10.4.5 Sample, Storage, and Tracking

In the field, samples may be stored temporarily in coolers with wet or dry ice (as appropriate). Security should be maintained and documentation of proper storage should be provided in the project field note book. Samples stored temporarily in coolers should be transported to a storage facility as soon as logistically possible. When possible, samples will be shipped directly to the appropriate laboratories from the field.

Prior to analysis, samples will be stored under appropriate conditions at the storage facility or laboratory (refrigerator or freezer). Security should be maintained at all times. A log book or inventory record typically is maintained for each sample storage facility refrigerator or freezer. The log books or inventory records are used to document sample movement in and out of the facility. In general, samples will be placed into a freezer and information regarding sample identification, matrix, and study will be recorded. Additional information in the record for each sample may include: (1) the date of the initial storage, (2) subsequent removal/return events with associated dates, and (3) initials of person(s) handling the samples. Additional information may include study name and special comments.

Documentation should allow for unambiguous tracking of the samples from the time of collection until shipment to the laboratory. The tracking system should include a record of all sample movement and provide identification and verification (initials) of the individuals responsible for the movement.

10.5 SAMPLE CUSTODY

Chain-of-custody (COC) procedures are adopted for samples throughout the field collection, handling, storage, and shipment process. Each individual sample will be assigned a unique identification label and have a separate entry on a COC record. A COC record should accompany every sample and every shipment to document sample possession from the time of collection through final disposal.

10.5.1 Definition of Custody

A sample is defined as being in a person's custody if one of the following conditions applies:

- The sample is in the person's actual possession or view.
- The sample was in the person's possession and then was locked in a secure area with restricted access.

- The person placed it in a container and sealed the container with a custody seal in such a way that it cannot be opened without breaking the seal.

10.5.2 Procedures

The following information typically will be included on COC forms:

- place of collection
- laboratory name and address
- sample receipt information (total number of containers; whether COC seals are intact; whether sample containers are intact; and whether the samples are cold when received)
- signature block with sufficient room for "relinquished by" and "received by" signatures for at least three groups (field sampler, intermediate handler, and lab)
- sample information (field sample identifier, date, time, matrix, lab sample identifier, and number of containers for that sample identifier)
- the name of the sampler
- airbill number of overnight carrier (if applicable)
- disposal information (to track sample from "cradle to grave")
- a block for special instructions
- analysis request information.

The sample identification, date and time of collection, and request for analysis on the sample label should correspond to the entries on the chain-of-custody form and in associated field log books or sampling forms.

The data quality manager or designated representative is responsible for reviewing the completed COC forms. Any inconsistencies, inaccuracies, or incompleteness in completing the forms must be brought to the attention of the field staff completing the form. If the problem is significant, corrective action should be taken and documented. Depending on the problem, this may involve informing the lab that a sample ID or analysis request needs to be changed, or notifying the Field Team Leader that retraining of field staff in COC procedures is indicated. The corrective action and its outcome should be documented.

10.6 ANALYTICAL PROCEDURES

A number of analytical methods or procedures may be used, including: quantification of Aroclors using Method 8081; quantification of Total PCBs using Method 8081; or quantification of PCB congeners and co-planars using gas chromatography with electron capture detection (GC/ECD) and/or gas chromatography with mass spectrophotometry (GC/MS). Co-planar PCB congeners may be analyzed and reported with the PCB congener analysis. Preconcentration steps (e.g., carbon column cleanup) may be required to obtain adequate detection limits for these compounds. General QC considerations and targets for analyses are described below, along with considerations for biological testing.

Laboratory method detection limit (MDL) studies should be conducted for each matrix per analytical method, according to specifications described in 40 CFR Part 136 or other comparable professionally accepted standards. The MDLs for each target analyte should be less than or equal to the required screening levels. The MDL is a statistically-derived, empirical value that may vary.

Laboratory QC samples, which include a method blank, replicate (matrix spike or duplicate) analyses, laboratory control sample, and a standard reference material (SRM), will be performed at a target frequency of one per twenty samples per matrix per analytical batch. Method blanks should be free of contamination of target analytes at concentrations greater than or equal to the MDL; or associated sample concentrations should be greater than 10 times the method blank values. The matrix spike/matrix spike duplicate and laboratory control sample analyses should meet accuracy and precision goals.

10.7 CALIBRATION PROCEDURES AND FREQUENCY

This section provides information on general calibration guidelines for laboratory and field methods.

10.7.1 Laboratory Equipment

All equipment and instruments used for laboratory analyses will be operated and maintained according to the manufacturer's recommendations, as well as by criteria defined in the laboratory's SOPs. Operation, maintenance, and calibration should be performed by personnel properly trained in these procedures. Documentation of all routine and special maintenance and calibration information should be recorded in appropriate log books and reference files.

Calibration curve requirements for all analytes and surrogate compounds should be met prior to sample analysis. Calibration verification standards, which should include the analytes that are

expected to be in the samples and the surrogate compounds, should be analyzed at a specified frequency and be within a percent difference or percent drift criterion.

10.7.2 Field Equipment

All equipment and instruments used to collect field measurements will be operated, maintained, and calibrated according to the manufacturer's recommendations, as well as by criteria defined in individual SOPs. Operation, calibration, and maintenance should be performed by personnel properly trained in these procedures. Documentation of all routine and special maintenance and calibration information should be recorded in appropriate log books or reference files. Field instruments that may be used include thermometers/ temperature probes, scales, pH meters, dissolved oxygen meters, and global positioning system units.

10.8 DATA REDUCTION, VALIDATION, AND REPORTING

10.8.1 General Approach

Data generated by the laboratory and during field measurements may undergo data review and validation by an External QA Reviewer. Laboratory data may be evaluated for compliance with data quality objectives, with functional guidelines for data validation, and with procedural requirements contained in this QAPjP.

10.8.2 Data Reporting

Laboratories should provide sufficient information to allow for independent validation of the sample identity and integrity, the laboratory measurement system, the resulting quantitative and qualitative raw data and all information relating to standards and sample preparation.

10.8.3 Data Review and Validation of Chemistry Data

Data review is an internal laboratory process in which data are reviewed and evaluated by laboratory supervisory or QA personnel. Data validation is an independent review process conducted by personnel not associated with data collection and generation activities. External and independent data validation may be performed for selected sample sets as determined by the PM and Data Quality Manager. Each data package chosen for review will be assessed to determine whether the required documentation is of known and documented quality. This includes evaluating whether:

- field chain-of-custody or project catalog records are present, complete, signed and dated
- the laboratory data report contains required deliverables to document procedures.

Two levels of data validation may be performed: full or cursory validation. Initial data packages received for each sample matrix may receive full validation. This consists of a review of the entire data package for compliance with documentation and quality control criteria for the following items:

- analytical holding times
- data package completeness
- preparation and calibration blank contamination
- initial and continuing calibration verifications
- internal standards
- instrument tuning standards
- analytical accuracy (matrix spike recoveries, laboratory control sample recoveries)
- analytical precision (comparison of replicate sample results)
- reported detection limits and compound quantitation
- review of raw data and other aspects of instrument performance
- review of preparation and analysis bench sheets and run logs.

Cursory validation may be performed on a subset of the data packages, at the discretion of the PM and Data Quality Manager. Cursory review includes the comparison of laboratory summarized QC and instrument performance standard results to the required control limits, including:

- analytical holding times
- data package completeness
- preparation and calibration blank contamination
- analytical accuracy (matrix spike recoveries, laboratory control sample recoveries)
- analytical precision (comparison of replicate sample results).

The full or cursory validation will follow documented quality control and review procedures as outlined in guidelines for data validation (U.S. EPA, 1993b) and documented in validation and method SOPs. Various qualifiers and/or comments or narratives may be applied to data during the validation process. These qualifier codes may be assigned to individual data points to explain deviations from quality control criteria and will not replace qualifiers or footnotes provided by the laboratory. Data validation reports summarizing findings will be submitted to the Data Quality Manager for review and approval.

Laboratory data will be evaluated for compliance with data quality objectives. Data useability, from an analytical standpoint, may be evaluated during the data evaluation. The data users (the Principal Investigator, PM, AM) will determine the ultimate useability of the data.

10.9 PERFORMANCE AND SYSTEM AUDITS

A Data Quality Manager or designee will be responsible for coordinating and implementing any QA audits that may be performed. Checklists may be prepared that reflect the system or components being audited, with references to source of questions or items on the checklist. Records of all audits and corrective actions should be maintained in the project files.

10.9.1 Technical System Audits

Technical System Audits (TSAs) are qualitative evaluations of components of field and laboratory measurement systems, including quality control procedures, technical personnel, and QA management. TSAs determine if the measurement systems are being used appropriately. TSAs are normally performed before or shortly after measurement systems are operational, and during the program on a regularly scheduled basis. TSAs involve a comparison of the activities described in the study plan and SOPs with those actually scheduled or performed. Coordination and implementation of any TSAs will be the responsibility of a Data Quality Manager or designee.

Analytical Data Generation (Laboratory Audit)

Laboratory audits may be performed to determine whether the laboratory is generating data according to all processes and procedures documented in associated project plans, QAPjP, SOPs, and analytical methods. Laboratory audits can be performed by an External QA Reviewer, a Data Quality Manager, or their designee.

Field Audits

Field Audits may be performed to determine whether field operations and sample collection is being performed according to processes and procedures documented in the study plan, QAPjP, and SOPs.

10.9.2 Performance Evaluation Audits

Performance Evaluation Audits are quantitative evaluations of the measurement systems of a program. Performance Evaluation Audits involve testing measurement systems with samples of known composition or behavior to evaluate precision and accuracy typically through the analysis of standard reference materials.

10.10 PREVENTATIVE MAINTENANCE PROCEDURES AND SCHEDULES

Preventative maintenance typically is implemented on a scheduled basis to minimize equipment failure and poor performance. In addition to scheduled calibration procedures described above, the following procedures may be followed:

- Thoroughly clean field equipment before returning to the office. The equipment generally should be stored clean and dry.
- Replaceable components, such as pH electrodes and dissolved oxygen membranes, should be inspected after and before each use, and replaced as needed to maintain acceptable performance.
- Equipment that is identified to be malfunctioning or out-of-calibration will be removed from operation until repaired or re-calibrated.

10.11 PROCEDURES USED TO ASSESS DATA USEABILITY

Data useability ultimately is a function of study methods, investigator expertise and competence, and intended uses. QA/QC procedures are designed to help ensure data useability but, in themselves, neither assure data useability nor — if not implemented — indicate that data are not useable or valid. Data validity and useability will ultimately be determined by the Principal Investigator, PM, and AM using best professional judgment. Independent data validation, consultations with Data Quality Managers, and review of project-wide databases for data compatibility and consistency can be used to support useability evaluations. The useability and validity of existing and historical data, which were not collected pursuant to the QAPjP presented in this Assessment Plan, will be determined by the AM, PM, Principal Investigators, and Trustee technical staff using best professional judgment.

10.12 CORRECTIVE ACTIONS

10.12.1 Definition

Corrective actions consist of the procedures and processes necessary to correct and/or document situations where data quality and or QA procedures fall outside of acceptance criteria or targets. (These criteria/targets may be numeric goals such as those discussed in Section 10.3, or procedural requirements such as those presented throughout the QAPjP and other project documents (e.g., SOPs)).

The goal of corrective action is to identify as early as possible a data quality problem and to eliminate or limit its impact on data quality. The corrective action information typically is provided to a Data Quality Manager for use in data assessment and long term quality management. Corrective action typically involves the following steps:

1. discovery of a nonconformance or deviations from data quality objectives or this plan
2. identification of the party with authority to correct the problem
3. planning and scheduling of appropriate corrective action
4. confirming that the corrective action produced the desired result
5. documenting the corrective action.

10.12.2 Discovery of Nonconformance

The initial responsibility of identifying nonconformance with procedures and QC criteria lies with the field personnel and bench-level analysts. Performance and system audits are also designed to detect these problems. However, anyone who identifies a problem or potential problem should initiate the corrective action process by, at least, notifying a Principal Investigator or Data Quality Manager of his/her concern.

Deviations from QAPjP or SOP procedures are sometimes required and appropriate due to field or sample conditions. Such deviations should be noted in field or laboratory logbooks and their effect on data quality evaluated by a Principal Investigator and Data Quality Manager. Occasionally, procedural changes are made during the course of an investigation because method improvements are identified and implemented. Even though these procedural improvements are not initiated due to nonconformance, they are procedural deviations and typically should be documented.

10.12.3 Planning, Scheduling, and Implementing Corrective Action

Appropriate corrective actions for routine problems depend on the situation and may range from documentation of the problem, to resampling and reanalysis, to the development of new methods. When the corrective action is within the scope of these potential actions, the bench-level analyst or the field staff can identify the appropriate corrective action and implement it. Otherwise, the corrective action should be identified and selected by the PM, the Field Team Leader, the Laboratory Manager, or the Data Quality Manager.

10.12.4 Confirmation of the Result

While a corrective action is being implemented, additional work dependent on the nonconforming data should not be performed. When the corrective action is complete, the situation should be evaluated to determine if the problem was corrected. If not, new corrective actions should be taken until no further action is warranted, either because the problem is now corrected or because no successful corrective action has been found.

10.12.5 Documentation and Reporting

Corrective action documentation may consist of the following reports or forms:

- Corrective action forms initiated by project staff. These forms will be collected, evaluated, and filed by the Data Quality Manager.
- Corrective action log maintained by the Data Quality Manager in order to track the types of nonconformance problems encountered and to track successful completion of corrective actions.
- Corrective action plans, if needed to address major nonconformance issues.
- Performance and systems audit reports, if such audits are performed.
- Corrective action narratives included as part of data reports from independent laboratories.
- Corrective action forms initiated by laboratory staff and summarized in the report narrative.

10.12.6 Laboratory-Specific Corrective Action

The need for corrective action in the analytical laboratory may come from several sources: equipment malfunction, failure of internal QA/QC checks, method blank contamination, or failure of performance or system audits; and/or noncompliance with QA requirements.

When measurement equipment or analytical methods fail QA/QC checks, the problem should immediately be brought to the attention of the appropriate laboratory supervisor in accordance with the laboratory's SOP or Quality Assurance Manual. If failure is due to equipment malfunction, the equipment should be repaired, precision and accuracy be reassessed, and the analysis rerun.

All incidents of QA failure and the corrective action tasks should be documented, and reports should be placed in the appropriate project file. Corrective action should also be taken promptly for deficiencies noted during spot-checks of raw data. As soon as sufficient time has elapsed for corrective action to be implemented, evidence of correction of deficiencies should be presented to a Data Quality Manager or PI.

Laboratory corrective actions may include, but are not limited to:

- reanalyzing the samples, if holding time criteria permits and sample volume is available
- resampling and analyzing
- evaluating and amending sampling analytical procedures
- accepting data and acknowledging the level of uncertainty.

CHAPTER 11
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