8.0 Sample Handling and Custody

A critical activity within any data collection phase involving physical samples is the handling of sample media prior to sampling, handling/transporting sample media to the field, handling samples from the field at the time of collection, storage of samples (at field or other locations), transport of samples from the field site, and the analysis of the samples. Documentation ensuring that proper handling has occurred throughout these activities is part of the custody record, which provides a mechanism for tracking samples through sample collection, processing and analysis. Custody records document the "chain of custody"; the date and person responsible for the various sample handling steps associated with each sample. Custody records also provide a reviewable trail for quality assurance purposes and as evidence in legal proceedings.

Prior to the start of an EDO, the various types of samples should be identified and the following questions asked:

- Does the sample need to be analyzed within a specified time period?
- What modes of sample transport are necessary and how secure should they be?
- What happens if a sample is collected on Friday? Is the sample shipped or stored at the field office and what are the procedures?
- Can the sample's integrity be affected by outside influences (e.g. temperature, pressure, humidity, jostling/dropping during shipment, other influences) and do these need to be monitored (e.g., max/min thermometers, pressure sensors)?
- How critical is it that sample integrity be known (e.g., is evidence tape necessary)?
- How can it be documented that sample integrity was maintained from the collection to reporting?
- What are the procedures when sample integrity is compromised (e.g., flag, don't analyze)?

These are some of the questions that should be answered and documented in the monitoring organization's QAPP and SOPs.

This section specifically addresses the handling and custody of physical environmental samples (e.g., exposed filters for particulate matter (PM) determinations and canisters containing whole air samples) that are collected at a field location and transported to a laboratory for analysis. For specific details of sample handling and custody (i.e., PAMS, NATTS, STN etc) monitoring organization should consult the appropriate technical assistance documents located in the National Programs summaries in Appendix A.

In addition to physical samples, some types of field data collected in hard copy (e.g., strip charts, sampler flow data, etc.) or electronic (e.g., data downloaded from a data logger with limited storage space) format are irreplaceable and represent primary information about physical samples or on-site measurements that are needed to report a final result. When such hard copy or electronic data are transported and/or change custody, it is advised that the same chain of custody practices described in this section for physical samples be employed to ensure that irreplaceable data can be tracked and are not altered or tampered with.

For additional information, an EPA on-line self-instructional course, "*Chain-of-Custody Procedures for Samples and Data*[1]" is available for review. The National Enforcement Investigation Center[2] (NEIC) also offers a course relevant to chain of custody issues.

Comment [ALS1]: It sounded to me like Mr. Shanis had a specific course in mind, but I wasn't able to find one on chain of custody.

[1] http://www.epa.gov/apti/coc/
[2] http://www.epa.gov/compliance/about/offices/division/neic.html

Laboratory Information Management Systems

A laboratory information management system or LIMS, is a computer system used in the laboratory for the management and tracking of samples, instruments, standards and other laboratory functions such as data reductions, data transfer and reporting. The goal is to create an EDO where:

- Instruments used are integrated in the lab network; receive instructions and worklists from the LIMS and return finished results including raw data back to a central repository where the LIMS can update relevant information to external systems (i.e., AIRNow or AQS).
- Lab personnel will perform calculations, documentation and review results using online information from connected instruments, reference databases and other resources using electronic lab notebooks connected to the LIMS.
- Management can supervise the lab process, react to bottlenecks in workflow and ensure regulatory demands are met.
- External participants can review results and print out analysis certificates and other documentation (QA Reports, quality control charts, outlier reports etc.).

For monitoring programs that are fairly stable, such as criteria pollutant monitoring, development of a LIMS system may be very cost effective and should be considered. There is an upfront cost in the development of these systems but monitoring organizations that have devoted resources to their development have seen pay offs in improved data quality, sample tracking and data reporting.

8.1 Sample Handling

In the Ambient Air Quality Monitoring Program, discrete samples from manual methods associated with SLAMS, PAMS, NATTS, and other networks, are physically handled prior to analysis. One must pay particular attention to the handling of filters for particulate matter and lead since it has been suggested that the process of filter handling may be the largest source of measurement error (especially low-volume methods). Due to the manner in which concentrations are determined, it is critical that samples are handled as specified in SOPs. The various phases of sample handling that should be documented in a QAPP and SOP include:

- Sample preparation, labeling and identification;
- sample collection;
- transportation;
- sample analysis; and
- storage and archival

8.1.1 Sample Preparation, Labeling and Identification

Sample containers or filters are cleaned and prepared (pre-weighing of filters) before being used to collect samples. SOPs should indicate the proper care and handling of the containers/filters to ensure their integrity. Proper lab documentation that tracks the disposition of containers/filters through preparation is just as important as the documentation after sampling. Care must be taken to properly mark all samples to ensure positive, unambiguous identification throughout the sample collection, handling, and analysis procedures. Figure 8.1 shows a standardized identification sticker that may be used to label physical samples. Additional information may be added as required, depending on the particular monitoring

program. The rules of evidence used in legal proceedings require that procedures for identification of samples used in analyses form the basis for future evidence. An admission by the laboratory analyst that he/she cannot be positive whether he/she analyzed sample No. 6 or sample No. 9, for example, could destroy the validity of the entire test report. Any information that can be used to assess sample integrity, such as the pressure of canisters or liquid level, should be recorded at the time of sample collection. Liquid levels for samples in non-graduated containers can be marked on the side of the container with a grease pencil or permanent marker.

Positive identification also must be provided for any filters used in the program. If ink is used for marking, it must be indelible and unaffected by the gases and temperatures to which it will be subjected. Other methods of identification can be used (e.g., bar coding), if they provide a positive means of identification and do not impair the capacity of the filter to function.

(Name of Sampling Organization)

Sample ID No: _____ Storage Conditions: _____

Sample Type:_____ Site Name:_____

Date/Time Collected: _____ Site Address:_____

Sampler:_____

Figure 8.1 Example Sample Label.

Comment [ALS2]: Is this meant to be an example sample label or shipping container label? The text indicates Shipping Container label, but this looks like a sample label.

8.1.2 Sample Collection

Comment [ALS3]: Should sample installation (e.g., for filters) specifically covered somewhere else in the handbook, or should it be covered here?

To reduce the possibility of invalidating the results, all collected samples must be carefully removed from the monitoring device, placed in labeled, nonreactive containers, and sealed. Use of tamper-evident custody seals are suggested and may be required in certain cases. The sample label must adhere firmly to the container to ensure that it cannot be accidentally removed. Custody seals on sample containers serve two purposes: to prevent accidental opening of the sample container and to provide visual evidence should the container be opened or tampered with. The best type of custody seal depends on the sample container; often, a piece of tape placed across the seal and signed by the operating technician is sufficient; for other containers, wire locks or tie wraps may be the best choice. In some cases, the opening of sample containers by unauthorized personnel, such as Transportation Security Administration officers, cannot be avoided. The proper use of custody seals minimizes the loss of samples and provides direct evidence whether sample containers have been opened and possibly compromised. Samples whose integrity is questioned should be qualified (flagged).

8.1.3 Sample Transportation

Samples should be delivered to the laboratory for analysis as soon as possible following sample collection. It is recommended that this be done on the same day that the sample is taken from the monitor. If this is impractical, all the samples should be placed in transport containers (e.g., carrying case, cooler, shipping box, etc.) for protection from breakage, contamination, and loss and in an appropriate controlled-temperature device (i.e., refrigerator or freezer) if the samples have specific temperature requirements. Each transport container should have a unique identification, such as sampling location, date, and transport container number (e.g., number 2 of 5) to avoid interchange and aid in tracking the complete shipment. The number of the transport containers should be subsequently recorded

on the chain of custody (COC) form (described in Section 8.2) along with the sample identification numbers of the samples included within each transport container. It is advised that the container be sealed using an appropriate tamper-evident method, such as with custody tape or a wire lock.

In transporting samples, it is important that precautions be taken to eliminate the possibility of tampering, accidental destruction, and/or physical and chemical action on the sample. The integrity of samples can be affected by temperature extremes, air pressure (air transportation), and the physical handling of samples (packing, jostling, etc.). These practical considerations must be dealt with on a site-by-site basis and should be documented in the organization's QAPP and site specific SOPs.

The person who has custody of the samples must be able to testify that no tampering occurred. Security must be continuous. If the samples are put in a vehicle, lock the vehicle. After delivery to the laboratory, the samples must be kept in a secured place with restricted access.

8.1.4 Sample Analysis

SOPs, if properly developed, have detailed information on the handling of samples at the analysis phase. Similar to the preparation step, if the sample undergoes a number of steps (preparation, equilibration, extraction, dilution, analysis, etc.), and these steps are performed by different individuals, there should be a mechanism in place to track the sample through the steps to ensure SOPs are followed and the integrity of the sample was maintained. Laboratories make extensive use of laboratory notebooks at the various steps (stations) of the analytical process to record the sample handling process and maintain sample integrity.

8.1.5 Storage and Archival

Samples must be properly handled to ensure that there is no contamination and that the sample analyzed is actually the sample taken under the conditions reported. For this reason, whenever samples are not under the direct control of the sample custodian, they should be kept in a secured location. This may be a locked vehicle, locked refrigerator, or locked laboratory with limited access. It is highly recommended that all samples be secured until discarded. These security measures should be documented by a written record signed by the handlers of the sample on the COC form or in a laboratory notebook, indicating the storage location and conditions. Any samples not destroyed during the analysis process (e.g., exposed filters for PM) should be archived as directed by the method requirements or applicable QAPP. 40 CFR Part 58.16 requires PM_{10}, $PM_{10-2.5}$ and $PM_{2.5}$ filters from SLAMS manual lo-volume samplers be archived for 1 year from collection. However, it is suggested that they be archived the first year in cold conditions (e.g., at 4° C) and at room temperature for 2 additional years. It is also suggested that non-destructive lead analysis and STN samples follow this guidance.

Comment [ALS4]: This text was altered in addition to being moved from 8.1.1.

8.2 Chain of Custody (COC)

In order to use the results of a sampling program as evidence, a written record must be available listing the location of the samples at all times. This is also an important component of good laboratory practices[3]. The COC record is necessary to make a prima facie showing of the integrity of the samples. Without it, one cannot be sure that the samples and sampling data analyzed were the same as the samples and data reported to have been taken at a particular time. Procedures may vary, but an actual COC record sheet with the names and signatures of the relinquishers/receivers works well for tracking physical

[3] http://www.fda.gov/ora/compliance_ref/bimo/glp/default.htm

samples. The samples should be handled only by persons associated in some way with the monitoring program. A good general rule to follow is "the fewer hands the better," even though a properly sealed sample may pass through a number of hands without affecting its integrity.

Each person handling the samples must be able to state from whom and when the item was received and to whom and when it was delivered. A COC form should be used to track the handling of the samples through various stages of storage, processing, and analysis at the laboratory. It is recommended practice to have each person who relinquishes or receives samples sign the COC form for the samples. An example of a form that may be used to establish the COC for samples generated in the field is shown in Figure 8.2. This form should accompany the samples at all times from the field to the laboratory. All persons who handle the samples should sign the form. Figure 8.3 is an example of a laboratory COC form. COC forms should be retained and archived as described in Section 5 (Documents and Records).

When using professional services to transport physical samples, only reliable services that provide a tracking number should be used. Information describing the enclosed samples should be placed on the bill of lading. A copy of the shipping receipt and tracking number should be kept as a record. The package should be addressed to the specific person authorized to receive the package, although it is recognized that staff not typically part of the COC may receive the samples and deliver them to the authorized addressee. A procedure must be in place to ensure that samples are delivered to the appropriate person without being opened or damaged. In this circumstance, the sample is considered still in transport until received by the authorized addressee. It may be necessary to ship and/or receive samples outside of normal business hours. A procedure should be developed in advance that considers staff availability, secure storage locations, and appropriate storage conditions (e.g., temperature-controlled).

8.2.1 Sample Inspection and Acceptance

Once the samples arrive at their destination and at every custody change, the samples should first be checked to ensure that their integrity is intact. The contents of the shipment should be checked against the COC form to ensure that all samples listed were included in the shipment. The levels of liquid samples should be compared to original levels (if marked on the container or recorded), to identify whether major leaks have occurred. When using passivated stainless steel canisters, the canister pressure, upon receipt, should be recorded and compared to the final sample collection pressure to indicate canister leakage and sample loss. It is recommended that this comparison be made using a certified gauge that is calibrated annually. Any samples whose integrity or identity are questionable should be brought to the attention of the relinquisher and flagged. All flags should be "carried" along with the samples until the validity of the samples can be proven. This information can be included in the remark section of the COC form.

Chain of Custody Record					
Project No.		**Project Title**			**Organization**
Shipping Container No.					**Contact**
Field Samplers: *print*		*signature*			**Address**
Date	**Time**	**Site/Location**	**Sample Type**	**Sample ID**	**Remarks**
Relinquished by (*print and signature*):			Received by (*print and signature*):		**Comments**

Figure 8.2 Example Field COC Form.

Chain of Custody Record					
Project No.		**Project Title**			**Organization**
Laboratory/Plant: _____					
Sample Number	Number of Container	Sample Description			
Person responsible for samples			Time:		Date:
Sample Number	Relinquished By:	Received By:	Time:	Date:	Reason for change in custody

Figure 8.3 Example Laboratory COC Form.