ANALYTICAL CHEMISTRY DATA MANAGEMENT AND REVIEW FOR SOIL, FOODSTUFFS AND BIOTA

Purpose This Meteorology and Air Quality Group (MAQ) procedure 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 from current sources.

Scope This procedure applies to the analytical chemistry coordinator assigned to evaluate Soil, Foodstuffs, and Biota analytical data.

In this procedure

Signatures

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01/03/06

General information about this procedure

Attachments   This procedure has the following attachments:

Number	Attachment Title	No. of pages
1	Checklist for Completeness of Data Package	1
2	Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Database Checklist	1

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

Revision	Date	Description of Changes
0	3/29/04	New document.
1	01/05/06	Revised to update checklists and reflect new database structure.

Who requires training to this procedure?   The following MAQ personnel require training before implementing this procedure:
- Analytical chemistry data reviewers
- Analytical Chemistry Coordinator

Training method   The initial training method for this procedure is **mentored** 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.

General information, continued

Prerequisites In addition to training to this procedure, the following training is also recommended prior to performing this procedure:
- Education and/or experience in compliance/surveillance-oriented analytical chemistry
- Familiarity with Microsoft Access
- Familiarity with the operation of the Soil, Foodstuffs, and Biota database

Definitions specific to this procedure

Statement of Work (SOW): A list of specifications and requirements which analytical laboratories must meet in order to do work for MAQ (prepared according to MAQ-036).

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,
- that the data had acceptable properties (such as accuracy, precision, MDA),
- where, when, and by whom the analyses were performed,
- that the analyses were done under acceptable conditions (such as calibration, control, custody, using approved procedures, and following generally approved good practices), and
- that the MAQ SOW was otherwise followed.

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.

General information, continued

Definitions,
continued

- Rejected: Data do not conform to some or all requirements, critical QC criteria are not met, methods were not followed or had significant deviations, or critical documentation is missing or incomplete. The results are unusable.

Electronic Data Deliverable (EDD): The computer-compatible file that is delivered to ENV-MAQ from the analytical laboratory, in the SOW-specified format, via Internet, e-mail, or diskette 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, 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 and are correctly represented.

References

The following documents are referenced in this procedure:
- MAQ-024, "Personnel Training"
- MAQ-026, "Deficiency Reporting and Correcting"
- MAQ-036, "Preparing Statements of Work for Analytical Chemistry"
- MAQ-SFB, "Quality Assurance Project Plan for the Soil, Foodstuffs, and Biota Monitoring Project"

Note

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

Background

Description of data upload and verification

Requirements for chemical analyses are described in the data quality objectives (DQO) section of the Quality Assurance Project Plan for the Soil, Foodstuffs, and Biota Monitoring Project (MAQ-SFB). Data quality objectives are translated into procurement needs and related Statements of Work (SOW) according to MAQ-036. Data received from all internal and external chemistry laboratories under these SOWs are uploaded electronically and inspected to determine if they meet MAQ specifications. This inspection includes checking the data package against checklists to ensure that:

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

All data are verified (100%) against the hard copy to ensure exact reproduction of all the analytical concentrations. The analytical data are evaluated to ensure usability. The electronic forms are archived to protect their integrity.

Description of validation

Following verification of the EDD results with the hard copy data package, the analytical results are validated by a chemist with at least two years experience in data validation or laboratory analysis. Validation procedures are loosely based on the National Functional Guidelines for Data Review provided by the U.S. Environmental Protection Agency for the Contract Laboratory Program. The validation procedures follow the Standard Operating Procedures for Data Validation of data for the ENV-ECR program. Currently only SOP-15.05, R1, ICN1 Routine Validation of Inorganic Data, SOP-15.06, R1, ICN1 Routine Validation of Gamma Spectroscopy Data, and SOP-15.07, R1, ICN1 Routine Validation of Chemical Separation Alpha Spectrometry, Gas Proportional Counting, and Liquid Scintillation Data have been incorporated into the data base. More SOPs may be added to the database as needed.

Validation procedures determine if the analytical data quality has been compromised during analysis and if so, how that affects the end use of the data. The outcome can vary. One outcome can be no effect on data quality, in which case no qualifier is applied to the data. One outcome can be that data has an increased uncertainty associated with the value, in which case a qualifier of J, J+, or J- may be applied to the data. The latter two qualifiers indicate that the error associated with the value is biased to the high side of the value or to the low side of the value. Finally, the outcome could be data quality has been seriously compromised by an analytical problem and the data are unusable, in which case the value is rejected.

Background, continued

Description of technical review validation

With the exception of soils (the soils are reported on a dry weight basis), all foodstuffs and biota are analyzed and reported on an ash weight basis and are converted to a dry weight basis for reporting purposes. Data will be compared to

- determine whether an on-site or perimeter value is detectable (when the result is higher than the minimum detectable concentration and greater than three times the analytical uncertainty) or nondetectable, and
- determine whether a detectable result is greater than the regional statistical reference level (RSRL) (the RSRL is based on the average and standard deviation of the last five years of regional data).

The technical reviewer determines whether a detectable soil result is greater than the screening action level (SAL) and the results 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 Soil, Foodstuffs, and Biota database.

Processing the EDD for Soil, Foodstuffs, and Biota analytical chemistry data

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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 prepared according to MAQ-036. Each EDD requires specific software to enable it to be incorporated into the existing databases. The **analytical chemistry coordinator** uploads and processes these EDDs according to these detailed processes as soon after receipt as practical. The EDD must be both uploaded and processed prior to verification. The data base will indicate if the upload was successful or if fatal errors requiring correction were detected in the EDD. Successful upload of the EDD will result in the chain of custody number for that EDD appearing in the list of data waiting to be verified.

Prepare checklist for deliverables When new services are procured, prepare a checklist as needed to evaluate the completeness of any deliverables. See the chapter "Preparing checklists for deliverables" in MAQ-036.

Resolution When expected components are missing or significant errors are detected, the **database administrator** or the **project leader** contacts the lab immediately and requests that a revised EDD be sent expeditiously. Also document the problem by preparing a deficiency report according to MAQ-026.

Data Verification

Evaluate data package completeness against SOW requirements and completeness checklist

After uploading data received electronically and upon receipt of the hard-copy data package, the **analytical chemistry coordinator** evaluates these deliverables using software to ensure that the major components are the same as those usually received or required by the SOW. These components are listed in Attachment 1. This step constitutes the first part of the data verification process.

Verify analytical data against hard-copy data

Having received the final hard-copy data package, the electronic data uploaded into the data base must be 100% verified against the hard copy reporting of the data. The **analytical chemistry coordinator** must do this prior to data validation. This constitutes the last part of the data verification process.

Resolution

When expected components are missing or analytical results are discrepant, contact the lab immediately and request that the missing components or corrections be sent expeditiously. Also document the problem by preparing a deficiency report according to MAQ-026.

Custody errors

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

- MAQ or lab staff not signing and dating chain of custody forms
- Loss or miscounting by MAQ or the lab
- Misidentifying by MAQ or the lab
- Lost samples
- Delivery to the wrong site or person

Document all custody errors with an MAQ Deficiency Report (MAQ-026). Resolution will require coordination with the lab. If new analyses are necessary, ship the new samples under a new chain of custody.

Data Validation

Purpose of analytical chemistry data validation

The data validation process determines whether data meet the data quality objectives specified in the quality plan (MAQ-SFB). All data will be evaluated for one of three outcomes: *accept*, *qualify*, or *reject* (see definitions). For qualified and rejected data, an explanation must be included in the database. This explanation occurs in the form of a reason code which can be linked to a standardized reason listed in the appropriate SOP.

Steps to validate data

Follow the steps below to validate the Soil, Foodstuffs, and Biota data:

Step	Action
1	Select a chain of custody number for data that has been successfully uploaded and verified. Do not validate any data that has not been verified.
2	Several queries have been written in the database that allow auto-validation where possible (e.g. comparing a detectable analytical sample result to any detected quantities in the blank sample, thus qualifying the analytical result as being blank contamination.) Run through all the queries to allow as complete auto-validation of the data as possible.
3	Once the data has been thoroughly auto-validated, the validation is checked by the **analytical chemistry coordinator** and any missing validation qualifiers are added while incorrect qualifications are removed. In all cases, many qualifiers can be applied to an analytical result but the first qualifier applied will be the only qualifier reported to the end user. The **analytical chemistry coordinator** must select the problem that most significantly affects the data and apply that qualifier first. The hierarchy of data quality parameters and their impact on the data is listed in the document "Hierarchy of LANL Final Reporting Qualifier/Reason Codes" (ERID 2005-91000). Once the data qualifiers are applied appropriately, check the button to accept the data validation and permanently record the final data validation results in the database.
4	Once data validation is completed, the **analytical chemistry coordinator** records any data qualifiers and their reasons on the hard copy analytical data result sheets (also called Form 1s). Print out a V&V report (which also lists these qualifiers) and submit it with the data package to the SFB project team leader or other end user of the data. In addition, complete and sign the Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Confirmation sheet (Attachment 2) and submit with the V&V report. This completes the data validation process.

Technical review of data

Run custom queries for data evaluation

Upon request of the project leader or technical reviewer for FSB data, develop and run custom queries of the database to automate the checking and calculation of the data against technical evaluation criteria.

Technical review

The **technical reviewer** responsible for routine review of these data conducts a review for the following:

- Data are properly converted to a dry weight basis or are otherwise properly converted to needed units.
- Determine whether an on-site or perimeter value is detectable (when the result is higher than the minimum detectable concentration and greater than three (3) times the analytical uncertainty).
- Determine whether a detectable result is greater than the regional statistical reference level (RSRL) (the RSRL is based on the average and standard deviation of the last five years of regional data).
- For soils, determine whether a detectable result is greater than the screening action level (SAL).
- Determine which data points may need to be qualified or rejected based on field collection data or other information.

Document needed changes and forward for implementation

If changes to the database are needed, the **reviewer** documents the needed changes in a memo or form and forwards the form to the **analytical chemistry coordinator** for implementation in the database.

Records resulting from this procedure

Records   The following records generated as a result of this procedure are to be submitted as records to the records coordinator:

- Soil, Foodstuffs, and Biota Field Data Validation and Verification Database inspection form; completed, signed and dated.
- Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Database Inspection form; completed, signed, and dated.
- Soil, Foodstuffs, and Biota Analytical Data Report from the data base listing all analytical results and qualifiers, if any
- Copy of final laboratory data package
- Deficiency reports resulting from chain-of-custody problems, if any
- MAQ internal memos documenting data quality evaluation, data validation, and initial concentration calculations

The following electronic records generated as a result of this procedure are to be contained within their respective Microsoft Access databases:

- entries in Soil, Foodstuffs, and Biota database for all accepted, qualified, and rejected data from both field and analytical processes.

Using a CRYPTOCard, click here to record "self-study" training to this procedure.
If you do not possess a CRYPTOCard or encounter problems, contact the ERSS training specialist.

ATTACHMENT 1

Meteorology and Air Quality Group **Checklist for Completeness of Data Package** This form is from MAQ-712

Soil, Foodstuffs, and Biota Sample Group #:_____

Inspection Criterion	**Criter met?**	**Comments**
Was analytical lab required to work to the above-listed standard by contract?	Y N NA	
Was an acceptable EDD received within 14 days of lab receipt of samples	Y N NA	
Final Data package received within 30 days of sample arrival at analytical lab?	Y N NA	Date sub: Date rcd:
Each page of each data package sequentially numbered.	Y N NA	
Narrative comments on the analysis of each sample group in cover letter or memo?	Y N NA	
Positive sample id in all tables and reports.	Y N NA	
Positive indication of signatures/initials at each work and review stage.	Y N NA	
Data received for each sample on C-of-C.	Y N NA	
Summary of sample results (to include customer id, sample delivery group or request number, lab id, isotope/analysis, analyte concentration, analyte uncertainty and MDA in the same appropriate units, counting times, and dates of analysis); an individual summary provided for each sample.	Y N NA	
Individual summary of each QA/QC sample (same parameters as sample results); QA/QC samples will include, at a minimum of one each of the following for every 20 field samples: a Laboratory Control Sample (LCS), a detector blank, a matrix blank and a matrix spike.	Y N NA	
Known values for all QA/QC samples?	Y N NA	
Individual sample raw data and individual spectral plots showing regions of interest (ROI) integrated for each gamma isotope.	Y N NA	
Individual QA/QC raw data and individual spectral plots showing ROI integrated for each isotope.	Y N NA	
Individual detector efficiencies and backgrounds.	Y N NA	
Laboratory bench sheets with sample of any manual calculations done.	Y N NA	
Evidence of NIST-traceable calibration standards.	Y N NA	
Copies of the most recent applicable MDA study results, initial calibration and recalibration.	Y N NA	
Chain of custody form.	Y N NA	
All equations used to calculate MDAs or sample results either in datapackage or published analytical procedures.	Y N NA	
Actual concentrations include negative values, rather than some form of "not detected" (less-thans are permitted).	Y N NA	
Uncertainties (identified appropriately as 1, 2, or 3 sigma in the final data package).	Y N NA	

Verified by: _____   Date: _____

ATTACHMENT 2

Meteorology and Air Quality Group
Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Confirmation

This form is from MAQ-712

This form serves as a hard copy record confirming that the verification and validation of data in the Soil, Foodstuffs, and Biota data base has been carried out according to the most recent revision of MAQ-712.

Chain of Custody validated and verified: _____

Project Name: _____

Analytical Laboratory: _____Paragon Analytics_____

Analytical Laboratory Work Order #: _____

Suite(s) validated and verified in the above Chain of Custody:

Isotopic Plutonium   Isotopic Uranium

Gamma Spectroscopy   Tritium

Strontium-90   Inorganics

Isotopic Americium   PCBs

Date of validation: _____

Validator: (name and Z number) ___Karen Schultz Paige_____111256_____

Validator's signature and date: _____

As project leader, I indicate with my signature that I have reviewed this validation and verification report and approve of its content.

Project Leader: (name and Z number) _____Phil Fresquez___084582___

Project Leader's signature and date: _____