaining oxalic acid is destroyed in a 400°C oven. The treated sample is quantitatively transferred into a liquid scintillation vial with 0.5M HCl and mixed with Ultima Gold AB cocktail. The sample activity is quantified using a liquid scintillation counter.

Analysis of Radionuclides in Foods by Liquid Scintillation Spectrometry

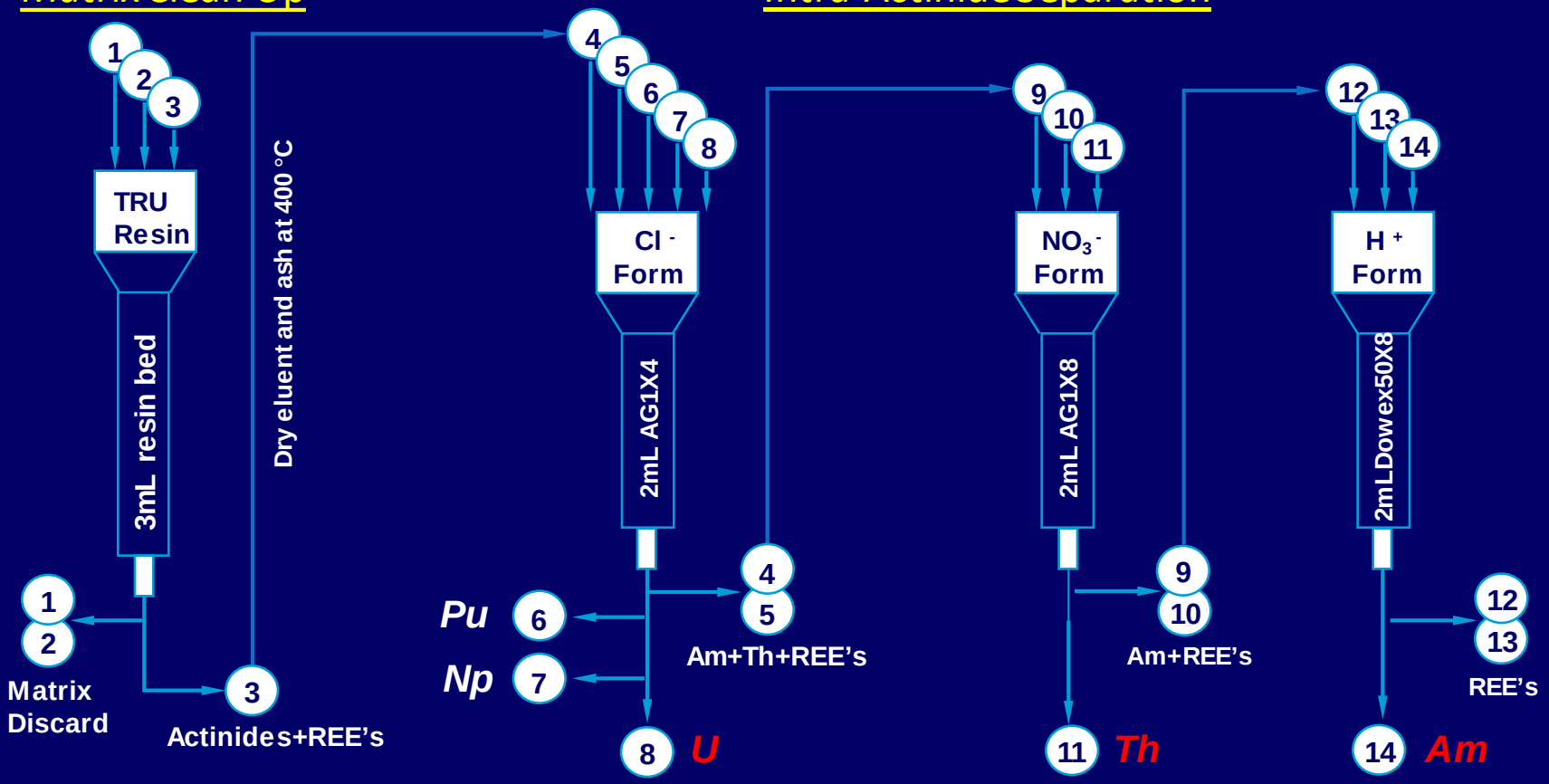
Calculation Models:

$$A_\alpha = \frac{G_\alpha - B_\alpha}{E_\alpha \times L_\alpha \times Y_\alpha \times W \times 60}$$

$$A_\beta = \frac{G_\beta - B_\beta}{E_\beta \times L_\beta \times Y_\beta \times W \times 60}$$



Sequential Separation of Actinides

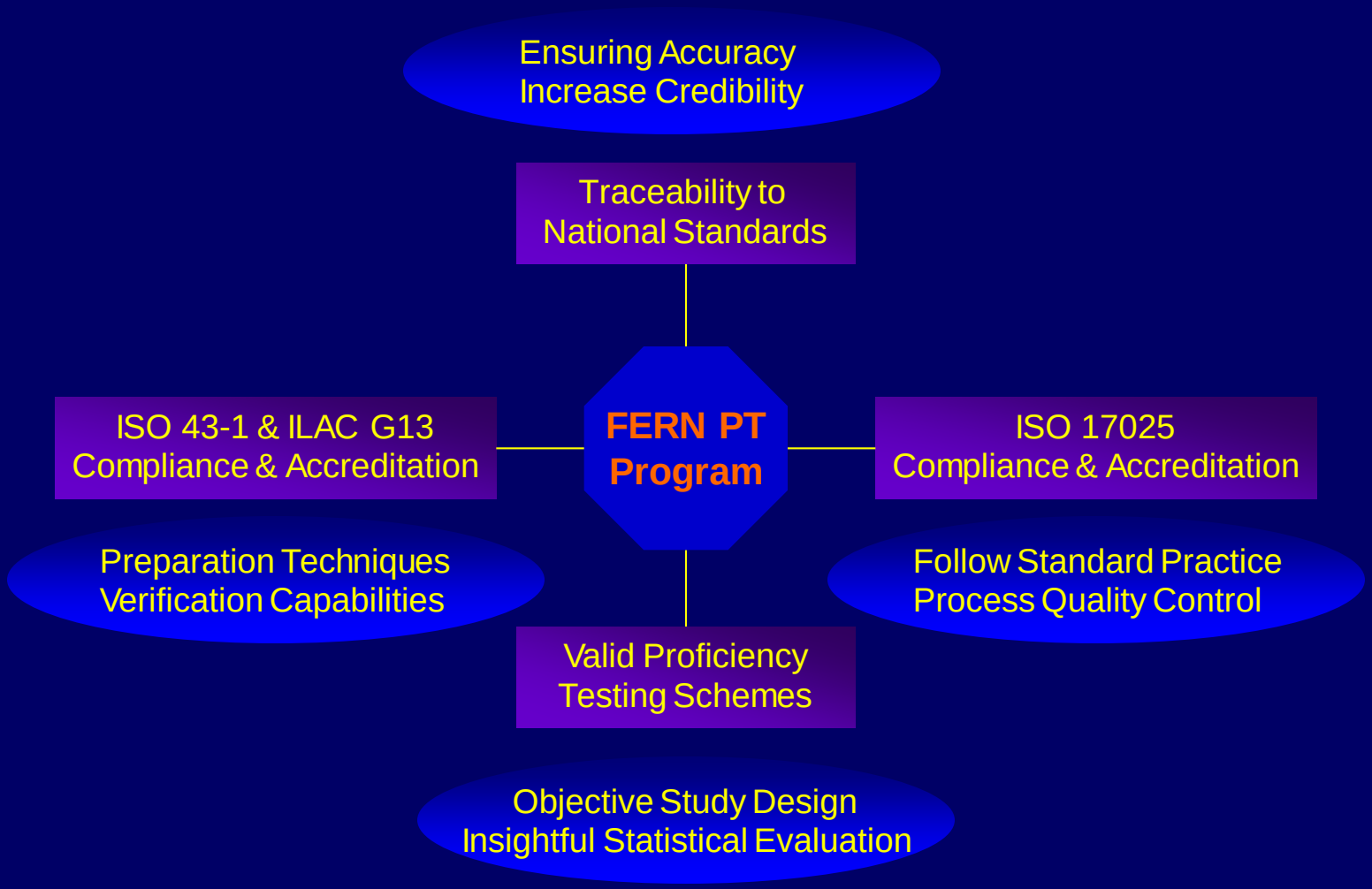


1. Sample loading, 50 mL of 3M HNO_3+0.3 g Ascorbic acid
2. Column washing, 20 mL of 3M HNO_3
3. Stripping, 40 mL of 0.1M $(NH_4)_2C_2O_4$ - 0.05M HNO_3
4. 1 mL of 9 M HCl
5. 10 mL of 9 M HCl
6. 18 mL of 9 M HCl - 0.05 M NH_4I
7. 8 mL of 4 M HCl - 0.1 M HF
8. 8 mL of 0.5 M HCl - 1 M HF
9. 1 mL of 8 M HNO_3
10. 10 mL of 8 M HNO_3
11. 20 mL of 2 M HNO_3
12. 1 mL of 1 M HCl
13. 20 mL of 1 M HCl
14. 20 mL of 6 M HCl

Rapid Detection of ^{239}Pu in Foods by DGA-ID-ICPMS

Analysis of Pu-239 in Foods by ICPMS 7700x w/o Mass Bias Correction							
		Food	Measured Pu-239		Known Pu-239		Bias
Food Group	Sample ID	Mass, g	ng/g	1s	ng/g	1s	%
Vegetable	MixedVegeT9	60	0.4704	0.0097	0.4725	0.0043	-0.44
	MixedVegeT10	60	0.4677	0.0092	0.4725	0.0043	-1.02
	MixedVegeT11	60	0.4719	0.0189	0.4725	0.0043	-0.13
	AppleSauce1	50	0.4718	0.0054	0.4725	0.0043	-0.15
	AppleSauce2	50	0.4686	0.0034	0.4725	0.0043	-0.83
	AppleSauce3	50	0.4686	0.0048	0.4725	0.0043	-0.83
Meat	RudderFishT9	60	0.4830	0.0073	0.4725	0.0043	2.22
	RudderFishT10	60	0.4772	0.0073	0.4725	0.0043	0.99
	RudderFishT11	60	0.4819	0.0090	0.4725	0.0043	1.99
	RudderFishT12	60	0.4765	0.0096	0.4725	0.0043	0.85
	Beef1	60	0.4732	0.0143	0.4725	0.0043	0.15
	Beef2	60	0.4655	0.0099	0.4725	0.0043	-1.48
	Beef3	60	0.4740	0.0137	0.4725	0.0043	0.32
	Scallop1	50	0.4581	0.0096	0.4725	0.0043	-3.05
	ScallopT11	50	0.4659	0.0114	0.4725	0.0043	-1.40
	Scallop3	50	0.4709	0.0088	0.4725	0.0043	-0.34
	ScallopT12	50	0.4632	0.0130	0.4725	0.0043	-1.97
Grain	RiceCracker1	30	0.4712	0.0044	0.4725	0.0043	-0.28
	RiceCracker2	30	0.4697	0.0066	0.4725	0.0043	-0.59
	RiceCracker3	30	0.4685	0.0041	0.4725	0.0043	-0.85
Dairy	DryMilk T9	100	0.4607	0.0197	0.4725	0.0043	-2.50
	DryMilk T9 Re-Run	100	0.4720	0.0116	0.4725	0.0043	-0.11
	DryMilk T10	100	0.4710	0.0111	0.4725	0.0043	-0.32
	DryMilk3	100	0.4763	0.0116	0.4725	0.0043	0.80
Composite Meal	HamburgerT9	50	0.4689	0.0190	0.4725	0.0043	-0.76
	HamburgerT10	50	0.4645	0.0166	0.4725	0.0043	-1.69
	HamburgerT10 Re-Run	50	0.4769	0.0144	0.4725	0.0043	0.93
	HamburgerT11	50	0.4705	0.0084	0.4725	0.0043	-0.42
Note: Each of the test samples was spiked with ~0.2 Bq of Pu-239.							

FERN/WEAC Radiological Laboratory Proficiency Evaluation



Establishment of FERN Standard Warehouse

Available

^{137}Cs	^{241}Pu	^{232}U
^{134}Cs	^{238}Pu	^{233}U
^{60}Co	^{239}Pu	Nat. U
^{65}Zn	^{242}Pu	Nat. Th
^{133}Ba	^{241}Am	Mixed γ
^{152}Eu	^{243}Am	
^{90}Sr	^{209}Po	
$^{210}Pb/^{210}Po$	^{243}Cm	
^{55}Fe	^{244}Cm	
^{129}I	^{229}Th	
^{228}Ra	^{230}Th	
^{226}Ra	^{3}H	

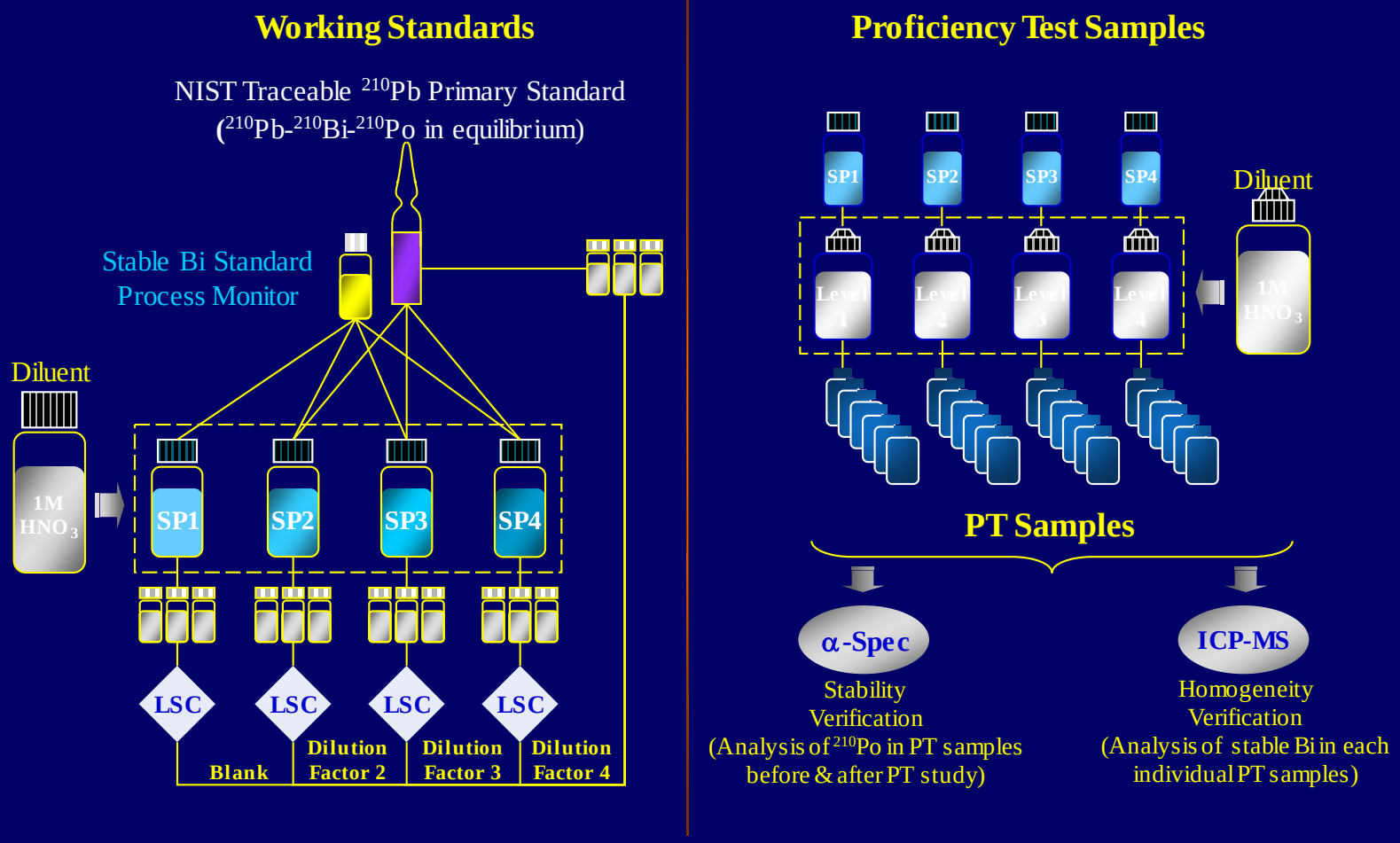
Unavailable

^{85}Sr	^{236}Pu
^{89}Sr	^{240}Pu
^{125}I	^{244}Pu
^{131}I	^{237}Np
^{192}Ir	^{228}Th
^{99}Mo	^{252}Cf
^{144}Ce	
^{140}Ba	
^{154}Eu	
^{99}Tc	
^{63}Ni	
^{236}U	

The standard warehouse is established as a part of network preparedness effort and to assist the following FERN activities: (The standards are free of charge to FERN member laboratories)

- Sample Analysis for Emergency Response
- Method Development and Validation
- Matrix Extension Study
- Laboratory Proficiency Testing and Evaluation
- Standard Method Verification
- Resolving Method and Measurement Discrepancies

Example of Preparation and Verification Scheme



Each Proficiency Test Sample with Assured Traceability

Performance Evaluation Criteria

Trueness: $$\left|V_{Lab} - V_{Ref}\right| \le 3 \times \sqrt{u_{Ref}^2 + u_{Lab}^2}$$

Precision: $$Z_{rep} = \frac{V_{Lab1} - V_{Lab2}}{\sqrt{u_{Lab1}^2 + u_{Lab2}^2}} \le \pm 3$$

False Positive Detection: $$\left|V_{blank} - u_{blank}\right| \begin{cases} \le 0.1 & \textit{No False Positive Detection (P)} \\ > 0.1 & \textit{False Positive Detection (F)} \end{cases}$$

Tolerable Bias: $$\left|\frac{V_{Lab} - V_{Ref}}{V_{Ref}} \times 100\right| \le B_{Exp}$$

Tolerable Uncertainty: $$\left[\sqrt{\left(\frac{u_{Ref}}{V_{Ref}}\right)^2 + \left(\frac{u_{Lab}}{V_{Lab}}\right)^2}\right] \times 100 \le P_{Exp}$$

B_{Exp} is the tolerable bias limit set by FDA, for instance 10% for ^{210}Po PT study

P_{Exp} is the tolerable uncertainty limit set by FDA, for instance 20 % (2s) for ^{210}Po PT study

The Reality Check

Radionuclide	Principal Emission
^{241}Am	α
^{238}Pu	α
^{239}Pu	α
^{240}Pu	α
^{210}Po	α
^{242}Cm	α
^{243}Cm	α
^{244}Cm	α
^{252}Cf	α
^{226}Ra	α
^{237}Np	α
^{228}Th	α
^{230}Th	α
^{232}Th	α
^{234}U	α
^{235}U	α
^{238}U	α

Radionuclide	Principal Emission
^{137}Cs	γ
^{60}Co	γ
^{153}Gd	γ
^{192}Ir	γ
^{75}Se	γ
^{170}Tm	γ
^{169}Yb	γ
^{141}Ce	γ
^{144}Ce	γ
^{57}Co	γ
^{134}Cs	γ
^{125}I	γ
^{129}I	γ
^{131}I	γ
^{99}Mo	γ
^{103}Pd	γ
^{103}Ru	γ
^{106}Ru	γ
^{198}Au	γ
^{109}Cd	γ
^{99m}Tc	γ
^{140}Ba	γ
^{140}La	γ
^{65}Zn	γ
^{85}Sr	γ
^{7}Be	γ

Radionuclide	Principal Emission
^{89}Sr	β
^{90}Sr/^{90}Y	β
^{147}Pm	β
^{227}Ac	β
^{3}H	β
^{32}P	β
^{241}Pu	β
^{228}Ra	β
^{63}Ni	β
^{99}Tc	β
^{204}Tl	β
^{14}C	β
^{85}Kr	β
^{68}Ge	*EC*
^{55}Fe	*EC*

- **Method Available**
- **Method Under Development**
- **Method Unavailable**

Thank you!

Any Questions?