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## Environmental Programs Directorate

### Standard Operating Procedure

for **AIRNET - ANALYTICAL CHEMISTRY DATA  
MANAGEMENT AND REVIEW**

#### APPROVAL SIGNATURES:

<b>Subject Matter Expert:</b>	<b>Organization</b>	<b>Signature</b>	<b>Date</b>
Karen Schultz Paige	WES-EDA	Signature on File	4/6/2009
<b>Responsible Line Manager:</b>	<b>Organization</b>	<b>Signature</b>	<b>Date</b>
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## 1.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) states the responsibilities and describes the process for receiving, uploading, and archiving analytical chemistry data; evaluating analytical chemistry quality; checking the resulting chemistry data packages for completeness and usability; and conducting validation/verification of both electronic and hardcopy data for the Los Alamos National Laboratory (LANL) Waste and Environmental Services Division (WES).

All **WES participants** shall implement this procedure when required to do the above-mentioned activities.

## 2.0 BACKGROUND AND PRECAUTIONS

### 2.1 Background

Requirements for chemical analyses are described in WES-SOP-5140, AIRNET Quality Assurance Project Plan. Data quality objectives are translated into procurement needs and related Statements of Work (SOW). Data received from all internal and external chemistry laboratories under these SOWs are uploaded electronically and inspected to determine if they meet WES specifications. This inspection includes checking the data package received from the laboratory to ensure that:

- the data package contains the components specified in statements of work,
- all of the requested analyses were performed for all samples, and
- the data are of a quality adequate for the use which WES intended.

The analytical data are evaluated to ensure usability and electronic forms are verified against hardcopy data packages, and then archived to protect their integrity. This is our overall verification and validation process (V&V). At this stage these data may be transferred to the WWW in static HTML tables.

Data are either manually entered into the Microsoft Access AIRNET (ambient air) database or uploaded from Electronic Data Deliverables specified in the SOWs. All data (100%) are verified against the hard copy to ensure exact reproduction of the analytical concentrations, and the data usability is evaluated for acceptance, qualification, or rejection. Initial gross air concentrations, net air concentrations (corrected for filter blank), bound water corrected tritium net air concentrations, and evaluation against action levels are performed and sent to the project technical reviewer, along with summaries of all analytical QC data. When documented data review and proposed actions are received back from the technical reviewer and project leader, these actions are posted to the Access databases. Ultimately, all electronic data are archived into limited-access tables to ensure their integrity. All stages of the process are tracked electronically within the AIRNET database.

### 2.2 Precautions

None

## 3.0 EQUIPMENT AND TOOLS

None

## 4.0 STEP-BY-STEP PROCESS DESCRIPTION

### 4.1 Processing and Evaluating the EDD for AIRNET Analytical Chemistry Data

#### Upload EDD:

EDDs may be received from both internal and external analytical chemistry laboratories. Format and content requirements are specified in each individual Statement of Work. Each EDD requires specific software to be incorporated into the existing databases. The uploading process is described in the Chemistry and AIRNET Database Users Guides. Upload these EDDs according to these processes as soon after receipt as practical.

#### Evaluate against SOW requirements:

After uploading data received electronically into the Chemistry database, perform referential integrity checking before moving these data into the AIRNET database. Continue the evaluation of these deliverables using software described in the AIRNET Database Users Guide to ensure that the major components are the same as those usually received or required by the SOW. When components are missing or errors are detected, contact the lab and request a revised EDD.

#### Calculate air concentrations:

When the field data and analytical chemistry data are completed for each biweekly or quarterly composite sample group, follow the steps below to run the database software that produces these reports and forward them to the technical reviewer for review of these data. The equations are in Attachment 2.

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- Worker
1. Ensure that AIRNET field data have been loaded, verified and validated, and that air volumes have been calculated (via database query) for each field record. Ensure that all analytical data have been uploaded, although the verification and validation process need not yet be complete.
  2. Run appropriate queries, using the procedures in the AIRNET Database Users Guide, to perform combined blank, initial gross air concentration, net air concentration (blank corrected), and bound water corrected tritium calculations. See the block below for more detail regarding the calculation and application of blank corrections.
  3. Perform initial QC sample evaluations using the procedures in the AIRNET Database Users Guide. See Attachment 1 for a description of the evaluation criteria employed by these queries.
  4. Prepare an internal memo that includes reports of QC evaluations performed and an overall data usability conclusion. The details on the content and preparation of this document are in the AIRNET Database Users Guide. Transmit these results to the technical reviewer for evaluation and review.
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#### Blank corrections:

Using appropriate database queries, calculate individual mean blanks for each of the following matrix types: Process Blank (PB), Trip Blank (TB), Matrix Blank (MB), and Laboratory Matrix Blank (LMB). Use professional judgment to evaluate to determine if any of these samples appears to be not representative of the true matrix blank. Flag such eliminated samples in the Archive table prior to calculating a Combined Matrix Blank (CMB) using all remaining values from the TB, MB and LMB categories (PB is already included in each of these so it is not added again). Subtract the CMB from the sample concentration PRIOR to calculating the Net Air Concentration. Propagate the standard deviation of the CMB onto the Sample uncertainty and report as the Net Air Concentration Uncertainty. Net Air Concentration units should be identical to the Air Concentration Units.

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Apply combined matrix blank correction:

When a CMB can be determined, apply it to all data (generally alpha/beta, tritium and quarterly isotopics and inorganics). When the sample result is a "less-than" value, no blank correction is appropriate and no Net Air Concentrations will be reported. In the event that the CMB is itself a "less-than" value, do not apply a blank correction and do not report a Net Air Concentration. Calculate additional uncertainty and report as Net Air Concentration uncertainty only where sample uncertainties have been reported and CMB standard deviation is > 0.

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Apply bound water correction to tritium collected on silica gel:

Atmospheric water vapor collected on silica gel and then thermally distilled for tritium analysis requires further correction. The collected water exchanges hydrogen atoms with bound water in the silica matrix, thereby effectively diluting the collected tritium. We determine the bound water content in each batch of silica gel and make a correction to the tritium net air concentrations to reflect this dilution. We call the result the "RevisedH-3 Net Air Concentration" and "RevisedH3 Net Air Concentration Uncertainty." We use these values for air dose calculations. Details are in memorandum RRES-MAQ:02-296 (Aug. 8, 2002) and in the AIRNET Database Users Guide.

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Apply decay correction to tritium spike sample results:

Three samples per period consist of a known concentration of tritiated water evaporated onto silica gel. The standard activity must be decay corrected. The current activity of the standard (AT) at any given time in years (T) will be calculated from the original activity of the standard (A0) with the following equation:  $AT = A0 * e^{-(\ln 2 / 12.32) * T}$  where 12.32 years is the half life of tritium.

## 4.2 Evaluate the AIRNET Data

Run-time requirements:

The FFCA stations, and several others, must meet 95% run-time per calendar year. Aim for 90% run time for all other stations.

Calculate the cumulative run-time for the calendar year to date using database queries for that purpose. Express as a percentage of total possible annual hours. Generate and attach this report to the technical review and QC evaluation biweekly memo for gross alpha/beta only. These queries are documented in the AIRNET Database Users Guide. Equations are in Attachment 2.

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Completeness requirements:

FFCA stations, and several others, must meet 80% annual completeness requirements. The goal is to achieve 80% sample completeness for all AIRNET stations.

Calculate the completeness for the year to date by dividing the total number of usable biweekly concentration values by the total number of sampling periods to date in the year, using database queries designed specifically for that purpose. Express as a percentage. Generate and attach this report to the HP Review and QC Evaluation bi-weekly memo for each analysis group. These queries are documented in the AIRNET Database Users Guide. Equations used are in Attachment 2.

When a sampler does not operate for the complete (biweekly) sample period, but the samples are collected and analyzed, use professional judgment to determine if the sample results are representative. Recalculate sample completeness as needed. The technical reviewer, the field team leader, and the analytical chemistry coordinator/data base manager decide jointly to reject or qualify data on these bases.

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Evaluate data package completeness:

When the final hard-copy data package arrives ensure that the major package components are the same as usual. Request the missing components from the lab.

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Custody errors:

Custody errors make it difficult to demonstrate that the samples shipped by WES were the same as those analyzed by the lab. Examples include:

- WES or lab staff not signing and dating chain-of-custody forms
- Loss or miscounting by WES or the lab
- Misidentification by WES or the lab
- Lost or incorrectly delivered

Document custody errors. Resolution will require coordination with the lab. If new analyses are necessary, ship the new samples under a new chain of custody.

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Analytical chemistry data evaluation

The data evaluation process determines whether chemical analyses data meet the data quality objectives specified in the quality plan (WES-SOP-5140). All data will be evaluated as accept, qualify, or reject. For qualified and rejected data, an explanation must be included in the database.

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Evaluate the data:

Follow the steps below to evaluate the AIRNET data:

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- Worker
1. Evaluate data for completeness. Each analytical data element should have a value.
    - For missing data, record an explanation in the database and label the record as “rejected.” If a missing datum is located, enter the correct value, label the record as “qualified” and state a reason.
    - If data errors are identified, contact the lab for a corrected report. Label data as “rejected” pending resolution with the laboratory.
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2. Look for values within the expected range. This may be based on historical ranges and/or on minimum detectable levels.
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3. If the element is outside its normal range or significantly above the required MDA, further validation and verification may be required. Consult with the lab to determine a possible cause. Study field records for suggestions of contamination. If investigation warrants, label analytical records as “qualified” by entering “Q” in the analytical data qualification field. State a reason in the comment field. Prepare and reference a separate memo if more detail is needed.
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4. If the data quality is seriously affected, then the data point may be labeled as “rejected” by entering “R” in the analytical data qualification field. State a reason in the comment field. Prepare and reference a separate memo if more detail is needed. Rejected data will not be used for any purposes.

### 4.3 Implementing Technical Review Input

The technical reviewer responsible for routine review of these data conducts review according to WES-SOP-5148 “AIRNET – Technical Evaluation of Data and Calculated Air Concentrations”, documents the outcome, and approves the data for use. Once changes in acceptance outcomes are implemented, both field data and analytical data are archived in limited access tables. At times data may be so different from normal or seem suspect based upon professional judgment. With Project Leader concurrence, confirming analyses may be requested from the analytical laboratory.

Perform the following steps to implement the recommendations and changes from the technical reviewer:

- Worker
1. After the technical reviewer returns a formal memo listing the changes to be made, implement the recommended actions in the database and document the reasons in the comment field.
  2. When both the validation and verification, and technical review process are complete, archive both field and analytical chemistry data using the procedures in the AIRNET Database Users Guide. These then become the official data..
  3. Publish fully approved data to the WES homepage using the detailed procedures documented in the AIRNET Database Users Guide.

#### 4.4 Implementing Confirming Chemical Analysis

- Worker
1. After the technical reviewer or other AIRNET project staff determines the desirability/need for confirming analyses, consult among the team to design the additional analytical request. Obtain Project Leader approval for analytical design and for pursuing negotiations with the analytical laboratory.
  2. Contact the analytical laboratory manager and negotiate costs and turnaround requirements for the confirming analyses. With Project Leader concurrence, submit these samples using the normal process. In many cases the residual portion of the samples involved will probably already be at the analytical laboratory. In that event, be sure to send the official cover letter detailing the reanalysis request.
  3. Conduct all confirming analyses as a special study.
  4. Process EDD and data packages similarly to routine samples.

#### 4.5 Post AIRNET Data

- Data Base Manager
1. Produce data reports for each open station and analyte group measured at that station using MS Internet Wizard. By selecting the proper destination subdirectory on the MS Access form, the files are published directly to the local network web page. This is described in the AIRNET Database Users Guide.
  2. Send an email to the external website webmaster saying the reports are ready to be posted to the external website. Once the reports are live, access the Active Sites Switchboard and check that the links work.
  3. Produce summary plots using MS Access and MS Excel. The plots must be moved as described in the AIRNET Database Users Guide.
  4. Follow step 3 above, substituting the local network and live versions of the Plots Switchboard for the Active Sites Switchboard.
  5. Notify AIRNET task leader that the new data and plots have been posted.

6. When new stations are opened, the webmaster adds links to the Active Sites Switchboard. The webmaster moves the links for closed stations to the Inactive Sites Switchboard.

#### 4.6 Records Management

- Worker
1. Maintain and submit records and/or documents generated to the Records Processing Facility according to EP-DIR-SOP-4004, Records Transmittal and Retrieval Process.

### 5.0 DEFINITIONS

**Statement of Work (SOW):** A list of specifications and requirements which analytical laboratories must meet in order to do work for WES.

**Data Package:** A hardcopy report from an analytical laboratory on a single set of chemical analyses, which contains the material specified in the SOW and sufficient documentation to allow an appropriate professional, at a substantially different time and location, to ascertain:

- What analyses were performed, and what results were obtained
- The data had acceptable properties (such as accuracy, precision, minimum detectable activity/ level)
- Where, when, and by whom the analyses were performed
- The analyses were done under acceptable conditions (such as calibration, control, custody, using approved procedures, and following generally approved good practices)
- The WES SOW was otherwise followed

**Completeness:** The ratio of the amount of valid data obtained from a measurement system to the amount that was expected to be obtained under ideal conditions.

**Usability:** A qualitative decision process whereby the decision-makers evaluate the achievement of data quality objectives and determine whether the data may be used for the intended purpose. Three levels or classes of data quality are used:

- Accepted: Data conform to all requirements, all quality control criteria are met, methods were followed, and documentation is complete.
- Qualified: Data conform to most, but not all, requirements, critical QC criteria are met, methods were followed or had only minor deviations, and critical documentation is complete.
- Rejected: Data do not conform to some or all requirements, critical QC criteria are not met, methods were not followed or had significant deviations, and critical documentation is missing or incomplete.

**Electronic Data Deliverable (EDD):** The computer-compatible file that is delivered to WES from the analytical laboratory, in the SOW-specified format from which analytical chemistry data may be uploaded directly into the databases.

**Validation:** A systematic process for reviewing a body of data or a report against a set of criteria to provide assurance that the data or report is adequate for their intended use. Validation consists of data reviewing, screening, checking, auditing, verification and certification.

**Verification:** The act of reviewing, inspecting, testing, checking, auditing, or otherwise determining and documenting whether items, processes, services or documents conform to specified requirements.

## 6.0 PROCESS FLOW CHART

None

## 7.0 ATTACHMENTS

Attachment 1 QC Evaluations Performed (1 page)

Attachment 2 Data Parameters (2 pages)

## 8.0 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	02/06/98	New document.
1	12/7/99	Extensive revision of process, inclusion of steps formerly in MAQ-208.
2	4/14/06	Describe blank corrections to calculated air concentrations, add formulas for calculating net air concentration and its uncertainty, and describe confirming chemical analysis process. Add calculation for decay correction of tritium spike. Update of data transfer to WWW.
0	4/8/2009	New document number and reformatted for WES division. Formerly ENV-MAQ-036.

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**ATTACHMENT 1**

**SOP-5141-1**

**QC EVALUATIONS PERFORMED**

Records Use only



Type of Data	Evaluation Performed	Acceptance Criteria
All	Laboratory Control Standard (LCS) recovery check	100 ±10%
All except Alpha/Beta	Process blank (PB)	See Control Criteria below
All	Matrix blank (MB)	See Control Criteria below
All	Trip blank (TB)	See Control Criteria below
Alpha, Beta, and H-3	Matrix replicate evaluation	For analytically significant, positive results, similar to control criteria below.
Gamma	Matrix replicate evaluation	Qualitative agreement (within a factor of 5) for analytically insignificant results (i.e. less-than values).
H-3	Matrix spike	100 ±10% of added spike
All	MDA achieved	All samples below SOW specification
All	Missing field or analytical data	No missing data for actual field samples
Tritium	Collection efficiency	Between 50 and 130 % of theoretical
Gamma	"Naturals"	All should have positive results
Gamma	"Artificials"	Compare calculated dose to 0.5 mrem target
Each bi-weekly period, reported with Z	Sampling Station run time completeness	95% up-time for FFCA and Consent Decree stations, 90% up-time for all others
All	Analytical completeness	80% successful analysis of valid samples
Alpha/Beta, H-3, Alpha Isotopics	Action level comparison	< 100% of target value

**General Control criteria:**

“Under control” is within  $\leq 2s$  of annual mean for that QC type

“Warning” is between  $2s$  and  $3s$  of annual mean for that QC type

“Out of control” is  $\geq 3s$  of annual mean for that QC type

**ATTACHMENT 2**

**SOP-5141-2**

**DATA PARAMETERS**

Records Use only



This table lists important equations used to calculate critical AIRNET data parameters. These equations are implemented in queries in the AIRNET MS Access database. The complete data evaluation process is described in SOP - 5140, SOP - 5148, and this procedure.

Parameter	Description	Equation
Alpha and beta air concentration	Average concentration of alpha and beta in the air for the sampling period.	$C (fCi/m^3) = A (pCi) * 10^3 / V (m^3)$ where A is the sample activity reported by the analytical laboratory.
Am, Pu, and U air concentration	Average concentration of Am, Pu, U in the air for the sampling period.	$C (aCi/m^3) = A (pCi) * 10^6 / [V (m^3) * (filter\ fraction)]$
Tritium air concentration	Average concentration of tritium in the air for the sampling period.	$C (pCi/m^3) = A (pCi/ml) * AH (ml/m^3)$
Absolute humidity <sup>1</sup>	Average of weekly absolute humidity (gm/m <sup>3</sup> ) measured by The Weather Machine during each week of sampling period.	$AH_1, AH_2 = \text{average humidity for } 1^{st} \text{ and } 2^{nd} \text{ weeks of period}$ $AH = (AH_1 + AH_2) / 2$
Possible run hours	Total hours within current sample year between start and stop of sample collection.	$T_{f,p}, T_{t,p} = [(\text{stop date of current sampling period in CY}) - (\text{start date of initial sampling period in CY})] * 24$ (Normally the same for both filter and gel samples.)
Actual run hours	Hours the samplers collected particles or tritium during current sample year.	$T_{f,a}, T_{t,a} = \text{Sum of } T_{f,a}, T_{t,a} \text{ over all sampling periods to date within current CY}$ (Normally the same for both filter and gel samples.)
Runtime percentage	The percent of total possible runtime that were sampled; for both the filter and tritium.	$RT_t = (T_{t,a} / T_{t,p}) * 100$ $RT_f = (T_{f,a} / T_{f,p}) * 100$ (Normally the same for both filter and gel samples.)
Actual filter air volume	Volume, in m <sup>3</sup> , of air sampled by the station.	$V_f = T_{f,a} * [(\text{start filter flow} + \text{end filter flow}) / 2] * 0.02832 (m^3/ft^3) * 60 (min/hr)$
Actual tritium air volume	Volume, in m <sup>3</sup> , of air sampled by the station.	$V_t = T_{t,a} * [(\text{start gel flow} + \text{end gel flow}) / 2] * 1e-6 (m^3/cc)$
Sample completeness	The percent of total possible samples taken that were analyzed successfully.	$C\% = \text{Sum of (number of sample data by analysis reported from lab)} / (\text{number of sampling periods in current sampling year})$

Parameter	Description	Equation
Mean Process Blank (PB)	Mean of all individual process blanks reported by the analytical laboratory for each analysis	$PB \text{ (mean)} = \text{Mean of PB} \pm \text{StDev of PB}$
Mean Trip Blank (TB)	Mean of all individual trip blanks reported by the analytical laboratory for each analysis and not rejected.	$TB \text{ (mean)} = \text{Mean of TB} \pm \text{StDev of TB}$
Mean Matrix Blank (MB)	Mean of all individual matrix blanks sent with sample batch from WES, reported by the analytical laboratory for each analysis and not rejected.	$MB \text{ (mean)} = \text{Mean of MB} \pm \text{StDev of MB}$
Mean Laboratory Matrix Blank (LMB)	Mean of all individual laboratory matrix blanks (filter material sent from WES in bulk and kept at analytical laboratory), reported by the analytical laboratory for each analysis and not rejected.	$LMB \text{ (mean)} = \text{Mean of LMB} \pm \text{StDev of LMB}$
Combined Matrix Blank (CMB)	Mean of individual TB, MB and LMB that have not been rejected.	$CMB \text{ (mean)} = \text{Mean of (LMB, MB and TB)} \pm \text{StDev of (LMB, MB and TB)}$
Gel Water Content (WC)	Mass (in g) of water bound in silica gel.	$WC = \text{final silica gel weight} - \text{initial silica gel weight}$
Net Air Concentration	Air concentration calculated from blank corrected results for filter samples.	$NAC_f = (A \text{ (pCi)} - CMB)/([\text{filter air volume}] * [\text{filter fraction}])$
Net Air Concentration (tritium only)	Air concentration calculated from blank corrected results for tritium samples.	$NAC_t = (A \text{ (pCi/ml)} - CMB) * AH$
Net Air Concentration Uncertainty	Propagated uncertainty calculated from uncertainty of result and uncertainty of CMB used to blank correct result for filter samples.	$NACU_f = \text{Square root (squared sample result uncertainty + squared } [2 * \text{StDev of CMB}]) / ([\text{filter air volume}] * [\text{filter fraction}])$
Net Air Concentration Uncertainty (trit)	Propagated uncertainty calculated from uncertainty of result and uncertainty of CMB used to blank correct result for tritium samples.	$NACU_t = \text{Square root (squared sample result uncertainty + squared } [2 * \text{StDev of CMB}]) * AH$
Revised H3 Net Air Concentration	Bound-water corrected Net Air concentration <sup>2</sup>	$RevNAC_t = NAC_t * 1.03 * ((WC + [\text{bound water in silica gel (g)}]) / WC)$
Revised H3 Net Air Concentration Uncertainty	Bound-water corrected Net Air concentration uncertainty (NO propagation of additional uncertainty from bound water measurement) <sup>2</sup>	$RevNACUnc_t = NACUnc_t * 1.03 * ((WC + [\text{bound water in silica gel (g)}]) / WC)$

Equation subscripts: f = filter; t = tritium; a = actual; p = possible

<sup>1</sup> AH calculations are documented in memo ESH-17:99-104.

<sup>2</sup> Constants are defined and bound water determination method is described in memo ESH-17:02-296.