FEDERAL RADIOLOGICAL MONITORING AND ASSESSMENT CENTER

Laboratory Analysis Manual

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PREFACE

In Homeland Security Presidential Directive (HSPD)-5, the President directed the development of a new National Response Plan (NRP) to align Federal coordination structures, capabilities, and resources into a unified, all-discipline, all-hazards approach to domestic incident management.

The NRP is built on the template of the National Incident Management System (NIMS), which provides a consistent doctrinal framework for incident management at all jurisdictional levels, regardless of the cause, size, or complexity of the incident. The activation of the NRP and its coordinating structures and protocols—either partially or fully—for specific Incidents of National Significance provides mechanisms for the coordination and implementation of a wide variety of incident management and emergency assistance activities. Included in these activities are Federal support to state, local, and tribal authorities; interaction with nongovernmental, private donor, and private-sector organizations; and the coordinated, direct exercise of Federal authorities, when appropriate.

The Nuclear / Radiological Incident Annex to the NRP addresses the response of Federal agencies to terrorist incidents involving nuclear or radioactive materials (Incidents of National Significance), and accidents or incidents involving such material that may or may not rise to the level of an Incident of National Significance.

In the event of a potential or existing major radiological incident, the U.S. Department of Energy's National Nuclear Security Administration Nevada Site Office (NNSA/NSO) has been charged with establishing and managing the Federal Radiological Monitoring and Assessment Center (FRMAC). The FRMAC provides coordinated federal assistance in the off-site areas to the impacted state(s) and the Coordinating Agency responsible for regulation and/or operation of the accident site.

This manual was written for those personnel who will be called upon to provide technical data, input, and decisions. Overall, this manual provides general guidance and some specific diagrams and forms. However, it is understood that site and event specific operational decisions and procedure parameters will need to be established and documented at the time of an emergency event. It is also understood that FRMAC sample tracking and analysis may be operating in an integrated or coordinated environment with other agencies and jurisdictions, including state or local agencies. This manual is intended to provide enough guidance for stand-alone use without limiting FRMAC's ability to integrate the work with other partners or stakeholders. Note that the some of the titles of management positions within the FRMAC have been changed in order to comply with the structure of the Incident Command System (ICS) under NIMS.

NNSA / NSO has the overall responsibility for maintaining the master copy of all FRMAC manuals. Please provide comments on this manual to:

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National Nuclear Security Administration
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ACRONYMS AND ABBREVIATIONS

CA	Coordinating Agency
CFR	Code of Federal Regulations
CMRT I	Consequence Management Response Team Phase I
CMRT II	Consequence Management Response Team Phase II
CMRT III	Consequence Management Response Team Phase III
COC	Chain of Custody
CPM	Counts per Minute
DoD	U.S. Department of Defense
DOELAP	U.S. Department of Energy Laboratory Accreditation Program
DOECAP	U.S. Department of Energy Consolidated Audit Program
DOT	U.S. Department of Transportation
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
ERDS	Emergency Response Data System
ES&H	Environmental Safety and Health
FRMAC	Federal Radiological Monitoring and Assessment Center
FWHM	Full Width Half Maximum
GPC	Gas Proportional Counter
HSOC	Homeland Security Operations Center
HSPD-5	Homeland Security Presidential Directive 5
ICP-MS	Inductively Coupled Plasma Mass Spectrometer
IIMG	Interagency Incident Management Group
JFO	Joint Field Office
KPA	Kinetic Phosphorescence Analyzer
LCS	Laboratory Control Sample
LEGe	Low Energy Germanium Detector
LIMS	Laboratory Information Management System
LLD	Lower Level of Detection
LSAM	Low Specific Activity Material
LSC	Liquid Scintillation Counter
M&O	Maintenance and Operations

MAPEP	Mixed Analyte Performance Evaluation Program
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration
MQO	Measurement Quality Objective
NAVLAP	National Voluntary Laboratory Accreditation Program
NCM	Non-Conformance Memo
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIMS	National Incident Management System
NIST	National Institute of Standards and Technology
NNSA	National Nuclear Security Administration
NNSA/NSO	National Nuclear Security Administration Nevada Site Office
NRC	U.S. Nuclear Regulatory Commission
NRP	National Response Plan
NUPIC	Nuclear Procurement Issues Committee
PFO	Principle Federal Official
POC	Point of Contact
PT	Proficiency Testing
QA	Quality Assurance
QAP	Quality Assurance Program
QC	Quality Control
RODEO	Radioanalytical Organization Database for Emergency Operations
SCF	Sample Control Form
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOQ	Statement of Qualifications
SPRM	Standard Performance Reference Material
SRL	Sample Receiving Line
SRM	Standard Reference Material
TLD	Thermoluminescent Dosimeter
TPU	Total Propagated Uncertainty
WCRM	Well characterized reference material

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1.0 INTRODUCTION

When the FRMAC responds to a radiological accident, monitoring, sampling, and radioanalytical support will arrive from a number of different sources. The respondents providing this support will, in all likelihood, have received varying levels of training and will have experience with a variety of monitoring, sampling, and radioanalytical equipment and procedures. It is important that an acceptable and established set of standard operating procedures (SOPs) be followed by all personnel having responsibilities for processing samples and analytical data during the emergency. Overall, this manual provides general guidance and some specific diagrams and forms. However, it is understood that site and event specific operational decisions and procedure parameters will need to be established and documented at the time of an emergency event. It is also understood that FRMAC sample tracking and analysis may be operating in an integrated or coordinated environment with other agencies and jurisdictions, including state or local agencies. This manual is intended to provide enough guidance for stand-alone use without limiting FRMAC's ability to integrate the work with other partners or stakeholders.

Early in an emergency, analytical data will be urgently needed as a basis for protective actions. FRMAC emergency response procedures are intended for use in processing relatively large numbers of samples in the shortest possible time. In the early stages of an emergency, when the impact on the health and safety of the public is not well defined, the resources dedicated to quality assurance (QA) activities must be sufficient to assure that appropriate radioanalytical Measurement Quality Objectives (MQOs) and assessment Data Quality Objectives (DQOs) are met. As the emergency stabilizes, QA activities will evolve commensurate with the need to reach appropriate DQOs. DQOs represent a compromise between precise analytical determinations and the timeliness for emergency response activities.

The intermediate phase will require a greater degree of data quality assurance as longer-term exposure risks are evaluated. The intermediate phase could last up to 30 days depending on the magnitude of the incident. During this phase, the role of field measurements and mobile laboratory assets may decline if they are not be able to meet the more rigorous data quality objectives and measurement quality objectives needed for assessment decisions. These more rigorous objectives will require the use of larger laboratories, with greater capacity and enhanced capabilities. The role of local analytical capability may also decline depending on its capacity and ability to adapt to these later phase DQOs. Larger capacity and greater capability laboratories across the country will likely become the mainstay of the analytical effort as the incident evolves into the recovery phase. These laboratories may be geographically distant from the incident, which will increase sample management challenges. The relative role of field measurements, mobile laboratories and fixed laboratories will depend on the radionuclides of concern for the specific incident or emergency. This manual addresses the processes and procedures for coordinating the analysis of samples during FRMAC operations.

These procedures are applicable to DOE/NNSA's Consequence Management Response Team (CMRT) Phases I, II, and III as well as a FRMAC response. They are designed for use during the emergency and intermediate phase of an incident and may or may not be continued by the Coordinating Agency (CA) during the post-emergency (recovery) phase.

2.0 LABORATORY ANALYSIS ORGANIZATION

The Laboratory Analysis Manager receives direction from the FRMAC Operations Manager and provides direction to the staff supporting the Laboratory Analysis group. Figure 1 shows the organization of the Laboratory Analysis group. Complete FRMAC organization charts are available in the *FRMAC Operations Manual.*

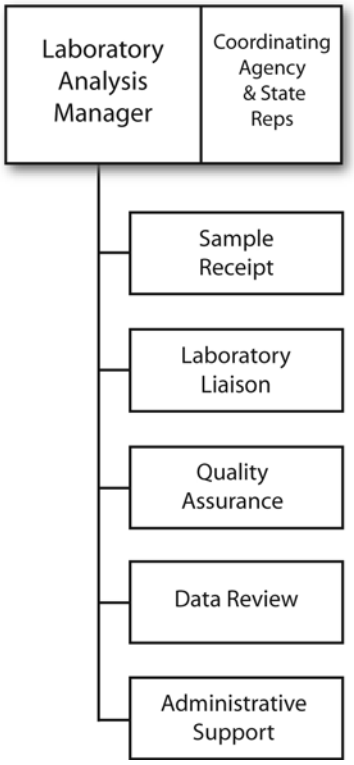


FIGURE 1. ***ORGANIZATION OF THE FRMAC LABORATORY ANALYSIS GROUP***

3.0 RESPONSIBILITIES

3.1 Laboratory Analysis Manager

1. Integrate operations with hotline operations (See Section 2.4.6 in FRMAC Health and Safety Manual) to assure that samples are surveyed for contamination and transferred to sample receipt personnel efficiently.
2. Communicate analytical capability and capacity to the monitoring manager and the assessment manager.
3. Evaluate the analytical capabilities of laboratories. Be able to identify what analyses can be performed by identified laboratories.
4. Ensure that the laboratories receiving samples can reach the detection limits required for assessment decisions. Ensure that the quantity of sample collected is adequate to meet detection limit requirements in a reasonable count time.

5. Monitor sample numbers and analysis types to maximize production and minimize turnaround time commensurate with DQOs. Track the sample load to each laboratory.
6. Coordinate sample identification, tracking and laboratory use with other state or local agencies involved in the emergency event.
7. Communicate expected turnaround time for results from laboratories and communicate analytical priorities to the laboratories based on assessment requirements.
8. Activate laboratories (fixed and mobile).
9. Direct samples to the appropriate laboratory.
10. Coordinate shipment of samples for offsite analysis. If qualified, ship samples as needed.
11. Designate an appropriate area to perform sample preparation/preservation (if necessary).
12. Act as Point of Contact (POC) for queries regarding the status of any sample or reprioritization submitted through a FRMAC Action Request Form (Figure 2).
13. Environment, Safety and Health oversight for sample handling and mobile laboratory areas.
14. Review data for accuracy and reasonableness.
15. Ensure that analysis results are forwarded to database in a usable format.
16. Resolve data quality issues.

3.2 Sample Control Technician

1. Receive samples that have been screened for contamination by hot line personnel. See the FRMAC Health and Safety Manual for specific screening procedures.
2. Review, validate and sign chain of custody documentation.
3. Inspect samples.
4. Log samples into sample tracking system.
5. Record sample tracking information.
6. Prepare samples for shipment and include shipping documentation.

If the sample control technician is a qualified shipper they may “ship” the sample, otherwise the Laboratory Analysis Manager will be responsible for using a qualified shipper.

FRMAC ACTION REQUEST

Reference No:

Report By Priority

Organization _____ 1. Emergency

Name _____ 2. Urgent

Date _____ **Time** _____ 3. Routing

Nature of Request:

Do not write below this line. For FRMAC Use Only.

FRMAC Response Action Group(s) Priority

Organization _____ 1. Emergency

Name _____ 2. Urgent

Date _____ **Time** _____ 3. Routing

Authroization;

Reply/Resolution

Distribution:

For FRMAC Tracking Use Only

Received By _____

Date/Time _____

Goldenrod (Originator) – Pink (file) – Green Response Group) – Yellow (originator Retain) – White (FRMAC Retention)

FIGURE 2. *FRMAC ACTION REQUEST FORM*

3.3 Laboratory Liaison

1. Communicates with DOE/NNSA's CM home team to activate existing laboratory service contracts.
2. If deployed, interface with mobile laboratory.
3. Advise Analysis Manager of laboratory capacity and track sample load per assigned laboratory.
4. Communicate sample priorities.

3.4 Quality Assurance Technician

1. Coordinates collection of quality assurance samples with the Monitoring Manager or designated monitoring staff.
2. Inject QA/Quality Control (QC) samples into the sample stream.
3. Compiles and reports results from QA samples and brings unusual results to the attention of the Laboratory Analysis Manager.
4. Investigates causes of unusual quality assurance results.

3.5 Data Review Technician

1. Performs review of laboratory results for completeness and inconsistencies.

3.6 Administrative Support Technician

1. Maintains records and files.

4.0 METHODS AND PROCEDURES

4.1 Laboratory Evaluation and Rating

4.1.1 Purpose

A Radioanalytical Organization Database for Emergency Operations (RODEO) resides on the FRMAC server. This procedure outlines the maintenance of this database and the guidelines to evaluate laboratories to produce a subjective rating. This procedure is applicable to all radioanalytical assets in RODEO.

4.1.2 Responsibilities

- Analysis Manager: Validates database information on a regular basis. This process may be automated and implemented by the ERDS Data Technician.
- ERDS Data Technician: Programs and prints reports for the Analysis Manager. Ensure that the current database is uploaded to deployment computers.

4.1.3 Procedure

Prior to utilizing a laboratory from the database, verify the database information that was used to create a subjective rating of an organization's capability and capacity to meet DQOs associated with the phases of a FRMAC response. When additional analytical assets are identified during an

event, the organization needs to complete the RODEO Questionnaire. Examples of the data collected are found in Figures 3 through 13.

Consider the following when making the subjective evaluation of a radioanalytical organization's ability to process samples:

- The analysis needed
- The sample matrix
- The MQO/DQO (minimum detectable concentration [MDC])
- Timeliness of results and sample capacity based on current sample workload
- Available bench space, hoods, staff, and nuclear instrumentation

For example, fairly simple, gross analysis needs minimal bench space, hoods, and nuclear instrumentation while, complicated low-level analyses need maximum bench space, hoods, staff, and nuclear instrumentation.

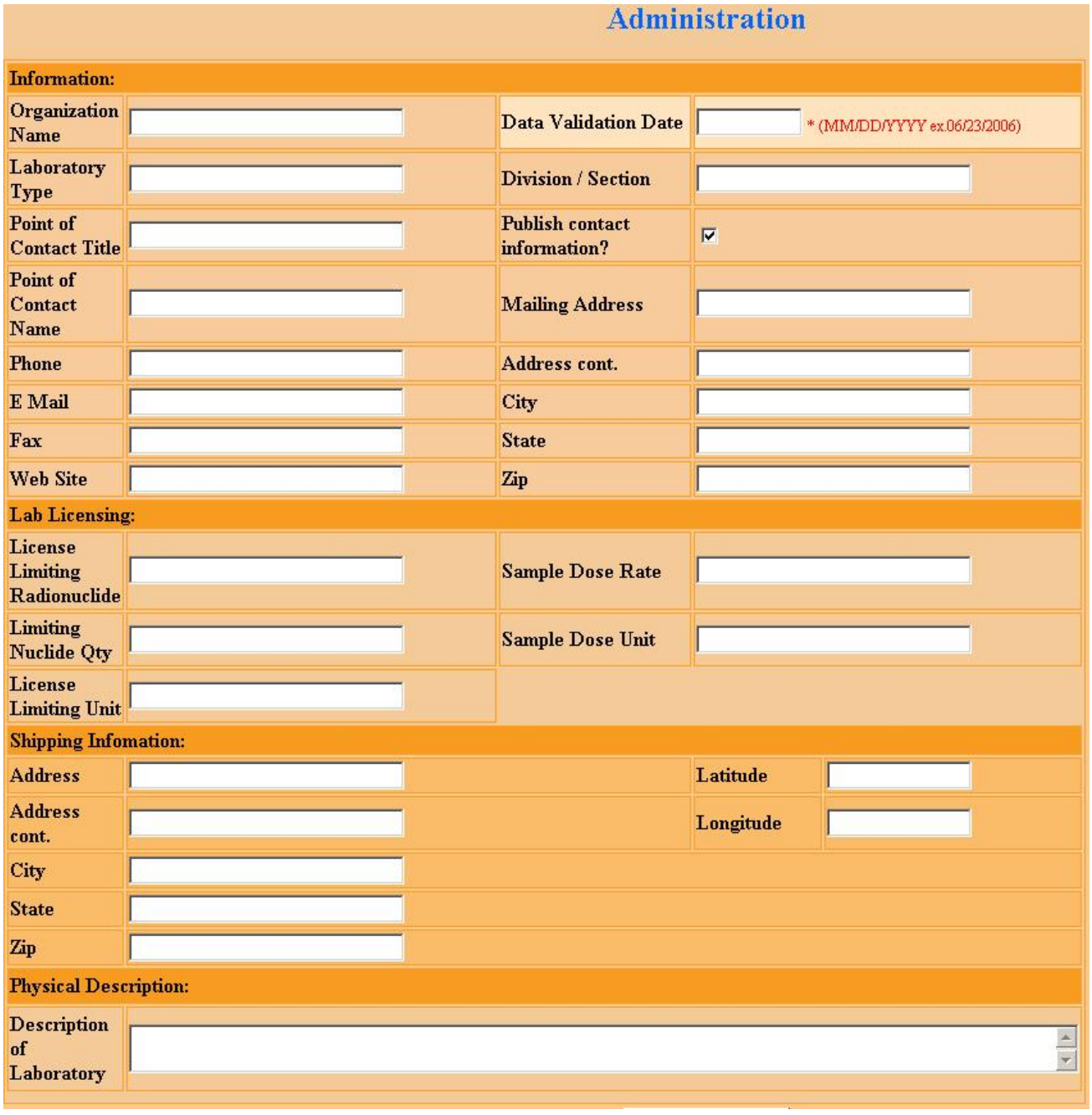
Equipment calibrations should be performed using National Institute of Standards and Technology (NIST) traceable reference radionuclide standards whenever possible. The adequacy of the facilities, instrumentation, and staff levels can be estimated by two general mechanisms: self reported information in the laboratory database or a statement of qualifications and external audit information. Information received from the prospective laboratory may provide an estimate of the laboratory's resources, but an initial onsite audit verifies the actual existence and maintenance of resources.

- Is the laboratory experienced in performing the same or similar analyses?
- Does the laboratory have satisfactory performance evaluation results from formal monitoring or accreditation programs?
    - The laboratory should be able to provide a summary of QA audits and proof of participation in inter-laboratory cross-check programs.
- Is there an adequate capacity to perform all analyses within the desired timeframe?
    - This criterion considers whether or not the laboratory possesses a radioactive materials handling license or permit for the samples to be analyzed. Large events will require more than one analytical laboratory to meet turn-around-time and DQO requirements.
- Does the laboratory provide an internal quality control review of all generated data that is independent of the data generators?
- Does the laboratory possess the appropriate well-documented procedures, instrumentation, and trained personnel to perform the necessary analyses?
    - Necessary analyses are defined by the data needs (radionuclide(s) of interest and target detection limits) identified by the DQO process.
- Are there adequate protocols for method performance documentation and sample security/chain of custody?

Providers of radioanalytical services should have an active and fully documented QA program in place. This program should comply with the objectives determined by the DQO. The QA program should include:

- Laboratory organizational structure.

- Personnel qualifications.
- Written standard operating procedures and instructions.
- Inter- and intra-laboratory performance analyses.
- Design control to define the flow of samples through the laboratory.
- A corrective action plan.
- An internal audit program.



Administration

Information:			
Organization Name		Data Validation Date	* (MM/DD/YYYY ex.06/23/2006)
Laboratory Type		Division / Section	
Point of Contact Title		Publish contact information?	☑
Point of Contact Name		Mailing Address	
Phone		Address cont.	
E Mail		City	
Fax		State	
Web Site		Zip	
Lab Licensing:			
License Limiting Radionuclide		Sample Dose Rate	
Limiting Nuclide Qty		Sample Dose Unit	
License Limiting Unit			
Shipping Infomation:			
Address		Latitude	
Address cont.		Longitude	
City			
State			
Zip			
Physical Description:			
Description of Laboratory			

FIGURE 3. ***RODEO – ADMINISTRATION***

FIGURE 4. *RODEO - STAFFING*

Analyte / Matrix Capability Chart

Analyte Matrix	Air Filter	Air Cartridge	Soil Sediment	Vegetation	Drinking Water	Surface Water	Ground Water	Milk	Meat/Fish Tissue
Gross Alpha	☐	☐	☐	☐	☐	☐	☐	☐	☐
Gross Beta	☐	☐	☐	☐	☐	☐	☐	☐	☐
Gamma_Emitters 59-2000 KeV	☐	☐	☐	☐	☐	☐	☐	☐	☐
H-3	☐	☐	☐	☐	☐	☐	☐	☐	☐
C-14	☐	☐	☐	☐	☐	☐	☐	☐	☐
Fe-55	☐	☐	☐	☐	☐	☐	☐	☐	☐
Ni-63	☐	☐	☐	☐	☐	☐	☐	☐	☐
Sr-89	☐	☐	☐	☐	☐	☐	☐	☐	☐
Sr-90	☐	☐	☐	☐	☐	☐	☐	☐	☐
Tc-99	☐	☐	☐	☐	☐	☐	☐	☐	☐
U-234/235/238	☐	☐	☐	☐	☐	☐	☐	☐	☐
Pu-238/239	☐	☐	☐	☐	☐	☐	☐	☐	☐
Am-241	☐	☐	☐	☐	☐	☐	☐	☐	☐
Pu-241	☐	☐	☐	☐	☐	☐	☐	☐	☐
I-129	☐	☐	☐	☐	☐	☐	☐	☐	☐
I-131	☐	☐	☐	☐	☐	☐	☐	☐	☐

Update Record

FIGURE 6. ***RODEO – SOP MATRIX***

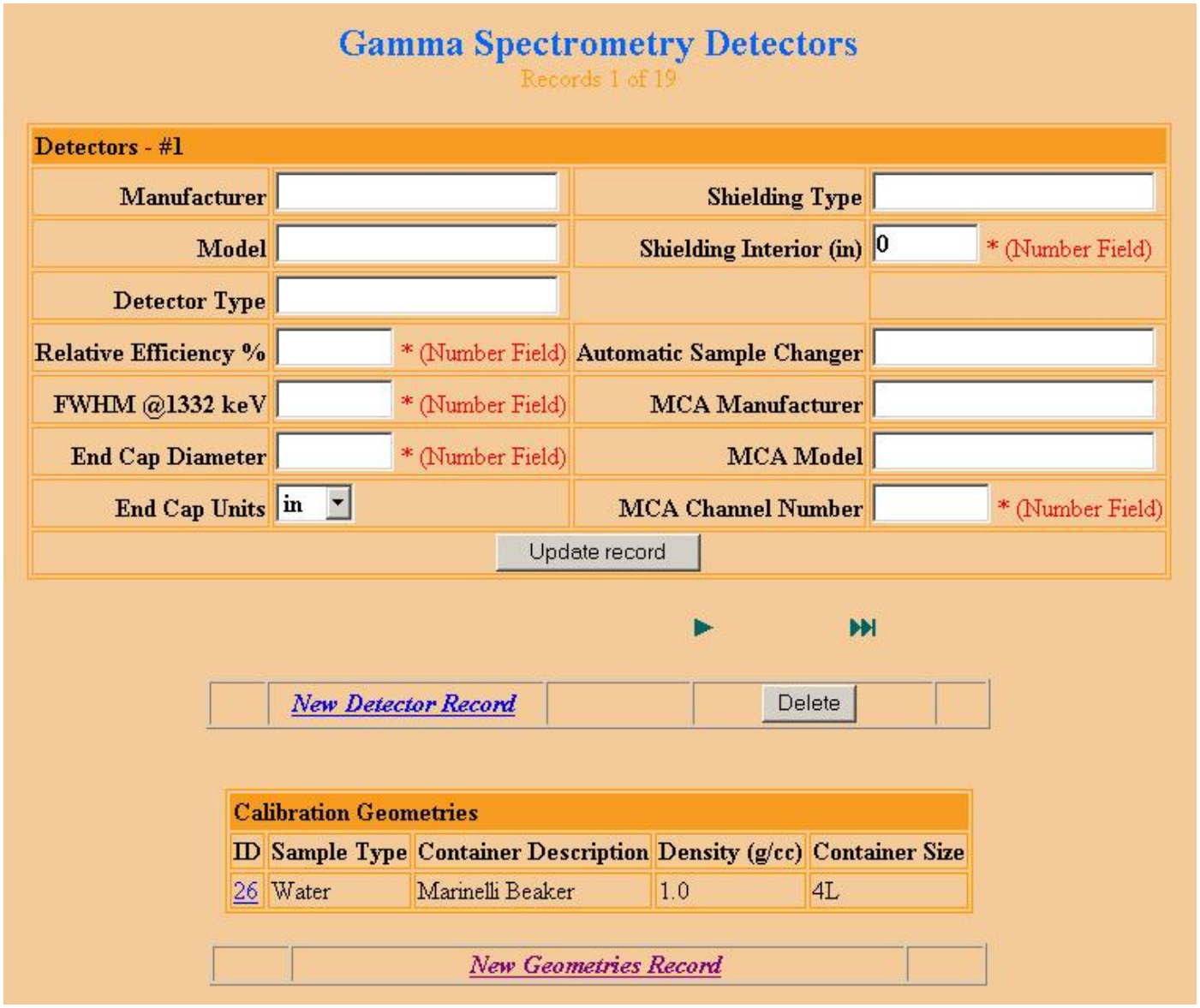


FIGURE 7. ***RODEO – GAMMA DETECTORS***

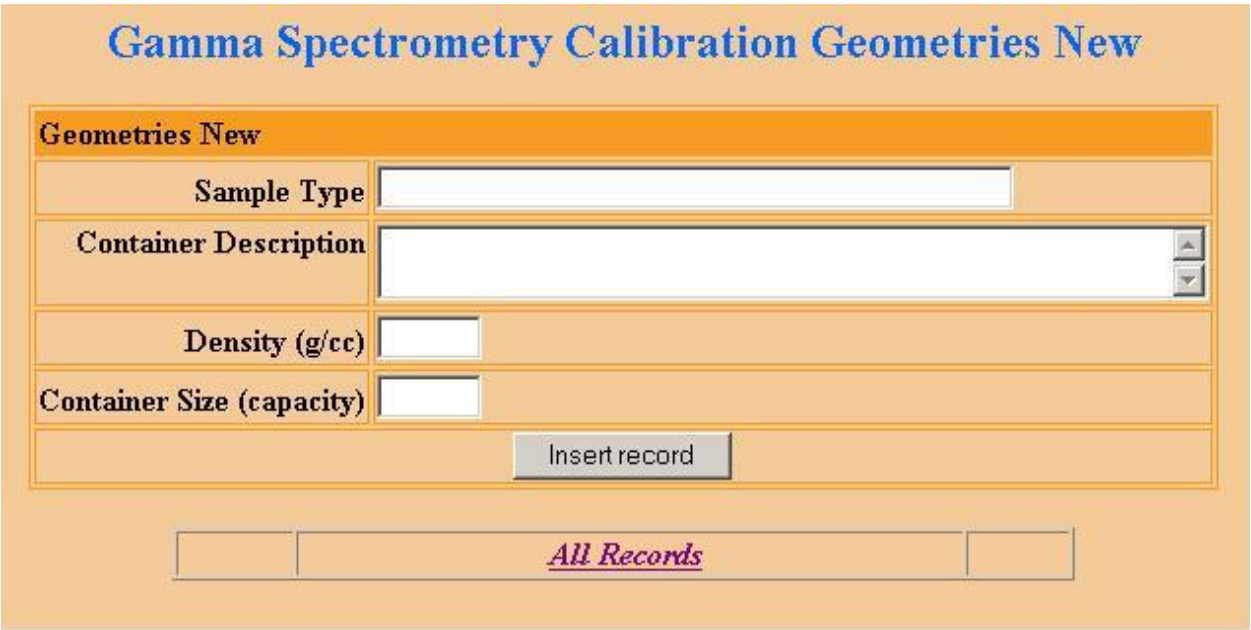


FIGURE 8. ***RODEO GAMMA GEOMETRIES***

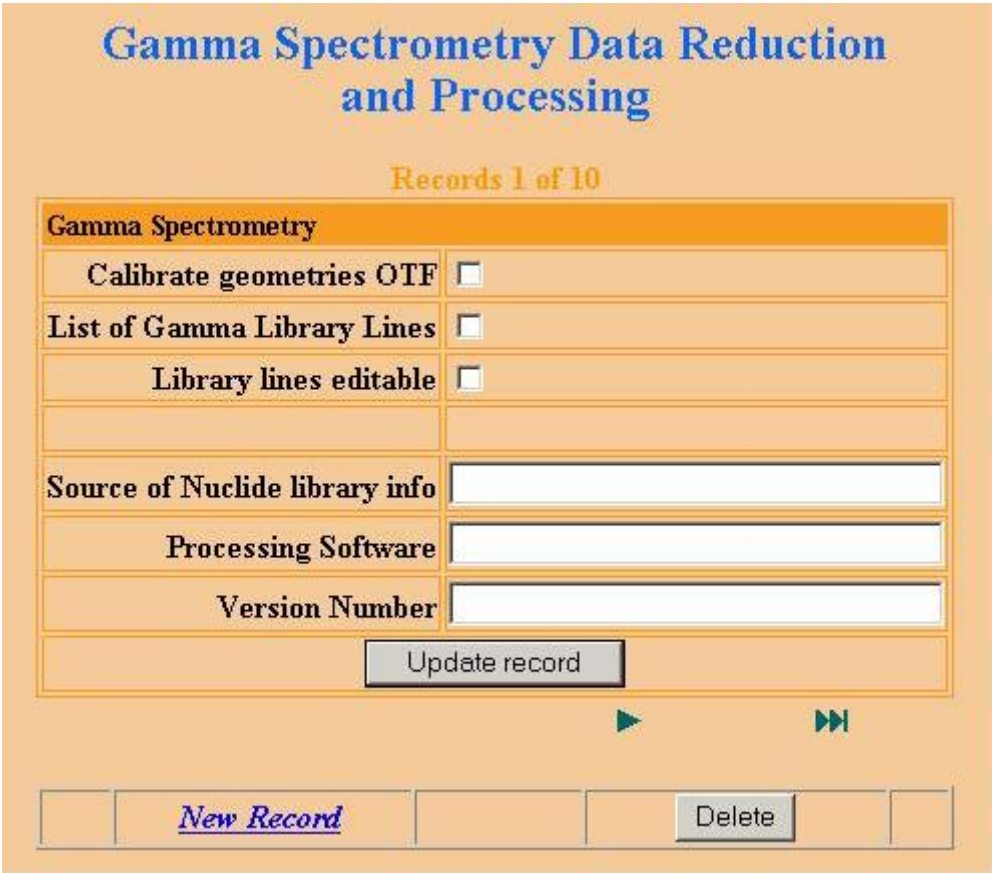


FIGURE 9. *RODEO GAMMA PROCESSING*

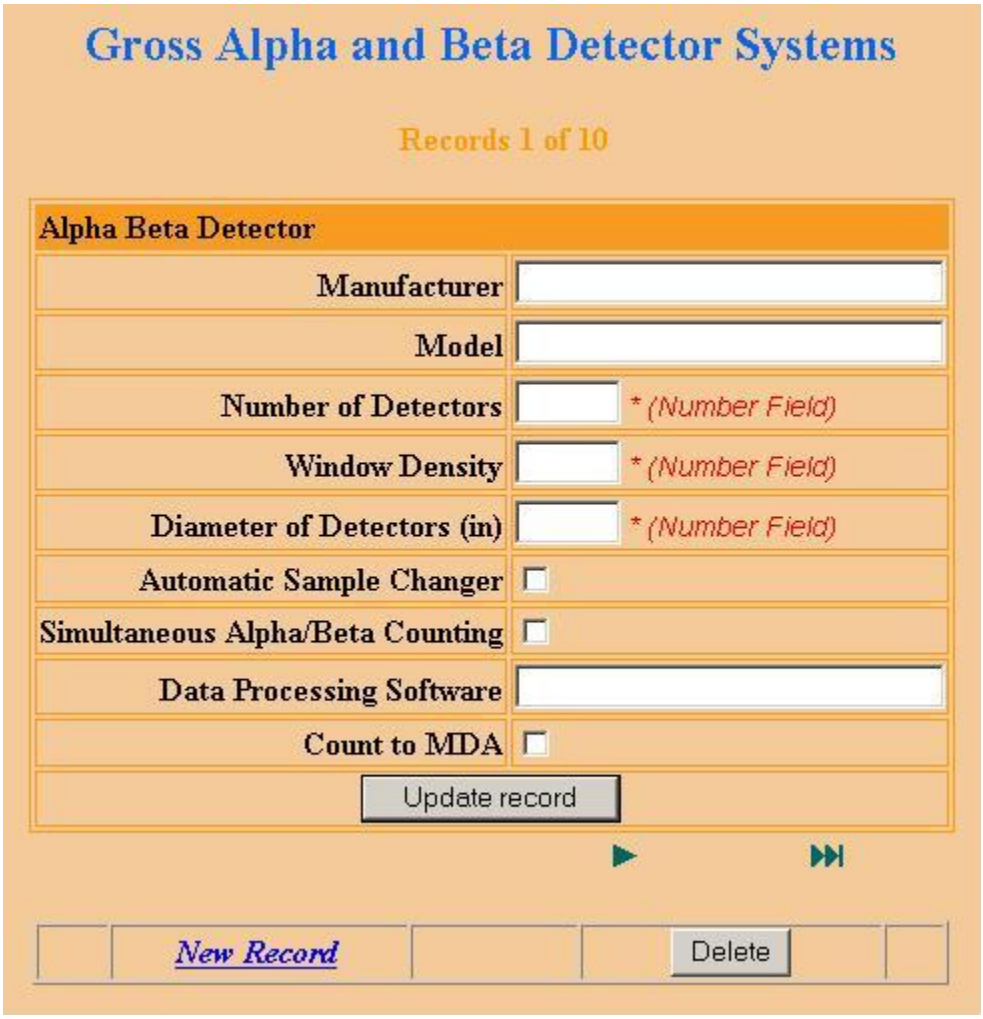


FIGURE 10. *RODEO – ALPHA AND BETA DETECTORS*

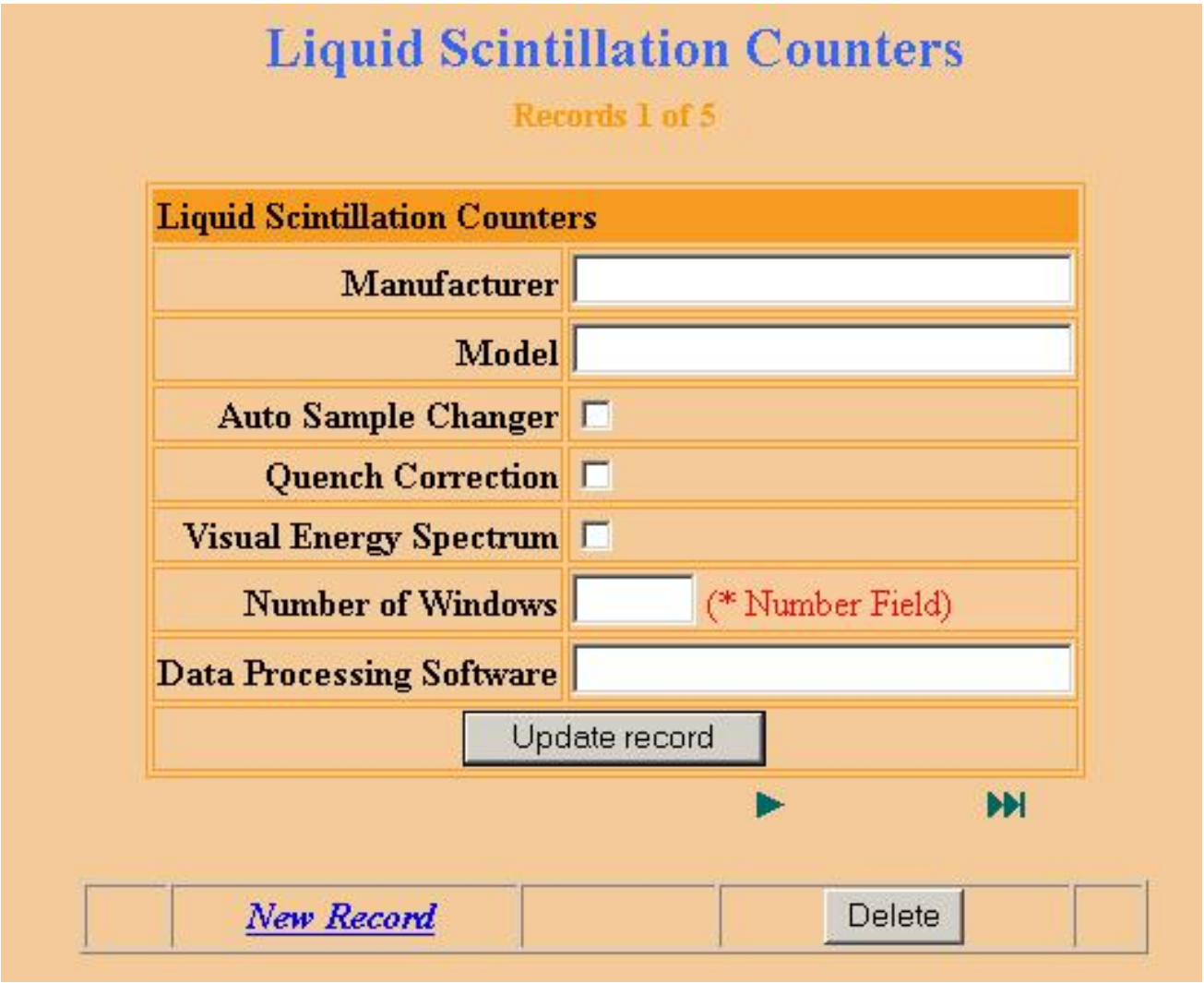


FIGURE 11. ***RODEO – LIQUID SCINTILLATION INSTRUMENTS***

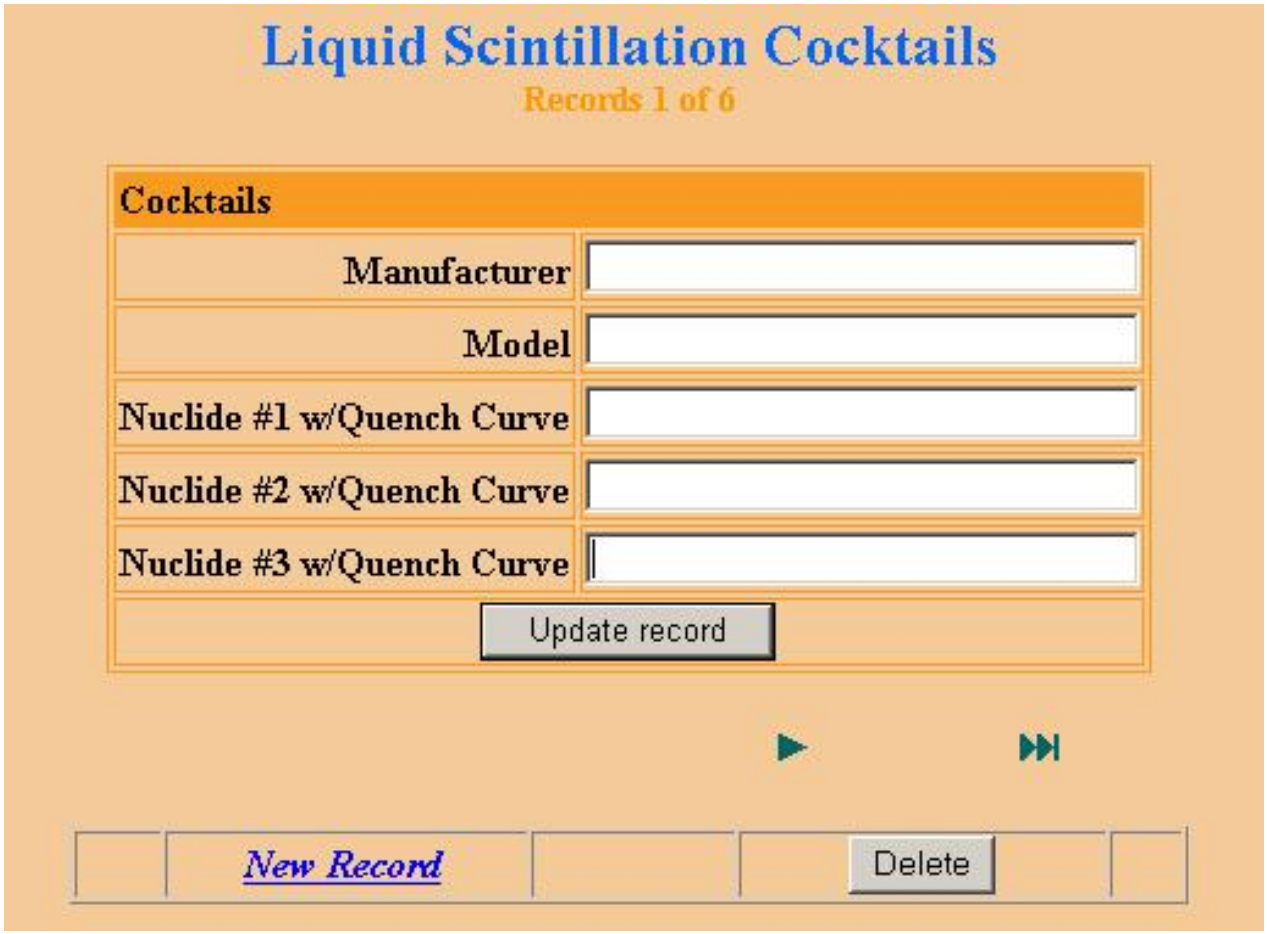


FIGURE 12. *RODEO – LIQUID SCINTILLATION COCKTAILS*

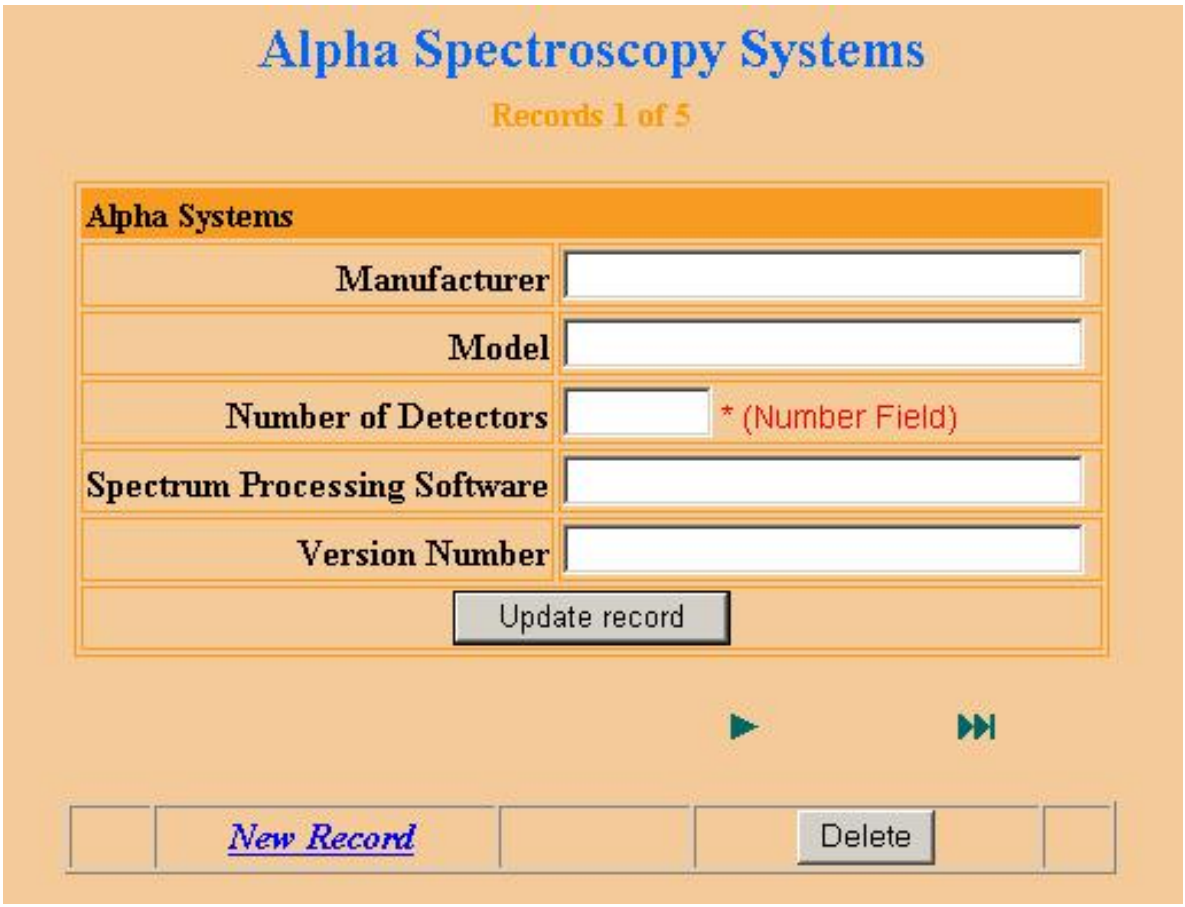


FIGURE 13. *RODEO – ALPHA SPECTROSCOPY SYSTEMS*

4.1.4 Records

The radioanalytical laboratory database is maintained on ERDS servers and deployable computers. Individual laboratory database entries should be validated by a laboratory representative semi-annually.

4.2 Laboratory Selection and Coordination

During the initial phase of the incident, the quantity of analytical data needed for assessment increases dramatically. DQOs are adjusted to be commensurate with the urgency of the decision at hand, and the risk of potential consequences from an incorrect decision. The intermediate phase will require more rigorous data quality as longer-term exposure risks are evaluated. The intermediate phase could last up to 30 days depending on the magnitude of the incident. During this phase, the role of field measurements and mobile laboratory assets will decline if they are not be able to meet the DQOs needed for assessment decisions. The role of local analytical capability may also decline depending on its capacity and ability to adapt to these increasing DQOs. The relative role of field measurements, mobile laboratories and fixed laboratories will depend on the radionuclides of concern for the specific incident or emergency.

Analytical assets are critical to provide timely data to FRMAC assessors. Those facilities that are not impacted by the event will be the first ones called upon. Some of these facilities may not normally analyze samples with the potential for significant levels of contamination but because of their proximity can still fill an analytical need during the earliest stages of an incident. Asset identification and qualification is an ongoing project, as new organizations are created and others dissolve, merge, relocate, or reorganize. The immediacy of an event and the need for a quick answer will determine the role of a local asset. There may be an expanded capability laboratory asset that is physically close to the event but not affected by the event.

Laboratories that have extended ability to conduct analysis for virtually any radionuclide occurring in a wide variety of media (soil, water, and vegetation) and have a thorough quality assurance program should be used as soon as possible. These laboratories are typically audited and accredited by several organizations (DOE's Laboratory Accreditation Program and Consolidated Audit Program, National Voluntary Laboratory Accreditation Program, and National Environmental Laboratory Accreditation Program) and participate in multiple radiological performance testing programs. Some of these laboratories have existing contracts with DOE field offices that provide for 24-hour turn-around time. The laboratories closest to the event would be utilized as early as feasible.

4.2.1 Purpose

Once the decision to perform sampling activities is made, the next step is to consider the type of analysis and determine the data needs for these analyses. It is advisable to select a radiochemical laboratory as early in the monitoring and sampling process as is practical. When available, mobile laboratories can provide on-site analytical capability. Obtaining laboratory or other services may involve a specific procurement process.

4.2.2 Procedure

This procedure covers steps that may be needed to identify and select laboratory resources that are currently available. Selection of laboratories will need to take into account the status of any existing agreements to perform work for FRMAC. The first steps of the procedure below may be performed before there is detailed information or a statement of work available, in order to gather current laboratory status and capacity information early in an emergency event:

Utilize the information in the Laboratory Database as discussed in Section 4.1 of this manual to identify laboratories that meet potential analysis needs taking into account the various evaluation items noted in Section 4.1 and taking into account the location of the laboratories relative to the emergency event.

Contact the laboratories identified above and verify the Laboratory Database information, gather information about their current capacity and workload and enter this information or reference it in the Laboratory Database for use during the emergency.

4.3 Sample Control Line Setup and Operation

4.3.1 Purpose

This method describes the setup and operation of the FRMAC sample receiving line.

Procedure: Set up a Sample Receiving Area. Using Figure 14 as an example, design a hotline setup and create a similar drawing for the specific event and site at which you will be working. Post a copy of the diagram in the work area and update it as needed.

Also:

- Cover work tables with plastic sheeting.
- Use yellow rope/tape to clearly mark areas.
- Make directional signs from durable materials such as plastic or cardboard.
- Monitor entire work area periodically for contamination.
- If possible, provide seating (with a protective coating) for waiting field monitors.

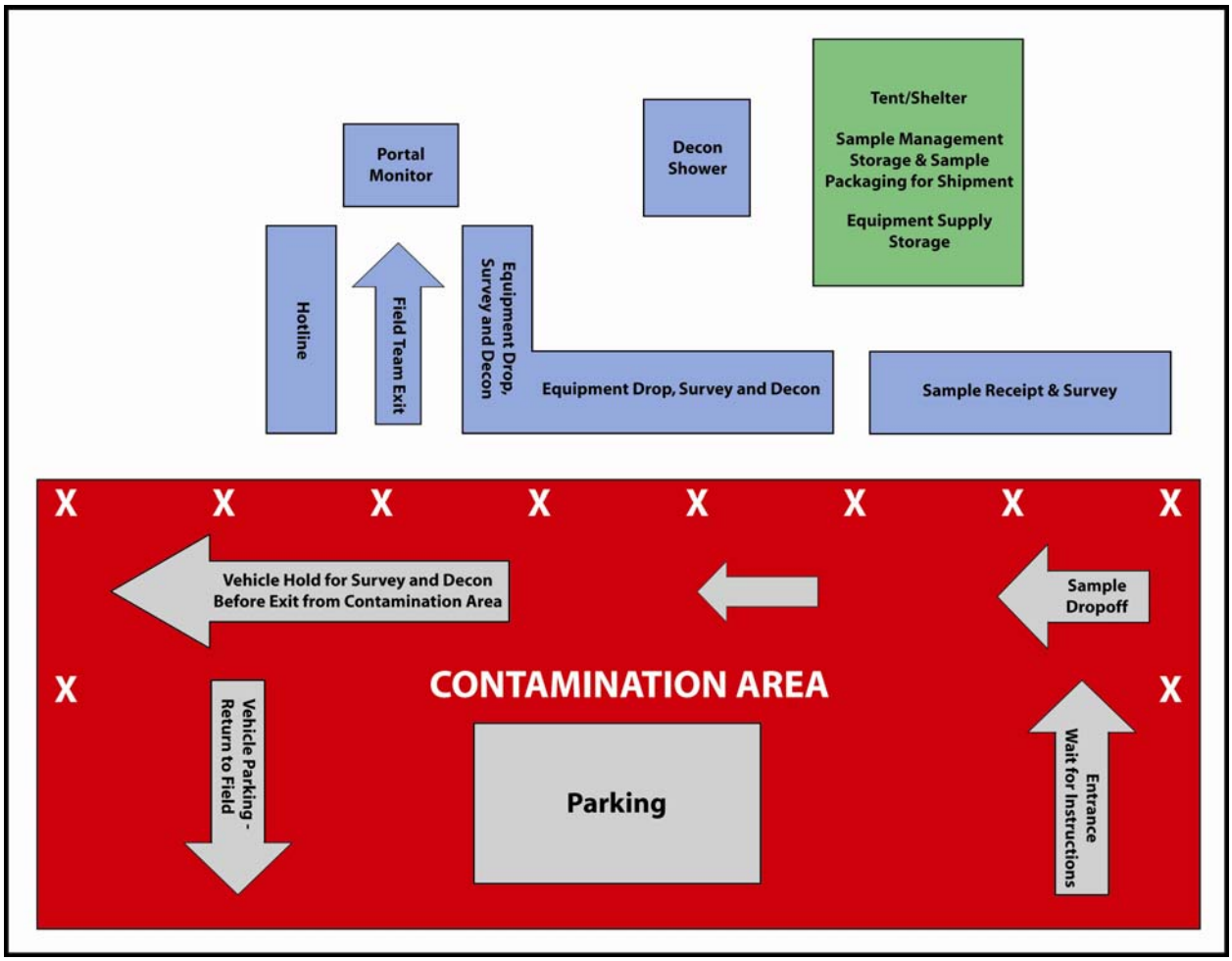


FIGURE 14. ***STYLIZED HOTLINE SETUP***

4.4 Sample Receipt and Handling

4.4.1 Purpose

This method describes the monitoring and receiving of samples from the field. All samples associated with the FRMAC operation will be received through the sample receiving line. Samples are surveyed and received from field monitoring personnel, packaged, and surveyed. Priorities and paperwork are checked and samples placed in a holding area, where they are segregated by activity, priority, media, collection location, or any other parameter.

- Sample container is placed into a clean outer plastic bag if determined to be contaminated by the wipe analysis.
- Survey reading is taken and recorded on the sample container or outer plastic bag.
- Sample Control Form (Figure 15) is surveyed and placed in a 9x12-inch sealable plastic bag if contaminated.
- Any additional labels indicating priorities for further sample processing and analysis are affixed to the outer bag.
- Security seal on original sample container is checked to be sure it is intact.
- All paperwork is properly checked before the field monitoring team is released. Any unresolved issues must be documented on a Sample Receipt Non-conformance Memo (Figure 16).
- Personnel at the Sample Control Area are notified that the sample is ready for transfer and login. Samples may be placed in a holding area by sample control.

SAMPLE CONTROL FORM & CHAIN OF CUSTODY | **SCF -**

Sampling Information (*to be filled out by the Field Team*)

Collection Team ID: | Collector's Name: | Org:

Location: ☐ GPS | Latitude: _____ | Description: _____

Longitude: _____

Collection Date: | Collection Time (Military): | # of Containers | Contact Dose Rate:

Remarks: _____

Sample Type (use only one)

Air	Sampler ID # / Type: / Filter size & Type:	
	Date ON: / Time ON: / Date OFF: / Time OFF:	
	Start Flow: / Stop Flow:	**OR** Total Volume: / Unit:
Milk	☐ Cow ☐ Goat ☐ Other _____	☐ Stored Feed ☐ Pasture ☐ Other _____
	Milking Date: / Milking Time: / Number of Animals sampled:	
Ground	Depth of soil sample: cm	Vegetation collected with soil samples? ☐ **Yes** ☐ **No**
	Sample surface area:	If vegetation in separate container, provide sample #:
Water	☐ Surface ☐ Ground / Well ☐ Potable / Tap ☐ Other:	
Other	☐ Vegetation ☐ Feed ☐ Produce ☐ Swipe ☐ Other:	
	Describe:	

Sample Receiving (*to be filled out by sample receiving technician*)

Processing Priority: | Dup Sample #: | Split Sample #:

Screening Value: | ☐ **Contamination Check:** Forms and sample bags surveyed.

Sample Remarks:

Analysis Requested: | ☐ **Sample Preparation Required**, send to sample preparation area before laboratory

Laboratory Assignment:

Special Instructions:

Custody Transfer (Signatures)

Relinquished By:	Date	Time	Received By:	Date	Time
Relinquished By:	Date	Time	Received By:	Date	Time
Relinquished By:	Date	Time	Received By:	Date	Time
Relinquished By:	Date	Time	Received By:	Date	Time

Original with Sample **Copy to Data Center** **Copy to Courier** **September 2002**

FIGURE 15. ***SAMPLE CONTROL FORM AND CHAIN OF CUSTODY***

Sample Receipt Non-Conformance Memo (NCM)

Sample Number: _____

Sample Processor: _____ Date/Time: _____

Check applicable boxes.

	Chain of Custody sheet missing/not signed			Bottles received broken or leaking
	Sample ID's do not match the chain-of custody			Insufficient sample received
	Label missing from container			Improper/unusual containers received
	Samples received past the EPA recommended holding time			Bottles not preserved correctly
	Separate layers in sample			Duplicate samples visibly different

Problems/Observations: _____

_____

_____

_____

Internal Resolution/Correction Action: _____

_____

_____

_____

Monitoring & Sampling Supervisor Contacted: _____

External Resolution/Corrective Action: _____

_____

_____

_____

FIGURE 16. ***Sample Receipt Non-Conformance Memo***

4.5 Sample Tracking, Protection and Storage

4.5.1 Purpose

Sample tracking refers to the identification of samples, their location, and the individuals responsible for their custody and transfer of the custody. This covers the entire process from collection of the samples and remains intact through the analysis and final holding or disposal. It begins with the collection of a sample where its identification and designation of the sample are critical to being able to relate the analytical result to a site location. Tracking samples from collection to receipt at the analytical laboratory is done through a Chain-of-Custody (COC) process, and documented on a COC record. Samples are received by the laboratory and tracked by their internal tracking (e.g., COC) procedures.

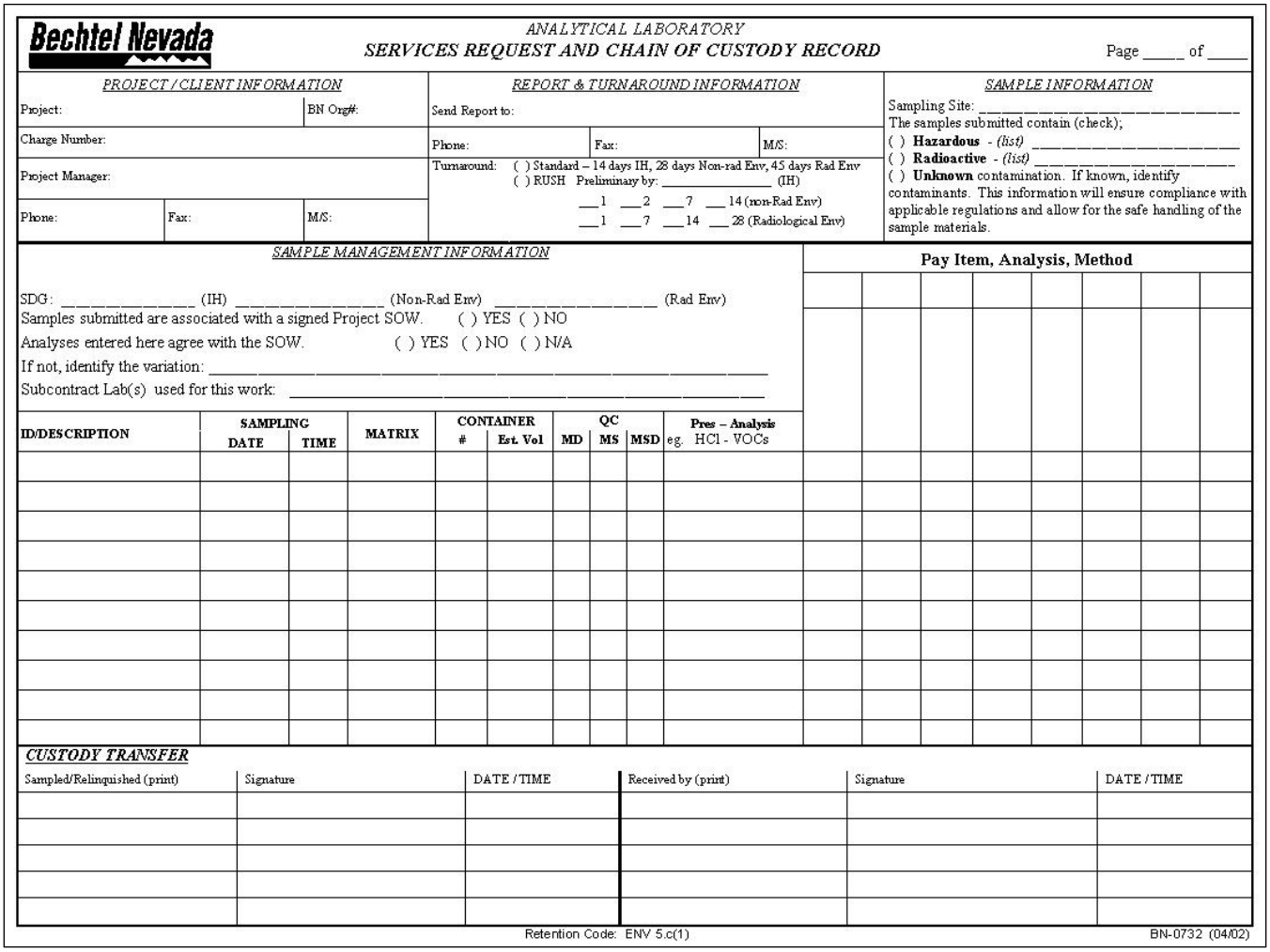
Documentation of the transfer of custody of a sample(s) is important. There must be sufficient evidence to demonstrate that the integrity of the sample is not compromised from the time it is collected to the time it is analyzed. During this time, the sample should either be under the positive control of a responsible individual or secured and protected from any activity that could change the true value of the results or the nature of the sample. Ensuring that a clear transfer of the custodial responsibility is well documented and no questions exist as to who is responsible for the sample at any time is critical.

4.5.2 Procedure

All samples leaving the site should be accompanied by a COC record. Bechtel Nevada Form BN-0732 (Figure 17) may be used initially. This documents sample custody transfer from the sampler, often through another person, to the laboratory. The individuals relinquishing the samples should sign and date the record. The record should include a list, including sample designation, number of samples in the shipping container, and the analysis requested for each sample.

Shipping containers should be sealed and include a tamper indicating seal that will indicate if the container seal has been disturbed. The method of shipment, courier name, or other pertinent information should be listed in the COC record.

The original COC record should accompany the samples. A copy of the record should be retained by the individual or organization relinquishing the samples. Discuss the custody objectives with the shipper to ensure that the objectives are met. For example, if the samples are sent by mail and the originator of the sample requires a record that the shipment was delivered, the package should be registered with return receipt requested. If, on the other hand, the objective is to simply provide a written record of the shipment, a certificate of mailing may be a less expensive and appropriate alternative.



Bechtel Nevada

ANALYTICAL LABORATORY
SERVICES REQUEST AND CHAIN OF CUSTODY RECORD

Page _____ of _____

PROJECT / CLIENT INFORMATION

Project: | BN Org#:
Charge Number:
Project Manager:
Phone: | Fax: | M/S:

REPORT & TURNAROUND INFORMATION

Send Report to:
Phone: | Fax: | M/S:
Turnaround: ( ) Standard – 14 days IH, 28 days Non-rad Env, 45 days Rad Env
( ) RUSH Preliminary by: _____ (IH)
___1 ___2 ___7 ___14 (non-Rad Env)
___1 ___7 ___14 ___28 (Radiological Env)

SAMPLE INFORMATION

Sampling Site: _____
The samples submitted contain (check);
( ) **Hazardous** - *(list)* _____
( ) **Radioactive** - *(list)* _____
( ) **Unknown** contamination. If known, identify contaminants. This information will ensure compliance with applicable regulations and allow for the safe handling of the sample materials.

SAMPLE MANAGEMENT INFORMATION

SDG: _____ (IH) _____ (Non-Rad Env) _____ (Rad Env)
Samples submitted are associated with a signed Project SOW. ( ) YES ( ) NO
Analyses entered here agree with the SOW. ( ) YES ( ) NO ( ) N/A
If not, identify the variation: _____
Subcontract Lab(s) used for this work: _____

Pay Item, Analysis, Method

ID/DESCRIPTION	SAMPLING DATE	SAMPLING TIME	MATRIX	CONTAINER #	CONTAINER Est. Vol	QC MD	QC MS	QC MSD	Pres – Analysis eg. HCl - VOCs

CUSTODY TRANSFER

Sampled/Relinquished (print)	Signature	DATE / TIME	Received by (print)	Signature	DATE / TIME

Retention Code: ENV 5.c(1)

BN-0732 (04/02)

FIGURE 17. *BN-0732 SERVICES REQUEST AND CHAIN-OF-CUSTODY RECORD FORM*

The individual receiving the samples should sign and date the record. The condition of the container and the tamper indicating seal should be noted on the Services Request and COC record. Any problems with the individual samples, such as a broken container, should be noted on the Sample Receipt Non-Conformance Memo (Figure 16).

A sample is defined as being in custody if any of the following conditions are met:

- It is within someone's possession.
- It is within someone's view, after being in someone's possession.
- It was in someone's possession and then was secured to prevent tampering.
- It is placed in a designated secure area.

4.6 Sample Transportation

4.6.1 Purpose

All samples being shipped for radiochemical analysis should be properly packaged and labeled before transport. The primary concern is the possibility of spills, leaks, or breakage of the sample containers. In addition to resulting in the loss of samples and cross-contamination, the possible release of hazardous material poses a threat to the safety of persons handling and transporting the package.

4.6.2 Scope

This procedure applies to movement of all potentially radioactive samples. All modes of transportation are covered.

4.6.3 References

- U.S. Nuclear Regulatory Commission (NRC) Regulations: NRC regulations for packaging, preparation, and shipment of licensed material are contained in 10 CFR Part 71: "Packaging and Transportation of Radioactive materials". Samples containing low levels of radioactivity are exempted as set forth in §§ 71.10. A licensee is exempt from all requirements of Part 71 if the specific activity of the sample being shipped is not greater than 74,000 Bq/kg (2,000 pCi/g). Low Specific Activity Material (LSAM) is defined in §§ 71.4: "Definitions." Samples classified as LSAM need only meet the requirements of the U.S. Department of Transportation (DOT), discussed below, and the requirements of §§ 71.88: "Air transport of plutonium." Most environmental samples will fall into this category.
- U.S. Department of Transportation Regulations: The U.S. Department of Transportation provides regulations governing the transport of hazardous materials under the Hazardous Materials Transportation Act of 1975 (88 Stat. 2156, Public Law 93-633). Applicable requirements of the regulations are found in 49 CFR Parts 170 through 189. Shippers of samples containing radioactivity should be aware of the current rules in the following areas.
- Accident Reporting - 49 CFR 17
- Marking and Labeling Packages for Shipment - 49 CFR 172
- Packaging - 49 CFR 173
- Placarding a Package - 49 CFR 172
- Registration of Shipper/Carrier - 49 CFR 107
- Shipper Required Training - 49 CFR 172
- Shipping Papers and Emergency Information - 49 CFR 172
- Transport by Air - 49 CFR 175
- Transport by Rail - 49 CFR 174
- Transport by Vessel - 49 CFR 176
- Transport on Public Highway - 49 CFR 177

4.6.4 Procedure

- Review NRC requirements (10 CFR part 71) and Department of Transportation (DOT) requirements (49 CFR parts 170 through 189) for packaging and shipping radioactive environmental samples.
- Visually inspect each sample container for indication of leaks or defects in the sample container.
- Liquid samples should be shipped in plastic containers, if possible, and the caps on the containers should be secured with tape. One exception to the use of plastic bottles is samples collected for 3H analyses which may require glass containers.
- Heavy plastic bags, with sealable tops, can be used to contain solid samples (e.g., soil, sediment, air filters). The zip-lock should be secured with tape. Heavy plastic lawn bags can be used to contain vegetation samples. The tops should be closed with a "tie" that is covered by tape to prevent it from loosening and slipping off.
- If glass sample containers are used, place sample containers inside individual plastic bags and seal in order to contain the sample in case of breakage
- Use packing material (e.g., paper, Styrofoam, "bubble wrap") to immobilize and isolate each sample container and buffer hard knocks on the outer container during shipping. This is especially important in cold weather when plastic containers may become brittle and water samples may freeze.
- When liquid samples are shipped, include a sufficient quantity of an absorbent material (e.g., vermiculite) to absorb all liquid packed in the shipping container in case of breakage. This absorbent material may suffice as the packing material described above.
- Include the original, signed and dated, Services Request and Chain of Custody Form (BN-0732) (Figure 17), identifying each sample in the package. It is good practice to place this COC form in a plastic bag to prevent it from becoming wet or contaminated in case of a spill during shipment. Multiple packages of samples may be covered by a single Services Request and Chain of Custody Form that reflects each package.
- Seal closed the package and apply COC tape in such a manner that it must be torn (broken) in order to open the package. The tape should carry the signature of the sender, and the date and time, so that it cannot be removed and replaced undetected.
- Ice chests, constructed of metal or hard plastic, make excellent shipping containers for radioactive environmental samples.
- If samples are sent offsite for analysis, the shipper is responsible for complying with all applicable federal, state, and local regulations. Applicable federal regulations are briefly described in the References section. Any state or local regulation will very likely reflect a federal regulation.

4.7 Sample Retention and Disposal

4.7.1 Purpose

This section describes the retention, management, and eventual disposal of analytical samples generated during an emergency response event. Samples may be retained both by radioanalytical laboratories (for potential reanalysis) and the FRMAC / Coordinating Agency response (for both reanalysis, and evidentiary purposes). All sample aliquots, including those returned from laboratories, must be secured and appropriately maintained until disposal is authorized by the Laboratory Analysis Manager, Operations Manager, and the FRMAC Executive Team which may include the Coordinating Agency, the FRMAC Director, the EPA Senior Official, and state and local representatives.

4.7.2 Scope

The scope of this requirement is the management of unused portions of potentially hazardous and radioactive analytical samples taken during an emergency response event, both at the locations of off-site and contract laboratories, and at the site of the event response itself. It is assumed that the majority of samples will be maintained briefly at the laboratory, then returned to the CA response organization. This document does not address the disposal of process waste generated during the analytical process, but adequate facilities and procedures for managing such waste shall be assured before samples are sent to any analytical facility.

4.7.3 References

Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 20, NUREG-1576, EPA 402-B-01-003, NTIS PB2001-106745. DRAFT July 2001.

4.7.4 Responsibilities

The Analysis Manager has primary responsibility for sample retention and disposal decisions. The task of sample archiving may be delegated to the sample control personnel. In a large-scale response, the Laboratory Liaison will communicate sample retention and disposal requirements to off-site and contract laboratories.

4.7.5 Procedure

- Off-site and contract laboratories shall retain all unused portions of analytical samples for at least 60 days, but no more than 1 year, after all analyses are complete. Unused samples shall be stored in a manner which allows rapid retrieval of any sample container, and all storage areas shall have sufficient access controls to ensure that the analytical facility has physical control and custody of the samples at all times.
- All laboratories performing analytical work for FRMAC shall either have approved waste handling procedures that comply with all applicable regulatory requirements, or shall be

capable of returning all unused samples to the Coordinating Agency exercising authority over any response.

- Any sample disposal shall comply with all applicable regulations and requirements. The sample COC form shall indicate the disposition of the sample (either disposal or return).
- Samples being returned shall be shipped in accordance with the requirements of section 4.6 (Sample Transportation). A COC form shall accompany the samples.
- Returned samples shall be retained by the Analysis Manager until written authorization for disposal is provided by the Coordinating Agency exercising authority over the response. Samples authorized for disposal by the Coordinating Agency will be disposed of in accordance with all applicable regulatory requirements. Unused samples shall be stored in a manner which allows rapid retrieval of any sample container, and all storage areas shall have sufficient access controls to ensure that the Coordinating Agency has physical control and custody of the samples at all times. Ultimate responsibility for final sample disposition is held by the Coordinating Agency.
- The sample's COC shall reflect the disposition of the sample.

4.7.6 Records

- The sample's COC form shall serve as the record of sample disposal.
- Copies shall be kept of all communications from off-site and contract laboratories assuming responsibility for sample disposal.
- Copies shall be kept of all communications from the Coordinating Agency authorizing sample disposal.

4.8 Quality Assurance and Performance Testing Samples

4.8.1 Purpose

Introduce performance testing samples with samples sent to analytical laboratories to monitor the performance of that laboratory. The performance of the analytical method should be assessed independently on a regular basis. This assessment is achieved through the use of blind samples that provide an objective means of evaluating the laboratory's performance for specific analytes and matrices. FRMAC Analysis Section will submit external single blind samples. External blind proficiency testing (PT) samples are used for QA purposes and also can provide information that is useful to determine a laboratory's quality processes.

4.8.2 Scope

Performance samples matching the matrix of normal samples are included with batches of similar samples sent to a radioanalytical laboratory. These samples are independently verified as to their radioactive content/concentration. QA samples may include various types of blank samples, standard reference material, spiked samples and duplicate samples as needed to estimate the variance of the sampling, sample processing and analysis of samples.

4.8.3 References

- National Environmental Laboratory Accreditation Program, Chapter 5, Quality Systems, Revision 15, National Environmental Laboratory Accreditation Council, May 2001.
- Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 18, NUREG-1576, EPA 402-B-01-003, NTIS PB2001-106745. DRAFT July 2001.

4.8.4 Responsibilities

- Laboratory Analysis Manager determines the frequency and matrix of PT samples. Responsible for documentation of PT sample certification. Purchase and plan for shipment of PT material with CMRT II deployment. Review PT sample results and acceptability calculations. Coordinates with the Monitoring Manager for the collection of QA/QC samples.
- Sample Control Technician creates chain of custody for PT material and includes PT samples with shipments to the laboratory designated by the Laboratory Analysis Manager.
- Quality Assurance Technician coordinates collection of QA samples with Laboratory Analysis Manager. Compiles and reports results from quality assurance samples and brings unusual results to the attention of the Laboratory Analysis Manager. Investigates causes of unusual quality assurance results.

4.8.5 Procedure

Analysis of quality assurance and performance testing samples is useful for estimating the variability and precision of analytical results. However, variability and contamination may be more influenced by sampling and sample processing techniques, so any quality assurance program needs to consider these parts of the overall process. Early in an emergency situation PT samples may not be readily available for introduction with routine samples. In this situation it is especially important to find out what calibration and QA samples have been recently analyzed by the laboratories that may be analyzing samples.

The procedure below uses a multi-faceted approach to cover a range of possible situations. The Laboratory Analysis Manager and Monitoring Manager, or their designees, are responsible for determining how much emphasis is placed on each facet of the approach.

In coordination with the Laboratory Analysis Manager and the Monitoring Manager, establish at least two sampling locations that are thought to be at background level for each radionuclide and sample media of concern. This may initially be based on plume models or air dispersion calculations. The purpose of these background locations is to have convenient sampling locations to which field teams may be sent to collect background samples. Once more information is available, establish additional reference sampling locations with elevated levels of contamination.

Whenever possible, prior to collecting and submitting any samples for an emergency event, each field team will be assigned to collect at least one sample at the background or reference locations and to complete the paperwork and submit these samples to the sample receiving line before

collection of any response samples. If there are problems with the sample collection procedures or the paperwork completion, the field team will be referred to the Monitoring Manager for additional training or resolution of problems.

Reference or background samples will be submitted as samples to laboratories, but the analysis priority will depend on the current workload and backlog of samples. Reference and background sampling locations will be assigned to each field team periodically.

Results from these background and reference samples will be used to estimate the overall variability of the sampling, sample processing and analysis. They will also be used to check for cross-contamination of samples.

Gathering initial laboratory information

- Contact each laboratory that is likely to analyze samples and find out the date of the last calibration and what PT samples have been analyzed recently for the types of samples planned for that laboratory.
- Request copies of laboratory results for recent QA samples and review the results. Note any potential problems and bring them to the attention of the Laboratory Manager as needed.
- Verify the sample geometries used for the types of samples to be sent to the laboratory and request current versions of their procedures for sample processing and analysis.

Background and reference samples

Blank samples are used to determine whether any radionuclide contamination is introduced by the sampling or measurement process. They assist in the control of any contamination introduced by the laboratory. Ideally, no target analytes should be present in the blank at detectable concentrations. If that is not possible (e.g., for naturally occurring radionuclides), those radionuclides should be well-characterized and tracked. Control charts can be used to track these radionuclide levels in blanks.

PT or Spiked Samples

- A laboratory control sample (LCS) is a QC sample of known composition (reference material) or an artificial sample, created by fortifying a clean material similar in nature to the environmental sample. The LCS is prepared and packaged in the same manner as the environmental samples going to the laboratory.
- Determine what PT samples, spiked samples or liquid standards are available.
- Determine how these materials may be used most effectively as quality assurance tools.
- Assign distribution of existing PT samples and spiked samples to the sample control technician.
- Order additional PT materials, spiked samples or liquid standards to meet the projected needs.

Duplicate or Split Samples

- Duplicate samples are two sample quantities collected at the same location and are packaged separately in the field. COC documents need to reflect the common sample number with an increased quantity collected or multiple containers.

- Duplicate or split samples capture the variability and reproducibility of the sampling process as well as the laboratory's reproducibility. The purpose for measuring precision is to determine whether the sampling teams and the analytical laboratory can execute a method consistently and obtain results of acceptable variability. Samples can cover a range of physical forms or matrices, from homogeneous samples like finished drinking water to complex soils or biological material, and each matrix has the potential to affect a sample's precision. Precision is a measure of agreement among replicate measurements of the same property under prescribed similar conditions. Precision is a fundamental aspect of the analytical process and should be evaluated routinely as part of FRMAC's quality system.

The QA/QC technician must assign separate sample numbers before the samples are submitted to an analytical asset. Duplicate samples may be sent to the same laboratory as a quality check on laboratory reproducibility or sent to a different laboratory as a quality check on each laboratory's intercomparability.

Acceptance Criteria

If differences between observed and known values typically arise outside of acceptable tolerances or control limits, these should be investigated thoroughly, as they indicate areas where important details of the analytical process may have been overlooked. Often a laboratory's observed values agree with the known value within acceptable tolerances, but are biased high or low. Careful documentation of the laboratory's performance in this regard can assist in characterizing the fluctuations of a measurement system or analytical method. Like other performance indicators, large or sudden changes in bias require scrutiny. Care must be used when evaluating results associated with complex and variable matrices. In general, aqueous samples tend to be less affected than other media like soils or heterogeneous materials. However, multi-phase fluids, high solid content, and brackish or saline waters may be more problematic.

Evaluation is typically performed using prepared samples consisting of media equivalent to a routine analytical sample with a known, measurable amount of the analyte of interest. Upon completion of the analysis, the results are compared to the known or accepted value, and the agreement is evaluated using a predetermined criterion. The range of sample types assayed in a laboratory may require that spikes are prepared using several sample media.

The numerical performance indicator for a blank sample used to monitor for unexpected contamination is:

where x denotes the measured blank activity and u_c (x) denotes the combined standard uncertainty of the blank result.

Recommended warning limits for Z Blank are +/-2 (standard deviations) and control limits are

$$Z_{Blank} = \frac{x}{u_c(x)}$$

+/-3 (standard deviations) which produce confidence levels of 95% and 99% respectively.

Duplicate sample evaluation typically is performed using multiple analysis of the same sample (blanks, spikes, blinds, reference materials, performance evaluation samples, etc.), and evaluating the analyses relative to a statistically based criterion. The reproducibility of analytical results should be evaluated by replicates to establish this uncertainty component.

All analytical batches should be evaluated with respect to precision, whether by using replicates or matrix spike duplicates. This is done typically by the use of an acceptance criterion that derives a statistic that quantifies the difference between two values obtained by analyzing the same sample. Limits are then placed on the criterion, and data for any batch in excess of the criterion require investigation and corrective action as appropriate. The numerical performance indicator for duplicates is:

$$Z_{Duplicate} = \frac{x_1 - x_2}{\sqrt{u_c^{\,2}(x_1) + u_c^{\,2}(x_2)}}$$

where x_1 and x_2 denote the two measured activity concentrations and $u_c^2(x_1)$ and $u_c^2(x_2)$ denote their respective combined standard uncertainties. Recommended warning limits for $Z_{Duplicate}$ are +/- 2 (standard deviations) and control limits are +/- 3 (standard deviations).

Performance testing samples are used to evaluate the ability of a laboratory produce quality data. PT samples are to be introduced into the analytical stream as a blind. Comparison of analytical results of well characterized reference material (WCRM) to their certified values provides linkage to the national scale of measurements and a measure of method accuracy. Such materials may be used in the evaluation of competing analytical methods, and also in the cross-comparison of inter-laboratory data – both at the national level and the international level. The numerical performance indicator for performance testing samples is:

$$Z_{CRM} = \frac{x - d}{\sqrt{u_c^{\,2}(x) + u_c^{\,2}(d)}}$$

where x is the measured value, d is the certified value, and $u_c^2(x)$ and $u_c^2(d)$ are the squares of the respective combined standard uncertainties. Warning limits for Z_{CRM} are +/- 2 (standard deviations) and the control limits are +/- 3 (standard deviations).

4.8.6 Records

Known values for PT samples will be retained for comparison to laboratory analysis results.

4.9 Laboratory Data Deliverables

4.9.1 Purpose

Specify the information that organizations providing radioanalytical support to a response need to provide. This information can range from simply the analytical result up to and including

documentation of every aspect of the analytical process. Electronic Data Deliverables are also specified.

4.9.2 Procedure

Deliverables are dependent on the phase of the response and the DQO/MQO needed for the data. If there are expectations of deliverables from the laboratory beyond those previously arranged or if no previous deliverable specifications have been established, be sure to specify deliverable requirements on the analysis request form or the chain of custody form.

4.10 Laboratory Data Verification and Validation

4.10.1 Purpose

The purpose is to provide a systematic approach to evaluate the analytical results along with the data deliverables that support the results.

4.10.2 Scope

This procedure covers review of data sets and laboratory reports. Verification and validation can be a long process. It may be that this work would not be done until some time after the data is delivered especially in the case of emergency samples.

4.10.3 Procedure

- This is the initial review of data sets and laboratory reports covering groups of samples. Skip any steps that have already been covered and documented previously for the sample group being reviewed and note those steps below as “done previously” and/or specify where the previous review information may be found.
- List the information that identifies the data set (sampling locations, type (s) of samples, date range of sample collection, site, laboratory, etc.) List the types of data that are included in the data set (e.g., field forms, all laboratory data related to samples, only final laboratory reports, etc.).
- Determine and specify whether or not the samples covered in the data set have been entered into the applicable database or sample tracking system.
- List any procedures or documents used or needed for review of the data set. Are the sampling and analysis procedures used to generate the data specified and available? If not covered in previous reviews, list any terms that are not defined that appear in the data set or laboratory report. Try to find a definition of these terms in the associated procedures or documents or by calling the laboratory that produced the report.
- Specify and request any missing documents or definitions needed for review of the data set.
- List or describe any calibration or QA/QC data that is present in the data set.

- Describe any obvious data trends related to sampling variables such as date of collection or sample location.
- Specify whether the data set or other available information indicates that additional sample or sample aliquots remain to be analyzed or are archived for later analysis.
- Review the QA/QC data and note any apparent inconsistencies or problems with this data. Specify whether the quality assurance and quality control data support or appear to be consistent with the associated routine data.
- Briefly specify or suggest any analysis of the data set that may be useful (e.g., plots, statistical analysis, etc.)
- Determine whether or not the applicable MQO has been met.

4.11 Recordkeeping

- Procedure: Any radiological emergency that requires a FRMAC response has substantial probability of leading to criminal and/or civil litigation. As such, the maintenance of associated records will be crucial. The FRMAC response shall be responsible for records retention until control of the event is assumed by the Coordinating Agency. At this time, the Coordinating Agency will assume responsibility for the records.
- Retention: All hard-copy records that relate to a FRMAC response shall be maintained for a period of at least 75 years.
- Storage: Records shall be stored in a manner that allows reasonably rapid retrieval, is protective of damage due to environmental conditions, and maintains positive control and custody over all information.
- Disposition/Disposal: The Coordinating Agency in charge of an event response shall determine if event records may be disposed of after 75 years.
- Electronic: Data that exists only in electronic form shall be saved in at least two locations, and shall be saved to non-volatile storage every month, at a minimum.

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APPENDIX A
MODEL SCOPE OF WORK

The following section is a model scope of work for contractual purposes. All of the provisions of the document are negotiable but should be used to insure that FRMAC and contract laboratory personnel fully understand the performance expectations of the other party.

General Information

This document defines the required Data Quality Objectives (DQO) for radiological analysis of samples collected by the DOE/NNSA Consequence Management (CM) program during a response for emergencies involving radioactive materials. This document applies to both mobile and fixed laboratories.

The laboratory's POC for any administrative or technical issues is the FRMAC Laboratory Analysis Manager.

Three processing categories required for samples are: emergency, rush, normal.

- The emergency samples are taken during the early phase of an emergency response and are for screening purposes. These samples may contain high radiation levels with unknown isotopes. The required turn around time for emergency samples are on the order of hours, detection limits are higher, and analytical methods are simple and rapid. The laboratory is required to be on high level of readiness and able to deploy on a short notice. It is expected that most laboratories providing emergency sample analysis support will be mobile laboratories or be fixed laboratories that are geographically close to the location of the incident and to be capable of operating 24 hours a day.
- The rush samples are taken four to thirty days after the incident (intermediate phase) and are for forensic purposes that require a higher level of data quality. The required turn around times are on the order of a few days, detection limits are lower than the emergency samples, and the analytical methods may be more complicated, including simple digestions. It is expected that most laboratories providing rush sample analysis support will be fixed laboratories, preferably close to the location of the incident.
- The normal samples are taken on the order of a few weeks to a year after the incident and are for site characterization and remediation. These samples require the highest level of data quality and legal defensibility. The required turn-around times are on the order of a few weeks, detection limits are very low, and the analytical methods are more complicated, including full radiochemical procedures. It is expected that all laboratories providing normal sample analysis support will be fixed laboratories.

It is expected that all support for emergency samples and some support for the rush samples will be provided by DOE-funded laboratories with a high level of readiness. In addition to DOE-funded laboratories, other federal laboratories, academic institutions, and the commercial sector will be asked to provide support for the rush and normal samples on a cost per sample basis as established by contracts. Note: Each stated requirement shall apply to all three sample categories, unless exempted.

The majority of the samples are air filters of various sizes and environmental samples (soil, vegetation, and water). The samples may also include bioassay specimen (urine, feces, tissue, or blood) and other ad hoc samples such as pieces of machinery.

Each sample is individually tamper-sealed and is assigned a unique sample number.

The type of processing (emergency, rush, normal) required for each sample is specified on the Chain of Custody (COC) form provided to the laboratory with each sample shipment.

Prior to submitting samples for analysis, FRMAC may perform a QA audit of the laboratory. In addition, the laboratory could be subject to periodic quality assurance audits. However, if requested by FRMAC, the laboratory shall allow the FRMAC representatives access for purposes of performing quality assurance audits at any time upon thirty (30) days notice by FRMAC to the laboratory.

FRMAC may submit blind or other audit samples to determine compliance with the minimum detectable activity (MDA), accuracy, and precision requirements.

Analysis results or any other written communication of information relating to the processing of samples should be sent to the FRMAC Analysis Manager at the field location.

Disposal of reagent solutions and process waste shall conform to the applicable federal, state, and local regulations. Unused sample portions and residues will not be disposed of without previous written authorization of the submitting organization.

Technical Criteria

Sample Receiving, Storage, and Handling

- Sample shipments originate from FRMAC in the field. The samples may be cooled pending transport and are shipped to the laboratory.
- Each shipment is accompanied by a COC form (Figure 18) listing designated POC, the samples in the shipment, and specifying the radionuclide(s), and desired analysis for each sample in the shipment. To expedite the paper work, the form will serve both as the COC and sample analysis request form, and will take precedence over any laboratory forms.
- The laboratory shall have procedures for sample receiving, radiation screening, processing, storage, and login.
- The laboratory shall inspect each sample upon receipt and indicate on the COC form the condition of the sample upon arrival and whether the tamper seal(s) were intact.
- The COC form shall then be signed on behalf of the laboratory.
- The laboratory shall record its login number(s) on the COC form and fax a copy to the POC. The cooler(s) in which the samples were shipped shall be returned separately. If the coolers are contaminated in transport, the receiving laboratory shall dispose of the coolers in accordance with applicable state and federal regulations.
- The laboratory shall have a designated area for storing samples. The laboratory shall maintain a system to store samples and remaining sample fractions such that a sample can be retrieved from storage in a timely manner.

- The laboratory shall, throughout all steps of the handling, storage, and analysis process, use containers, handling procedures, etc. to prevent loss, degradation, or contamination of samples.
- The laboratory shall store all remaining portions of an analyzed sample for at least six months after the final results are reported to the FRMAC. The laboratory may be required to retain samples after this period. No samples are to be disposed of unless directed by FRMAC Executive Team which may include the Coordinating Agency, EPA Senior Official, and state.
- The laboratory shall have a documented process for positive sample control and custody during the various processing steps (i.e., the samples will either be under direct control of a laboratory employee or kept in a secure location).

Sample Preparation and Chemistry

- The laboratory shall have detailed procedures for preparing samples, including any required radiochemistry, for all the requested analyses.
- The laboratory shall document the calibration and maintenance of various items such as fume hoods, pipettes, balances, and other equipment used during sample processing.
- Each batch of samples that are processed together shall include a blank (reagent or matrix, as appropriate) and a Lab Control Sample (LCS) (applicable to rush and normal samples only).
- The laboratory shall analyze a blank sample with each batch to determine the existence and magnitude of contamination problems, if any, as determined by best laboratory practices (applicable to rush and normal samples only).
- At least one of every 20 samples (5% of samples) shall be a blank. De-ionized water may be used for blank where an appropriate blank matrix is not available (e.g., soil).
- The laboratory shall analyze an LCS with each batch to monitor the overall performance of all steps in the analysis, including the sample preparation. The laboratory shall use an LCS with a matrix as similar as possible to that of the samples (applicable to rush and normal samples only).
- The LCS shall be spiked at a level to provide a counting uncertainty of less than 10% at the 2-sigma confidence level (applicable to rush and normal samples only).
- At least one of every 20 samples (5% of samples) shall be an LCS (applicable to rush and normal samples only).
- Chemical recovery of individual samples subject to chemical process and separation shall be established by means of spiking with tracer quantities of other radioisotopes of the same element or carrier quantities of the inactive isotope of the same or a chemically similar element (applicable to rush and normal samples only).
- All samples that require the addition of a tracer shall be tracer-spiked prior to sample preparation unless this is impossible for a technically feasible reason (applicable to rush and normal samples only).
- The tracer levels, or count times, shall be sufficient to provide a counting uncertainty of less than 10% at the 2-sigma confidence level (applicable to rush and normal samples only).

Counting Systems

- Requirements for satisfactory instrument calibration shall be in place to ensure that instruments are capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the calibration period, and routine performance checks document that the initial calibration is still valid. The laboratory shall use NIST traceable standards for all calibration. For routine instrument checks, non-NIST standards may be used as long as they are not used for computing sample results.
- The laboratory shall have system specific procedures for calibration, routine instrument checks, system maintenance, sample counting, data analysis, and report generation.
- Laboratory shall document instrument performance indicators on control charts that will be available to FRMAC upon request.
- The laboratory shall establish technically defendable warning and control limits for each control chart.
- When control limits for a system parameter are exceeded, the system shall not be used for counting samples until the problem is investigated, documented, and resolved.
- The laboratory shall maintain records for each system documenting repairs, software upgrades, and any other miscellaneous actions that affects the system.

Alpha Spectroscopy System (Alpha Spec)

- Energy versus channel calibration shall be established every two years or when the routine performance check indicates an out of statistical control change in energy gain or zero offset.
- System resolution shall be established every two years or when the routine performance check indicates an out of statistical control change in system resolution.
- Efficiency calibration for each counting geometry shall be established every two years or when the routine performance check indicates an out of statistical change in system efficiency.
- Detector backgrounds shall be established at a minimum, monthly, or during deployment for mobile laboratories.
- The system's energy calibration, resolution, and efficiency shall be checked at least once a week, or during deployment for mobile laboratories.

Gamma Spectroscopy System (Gamma Spec)

- Energy versus channel calibration shall be established every two years or when the routine performance check indicates an out of statistical control change in energy gain or zero offset.
- Resolution versus energy calibration shall be established every two years or when the routine performance check indicates an out of statistical control change in system resolution.
- Efficiency calibration (efficiency versus energy) for each counting geometry shall be established every two years or when the routine performance check indicates an out of statistical control change in system efficiency.
- Detector background shall be established at minimum, monthly, or during deployment for mobile laboratories.

- The system's energy calibration, resolution, and efficiency shall be checked with a source that contains low, medium, and high energy peaks each day prior to sample analysis, or during deployment for mobile laboratories.

Gas Proportional Counters

- A plateau curve shall be established every three years, or when the routine performance check indicates an out of statistical control change in system response, to determine the optimum voltages for alpha only and simultaneous alpha and beta counting. Instrument crosstalk will be determined simultaneously with the plateau curve.
- The gross counting systems shall be efficiency calibrated for each alpha and beta counting geometry every three years or when the routine performance check indicates an out of statistical control change in system efficiency.
- Self-absorption curves shall be developed every three years or when the routine performance check indicates an out of statistical control change in system efficiency.
- Detector background shall be established annually or when the routine performance check indicates an out of statistical control change in system background.
- The system's efficiency and background shall be checked each day that the system is used.

Liquid Scintillation Counters

- For Liquid Scintillation Counters with alpha/beta separation capability, and if used for alpha/beta counting, the optimum pulse shape discriminator setting shall be determined every three years or when the routine performance check indicates an out of statistical control change in system performance.
- Efficiency quench curves shall be established every three years for each radionuclide and cocktail type to be counted or when the routine performance check indicates an out of statistical control change in system efficiency.
- Background quench curves shall be established annually for each radionuclide to be counted unless matrix or batch blanks are used for background subtraction.
- The system's efficiency and background shall be checked each day that the system is used, or during deployment for mobile laboratories.

Kinetic Phosphorescence Analysis

- Background shall be established immediately prior to sample analysis.
- The unit shall be calibrated with a minimum of three standards with concentrations spanning the range of interest, immediately prior to analyzing samples.
- The background and calibration standards shall be analyzed as samples after the samples are analyzed to verify system stability.

Inductively Coupled Plasma Mass Spectrometry

- Background for each isotope shall be established immediately prior to sample analysis.
- The unit shall be calibrated for each isotope with a minimum of three standards with concentrations spanning the range of interest immediately prior to analyzing samples. For

multi-isotope analysis, the calibration curve for another isotope that is close in mass (within 25 amu) to the target isotope may be used.

- The background and calibration standards shall be analyzed as samples after the samples are analyzed to verify system stability.

Data Analysis and Review

- The laboratory shall have procedures for analyzing raw data and reviewing data.
- The minimum detectable activity (MDA) shall be within the limits listed in Appendix B. The MDA shall be calculated in accordance with American National Standards Institute Standard 13.30, Performance Criteria for Radiobioassay. 1996.
- Analytical results for rush and normal samples shall not contain a bias less than -25% or greater than +25% at 10 times the required minimum detectable activities. For gross alpha and beta measurements and emergency samples analytical results shall not contain a bias less than -50% or greater than +50% at 10 times the required MDA.
- The accuracy shall be demonstrated by comparison of the results of samples containing known standard reference material with the "true" value for that standard.
- Blank analysis results are assessed to determine the existence and magnitude of contamination problems, if any. The criteria for evaluation, applies to any blank associated with the samples. If problems with any blank exist, all data associated with the case shall be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data As mentioned earlier, if a reagent blank is used to blank-correct sample results, the blank results should be evaluated using control charts. Typically, one method blank and/or reagent blank is analyzed with each batch or grouping of analytical samples regardless of batch size. Situations may occur where more frequent blanks are required to ensure that analytical conditions are stable, particularly when analyzing high and low concentration samples in the same analytical batch, or when instruments, reagents, or analytical method are suspect.
- Chemical tracer recoveries shall be within 20% to 120% (applicable to rush and normal samples only).
- If the tracer recovery for a sample is less than 20%, and if possible, the laboratory shall reprocess the sample or analyze an additional sample aliquot, if available. If another sample aliquot is not available, the laboratory shall immediately notify FRMAC so that they may consider re-sampling or modification of procedures to provide additional sample volume in the future (applicable to rush and normal samples only).
- The laboratory shall meet the MDA for the analysis method and may compensate for low chemical recovery by increasing the count time.
- The LCS serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation. All LCS results shall fall within the control limits of 75-125% recovery of the known value (applicable to rush and normal samples only). The analyst should carefully consider the spiking levels for laboratory control samples and matrix spikes. Spikes and LCSs may be prepared near the lower limits of detection to test the methods performance on clean or slightly contaminated samples. Conversely, matrix spikes and LCSs may be spiked at high levels for groups of highly contaminated samples. The laboratory should try to spike at or near the action level or level of interest for the project.

- The total uncertainty shall include all uncertainties associated with the analysis (combined counting and established systematic uncertainty).
- The combined standard uncertainty shall be reported at the 1.96 sigma level (95% confidence limit).

Gamma Spectroscopy

- The laboratory shall identify and quantify all significant full energy peaks. The analysis gamma library shall at minimum contain Am-241, Cs-137, Co-60, and K-40.
- Each identified radionuclide peak energy shall be within 2 keV, or 1 full width half maximum (FWHM) at the observed energy.
- For radioisotopes that are found, the laboratory shall report an activity, uncertainty, and L_c(Critial Level test) for the isotope. A post-priori MDA, LLD or MDC may be substituted if the critical level test is not available.
- For target radionuclides that are not identified in the peak search and identification, the L_c(Critial Level test for that radionuclide shall be reported. A post-priori MDA, LLD or MDC may be substituted if the critical level test is not available.
- If the library search yields several candidate radionuclides having close matching peaks, all reasonable choices shall be considered. In deciding whether a library search result for a peak represents a realistic identification, professional judgment shall be exercised.
- If there is more than one reasonable match for peak, the result shall be reported as “either radionuclide X or radionuclide Y”.
- The reviewer shall be aware of common laboratory artifacts/contaminants and their sources (Radon and Thoron daughters in the air, etc.).
- For all the significant peaks (net cpm > 2-sigma uncertainty) that are not identified, the laboratory shall report the peak energy and its activity in gammas per second (gps = cps/efficiency).
- The laboratory shall be able to re-evaluate the analytical results of the all samples for one year following submission of the final analytical results to the FRMAC.

Delivery and Reporting of Results

- Results of analyses shall remain confidential and shall not be released to any third party, used to provide examples of the laboratory's work, or in any other way to provide data that would violate the confidentiality of sample results.
- The turn-around-time (TAT) shall start when the sample arrives at the laboratory’s facility and the laboratory has complete instructions for the analysis of the sample and ends when the results are reported to the FRMAC.
- The FRMAC shall be notified immediately of completion of emergency or rush samples, so that the results may be faxed. The results of normal samples shall be mailed.
- Results for each Sample Group shall be submitted together (Data Package).
- The following hard copy minimum information shall be provided for each result reported:
    - FRMAC Sample Number.
    - Laboratory’s Sample Number.

  - The type of sample (2″ AF, Water, Urine, etc.).
  - The volume of the sample shall be determined and reported. (n.nnE±nn Liters, Grams, etc.).
  - The aliquot used for analysis (n.nnE±nn Liters, Grams, etc.).
  - The type of analysis performed on the sample (alpha spectroscopy, liquid scintillation, etc.). This shall be the same as that listed on the Request for Analysis form.
  - The radionuclide(s) specified for analysis on the Request for Analysis form.
  - Results – The measured activity shall be in pCi/Liter, pCi/gram, and pCi/Filter for liquid, solid, and filter samples. For mass measurements (KPA or ICP-MS), the measured concentration shall be in µg/Liter, µg/gram, µg/Filter for liquid, solid, and filter samples. In addition, non-standard samples shall be reported in pCi/Sample or µg/Sample, as appropriate (n.nnE±nn pCi/F, pCi/g, etc.).
  - The total propagated uncertainty at the 1.95 sigma level (95% confidence) reported in the same units as the result. The format for this field is n.nnE±nn.
  - The recovery of the radio-tracer shall be reported with each associated sample.
  - The MDA is calculated according to ANSI 13.30 and is reported in the same units as the result. The format for this field is n.nnE±nn.

- Electronic deliverables shall include - in this order (as described above):
  - FRMAC Sample Number
  - Laboratory Sample ID
  - Sample Type
  - Sample Volume
  - Sample Aliquot
  - Analytical Method
  - Radionuclide
  - Result
  - Uncertainty
  - Chemical Tracer Yield
  - MDA

Each set of results submitted to the FRMAC for normal samples shall be accompanied by a case narrative report which includes for each sample the following information as applicable (this requirement is waived for emergency and rush samples):

- Result of associated blank(s).
- Result of associated Laboratory Control Sample(s).
- Procedures used for sample analysis.
- Commentary explaining any problems encountered during the analysis of the samples.

The laboratory shall submit periodic quality assurance reports which include:

- List all instrument, processing, and other quality related problems and a brief description of how it was resolved.
- Comparison results from programs participated in by the laboratory.
- Commentary explaining any unusual problems or events encountered by the laboratory (key personnel changes, funding issues, organizational changes, etc.).

Whenever the laboratory determines that a correction needs to be made to a previously reported result, the following is required:

- The corrected result and the reason for the change shall be promptly reported verbally to the FRMAC.
- The previous and revised result and the reason for the change shall be documented by memo within five business days.
- The laboratory shall immediately notify the FRMAC by telephone if the analysis requested for a sample can not be performed due to special circumstances such as unacceptable sample, lost sample, no sample received, invalid analysis, etc.

Quality Assurance

- The laboratory shall maintain a Quality Assurance (QA) Program that meets the accepted laboratory practices.
- The QA document shall contain, or point to, procedures for implementation of the QA requirements and shall be maintained current.
- Written procedures shall be developed and implemented for all steps in each analytical process.
- Preparation, identification, and use of procedures shall be controlled (i.e., changes are reviewed and approved)
- Procedures shall be reviewed and revised as needed on a periodic basis, as stated in the QA document.

The following specific QA requirements shall be included in the laboratory's QA Manual:

- Written QA Procedures - Written QA procedures shall be developed and implemented to control activities that could have a significant impact on the accuracy or validity of data. Preparation, identification, and use of such procedures shall be controlled for critical steps or tests in the analytical process.
- Document Control - Documents containing information that could have a significant impact upon quality (i.e., written procedures) shall be reviewed, identified, approved, updated, and distributed in a controlled manner.
- Material Identification and Control - Positive identification and control measures shall be used to assure that samples and other critical items are identifiable at all stages of the analysis and traceable to their source and the resultant data.
- Control of Measuring and Test Equipment - Measuring and test equipment shall be controlled and calibrated to assure the accuracy and reliability of required data. Traceability to the NIST or other recognized standards agencies shall be maintained.

- Non-conformances and Corrective Action - Nonconforming materials, components, or parts (including samples and data) shall be reported, controlled and disposed of in accordance with procedures in the QA Manual. A description of the corrective actions shall be included.
- Quality Assurance Records - Evidence that QA and QC activities were performed shall be documented, reported to the FRMAC as requested, and preserved.
- Quality Assurance Audits - The laboratory shall maintain a formal internal QA audit program. In addition, audits may be scheduled by the FRMAC on an as-needed basis.
- Staff shall receive QA and job specific training appropriate to their participation. All training, whether specific to QA or in other areas related to the work, shall be documented and accurate records kept.

The quality assurance program shall include:

- The analysis of blank and spiked samples with each batch of samples processed at one time.
- At least five percent (5%) of the total number of samples analyzed shall be quality control samples prepared by the laboratory to demonstrate compliance with accuracy and precision requirements (applicable to rush and normal samples only).
- The quality control samples shall have, insofar as possible, a matrix, volume, and other relevant characteristics of the actual samples being analyzed (applicable to rush and normal samples only).
- A system of reviewing and analyzing the results of these samples shall be maintained to detect current problems due to contamination, calibration, calculations, inadequate procedures, or other causes.

Documented and laboratory validated analytical methods shall be used whenever possible with all deviations tested and documented. FRMAC reserves the right to request specific analytical methods.

- The laboratory shall assure that its facilities and equipment are constructed and operated to emphasize radiological control to minimize the possibility of cross-contamination.
- The laboratory shall provide reasonable access to all facilities and data for the purpose of verification of performance and to ensure that the conditions of this Statement of Work are being met.
- The laboratory shall participate in any available, and relevant, comparison programs.

Software Activities

- All computer software, including any modifications, which has the potential to affect the quality of analyses shall be adequately tested and documented prior to release for use.
- Verification of the computer code shall be established using data for which the correct result is known.
- Methods shall be established to assure that changes in software developed in-house are properly documented, controlled, and approved.
- Methods shall be developed to evaluate, control, and correct data entry errors.

Records Management

The laboratory shall have a records management program for all record material and data generated by the processes necessary to perform the analyses.

The records management program shall have as a minimum:

- Written procedures for handling bioassay laboratory records and data throughout their life cycle.
- A system for rapid retrieval of records.
- Written records retention and disposition schedules which meet all federal, state, and local legislative and regulatory requirements.

  These records shall, as a minimum, include:

  - Program and policy manuals.
  - Procedures.
  - Equipment calibration and maintenance.
  - Results of all quality control performance checks.
  - Audit case files, including records management and quality assurance audits.
  - Raw data used in the determination of sample results.

The laboratory shall keep all records pertaining to the analysis of FRMAC samples (including QA/QC records and program and policy manuals in effect at the time of sample analysis) for a minimum of five years from the reporting date of the sample results.

The laboratory shall give FRMAC prior notice of its intent to dispose of any records pertaining to the analysis of FRMAC samples.

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APPENDIX B
REQUIREMENTS TABLES FOR MINIMUM DETECTABLE ACTIVITY

Early Phase Detection Limit Requirements

The following figures (B.1 – B.4) and tables (B.1 – B.4) indicate the required laboratory detection limits for the specific nuclide and matrix. This is not an exhaustive list but is indicative of the lower Minimum Detectable Activity (Concentration) needed for assessment.

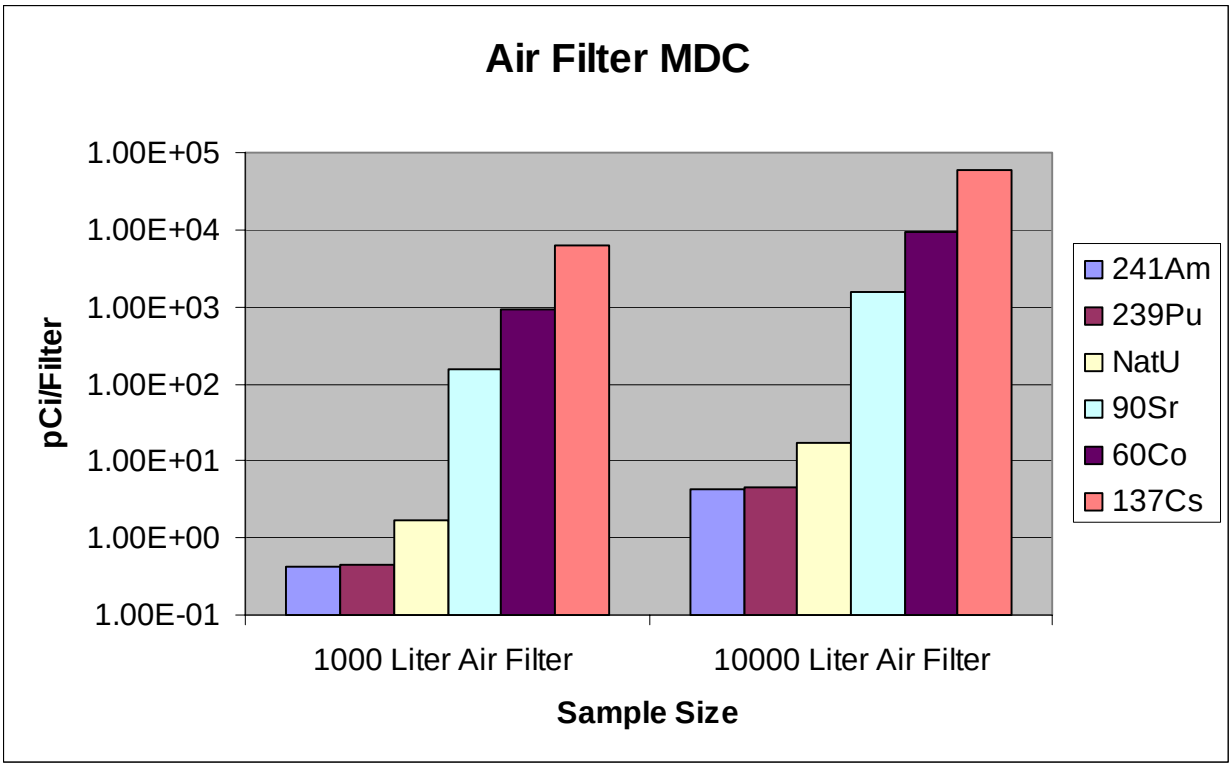

FIGURE B-1. ***AIR FILTER MINIMUM DETECTABLE CONCENTRATION LIMITS***

Table B-1. Air Filter Minimum Detectable Concentration Limits

Nuclide	1000 Liter Air Filter (pCi/Filter)	10000 Liter Air Filter (pCi/Filter)
^{241}Am	4.26E-01	4.26E+00
^{239}Pu	4.44E-01	4.44E+00
^{Nat}U	1.67E+00	1.67E+01
^{90}Sr	1.54E+02	1.54E+03
^{60}Co	9.09E+02	9.09E+03
^{137}Cs	6.06E+03	6.06E+04

Deposition Detection Limit Requirements: The following table indicates the required laboratory detection limits for the specific nuclide and matrix combination. It reflects deposition concentrations from the early phase through restoration phase needed for relocation assessment.

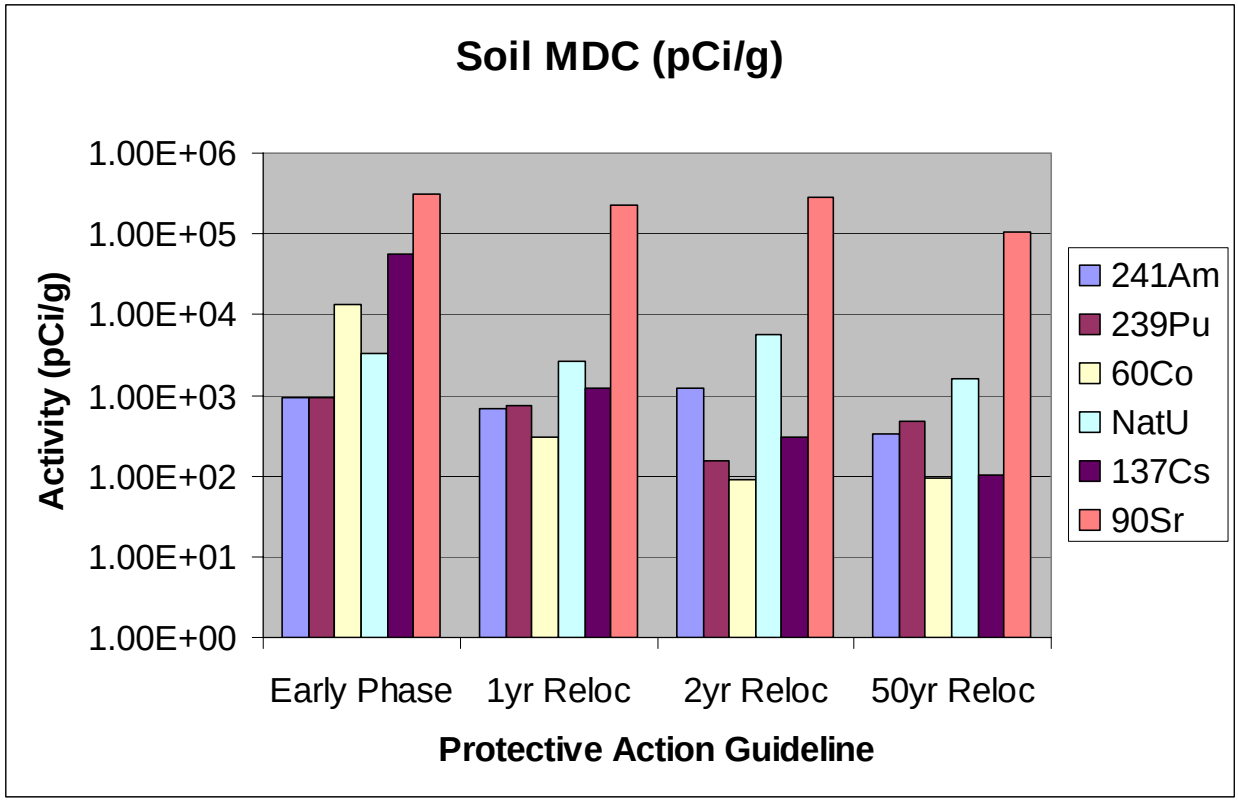

FIGURE B-2. ***SOIL MINIMUM DETECTABLE CONCENTRATION LIMITS***

Table B-2. Soil Minimum Detectable Concentration Limits

Nuclide	Early Phase (pCi/g)	1yr Relock (pCi/g)	2yr Relocation (pCi/g)	50yr Relocation (pCi/g)
^{241}Am	9.17E+02	6.94E+02	1.22E+03	3.33E+02
^{239}Pu	9.44E+02	7.50E+02	1.56E+02	4.70E+02
^{60}Co	1.31E+04	3.06E+02	8.89E+01	9.44E+01
^{Nat}U	3.33E+03	2.69E+03	5.56E+03	1.64E+03
^{137}Cs	5.56E+04	1.25E+03	3.06E+02	1.03E+02
^{90}Sr	3.06E+05	2.25E+05	2.78E+05	1.06E+05

Ingestion Detection Limit Requirements: The following table indicates the required laboratory detection limits for the specific nuclide and matrix combinations listed. The cow forage detection limit is needed for the grass-cow-milk-infant pathway. The fresh produce deposition reflects a value that may result in radioactivity concentrations in the edible portions equal to or exceeding the FDA DIL.

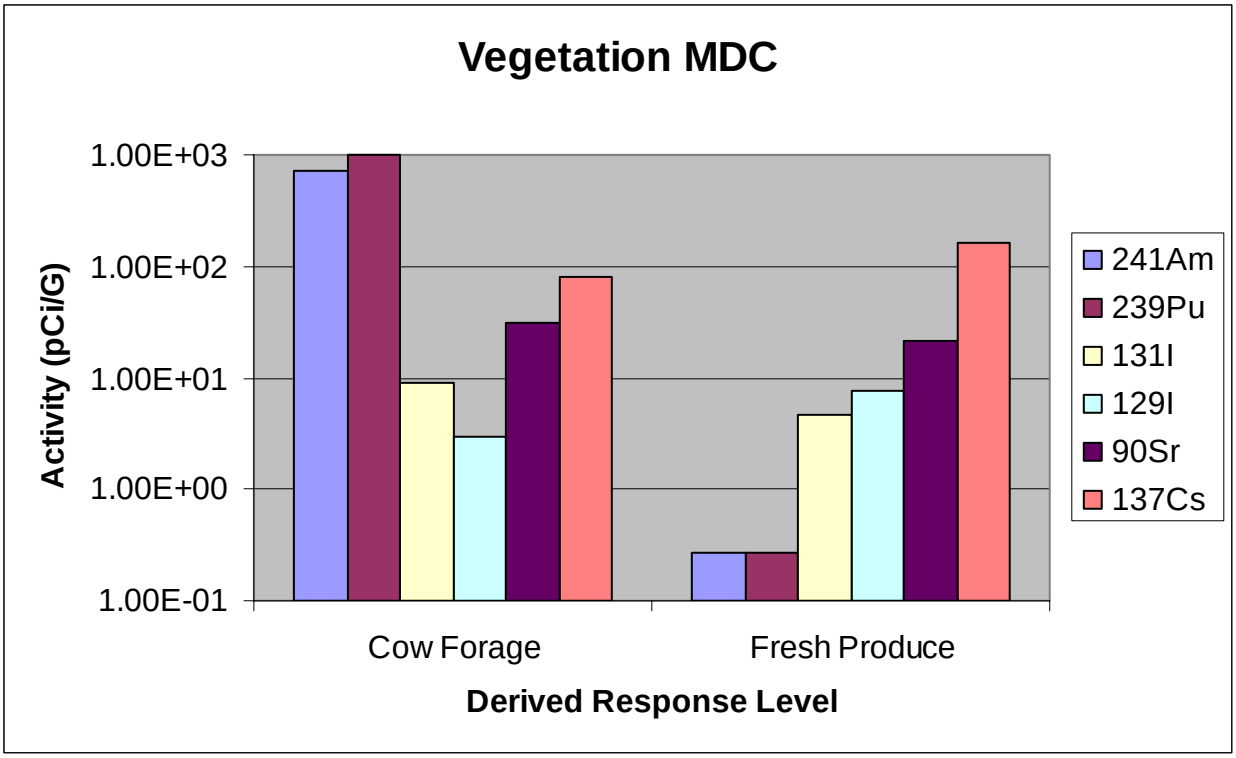

FIGURE B-3. ***VEGETATION MINIMUM DETECTABLE CONCENTRATION LIMITS***

Table B-3. Vegetation Minimum Detectable Concentration Limits.

Nuclide	Cow Forage (pCi/g)	Fresh Produce (pCi/g)
^{241}Am	7.20E+02	2.70E-01
^{239}Pu	9.80E+02	2.70E-01
^{131}I	9.20E+00	4.60E+00
^{129}I	3.00E+00	7.50E+00
^{90}Sr	3.10E+01	2.15E+01
^{137}Cs	8.10E+01	1.60E+02

Food and Drug Administration (FDA) Derived Ingestion Limit detection limit requirements: These are the detection limits needed for the edible portion of foodstuffs. The EPA is currently determining water limits. As an interim measure, the food limits are being converted to water detection limits.

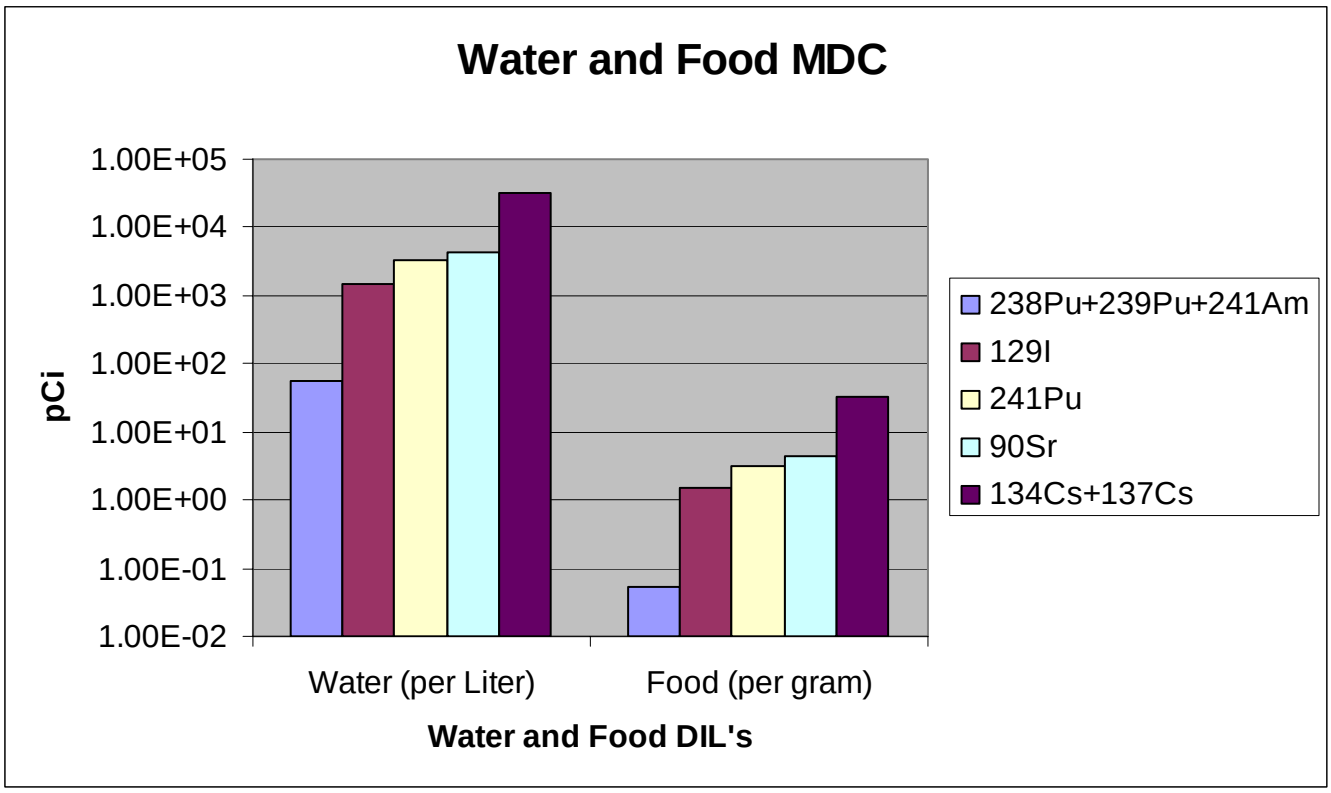

FIGURE B-4. ***WATER AND FOOD MINIMUM DETECTABLE CONCENTRATION LIMITS***

Table B-4. Water and Food Minimum Detectable Concentration Limits

Nuclide	pCi/L Water	pCi/g Food
$^{238}Pu+^{239}Pu+^{241}Am$	5.40E+01	5.40E-02
^{129}I	1.50E+03	1.50E+00
^{241}Pu	3.20E+03	3.20E+00
^{90}Sr	4.30E+03	4.30E+00
$^{134}Cs+^{137}Cs$	3.20E+04	3.20E+01

APPENDIX C

LABORATORY INFORMATION MANAGEMENT SYSTEM

A laboratory information management software system is being integrated into the operation of the laboratory analysis section. This software provides for electronic management of sample chain of custody, shipment, analytical results, quality assurance, data review and electronic data transfer into LIMS as well as data transfer out to the ERDS (Emergency Response Database System). This commercial product is Matrix LIMS Plus (version 4) by Autoscribe.

This future appendix will provide an operational overview of the software's capabilities and user interface's.

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APPENDIX D
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APPENDIX E
GLOSSARY

Accuracy — The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator (QAMS).

Affected Sample Result — A sample result that is considered to be significantly influenced by a quality deficiency, and is qualified, accordingly, through analytical data validation.

Analyst — The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Batch — An analytical batch is a group of sample aliquots analyzed together on the same instrument detector system.

Analytical Data Validation — A technically based analyte and sample specific process that extends the qualification process beyond method or contractual compliance and provides level of confidence in the data that an analyte is present or absent and if present, the associated variability. Data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification — A process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract. Data verification is defined as a systematic process, performed by either the data generator or by an entity external to the data generator.

Audit — A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Batch

Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same National Environmental Laboratory Accreditation Conference (NELAC)-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestives or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Blank

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Equipment Blank. A sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

Field Blank. Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken.

Instrument Blank. A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.

Method Blank. A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Reagent Blank. (Method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Blind Sample

A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the

	sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
Calibration Verification	Calibration verification, as described in this procedure, is defined as a periodic evaluation of instrument standardization established during initial calibration. Using tolerance or statistical control charts, calibration verification can alert the instrument user of the occurrence of out-of-control instrumental conditions.
Carrier	A carrier is a stable element/compound, introduced into the sample preparation/analysis process that will behave chemically similar to the analyte isotope. It is by virtue of this chemical similarity that the carrier will "carry" the analyte isotope(s)through the sample preparation/analysis process. The amount of the carrier recovered at the end of the analysis compared to that added initially is often used in the calculation of the final result.
Chain of Custody Form	Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See forms for an example.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Corrective Action	The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
Correctable Problem	Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Counting uncertainty	Counting uncertainty, as described in this procedure, is defined as the statistical sample standard deviation, which is an approximation of the population standard deviation, and is numerically defined as the square root of the number of counts obtained from a detector. This relationship holds true, provided that the distribution that the counts follows the Poisson distribution. Units for counting uncertainty are the same as for the reported result and the MDC.
Detection Limit	The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.
Initial Calibration	Initial calibration, as described in this procedure, is defined as the standardization of an instrument used in radioactivity detection against a traceable radioactive source(s) of known identity and quantity. This standardization prevails until such time as analytical conditions are deemed out of acceptable tolerance or statistical control limits.
Holding Time	Holding time, as described in this procedure, is defined as the period of time between sample collection and sample activity detection.
Inspection	An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic.
Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample)	A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System	A computerized system for tracking workflows and sample custody through the analytical process.

Matrix

The substrate of a test sample. These matrix definitions shall be used to describe QA/QC performance testing samples:

- **Drinking Water**: Any aqueous sample that has been designated a potable or potential potable water source.
- **Non-Potable Water**: Any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.
- **Solid and Chemical Materials**: Includes soils, sediments, sludge, products and by-products of an industrial process that results in a matrix not previously defined.
- **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Matrix Spike (spiked sample or fortified sample)

A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate)

A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Detection Limit

The minimum concentration of a substance (an analyte) that can be measured and reported with 95% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Minimum Detectable Activity (MDA)

The amount of a radionuclide, which if present in a sample, would be detected with a X probability of non-detection while accepting a probability, Y, of erroneously detecting that radionuclide in a appropriate blank sample. For this procedure, the X and Y probabilities are both set at 0.05.

Minimum Detectable Concentration (MDC)

The MDA expressed in concentration units relative to the sample weight or volume.

National Institute of Standards and Technology (NIST)	An agency of the US Department of Commerce's Technology Administration that is working with EPA, states, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested states can be accredited by NIST to provide NIST-traceable PT to those laboratories testing drinking water and wastewater.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Non-correctable problem	Non-correctable problems are those deficiencies, within data packages that cannot be addressed through additional laboratory submittals, and sample results must stand as-is. Non-correctable problems are deficiencies within data packages which preclude the evaluation of data quality by predefined criteria. Non-correctable problems may be revealed during both data verification and data validation.
Performance Audit	The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preparation Batch	A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality-indicator samples.
Preservation	Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Program	The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Test Sample (PT)	A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.
Quality Assurance	An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
Quality Control	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.
Quality Control Chart	For purposes of this procedure, a quality control chart is used to determine if the response of the instrument has changed statistically; the magnitude the statistical response change may or may not be significant when compared to the required precision and accuracy criteria for the overall analytical technique.
Quality Control Sample	An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.
Quality-indicator Sample	Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.
Quantitation Limits	Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specified degree of confidence.

Reference Standard	A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.
Replicate Analyses	The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.
Reporting Batch	A reporting batch is a group of sample results reported together in a single data package. The reporting batch may be comprised of samples prepared and analyzed together in the same preparation batch or samples prepared and analyzed in different preparation or analytical batches.
Required Detection Limit (RDL)	The RDL is a contractually-specified detection limit (MDA or MDC) which, under typical analytical circumstances, should be achievable.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a COC Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard Operating Procedures (SOPs)	A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A material or substance of one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.
Tolerance Chart	For purposes of this manual, a tolerance chart is based upon maintaining a change of instrument response to a tolerance level judged acceptable to meet overall quality requirements for the technique; a tolerance level should never be more restrictive than what is statistically possible.
Total Propagated Uncertainty (TPU)	The addition of the square root of the sum of the squares of random components of the individual uncertainties, plus the magnitude of the estimated individual systematic relative uncertainties. TPU may include uncertainties introduced through field sampling and analytical laboratory procedures. For the purposes of this manual, TPU includes only those random and systematic uncertainties associated only with laboratory preparation and analysis.
Traceability	The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
Traceable Reference Material (TRM)	A NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).
Tracer	A tracer is a radioactive isotope, introduced into the sample preparation/analysis process that will behave chemically similar to the analyte isotope. The tracer isotope is of the same element as the analyte isotope(s) except where the decay mode, half-life, or availability dictates the use of the isotope of a different element. The activity of tracer detected at the end of the analysis compared to that added initially is used in the calculation of the final result.
Turn-around Time	Turn-around time is contractually-specified as the amount of time which elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.
Validation	The process of substantiating specified performance criteria.
Verification	Confirmation by examination and provision of evidence that specified requirements have been met. NOTE: In connection with the management of measuring equipment, verification provides a

	means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Well Characterized Reference Material (WCRM)	The WCRM may be derived from a field sample which has been well characterized through multiple analyses providing a high level of confidence of the activity level in the sample. The WCRM may be submitted to NIST for characterization and classification as a certified reference material.