NHSRC & ORIA's Efforts to Enhance US Radiochemistry Capacity and Capability

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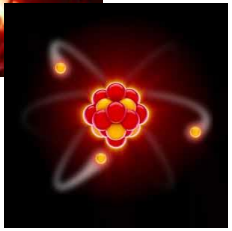
Overview

- Research Focus
- Laboratory Challenges
- Method Development
- Laboratory Proficiency Test Program
- Selected Analytical Methods (SAM)
- ORIA Response Guides
- Research Impact

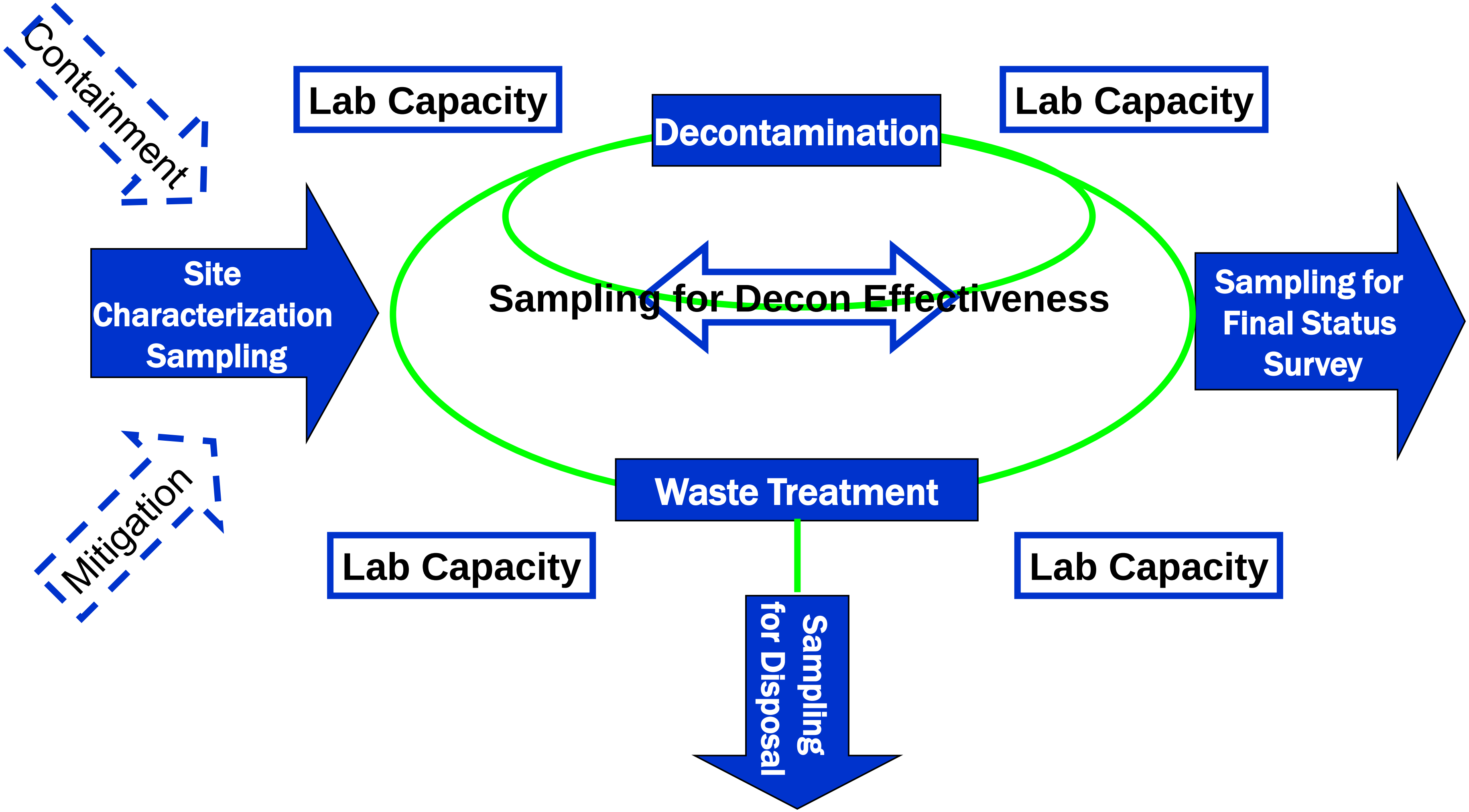
Why Are Rapid Methods Necessary?



"Dirty Bomb" in major urban area

- Estimated thousands of people exposed to detectable contamination
  - Potential: > 100,000 clinical samples.
- Hundreds of thousands of environmental samples in the first year
- The analysis of the clinical and environmental samples (depending on the radionuclides) would take *years* to complete, review and verify – causing significant delays in consequence management activities

Remediation & Recovery Process



Reasons for the Research

- Lack of validated *rapid* methods for select environmental matrices and urban materials
- Certain isotopes of interest cannot be reliably detected and quantified with hand-held instruments
- Some methods listed in the *Selected Analytical Methods for Remediation and Recovery (SAM)* document are not specific or verified for the isotope/matrix pair of interest
- Many labs do not perform listed SAM methods or have the laboratory personnel with the knowledge and expertise to do so

Research Focus

Current Focus - Urban Material Analytical Methods

To begin to address a radiological emergency, there is a need for quick and validated analytical methods for traditional environmental matrices (e.g., water, air filters, swipes, etc.) and contaminated building materials in an urban environment that result from a radiological event

Lab Capacity

Insufficiency of laboratory surge capacity supporting environmental, clinical, and longitudinal public health sample analyses (CBR) Tier 1

Collaboration

Method development is in collaboration with NAREL and in support of the Environmental Response Laboratory Network

Integrated Consortium of Laboratory Networks (ICLN)

ICLN

- Laboratory Response Network
- National Plant Diagnostics Network
- National Animal Health Laboratory Network
- Environmental Response Laboratory Network
- Food Emergency Response Network
- DoD Laboratory Network

List of Threat Radionuclides

This list includes radionuclides that are most likely to be encountered in a radiological event

NOTE: Nuclides listed by isotope number, not priority

Gamma Emitters	Alpha/Beta Emitters
Cobalt-60 (Co-60)	Tritium (H3)
Selenium-75 (Se-75)	Phosphorus-32 (P-32)
Molybdenum-99 (Mo-99)	Strontium-89 (Sr-89)
Ruthenium-103 (Ru-103)	Strontium-90 (90)
Ruthenium-106 (Ru-106)	Technetium-99 (Tc-99)
Iodine-125 (I-125)	Polonium-210 (Po-210)
Iodine-131 (I-131)	Radium-226 (Ra-226)
Cesium-137 (Cs-137)	Uranium-234 (U-234)
Europium-154 (Eu-154)	Uranium-235 (U-235)
Iridium-192 (Ir-192)	Uranium-238 (U-238)
	Plutonium-238 (Pu-238)
	Plutonium-239 (Pu-239)
	Americium-241 (Am-241)
	Curium-244 (Cm-244)
	Californium-252 (Cf-252)

Challenges for Laboratories Following a Radiological Incident

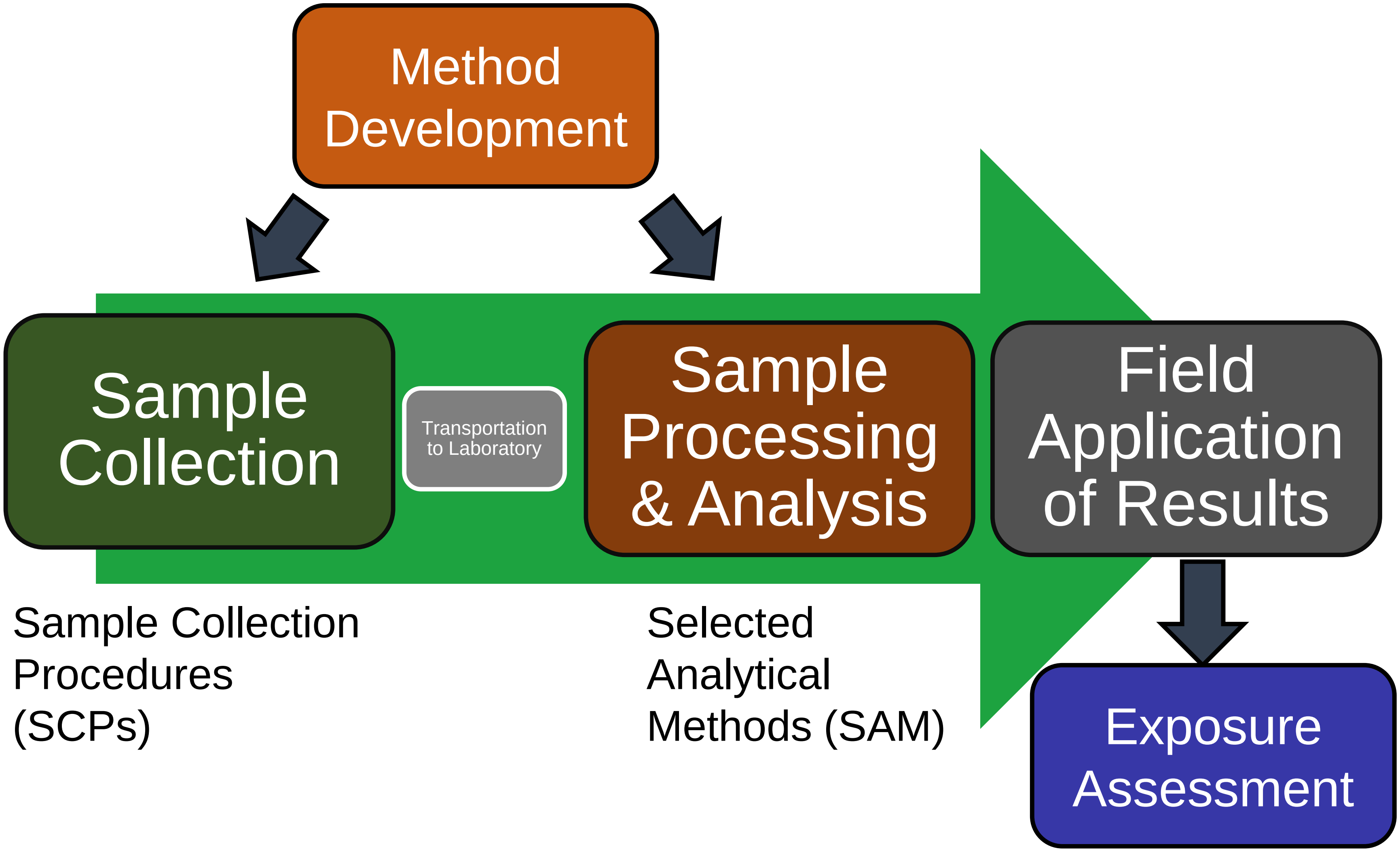
- Sudden influx of large numbers of potentially radioactive samples
  - Wide range of activities possible
  - Multiple and unique matrices
  - Very short turnaround and reporting times required

- Data quality objectives (DQOs) and measurement quality objectives (MQOs) associated with the protection of human life and the environment will change based on the phase of the event

- Most laboratories have little if any experience with shift from routine operations to incident response mode – general lack of preparedness will lead to a number of problems – unforeseen bottlenecks, lab contamination, potential exposure to lab personnel, etc.

What Has Been Done to Address Laboratory Challenges?

- Development of Rapid Methods for Radiological Analysis
- Implementation of Proficiency Test Studies
- Publishing of:
  - SAM and companion documents
  - Radiological Sample Collection Procedures
  - Radiological Laboratory Emergency Response Guides


Method Development
Sample Collection
Transportation to Laboratory
Sample Processing & Analysis
Field Application of Results
Exposure Assessment
Sample Collection Procedures (SCPs)
Selected Analytical Methods (SAM)

Rapid Radiochemical Methods

Previously:

- Many radiological analyses require turn-around-times (TAT) of days to weeks
- Validation of many methods not performed and method performance unknown for different matrices
- Many methods have not been tested for 'ruggedness'

Method Design Criteria

- Use modern technology
- Use readily available chemical standards and equipment
- Have reportable results in 1 to 2 days
- Specific for the radionuclides of interest
- Accurate estimates of measurement uncertainty
- Generation of accurate defensible data

Validation Criteria

- Method Uncertainty
- Detection Capability
  - Difference between blanks and samples spiked at the minimum detectable concentration (MDC)
- Specificity
  - Determined by spiking with non-target nuclides
- Ruggedness
  - Tracer yields, spectral quality
- Evaluation for bias

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

Basis of Method Evaluation and Validation

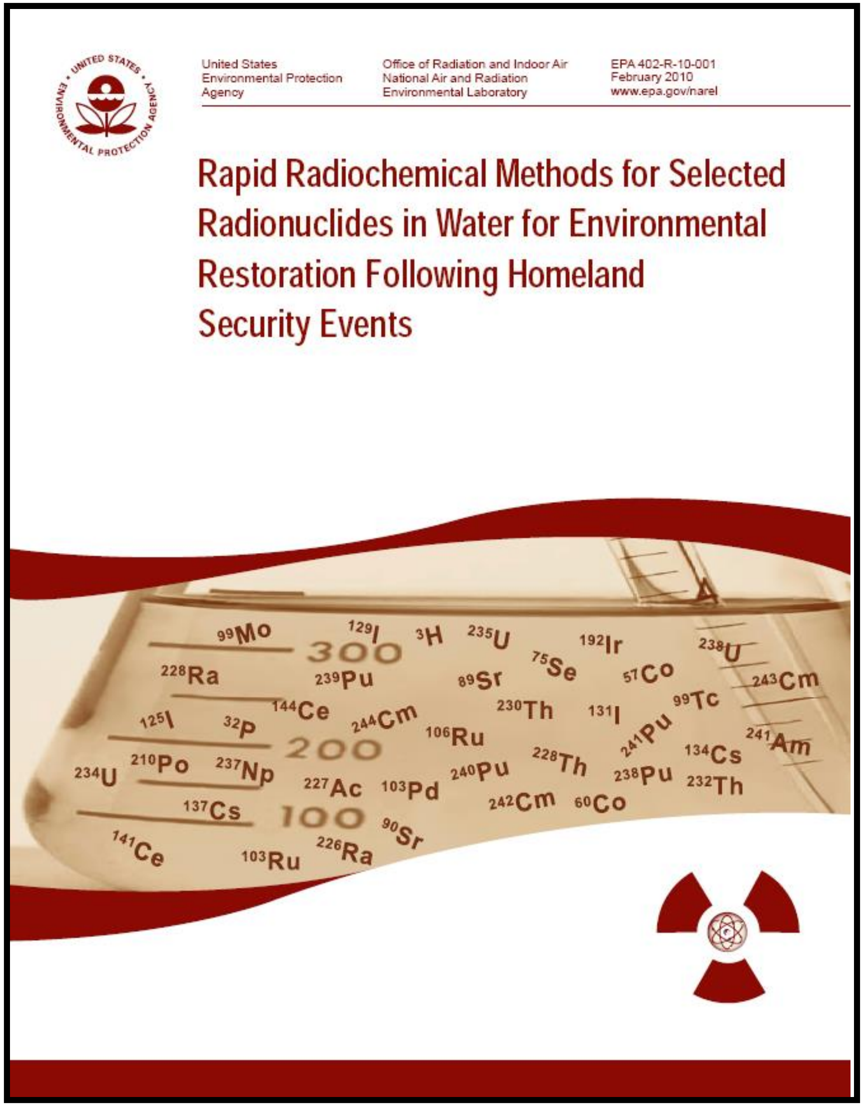
- *Method Validation Requirements for Qualifying Methods Used by Radioanalytical Laboratories Participating in Incident Response Activities (EPA 402-R-09-006)*
- *Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP); Chapters 6 and 20 (NUREG-1576; EPA 402-B-04-001A/B/C)*
- *Quality Assurance Project Plan Validation of Rapid Radiochemical Methods for Radionuclides Listed for EPA's Standardized Analytical Methods (SAM) for Use During Homeland Security Events*

Measurement Quality Objectives

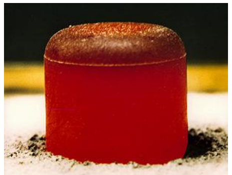
- Consistent with recovery phase objectives
- Based on Analytical Action Level (AAL)
- Required method uncertainty
- Required MDC

First Methods - Water

- Target radionuclides:
  - ^{241}Am
  - $^{239/240}Pu$ & ^{238}Pu
  - U isotopes
  - ^{226}Ra
  - ^{90}Sr (Total Radiostrontium)
  - ^{32}P
- Used more rapid technology (e.g., extraction chromatography, vacuum box processing)
- Achieved TATs for radionuclides in hours to 1 to 2 days



Method	Turn Around Time
Rapid Radiochemical Methods for Selected Radionuclides in **Water** For Environmental Restoration Following Homeland Security Events	5 to 37 Hours
Rapid Method for Acid Digestion of Glass-Fiber and Organic/Polymeric Composition **Filters and Swipes**, Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses	5.25 to 36 Hours
Rapid Method for Sodium Carbonate Fusion of Glass-Fiber and Organic/Polymeric Composition **Filters and Swipes**, Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses	10 to 31.75 Hours
Rapid Method for Fusion of **Soil and Soil Related Matrices** Prior to Analyses	9 to 36 Hours
Rapid Radiochemical Method for **Phosphorus-32** in Water Samples for Environmental Remediation Following Homeland Security Events	5 Hours



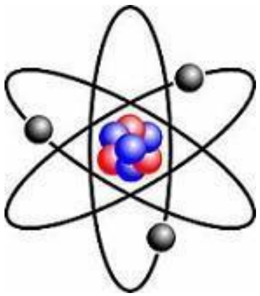
Status

- Soil Methods - Published
- Acid Digestion and Carbonate Fusion for Swipes - Published
- Phosphorus-32 in Water – Published
- Radioisotope Thermoelectric Generator (RTG) Methods (air filters and water) – Published
- Building Materials Methods –
  - Concrete: Published
  - Brick: In review

Additional Development Planned and In-Progress

- Rapid Methods – Building Materials
  - Building roofing materials (in-progress)
  - Asphalt – roads (in progress)
  - Limestone (method drafted)
  - Stucco (method drafted)
  - Granite
  - Clay roof piping

Additional Development Planned and In-Progress



- Analysis of ^{89}Sr in fresh fission product mixtures (in progress)
- Rapid method for ^{252}Cf in various matrices (in progress)
- Rapid method for $^{243/244}Cm$ in various matrices (in progress)
- Rapid method for ^{210}Po on swipes
- Rapid methods for gross alpha and gross beta swipes
- Rapid method for ^{99}Tc on air filters
- Rapid method for Tritium in aqueous and liquid phase samples
- Preliminary research on decon chemical interferences (in progress)

How Laboratories Practice

EPA recognized that just publishing the rapid methods was not enough

- Current PT Programs, relative to incident response testing, have the following limitations:
  - Activity concentrations are very limited
  - Uses same short list of long-lived radionuclides most of the time
  - Do not cover key improvised nuclear device (IND) or radiological dispersion device (RDD)-type radionuclides
  - Don’t stress changing MQOs as would occur in an incident

Proficiency Test Program

EPA has implemented six rounds of “No Fault” PT studies:

- First three rounds were spikes of various isotopes in different media
- Last three rounds have been samples created from freshly irradiated uranium – mixed fission products
  - Most laboratories have not analyzed samples containing mixed fission products in many years

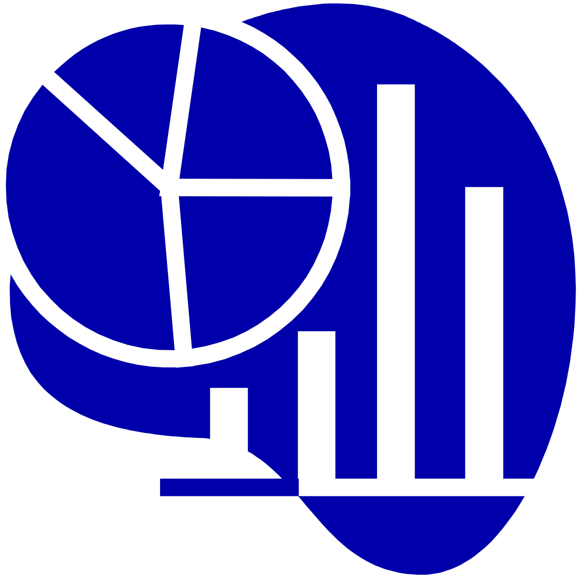
Project Phases for Mixed Fission PT Program Studies

Supply participating laboratories with

- Phase I
  - A single water sample – 8 fission products, 2 activation products (AP), 1 radionuclide not commonly encountered (all added as individual radionuclides in a ratio approximately of that from a fission event)
- Phase II
  - Single sample of freshly irradiated uranium (natural) dissolved in acid
  - Initial focus – gamma ray emitters
  - Round 2 – Pu and Sr analysis also requested
- Phase III (still in development)
  - Computer simulated, electronic spectra of various fission product radionuclides

Participation

- Federal, State, and Commercial Laboratories
- Phase I
  - First round (13 Participants)
  - Second round (19 participants)
- Phase II
  - First round (27 participants)
  - Second round (39 participants)



PT Program Lessons Learned – So Far

- Phase I
  - Many fission products not in gamma libraries of many of the tested labs
  - Many laboratories don't have sample geometries necessary for high activity samples
- Phase II
  - Fission product spectra provided a significant challenge to software algorithms
  - Some short-lived radionuclides ($t_{1/2}$ <1-2 d) are present even after four weeks
  - Significantly higher activity creates significant level of interference
  - Sample screening at the laboratory yields information that conflicts with or contradicts the activity concentrations within the sample

SAM History

In response to 9/11: EPA's Homeland Security Laboratory Capacity Workgroup was formed:

> Identified critical need for a list of pre-selected, pre-evaluated, analytical methods to be used by all laboratories when analyzing homeland security incident samples

September 2004: *Standardized Analytical Methods for Use During Homeland Security Events* was published focusing on Chemical and Biological agents of interest

July 2012: Name changed to *Selected Analytical Methods for Environmental Remediation and Recovery (SAM)* to better reflect focus on providing selected analytical methods for use across multiple laboratories during environmental remediation and recovery

SAM Progress

SAM 1.0

Published September 2004

Chemical Methods

82 analytes (10 biotoxins)
4 matrices

Pathogen Methods

27 analytes
3 matrices

www.epa.gov/sam

SAM 2012

Published July 2012

Chemical Methods

142 analytes
5 matrices

Radiochemical Methods

25 analytes
6 matrices

Pathogen Methods

31 analytes
4 matrices

Biotoxin Methods

18 analytes
5 matrices

SAM Impact

- Identifies a single selected method for each analyte/sample type pairing. Using the same set of methods would:
  - Permit sharing of sample load between laboratories
  - Potentially increase the speed of analysis
  - Improve data comparability
  - Simplify the task of outsourcing analytical support to the public and commercial laboratory sector
- Use of such methods would also improve follow-up activities of :
  - validating results
  - evaluating data
  - making decontamination and re-occupancy decisions

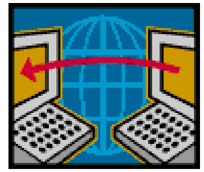
SAM 2012

Radiochemical Methods

25 analytes
6 matrices
45 methods

SAM 2012 Rad Section Additions:
Total Activity Screening analyte class
Non-food vegetation as a sample type
Newly developed rapid methods for selected radionuclides in water, air filters, and surface wipes
Replaced the ^{90}Sr method for aqueous and liquid phase samples 7500-Sr-B with D5811-08
Replaced the ^{99}Tc method for aqueous and liquid phase samples Tc-02-RC with D7168-05
Added Gamma Spectrometry methods for ^{241}Am

SAM Website



www.epa.gov/sam

SAM Methods Query - On-line tool for identifying analytical methods to be used by multiple laboratories measuring target chemical, radiochemical, and biological analytes in environmental samples

Technical Contacts - Contacts for information regarding use of SAM methods

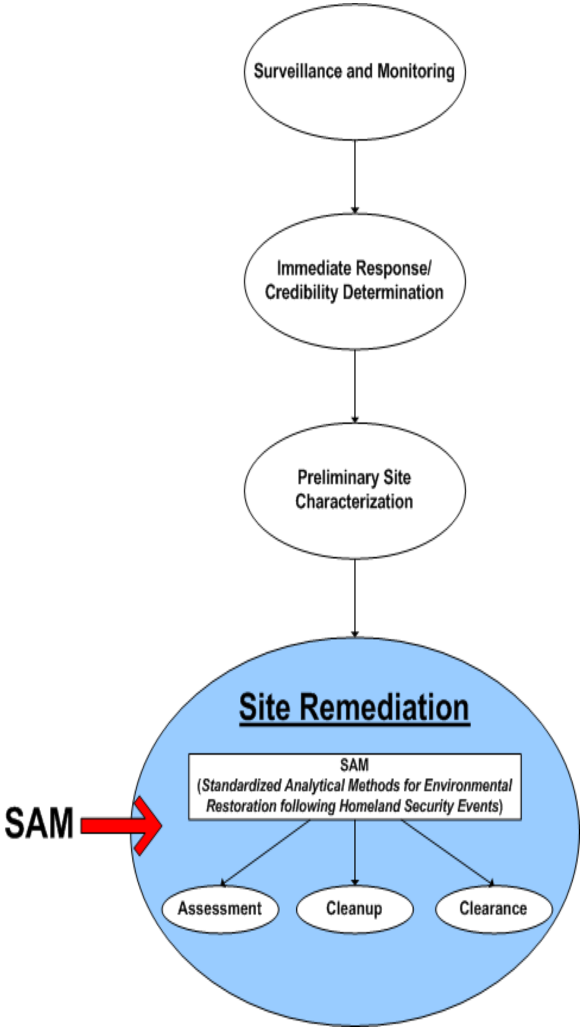
Selected Methods - Summary descriptions and sources of SAM methods

SAM Companion Documents and Sample Collection Procedures - Links to SAM companion documents and other related sample collection procedures

SAM Addendums – Summary descriptions and links to the most recently published rapid methods

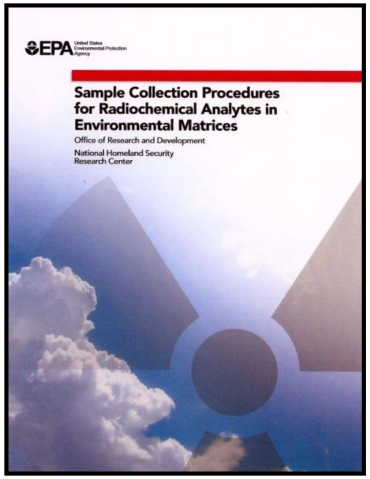
Future of SAM

- SAM is scheduled to be updated and published in 2017
- Newly developed methods will be included
  - Currently using the addendum process to address the need of making newly published methods available to the laboratories and the ERLN allowing for less frequent updates of the entire SAM document, and provides a longer timeframe for method development activities to be completed
- Small workgroups will begin the revision process in early FY16

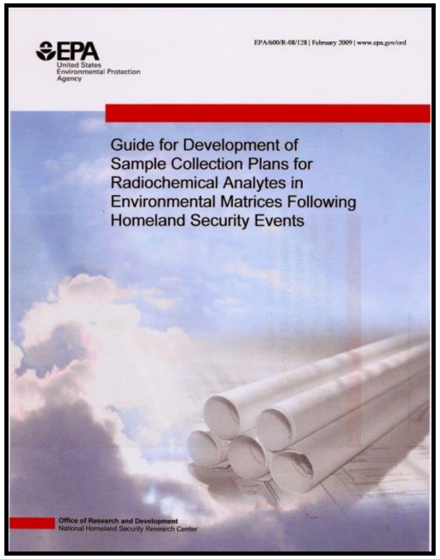


SAM Radiochemical Support Documents

Sample Collection Procedures for Radiochemical Analytes in Environmental Matrices, EPA/600/R-12/566 July 2012



- Procedures for sample collection during site characterization, remediation, and final status phases in support of the SAM selected methods



Guide for Development of Sample Collection Plans for Radiochemical Analytes in Environmental Matrices Following Homeland Security Events, EPA/600/R-08/128 February 2009

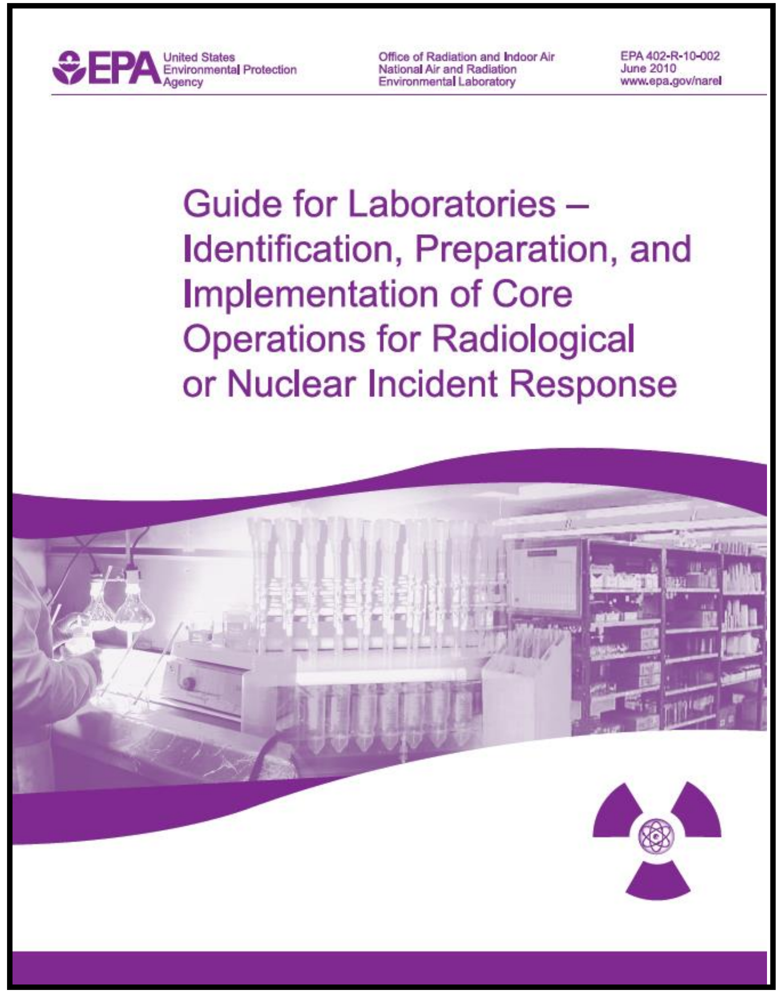
- Framework to assist incident commanders, project managers, state and local authorities, contractors, and enforcement divisions in developing a sample collection plan

ORIA Radiological Incident Response Guides-Laboratory and Management Technical Information

- Documents provide guidance in many areas including:
  - Laboratory operations when switching to incident response mode
  - Technical and statistical bases for Quality Assurance aspects of method validation
  - Comparison of laboratory and field measurements
  - Background information on the types of radionuclides to expect and potential chemical forms
  - Found at http://www.epa.gov/narel/

Key components:

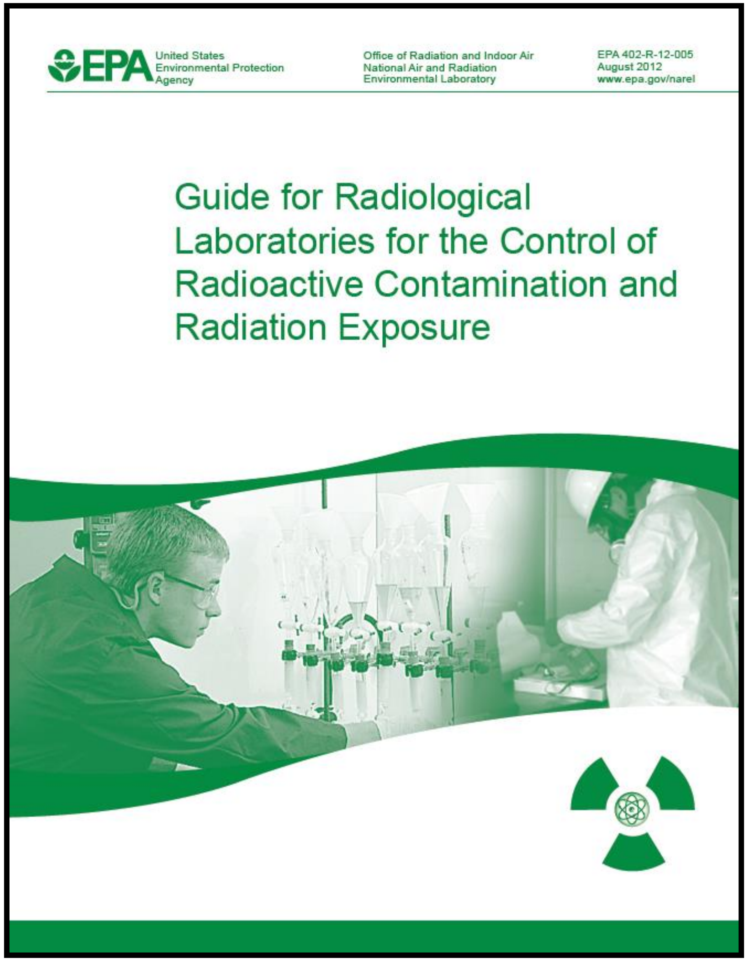
- How to develop a laboratory incident response plan that addresses:
- How to switch from routine environmental laboratory to incident response operations
- Increased contamination controls
- Impacts on the Quality System and QC program
- Identifying needs and managing resources



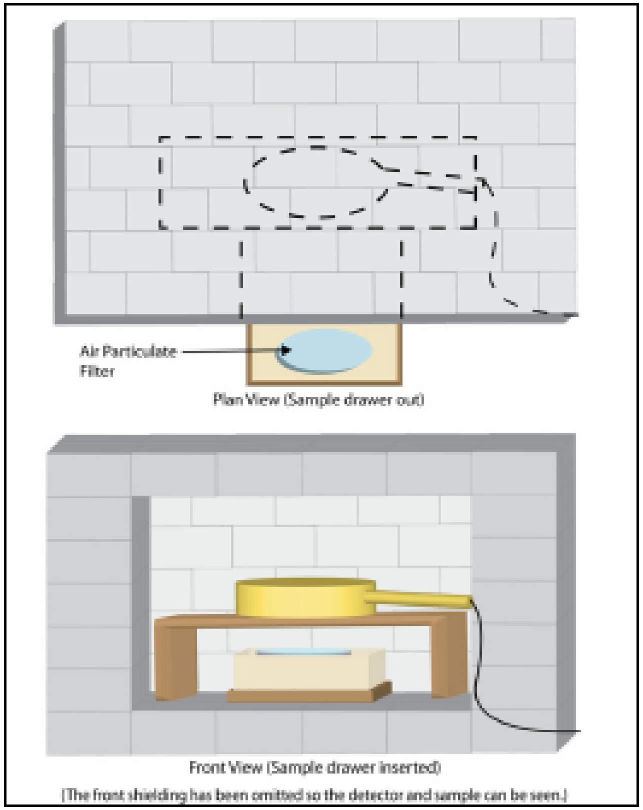
EPA 402-R-10-002, June 2010

Key components:

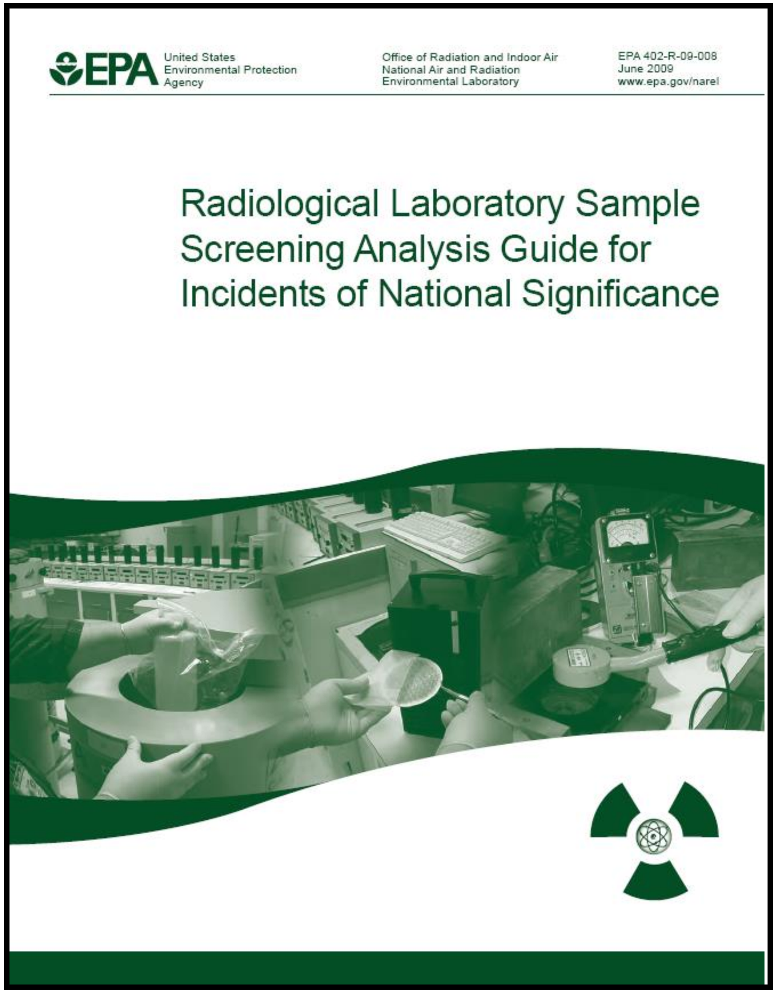
- Use together with Core Operations Guide
- Contrasts radiological and radioanalytical contamination
- Identifies practices and approaches to contamination control
- Examples of setting up process flow and sample handling for incident response
- Examples on how to develop MQOs appropriate to radioanalytical contamination monitoring



EPA 402-R-12-005, August 2012



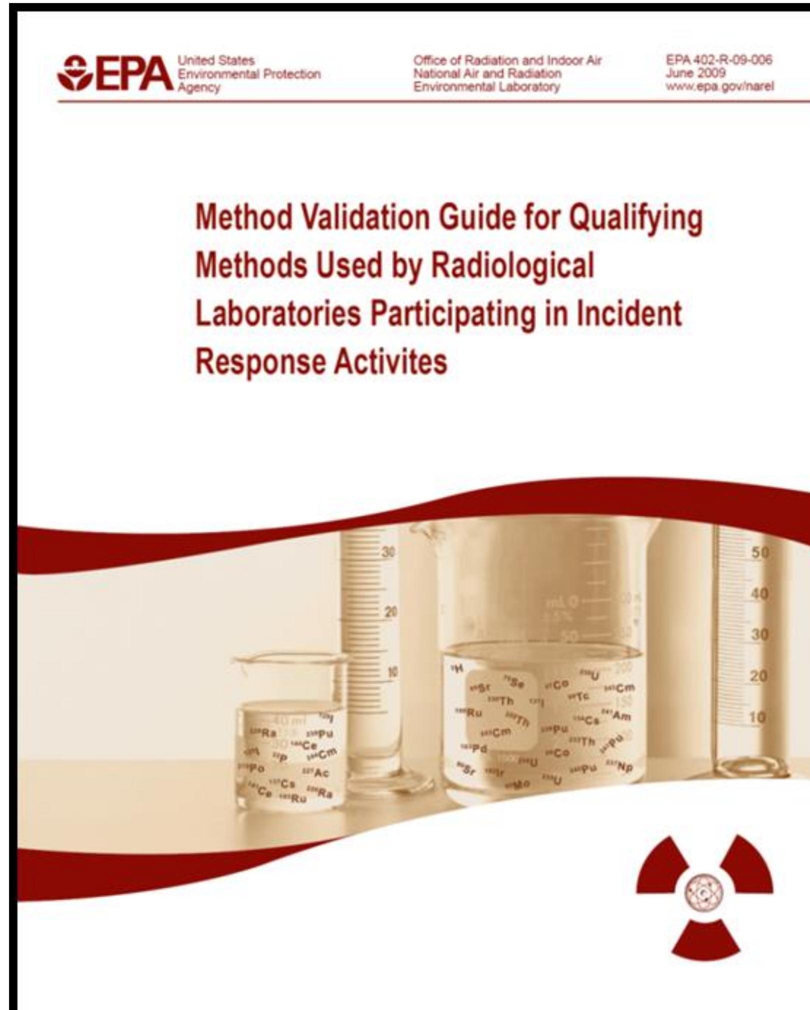
Suggested configuration for rapid screening of air particulate filters



EPA 402-R-09-008, June 2009

Key components:

- Statistical approach to demonstrate when MQOs specified in the method validation plan are met
- Implements MARLAP Chapter 6 method validation protocol
- Graded approach with 5 validation levels
- Number of samples varies by validation level
- Step-by-step process with clear examples



EPA United States Environmental Protection Agency

Office of Radiation and Indoor Air
National Air and Radiation Environmental Laboratory

EPA 402-R-09-006
June 2009
www.epa.gov/narel

Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activites

EPA 402-R-09-006, June 2009

Key components:

- Describes complementary aspects of field and lab measurement techniques
- Developing MQOs
- Validation of lab and field methods
- Example Scenarios: Approaches to Integrating Field and Laboratory Measurements During Response to a Radiological or Nuclear Incident

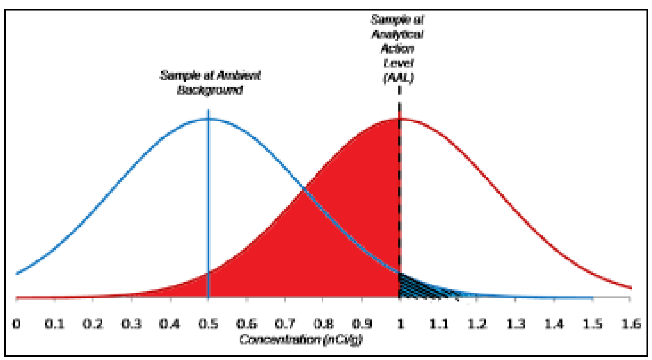
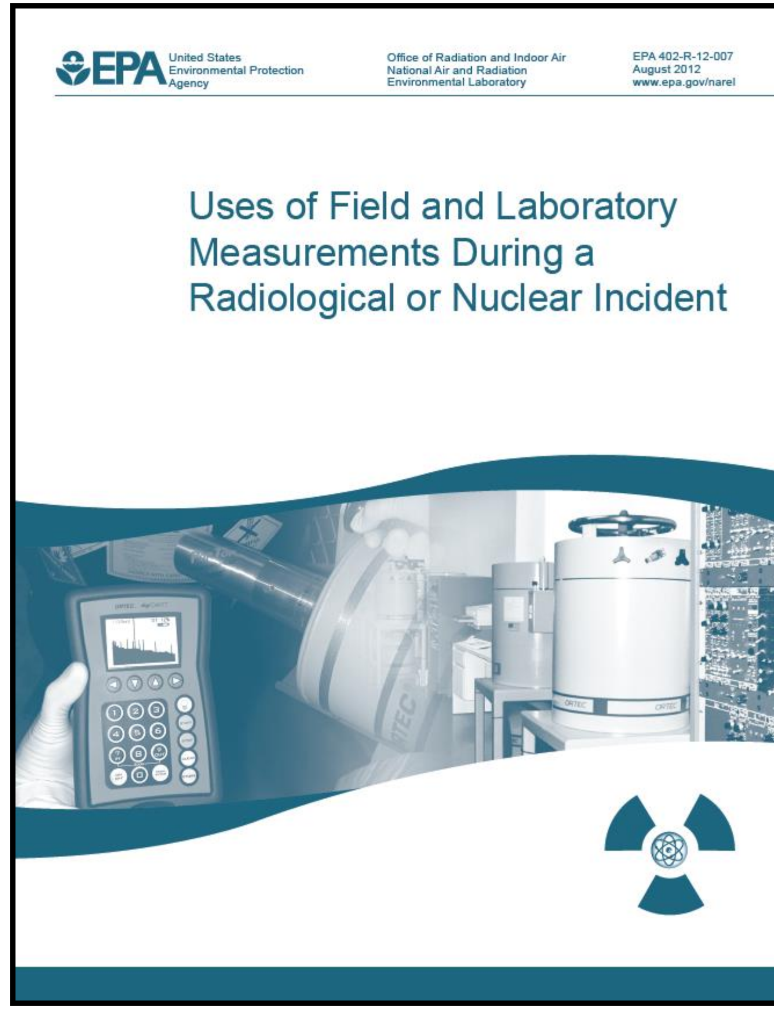
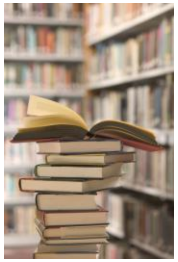


Figure 1 – Uncontrolled Decision Error at the AAL



EPA 402-R-12-007, August 2012

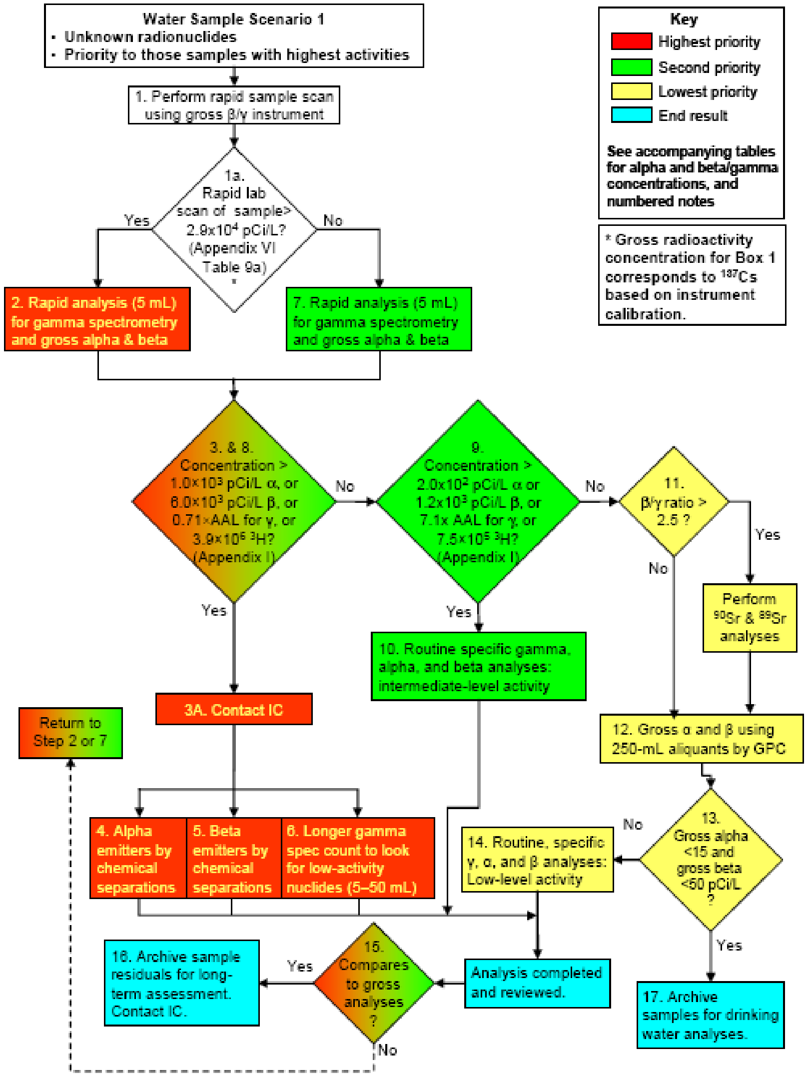
Radiological Incident Response Guides- Different Matrices

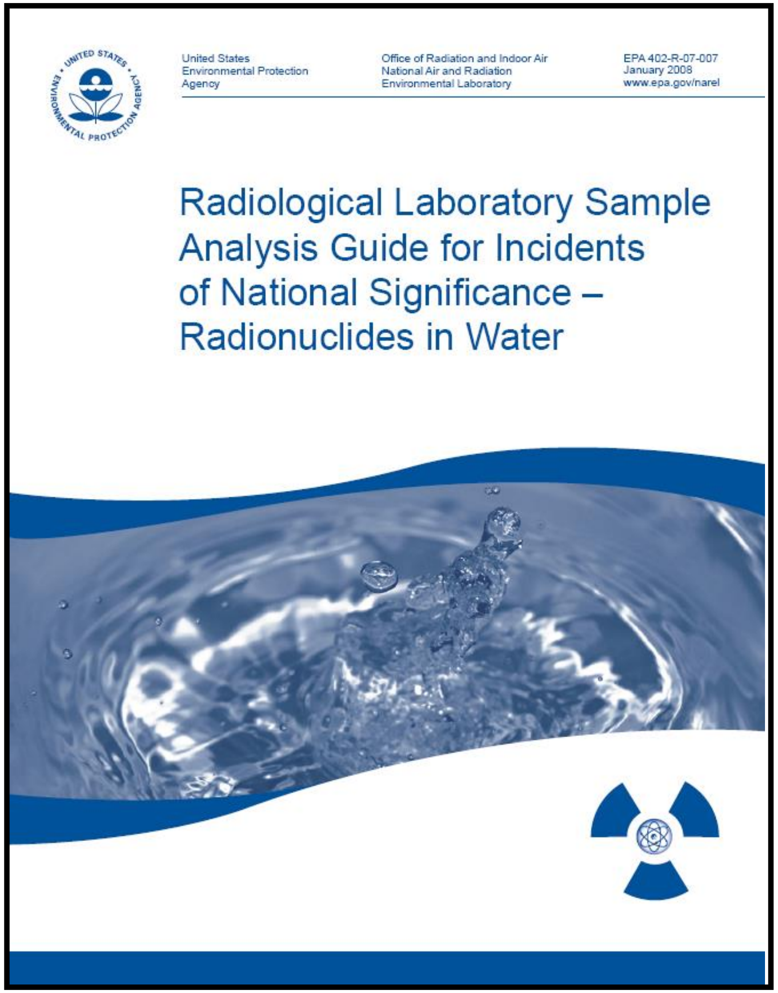


- Purpose of the guides
  - Provide MQOs to help labs plan for incident response
    - e.g., required method uncertainty at Analytical Action Levels (AAL)
    - Conversion of Protective Action Guides (PAGs) and Risk Levels to radionuclide concentrations in different matrices
  - Discuss potential problems when processing high activity concentration samples
  - Flow charts for sample processing optimized to the phase of the incident
  - Detailed radiological background information for the particular matrix
- Found at http://www.epa.gov/narel/

All Guides Contain

- List of potential threat radionuclides
- Specific radionuclide concentrations at PAGs and AAL for each matrix
- How to calculate the required method uncertainty
- Flow charts for sample processing
- Other requirements specific to the matrix

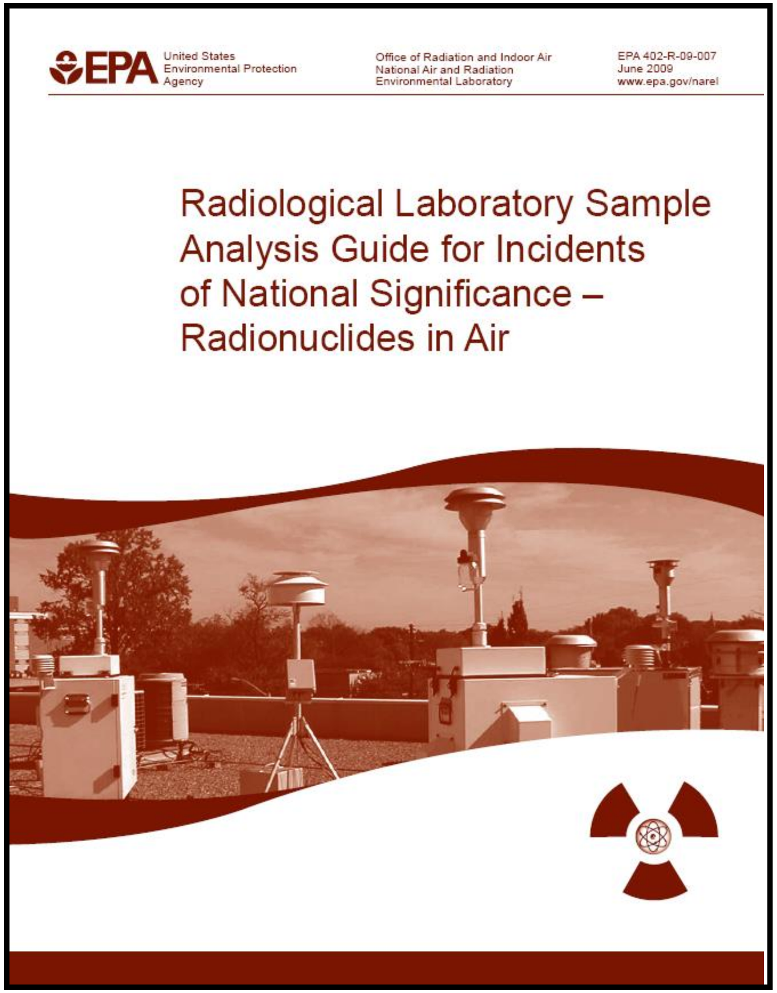




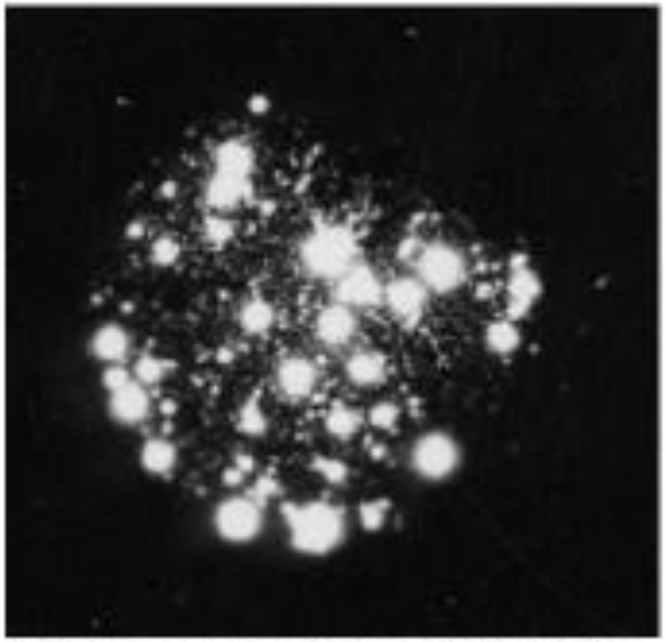
EPA 402-R-07-007, January 2008

Specific information for air filters the lab should know:

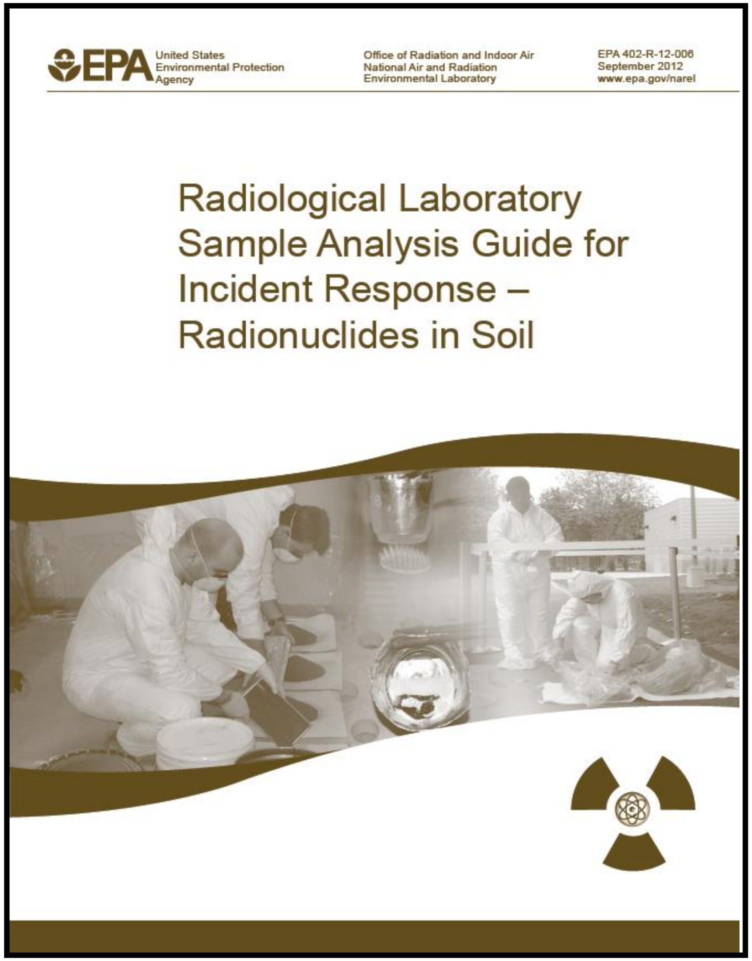
- Volume of air sampled.
- Beginning and ending times of the sampling period.
- Type of filter medium.
- Percent area of the filter sent to the laboratory (e.g., if the filter was split or “punched” prior to shipment to the laboratory).
- Contact activity or dose reading of the filter at the end of the sampling period.



EPA 402-R-09-007, June 2009



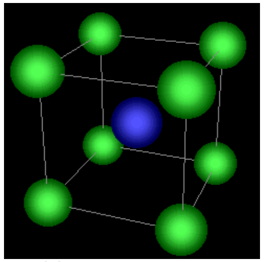
The autoradiograph shows the presence of hot particles of various sizes in a soil sample



EPA 402-R-12-006, September 2012

Impact of Research

Addresses Laboratory Capacity by: Turn around time reductions of 50% or more when compared to existing traditional radiochemical methods



Supports the ERLN by:

- Providing quick results to the field
- Supporting remediation activities
- Informing decision makers on risk encountered in radiologically contaminated urban environments
- Facilitating radiochemistry laboratory preparedness

Addresses the need for publically available urban material methods

Addresses chemical decon interferences on radioanalytical methods

QUESTIONS?

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