PROCESSING AND SUBMITTING SAMPLES

Purpose

This Meteorology and Air Quality Group (MAQ) procedure describes the methods of processing soil, foodstuffs (produce, eggs, milk, honey, fish, and game animals), and nonfoodstuffs (vegetation, mice, bees, birds) and submitting all samples to a laboratory for analysis.

Scope

This procedure applies to the individual(s) assigned to processing samples as part of the Soil, Foodstuffs, and Biota Monitoring Program.

In this Procedure

Hazard Control Plan

The hazard evaluation associated with this work is documented in Attachment 1: Initial risk = **low**. Residual risk = **low.** Work permits required: none. First authorization review date is one year from group leader signature below; subsequent authorizations are on file in group office.

Signatures

Prepared by: Phil Fresquez, Environmental Surveillance Team Leader	Date: 6/23/04
Approved by: Terry Morgan, QA Officer	Date: 6/23/04
Work authorized by: Jean Dewart, MAQ Group Leader	Date: 6/24/04

07/13/04

General information about this procedure

Attachments

This procedure has the following attachments:

Number	Attachment Title	No. of pages
1	Hazard Control Plan	2
2	Chain-of-Custody Record	1
3	Schematic of Distillation Setup	1

History of revision

This table lists the revision history and effective dates of this procedure.

Revision	Date	Description Of Changes
0	10/4/96	New document.
1	3/99	Reformatted in accordance with LIR300-00-01, Safe Work Practices.
2	4/01	Added new Section 9.0, Training.
3	4/02	Change in directorate.
4	4/03	Team name change to Environmental Surveillance.
5	7/13/04	Updated and reformatted document to conform with MAQ procedures.

Who requires training to this procedure?

The following personnel require training before implementing this procedure: MAQ personnel assigned to process and submit samples to an analytical laboratory for analysis.

Training method

The training method for this procedure is **on-the-job** training by a previously-trained individual and is documented in accordance with the procedure for training (MAQ-024).

Annual retraining is required and will be by self-study (“reading”) training.

Prerequisites

In addition to training to this procedure, the following training is also required prior to performing this procedure:

- First Aid
- Cardiopulmonary Resuscitation (CPR)

General information, continued

Definitions specific to this procedure

Foodstuffs: produce (fruits, vegetables, and grains), fish (surface feeders and bottom feeders), eggs, milk, honey, and game animals.

Nonfoodstuffs: vegetation, mice, bees, and birds.

Produce: any fruit, vegetable, and/or grain that could be consumed directly from a garden or an orchard after simple washing.

Soil: Surface soil includes material down to 5-cm (0- to 2-in.) depth.

Composite sample: Samples composed of the five sub-samples taken from an area.

References

The following documents are referenced in this procedure:

- MAQ-024, "Personnel Training"
- MAQ-026, "Deficiency Reporting and Correcting"
- MAQ-Field, "General Field Safety All Employees"

Note

Actions specified within this procedure, unless preceded with "should" or "may," are to be considered mandatory guidance (i.e., "shall").

Worker Safety

Precautions and limitations

This document establishes the basic requirements for processing samples and applies to all personnel performing laboratory procedures described in this document. Work performed under this procedure by LANL personnel will occur only after required training to applicable documents has been completed and documented.

Safe work practices requirements

Personal Protective Equipment - For processing samples, the following personal protective equipment must be worn: safety glasses, safety shoes, lab coat, rubber gloves, cut-resistant (Kevlar) gloves when using knife, and face shield when cutting up game samples.

Do not perform work under conditions you consider unsafe. Before beginning work described in this procedure, review safety needs and requirements, identify hazards, and develop hazard mitigation measures.

Equipment Needed

Equipment needed

The following equipment is required for preparing and processing samples for analysis:

- cutting boards, knives, and cut-resistant (Kevlar) gloves
- face shield
- balance
- glass beakers (50-mL, 100-mL, 1-L, and 2-L volumes; one for each sample)
- aluminum foil
- hot-mitts/pot holders
- hot plate
- watch glass (one for each tritium sample)
- plastic wrap (*e.g.*, Saran wrap™)
- ice cubes
- small paper bags (*i.e.*, lunch bags; one for each sample)
- Wiley mill with a 40-mm screen
- drying and ashing ovens
- polyethylene bottles (20-mL and 500-mL volumes; one for each sample)
- zip-lock bags (gallon size) and labeling pens
- chain-of-custody tape
- laboratory notebook
- blue ice
- insulated coolers for shipping

Processing samples for tritium analysis

Preparing samples for tritium analysis

Within two days of collection, prepare samples for tritium analysis by following the steps below.

Step	Action
Produce	
1	Separate collected produce by variety, assembling composite samples of each type of fruit, vegetable, or grain from on-site, perimeter, and background sites.
2	Assign ID numbers to samples, and label 1-L and 2-L beakers, poly-bottles, and bags with these numbers. Label larger bags, used for submitting samples, with sample location, date, time, and then initial.
3	Remove a subsample (approximately one-third) of produce from each composite for analysis of tritium. Dice the subsample and place in 1-L sample beakers as described in Step 1 of the following chapter *Processing samples for tritium analysis*.
Fish	
1	Separate the fish by species and clean the fish as though being prepared for human consumption.
2	Assign ID numbers to samples, and label 1-L and 2-L beakers, poly-bottles, and bags with these numbers. Label larger bags, used for submitting samples, with sample location, date, time, and then initial.
3	Remove viscera, and discard; remove head, tails, and fins and place them in a 1-L sample beaker as described in Step 1 of the following chapter *Processing samples for tritium analysis*. Prepare approximately 10 beakers for each lake sampled. **Do not fillet** (remove bones) because some consumers may use them for fish meal, and the efficiency of bone removal varies among individual preparers.
Game Animals	
1	Carefully remove the skin and discard at the Los Alamos landfill. Wear face mask when cutting up game samples.Remove the muscle from the bone. Muscle, bone, and organ samples will be processed according to the same procedures, but will remain as separate samples.

Steps continued on next page.

Processing samples for tritium analysis, continued

Step	Action
Game Animals *(continued)*	
2	Assign ID numbers to samples, and label 1-L and 2-L beakers, poly-bottles, and bags with these numbers. Label larger bags, used for submitting samples, with sample location, date, time, and then initial.
3	Place samples in a 1-L sample beaker as described in Step 1 of the following chapter *Processing samples for tritium analysis*.
Vegetation	
1	Separate collected vegetation by variety, assembling composite samples of each type of vegetation. Cut or break vegetation into smaller pieces to facilitate handling.
2	Assign ID numbers to samples, and label 1-L and 2-L beakers, poly-bottles, and bags with these numbers. Label larger bags, used for submitting samples, with sample location, date, time, and then initial.
3	Remove a subsample (approximately one-third) of vegetation from each composite and place in 1-L sample beakers as described in Step 1 of the following chapter *Processing samples for tritium analysis*.
Mice, bees, and birds	
1	Assign ID numbers to samples, and label 1-L and 2-L beakers, poly-bottles, and bags with these numbers. Label larger bags, used for submitting samples, with sample location, date, time, and then initial.
2	Place complete carcass in 1-L sample beakers as described in Step 1 of the following chapter *Processing samples for tritium analysis*.

Processing samples for tritium analysis

To process samples in preparation for tritium analysis, follow the steps below (refer to Attachment 3 for schematic of setup). The same process is used for produce, fish, game animals, vegetation, mice, bees, and birds.

Step	Action
1	Place a 100-mL beaker upside-down in the center of a 1L sample beaker, with a 50-mL beaker right-side-up on top of it. Then place samples in the beaker. Refer to Schematic of Distillation Setup (Attachment 3).
2	Clean the table top with soap and water after the dissecting of the samples. Follow with diluted bleach to ensure (pathogen) contamination control.
3	Cover the top of the large beaker with a watch glass and seal with plastic wrap.
4	To aid in condensation of the water-sample, fill a beaker with ice and place it on top of the watch glass.

Processing steps continued on next page.

Processing samples for tritium analysis, continued

Step	Action
5	Place the sample on a hot plate, warming at a low temperature until water begins to condense on the watch glass. Be certain that the condensation drips into the 50-mL sampling beaker. **CAUTION!! Hot plate and glassware will become hot! Use care when handling these items.**
6	Collect about 10mL of distillate from each sample, and carefully place sample into labeled 20-mL polyethylene bottles.
7	Seal each bottle with chain-of-custody tape, and record each sample on the appropriate chain-of-custody form.
8	Place all tritium samples and the chain-of-custody form into a labeled zip-lock bag and refrigerate. Maintain chain-of-custody on the samples (see chapter *Chain-of-Custody for Samples*) until they are submitted to Paragon Analytics, Inc., Fort Collins, CO.

Processing samples for heavy metals analysis

Preparing and processing samples for heavy metals analysis

Produce and vegetation are processed using the same method and stored dry. Fish and game animals are processed using the same method and frozen.

Step	Action
Produce and vegetation	
1	Produce Remove approximately 100g (fresh weight) of produce from each composite and rinse as though being washed for human consumption. Pat the produce dry with paper towels, and cut it into pieces to facilitate oven drying. Place samples into labeled paper bags. Vegetation Remove approximately 100 g (fresh weight) of vegetation from each composite of each type of vegetation and cut or break vegetation into smaller pieces to facilitate oven drying Place individual samples into labeled paper bags.
2	Place paper bags into ovens and dry the samples in the oven at 75°C for 48 hours.
3	Remove the samples from the oven, and grind each through a 40mm screen using the Wiley mill. CAUTION: Do not operate mill unless you have received personal instruction from a previously trained group member.
4	Place ground samples into labeled 20-mL polyethylene bottles, and then seal the bottles with chain-of-custody tape.
5	Place the sealed bottles into a labeled zip-lock bag
6	Record all samples on a chain-of-custody form (Attachment 2), and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory. See chapter *Chain-of-custody for samples.*

Steps continued on next page.

Processing samples for heavy metals analysis, continued

Step	Action
Fish and game animals	
1	Fish Clean and rinse five fish with water. Game Animals Thoroughly wash muscle, bone, and organs to remove excess blood and/or debris. (Wear face mask when cutting up game samples.) Use paper towels to pat-dry.
2	Fish Remove a 10-g (fresh weight) sample of meat from the each of the five individual fish. Game Animals Remove a 10-g (fresh weight) sample of muscle, bone, or organ
3	Put samples into individually labeled zip-lock plastic bags and place in freezer. Keep samples frozen until submitted to analytical laboratory.
4	Record all samples on a chain-of-custody form (Attachment 2), and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory (Paragon Analytical, Inc., Ft Collins, CO). See chapter *Chain-of-custody for samples.*
5	Clean the table top with soap and water after the dissecting of the game and fish tissue. Follow with diluted bleach to ensure (pathogen) contamination control.

Processing samples for radiochemical analysis

Processing samples for radiochemical analysis

All remaining samples will be used for radiochemistry analysis. (Except for the fish collected specifically for organics analysis.)The minimum ash weight for submittal to the analytical laboratory is 5g, which takes about 500g wet weight. **CAUTION!!** During drying and ashing procedures, ovens and glassware will become hot! Use care when handling these items.
The same process is used for produce, fish, game animals, vegetation, mice, and birds.

Step	Action
1	Prepare the sample beakers by weighing the 2-L beaker to determine the tare weight and record this value in the laboratory notebook.
2	Place ~ 500 to 2,000g of produce, fish, game animal (muscle, bone, or organ meat), vegetation, mice, and birds into labeled 2-L tared beakers and weigh to the nearest 0.01g to determine gross weight. Split a large sample into two beakers to serve as replicates for analysis.
3	Record the fresh weight of the samples (subtract the tare weight from the gross weight) in the laboratory notebook.
4	To dry the samples, cover each beaker with vented aluminum foil (poke holes) and place in the drying oven. Carefully note the placement-order of the beakers in the lab notebook.
5	Dry the samples in the beakers at about 75^{o}C for 5 days.
6	After the fifth day, weigh the samples to the nearest 0.01g. Continue drying and weighing the beakers each day until sample weights are constant (+10%) in two successive weighings—samples are dry.
7	Remove dry samples from the oven and weigh them to the nearest 0.01g. Subtract the original tare weight from this gross weight to calculate the dry weight of each produce sample. Enter this data in the laboratory notebook.
8	To ash the samples, place samples in the ashing oven, carefully note placement of beakers, and ash the samples for 5 days. During ashing, raise the temperature step-wise from 75^{o}C to 500^{o}C to avoid explosive combustion of the organic materials in the early stages of the process.
9	After ashing is complete, reweigh the samples to the nearest 0.01g. Calculate ash weights by subtracting tare weights from gross ash-weights. Record ash weights in the laboratory notebook.
10	Transfer each ash sample to a 500-mL polyethylene bottle and label the bottle.
11	Seal the bottles with chain-of-custody tape and record all samples on a chain-of-custody form (Attachment 2), and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory (Paragon Analytics, Inc.). See chapter *Chain-of-custody for samples.*

Processing samples for organics analysis

Preparing fish samples for organics analysis

Fish samples for organics analysis must be kept cool.

Step	Action
1	Rinse and weigh the whole fish and record weight in laboratory notebook. Do not skin. Leave head and tail attached.
2	Complete Sample Location and Fish Physical Characteristics Form (Attachment 4) after weighing and measuring fish.
3	Gut and separate viscera (organs, fatty deposit). If necessary, combine subsamples of viscera to meet minimum sample quantity requirements. Weigh the viscera and record weight in laboratory notebook.
4	Put fish in pre-labeled amber screw-top jars and place in refrigerator or freezer until submitted to analytical laboratory.
5	Record all samples on a chain-of-custody form (Attachment 2), and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory (Paragon Analytics, Inc.). See chapter *Chain-of-custody for samples*

Collecting honey, milk, and eggs

Sample collection

Honey, milk, and eggs do not require field work. Samples are purchased from honey producers and from farmers.

Equipment needed

The following equipment is required for collecting milk and egg samples:

- ice chest with ice
- large zip-loc bags
- marker for labeling bags
- chain-of-custody forms (Attachment 2) and tape

Honey

Honey producers bring honey in glass jars directly to the laboratory at TA-21, Bldg. 210. Perform the following steps to prepare samples for submittal to the analytical laboratory.

Step	Action
1	Seal jars with chain-of-custody tape.
2	Record all samples on a chain-of-custody form (Attachment 2), and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory. See chapter *Chain-of-custody for samples.*

Milk sample locations

Milk is collected (purchased) from several farmers raising goats in the Los Alamos and White Rock areas and from the Albuquerque area. Locations vary from year to year depending on availability of goats. Milk will be analyzed for various radionuclides and local milk is compared to milk produced from Albuquerque, NM.

Number of samples

The approximate number of samples collected at perimeter locations is two and one from regional locations.

Steps to prepare milk samples for submittal

Perform the following steps to prepare samples for submittal to the analytical laboratory.

Step	Action
1	Collect (purchase) a 1-gallon sample of milk and place in cooler with ice for transport back to laboratory.
2	Complete a chain-of-custody form (Attachment 2) and maintain proper chain-of-custody on the samples until submitted to the analytical laboratory. See chapter *Chain-of-custody for samples.*

Steps continued on next page.

Collecting honey, milk, and eggs, continued

Step	Action
3	Seal the bottles/jars with chain-of-custody tape. Place samples on ice or in refrigerator until submitted to the analytical laboratory.

Eggs

Sample Locations - Eggs are collected in the summer from the closest free-ranging chicken facility in perimeter areas (e.g., Los Alamos, White Rock/Pajarito Acres, and/or San Ildefonso) and from regional background areas (Española, Santa Fe, or Jemez). Actual locations will vary from year to year depending on availability of eggs.

Number of samples

Two dozen samples are collected at each perimeter and regional location

Steps to prepare samples for submittal

Perform the following steps to prepare samples for submittal to the analytical laboratory

Step	Action
1	Collect two dozen eggs directly from the producer (farmer); eggs are usually placed in egg cartons.
2	Place cartons in labeled plastic bags (date, location, and sample number), and pack on ice for transport back to the laboratory. Complete a chain-of-custody form (Attachment 2) with the appropriate sampling information.
3	Once at the lab, seal the cartons with chain-of-custody tape, and store the eggs on ice or in a refrigerator and maintain proper chain-of-custody on the samples until they are submitted to the analytical laboratory for analysis (normally within two working days). See chapter *Chain-of-custody for samples.*

Sample Processing

No processing is required for honey, milk, and egg samples. Samples are shipped to Paragon Analytics, Inc. following the steps in the next chapter *Submitting the Samples.*

Submitting the samples

Submitting samples for analysis

Submit all samples for tritium, heavy metal, radiochemical, and organic analyses to Paragon Analytics, Inc., Ft. Collins, CO, using the following steps.

Step	Action
1	Place all samples in unsealed insulated coolers: • Pack dry samples (including ground produce and soils) in a cooler without ice. • Pack refrigerated samples (liquid and eggs) and frozen samples in a cooler with blue ice.
2	Request the following analyses on the appropriate chain-of-custody forms (see Attachment 2): • analysis of tritium content; reported in pCi/L of moisture • analysis of the following heavy metals: Ag, As, Be, Cd, Cr, Hg, Ni, Pb, Sb, Se, Tl, and Zn (plus the others on EPA's Target Analyte List); reported in μg/g (dry weight) • analysis of the following radionuclides: strontium-90, isotopic uranium, cesium-137, plutonium-238, plutonium-239/240, and americium-241; reported in pCi/g (dry weight)
3	Place the bags containing the sealed and labeled bottles or egg cartons and the chain-of-custody form into a cooler (each medium in its own cooler.
4	Fill out a Shipping Manifest (Laboratory form 1768) with appropriate information.
5	Take unsealed cooler(s) and properly filled out Shipping Manifest (Laboratory form 1768) to the procurement specialist at the Pueblo Complex. The procurement specialist must sign the shipping manifest.
6	Take unsealed cooler(s) and the properly signed Shipping Manifest to LANL's Shipping & Receiving office at TA-3, Bldg. 30. Shipping personnel will inspect the contents of the coolers and then seal and ship them to Paragon Analytics, Inc., Ft. Collins, CO.

Chain-of-custody for samples

Maintaining custody of samples

A sample is physical evidence collected from a facility or the environment. Chain-of-custody must be documented for all samples used to demonstrate compliance. Verify that the possession and handling of samples is traceable at all times. A sample is considered in custody if it is one of the following:

- In one's physical possession.
- In one's view after being in one's physical possession.
- In one's physical possession and then locked up so that no one can tamper with it.
- Kept in a secure area where access is restricted to authorized and accountable personnel only.

NOTE: A secured area is an area that is locked, such as a room, cooler, vehicle, or refrigerator. If the area cannot be secured by locking, use a custody seal to secure the area or the sample container.

Transferring custody of samples

Whenever samples are transferred into the custody of another person or organization, complete the "relinquished by/received by" and "date" sections of the form (Attachment 2). These sections of the form must provide a complete history of custody of the samples from collection to transfer to the analytical laboratory.

If chain-of-custody is broken

Whenever there is a break in the chain of custody of a sample, document the failure by initiating a deficiency report in accordance with the procedure for deficiencies (MAQ-026). [The deficiency process will document the occurrence, evaluate the potential impact (if any) on the samples, and propose a fix to prevent recurrence.]

Records resulting from this procedure

Records

The following records generated as a result of this procedure are to be submitted **within three weeks of shipment** as records to the records coordinator:

- Chain-of-custody record
- Copy of Shipping Manifest

HAZARD CONTROL PLAN

1. The work to be performed is described in this procedure.
"Processing and Submitting Samples"

2. Describe potential hazards associated with the work (use continuation page if needed).

Use of electrical appliances (hot plates and ovens)
Hot glass
Broken glass
Splattering of hot water for making tea from plants
Drying and ashing ovens
The Wiley Mill
Use of knives
Ergonomic injuries (repetitive motion)
Blood-borne pathogens

3. For each hazard, list the likelihood and severity, and the resulting initial risk level (before any work controls are applied, as determined according to LIR300-00-01, section 7.2)

Use of electrical appliances (hot plates and ovens)—occasional/moderate = low
Hot glass—occasional/moderate = low
Broken glass—occasional/moderate = low
Splattering of hot water—occasional/moderate = low
Drying and ashing ovens—occasional/moderate = low
The Wiley Mill—remote/negligible = minimal
Use of knives—improbable/moderate = minimal
Ergonomic injuries (repetitive motion)—remote/negligible = low
Blood-borne pathogens—improbable/critical = low

Overall *initial* risk: ☐ Minimal ☒ Low ☐ Medium ☐ High

4. Applicable Laboratory, facility, or activity operational requirements directly related to the work:
☒ None ☐ List: Work Permits required? ☒ No ☐ List:

HAZARD CONTROL PLAN, continued

5. Describe how the hazards listed above will be mitigated (e.g., safety equipment, administrative controls, etc.):

Use of electrical appliances (hot plates and ovens)—Wear safety glasses, lab coat, and rubber gloves. Be familiar with the operator's manuals for each piece of equipment.
Hot glass—Use hot mits.
Broken glass—Wear safety glasses, lab coat, and cut-resistant (Kevlar) gloves
Splattering of hot water—Wear safety glasses, lab coat, and heavy rubber gloves.
Drying and ashing ovens—Use hot-mitts or pot holders when working with the ovens, hot plates, or hot beakers.
The Wiley Mill—Wear safety glasses, lab coat, and heavy rubber gloves. Be familiar with the operator's manual.
Use of knives—When knives are being used, wear cut-resistant (Kevlar) gloves to prevent injuries
Ergonomic injuries (repetitive motion)—Take a short break every hour.
Blood-borne pathogens—wear a face shield.

6. Knowledge, skills, abilities, and training necessary to safely perform this work (check one or both):

☒ Group-level orientation (per MAQ-032) and training to this procedure.
☒ Other → See training prerequisites on procedure page 3. Any additional describe here:

7. Any wastes and/or residual materials? (check one) ☒ None ☐ List:

8. Considering the administrative and engineering controls to be used, the *residual* risk level (as determined according to LIR300-00-01, section 7.3.3) is (check one):

☐ Minimal ☒ Low ☐ Medium (requires approval by Division Director)

9. Emergency actions to take in event of control failures or abnormal operation (check one):

☒ None ☐ List:

For all injuries, provide first aid and see that injured person is taken to Occupation Medicine (only if immediate medical attention is not required) or the hospital.

Signature of preparer of this HCP: This HCP was prepared by a knowledgeable individual and reviewed in accordance with requirements in LIR 300-00-01 and LIR 300-00-02.

Preparer(s) signature(s)	Name(s) (print) /Position	Date

Signature by group leader on procedure title page signifies authorization to perform work for personnel properly trained to this procedure. This authorization will be renewed annually and documented in ESH-17 records. Controlled copies are considered authorized. Work will be performed to controlled copies only. This plan and procedure will be revised according to MAQ-022 and distributed according to MAQ-030.

Environmental Surveillance Team Chain-of-Custody Record

This form is from MAQ-706

Project Contact _____

Contact Phone No. _____ **MS**_____

Project Name Foodstuffs Sampling

_____

_____

Account Code _____

Cost Center _____

Program Code _____

Date Collected	Time Collected	Station Name/Number	Number of Samples	Analysis Requested	Remarks

Relinquished by (print and sign)	Date
	Time
Received by (print and sign)	
	Date
	Time

Relinquished by (print and sign)	
	Date
	Time
Received by (print and sign)	
	Date
	Time

Relinquished by (print and sign)	
	Date
	Time
Received by (print and sign)	
	Date
	Time

Samplers (print names and initial) _____

Comments

SCHEMATIC OF DISTILLATION SETUP

For processing samples for tritium analysis