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Environmental Programs Directorate

Waste & Environmental Services

Standard Operating Procedure

for **AIRNET - SAMPLING OF AMBIENT AIRBORNE TRITIUM**

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1.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) states the responsibilities and describes the field sampling of water vapor from the air and the submission of a sample for tritium analysis for the Los Alamos National Laboratory (LANL) Waste and Environmental Services Division (WES)

All **WES participants** shall implement this procedure when performing field sampling of water vapor from the air and the submission of a sample for tritium analysis.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

Los Alamos National Laboratory (Laboratory or LANL) routinely handles tritium as part of its operations. The air monitoring network (AIRNET) system monitors levels of ambient tritium. The AIRNET samplers continuously collect water vapor from the atmosphere that is analyzed to determine airborne tritium concentrations. See SOP-5140, Quality Assurance Project Plan for the Radiological Air Sampling Network (AIRNET), for a detailed description of the project purpose and requirements.

Water vapor is collected by silica gel (silica acid) in a plastic cartridge. A pump continuously pulls air through the cartridge. Cartridges are exchanged every two weeks.

Tritium is an isotope of hydrogen with a half-life of 12.3 years. Tritium gas can occur as T₂ or HT. Tritium oxide is usually in the form of HTO, but T₂O can also occur. In the environment, tritium gas will eventually oxidize into tritiated water vapor. Sampling of tritium oxide is addressed in this procedure.

The Laboratory handles tritium and periodically releases small amounts of tritium to the environment. In addition, there have been some unplanned releases to the environment of elemental tritium gas and tritiated water vapor.

The following four primary sources of tritium are sampled by this method:

- natural tritium produced by the interaction of cosmic rays with hydrogen atoms in the atmosphere,
- tritium produced by atmospheric nuclear testing,
- tritium released from Laboratory operations and other operations, and
- tritium from commercial products such as exit signs and illuminated markers.

2.2 Precautions

N/A

3.0 EQUIPMENT AND TOOLS

Equipment and tools are defined in individual steps in section 4.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Preparation of Silica Gel Cartridges

Worker	1. Change air filters (SOP-5143, AIRNET—Environmental Sampling of Airborne Particulate Radionuclides) and tritium cartridges every two weeks (three at times). Filters and cartridges may be changed sooner as directed by AIRNET task leader. Prepare sample cartridges in the week preceding “changeout” week. Sample changeout usually takes three days.
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Worker

2. Dry the silica gel as follows.

- Dry gel in a tray or in the original metal cans with the lid removed.
- Dry gel in a drying oven at 145°C for at least two days.
- Remove the hot cans with the orange terry cloth gloves.
- Replace the lids tightly immediately after removal from oven.
- Allow gel to cool before proceeding to the following steps. Dried silica gel can be stored for several months before use in the field.
- Perform a bound water calculation following SOP-5178 "[AIRNET - Determining Water Content of Silica Gel using the Lindberg Furnace](#)" once for each lot of silica gel.

3. Collect the following materials and tools at Technical Area (TA)-54-1001:

- dried silica gel, 6–12 mesh;
- balance, 1000 g capacity (minimum);
- plastic funnel; and
- empty silica gel cartridges for all sample locations, including trip "blank" cartridges (all cartridges should be the same color to identify the sample week).

NOTE: Cartridges are labeled and dedicated for reuse at a single location.

4. Make sure all the material and equipment that will contact the silica gel are clean and dry before proceeding. To fill the cartridges, perform the following steps.

- a. Ensure all cartridges have the same color for that week to identify the sampling week to which these cartridges belong.
 - b. Turn on the hood. In the hood, fill all the sampling cartridges to the top with dry silica gel. Each cartridge will hold about 135 g of silica gel. Replace the top.
 - c. If not already running on the PC, launch the Mettler Toledo BalanceLink program and minimize it to an icon.
 - d. Open the AIRNET access database and go to the appropriate form for entering initial gel weights. Ensure that the correct sample period ID is selected.
 - e. Zero the balance with any supports to be used in place. Use the 500 g and the 1 kilogram (kg) check weights to check the balance function. Put on a cloth glove to handle the weights. Enter the results on the initial gel weight entry form. Ensure the balance reads zero with no weights but WITH any supports to be used.
 - f. Place a cartridge on the center of the balance pan so that the fittings do not touch the table or the sides of the balance (you may use a tared support if appropriate). Press F2 to transfer the weight from the balance into the selected cell of a displayed table or query. Repeat to weigh all cartridges.
 - g. Print out the just-recorded weight data and have another worker re-weigh all the cartridges. Compare the weights with the printout data to verify proper data entry and initial and date. File this record as described in section 4.7.
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- Worker
- e. Zero the balance with any supports to be used in place. Use the 500 g and the 1 kilogram (kg) check weights to check the balance function. Put on a cloth glove to handle the weights. Enter the results on the initial gel weight entry form. Ensure the balance reads zero with no weights but WITH any supports to be used.
 - f. Place a cartridge on the center of the balance pan so that the fittings do not touch the table or the sides of the balance (you may use a tared support if appropriate). Press F2 to transfer the weight from the balance into the selected cell of a displayed table or query. Repeat to weigh all cartridges.
 - g. Print out the just-recorded weight data and have another worker re-weigh all the cartridges. Compare the weights with the printout data to verify proper data entry and initial and date. File this record as described in section 4.7.
 - h. Add 10 g of deep well water to the trip blanks.
 - i. Take the trip blanks with the cartridges during changeout; between collections, store the cartridges in the lab. Ensure these cartridges are processed (in about 24 days) with the cartridges just prepared, not with the samples to be collected the next week. Maintain proper chain-of-custody on these samples (see section 4.6, Chain-of-Custody for Samples).
5. If the connection to the AIRNET database is not working and the gel weight data cannot be loaded directly into the database, follow the steps for obtaining initial (above) or final (pg 12) weights, EXCEPT load the Excel spreadsheet d:\GelWeights\GelWeightsTemplate.xls from the local computer hard drive and enter the data into this instead of the AIRNET database.
 6. After data entry into the spreadsheet, save the file and give it a name (e.g., "Final020318" or "Initial020318", where "Final" is for final gel weights and "Initial" is for initial gel weights; "020318" is the period ID). Send the file (via e-mail if available or on a diskette) to the AIRNET database manager for loading into the AIRNET database.

4.2 Field Changeout

- Worker
1. It is preferred that two people travel together to collect AIRNET samples. However, it is acceptable for one person to collect samples. In either case, ensure a working cellular phone and a radio are in the vehicle.
 2. Complete all facility-specific training requirements and follow facility-specific work rules, including access requirements. Work control in a laboratory facility is the responsibility of the Facility Manager. All work must be performed with an approved IWD and be on the Plan of the Day.
 3. Check with the pueblo authorities before doing any type of work at a pueblo. Work control is the responsibility of the pueblo authorities. Arrangements with some pueblos provide for them to collect their own samples and deliver them to the field team for inclusion in the current sample shipment.
 4. Check with project leaders for any special requirements before working on private property. Each private property owner has a separate arrangement with the group.
- Worker
5. If an AIRNET station housing appears damaged in such a way that electrical wires are exposed or could be shorted to the housing or conduit, do not touch the station! Immediately report the damage and request that an electrician repair the electrical damage.

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6. Consult Attachment 1 of SOP-5143 for the locations and directions to the AIRNET sample locations. Perform the following steps at each station. These steps may be performed in conjunction with SOP-5143. Two people may perform the following steps, in which case the recording of the data may be done by the second person and the timer reset as soon as the data are recorded.
 - a. Open the housing and read the timer. Record the timer reading in the palm computer, in the field notebook, or on the chain of custody. If both timer and pump are operating properly, skip to step d.
 - b. If the pump is not running, attempt to restart it by resetting the ground fault circuit interrupter (GFCI) breaker and then checking the power source breaker, or taking other actions.
 - c. If these actions fail or cannot be completed, contact the facility manager or the electrician. At pueblo sites, notify the appropriate pueblo contact. If the pump is started, continue to the next step. If the pump cannot be restarted, replace the pump by following SOP-5145 or troubleshoot the station as necessary. If the station cannot be restarted during the visit, collect the current sample but do not install the new sample until the pump can be restarted.
 - d. Read the as-found flow rate for the silica gel. If, for some reason, a flow reading cannot be obtained when the pump is running, record a final flow rate of 0. If the flow meter indicates >250 cubic centimeters per minute (cc/min), use the Buck calibrator (see SOP-5145, Calibration of Air Flow Rates at Sampling Stations) to obtain a reading or note that flow is >250 cc/min. Make a note in the comments field. Record the final flow rate on the palm computer, the field notebook, or on the chain of custody.
 - e. Change the sample by removing the used cartridge from the quick-connect fitting and installing the new cartridge. Ensure that the new cartridge is securely fastened to the quick-connect fitting, "clicked". Make sure the label on the cartridge correctly matches the location and the color on the cartridge is correct for the sample period.
 - f. Set the flow rate to 200 cc/min (± 20 cc/min) after the new cartridge is installed.
 - g. Enter or verify the following data has been recorded electronically, in the field notebook, or on the chain of custody:
 - date and time (recorded automatically by the field computer);
 - timer reading (hours);
 - initial (as-found) air flow rate;
 - final (after new filter and cartridge installed) air flow rate;
 - sample I.D., in the following format: a sample collected the week of February 14, 2000 (always a Monday) at station 13 would be 000214.13. Label the trip blanks as "station" numbers 87, 88, 89, 98, and 99 (recorded automatically by the field computer); and
 - any comments on difficulties encountered, "unusable" sample, estimated data, or other conditions.

Worker

- h. Reset the digital timer by pressing the reset button. (This can be done at any time after recording the timer reading).
- i. Close and secure the sample housing.
- j. Follow the requirements in section 4.6, for chain-of-custody documentation and handling.

7. If a sampler is found in an inoperable condition, or if there are other problems that have resulted in a total or partial loss of sampled material or affected the integrity or reliability of the sample, the condition must be documented by recording the condition in the field notebook or palmtop computer logbook.

Record in the comments whether a datum was estimated (e.g., enter "Estimated timer reading" in comments if timer was out and hours were calculated from other sampler information).

In all cases of inoperable or damaged stations, document the condition by recording in the field notebook or field computer.

8. After returning to TA-54-1001, download the data from the field computers to the desktop computer database and print the tritium chain-of-custody forms according to SOP-5149. Check the data on the forms for errors and keep them in a safe place until samples are shipped.

4.3 Processing the Silica Gel

- Worker
1. Prepare the silica gel within a few days after collection to limit further moisture absorption. If the network connection to the database is down, see the instructions in section 4.1 step 7.
 2. Weigh the samples by performing the following steps.
 - a. If not already running, launch the Mettler Toledo BalanceLink program and minimize it to an icon.
 - b. Open the AIRNET access database and go to the appropriate form for entering final gel weights. Ensure that the correct sample period ID is selected.
 - c. Zero the scale with any cartridge props used in place. Use the 500 g and the 1 kg check weights to check the balance function. Put on a cloth glove to handle the weights. Note the results on the final gel weight entry form. Ensure the balance reads zero with no weights.
 - d. Place the cartridge on the center of the balance pan so that the fittings do not touch the table or the sides of the balance (you may use a tared support if appropriate).; Press F2 to transfer the weight from the balance into the database. Repeat for each cartridge.
 - e. Print out the just-recorded weight data and have a coworker re-weigh all the cartridges. Compare the weights with the printout data to verify proper data entry, then initial and date.
 - f. Prepare three matrix spike samples and three blank samples for shipping by following section 4.5. Perform final weighing of the spikes and blanks and enter data into the gel weight table.
 - g. Calculate the water content of the samples and email the water content sheet to the analytical laboratory.

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- Worker
3. Collect the following equipment and supplies:
 - 250 milliliter (ml) polyethylene bottles (can be obtained from the SMO),
 - 12 × 12 × 6 heavy duty box,
 - security tape,
 - permanent marker,
 - packing tape, and
 - pre-printed labels with sample numbers and barcodes (from AIRNET database).
 4. To unload the silica gel cartridges, perform the following steps.
 - a. Pre-label all the bottles by writing the station number on the bottle (not the cap) with a marker.
 - b. Put the pre-printed labels on the bottle.
 - c. In the laboratory hood, line up as many bottles with their respective cartridges as will easily fit in the hood.
 - d. Unscrew the tops from the cartridges and pour the silica gel into the appropriate pre-labeled bottle.
 - e. Before removing the cartridges and bottles from the hood, double-check that the cartridge and bottle numbers match and that there were no errors in labeling.
 - f. Repeat steps a through e for the remaining cartridges.
 - g. Package all the bottles in the box. Use bubble wrap to pad the shipment. Include the paperwork generated in Section 4.4. Use custody tape in appropriate places to indicate tamperin.

4.4 Submitting Silica Gel Samples for Analysis

- Worker
1. To complete the appropriate paperwork and submit the samples for analysis, perform the following steps:
 - a. Run AIRNET database and follow the database menus to print the Gel Agreement Shipping Report.
 - b. Select the Period # (example: ID 080218). Then select Sample Shipping. Select Period # again (example: ID 080218). Then select Gel Agreement Shipping Report.
 - c. Print the Gel Agreement Shipping Report and place all chain-of-custody forms with the Gel Agreement Shipping Report.
 - d. Get a second AIRNET team member to double-check the shipping letter, sample ID number, dates, and number of samples shipped.
 - e. Complete the AIRNET Sample Shipping Checklist (SOP-5143, Attachment 4).
 - f. Make one copy of the shipping letter and chain of custody forms. Make a copy of the chains of custody for the validation and verification notebook. Send the original copies of all paperwork with the sample shipment.
 - g. Take samples to the Sample Management Office for shipping.
 - h. Enter shipping date in Sample and Data Tracking Table of the AIRNET database.

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4.5 Preparing Tritium Matrix Spikes and Blanks

- Worker
1. Obtain tritium spikes from o2Si or other approved vendor.
 2. If a tritium matrix spike is spilled, call a radiation control technician (RCT) immediately. Avoid touching the liquid. Spike samples with concentrations up to 2 microcuries per liter ($\mu\text{Ci/l}$) may be transported without special requirements. If a spike is over this level, use the special transport container with required labeling.
 3. When a new batch of spiked samples is received from the lab, the spiked water must be adsorbed onto silica gel and prepped for future sampling periods. Follow step 5 below.
 4. Collect the following equipment and supplies:
 - sealable jars
 - tritiated water spikes
 - deep well water (water from a regional aquifer well that has not shown analytical results for tritium)
 - small vials (e.g., 20 ml scintillation vials)
 - dried silica gel, 6–12 mesh
 - balance, 1000 g capacity (minimum)
 - shipping containers
 - LubriSeal® stopcock grease
 5. To process and label the blanks and spikes, perform the following steps.
 - a. Weigh out 125-145g of silica gel and pour into each sealable jar. Record the initial gel weight on a sticker and place the sticker on the jar, along with the date.
 - b. For the blanks, weigh approximately 10 g of deep well water into the small vials.
 - c. Set the spiked sample or a blank with lid removed into the a jar, seal the jar, and allow the liquid to be absorbed into the silica gel. Blanks may be absorbed using 500 ml glass sample jars. Tritium spikes must be absorbed in sealable glass jars that have been sealed with stopcock grease to avoid any loss of tritium during the absorption period. This process takes about a month.
 - d. Label the shipping containers with the sample identification numbers for upcoming sample periods, with sample numbers 91, 92, and 93 (for the blanks) and 94, 95, and 96 (for the spikes).
 - e. Place the shipping container on the scale and tare the scale.
 - f. Pour the silica gel from the sealable jar into the numbered shipping container. Be sure to keep the spike vial next to the appropriate shipping container until the numbers are recorded.
 - g. Weigh the shipping container with the silica gel to get a final gel weight. Enter initial and final gel weights into the initial and final gel weight tables for the sample period being sent for analysis.
 - h. Close the shipping containers tightly. Wrap Parafilm wax around the cover of the tritium spike shipping container and seal with custody tape.
 - i. Have a second person record both the number on the spike vial and the sample period ID into the database according to step j below as each container is poured. If a second person is not available, record the data on a sheet for later entry. Leave each spike vial next to the shipping container until step k below is completed.
 - j. In the AIRNET database, open the Field Data Management form and select H-3 Matrix spikes. Highlight the AIRNET period number in the scrolling window and then click the H-3 Matrix Spike AIRNET number to CST QC. Enter the lab's spike number and the corresponding WES sample ID.
- Worker

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k. Print out the entered data and have a second person compare the entries in the database with the containers arranged on the work bench. Both workers should initial the print out after data is verified.

6. When a new shipment of tritium matrix spikes arrives, enter the lot numbers and concentrations into the AIRNET database. In the AIRNET database, run the form, Field Data Management, and select H-3 Matrix spikes, then enter new H-3 Quality Control Sample Spike Concentrations. Type in the lab's identifying numbers for each unique spike and the corresponding tritium concentration. Next, select the appropriate button on the form to enter the date of preparation of the spikes. Print out the entered data and have a second person verify correct entry. H-3 matrix spike vials should be stored in a locked cabinet. Empty vials need to be disposed of according to an approved waste profile form.

4.6 Chain-of-Custody for Samples

- Worker
1. Verify that the possession and handling of samples is traceable at all times. 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. 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, or
 - 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.
 2. Complete the "relinquished by/received by" and "date" sections of Attachment 2 of SOP-5143 whenever samples are transferred into the custody of another person (other than an AIRNET field team member) or organization. These sections of the form must provide a complete history of custody of the samples from collection to transfer to the analytical laboratory.
 3. Whenever there is a break in the chain of custody of a sample, document the failure and report it to the project leader.

4.7 Waste Management

- Worker
1. All samples not sent for analysis and samples returned from the analytical laboratory will be handled according to an approved waste profile form when they are no longer considered archive samples and are ready for disposal. Any waste created due to an unexpected release or emergency situation will be evaluated by the waste management coordinator prior to disposal.

4.8 Records Management

- Worker 1. The following records will be submitted to the Records Processing Facility according to EP-DIR-SOP-4004, Records Transmittal and Retrieval Process:
- Air Monitoring Field Data Form and Chain of Custody Record (SOP-5143 Attachment 2)
 - AIRNET Silica Gel Shipping Letter original with samples shipped to analytical lab.
 - Field notes.

5.0 REVISION HISTORY

Revision No. <i>[Enter current revision number, beginning with Rev.0]</i>	Effective Date <i>[DCC inserts effective date for revision]</i>	Description of Changes <i>[List specific changes made since the previous revision]</i>
0	7/19/94	New document issued as LANL-ESH-8-204.
1	12/18/95	Revised to reflect changes in process and organization.
2	05/15/96	Added electronic recording of field data; changed the tracking of sample period by color of cartridge head.
3	09/24/96	Expanded steps for inoperative pumps, added attachment for example of electronic data recording, added actions for #90, added field safety information to prerequisites.
4	2/20/97	Add steps and time limits for submitting records when completed, other editorial changes.
5	6/9/98	Added balance check sheet, added steps for recording balance checks, revised steps for submitting samples for analyses.
6	02/26/99	Revised steps for describing weighing of cartridges and recording data, changed attachments.
7	04/21/00	Added HCP as Attachment 1, described use of BalanceLink program, revised steps describing use of queries for entering distillate data, add text for handling matrix spikes, removed attachment "Example of Electronic Silica Gel Mass Data Entry," editorial changes.
8	09/7/00	Revised to allow processing of silica gel by either on-site distillation or shipment to off-site lab.
9	03/13/01	Added steps for new trip blanks, changed process for processing and packaging spikes and trip blanks, removed form for balance checks.
10	04/15/02	Revised to reflect use of Palm computer devices, revised some steps about sample handling, and revised handling of station 90 filters.
11	08/30/02	Add new steps for adding water to trip blanks after initial weights and to weigh trip blanks with other samples.
12	12/14/04	"JCNNM" became "electrician," removed step to estimate timer reading in field, removed requirement to write DR on inoperative stations, no use of tweezers to handle check weights, converted HCP to HR, minor edits.
13	01/12/06	Added steps on use of new drum to mix silica gel.
0	04/02/09	Division/Group name change, new Procedure number supersedes ENV-MAQ-204, R13.
1	3/5/10	Removed steps for using blue caps on silica gel cartridges. This procedure supersedes SOP-5144, R0.
2	6/2/11	Removed steps for homogenizing silica gel in a drum prior to drying. Step was not needed.

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