

Archive. When sample(s) are placed in a separate location than active samples undergoing investigation, until it is determined that further investigations are not required

Calibration. A calibration is a set of operations which establish, under specific conditions, the relationship between values indicated by a measuring instrument or measuring system, and the corresponding standard or known values derived from the standard.

Calibration Check. A calibration check verifies that an instrument is calibrated. A calibration check is a set of operations which verify, under specific conditions, the agreement between values indicated by a measuring instrument or measuring system, and the corresponding standard or known values derived from the standard.

Secure Area. An area (e.g. secure facility) where access to samples and standards is limited and controlled.

Standard. A standard is a solution containing analytes of known parameters (e.g. concentration, mineralogy) specifically intended for the calibration or calibration check of instruments.

Termination. The sample(s) are no longer under the prescribed experimental conditions.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

2.0 Implementation Actions

2.1 Controlling Documents

Test Plans (NP 20-1) and/or Activity/Project Specific Procedures (SP) may be used to define sample and standard preparation, handling, preservation, cleaning, shipment, transfer, analysis, storage, and final disposition. Sample collection/creation and analyses may also be documented in scientific notebooks as described in NP 20-2. Planning documents are required to ensure that sample collection/creation methods, controls, and identification will result in samples which are appropriate for their intended use.

2.2 Samples, Standards and Chemicals Identification

Samples and standards shall be collected or created in accordance with the appropriate controlling work documents. Sample and standards control measures, shall ensure traceability continuously from collection through final disposition. These measures shall include a unique sample or standard identifier (i.e. the concentration of the standard, along with the combination of the date of fabrication and SN information would be a unique identifier), pertinent information concerning the sample or standard (e.g. environmental conditions), and recorded in the drilling log, scientific notebook, on sample or standard information forms (i.e. Chain of Custody), or other appropriate records format. If a sample or a standard has a maximum life expectancy or expiration date, that date shall be documented on the sample or standard label.

In addition, a standards label shall contain

- 1) a unique identification (e.g. 10 PPM Sc);
- 2) the date of fabrication;
- 3) the initials or name of the person who created it;

- 4) the document name and page number (e.g. notebook) showing the record of the material preparation;
- 5) special requirements (e.g. storage requirements) as applicable.

The PI is responsible for the sample and standard from initiation to final disposition by verifying that sample and standard identification is documented in the SN. Additionally, the PI is responsible for all documentation recorded in the SN to ensure traceability of the sample and standard through the lifetime of the experiment. This documentation will include, but is not limited to the analysis, anomalies that occurred during the investigation (e.g. a broken sample), the preparation of the standards, and the termination of the experiment. Termination is defined as: the sample(s) are no longer under the prescribed experimental conditions (see sect 1.1). Upon termination the sample(s) may be placed in archive until it is determined that further investigations are not required; sample(s) will be archived according to Section 2.5. When it is determined that further investigations are not required, the sample(s) will be disposed to according to Section 2.6.

Sample and standards identification shall be maintained by placing clear, legible, and permanent identification directly on the samples and standards, if possible, or in a manner that ensures that identification is maintained. If direct physical markings are either impractical or insufficient, other appropriate means shall be employed e.g., physical separation, labels or tags attached to containers. Label markings must not detrimentally affect the sample or standard content, integrity, or form. If sample or standard labels become obliterated or hidden by surface treatments or sample or standard preparation, other means of identification must be substituted. If the identification cannot be determined a deviation will be identified in accordance with section 2.3.

Markings and labels shall indicate the need for special environments or other special controls. If samples or standards are sub-divided, the unique identification must be transferred to each sub-sample or sub-standard part or sub-sample or sub-standard container.

The chemicals used to prepare samples and standards shall be identified by their name, manufacture name, lot number, and expiration date when applicable.

2.3 Conditions Adverse to Quality or Significant Conditions Adverse to Quality Samples and Standards

Deviations from sample and standards control requirements shall be documented as a Condition Adverse to Quality (CAQ) or Significant Condition Adverse to Quality (SCAQ) in accordance with the process specified in NP 16-1 (Corrective Action). Deviations include, but are not limited to, the following:

- Improper handling and/or shipping
- Loss of traceability
- Loss of identity
- Lost samples or standards
- Use of samples, standards, or chemicals after expired lifetimes
- Chain of custody violations (Note: refer to SP 13-1 [Chain of Custody] for requirements)
- Damaged samples or standards (e.g., exceeding temperature requirements)

A CAQ or SCAQ sample, standard, or chemical shall be segregated or appropriately identified (tags, markings) to prevent inadvertent use. Further processing of the sample or standard shall be controlled until the deviation is evaluated, and the situation resolved. A CAQ or SCAQ sample or standard may be used if the person requesting the sample or standard data evaluates the situation and concludes that use of the sample or standard is appropriate, however, the sample's or standard's deviation status must accompany the sample or standard data. This evaluation is documented as

part of the NP 16-1 Corrective Action process. Samples and standards evaluated through this process shall be categorized in one of the following three ways: "Use-As-Is," "Limited Use," or "Discard." This categorization shall be documented as part of the deviation. Samples and standards that have lost their identity or when sample or standard identification cannot be determined *shall not be used* in further investigations and documented as a CAQ or a SCAQ.

2.4 Use, Handling, Storing, and Shipping Samples and Standards

The PI shall ensure that samples and standards are protected during shipping and handling to prevent damage or deterioration that would compromise the intended use of the samples and standards, and therefore the data derived from sample and/or standard analysis. Samples and standards collected by an organization other than SNL WIPP shall be handled and shipped in accordance with the requirements of that organization until the sample or standard becomes the responsibility of the SNL WIPP program.

Storage methods for samples and standards shall include: sample or standard identification, duration of storage (e.g. expiration), maintenance or replacement of identification markings and tags from damaged, aging or deterioration due to environmental conditions. (SP 13-1 Sect. 2.3)

Controlling documents shall be developed to specify any special protective environments (e.g., inert atmospheres, specific moisture content levels, or temperature levels) and equipment (e.g., containers) required for the samples and standards. These documents or procedures shall be issued *prior to* collecting, handling, and shipping samples or standards; and shall address the creation and maintenance of such environments.

Specific requirements for handling, storage, cleaning, packaging, shipping, and preservation of critical, sensitive, perishable, or high-value samples and standards shall be developed and implemented if necessary.

Chemicals determined to be unacceptable (expired or indeterminate) shall be segregated immediately upon discovery, to prevent inadvertent use. The unacceptable chemical will be labeled or tagged with explanation for the segregation. If an expired chemical or standard has inadvertently been used in data collection, a deviation will be identified in accordance with section 2.3.

When performing a calibration, it is recommended to use at least three NIST traceable standards which cover the range of the expected measurements. However, if procedures relevant to the instrument or measuring system advises to use fewer than three standards (e.g. manufacturer recommendations), the calibration or calibration check should be performed according to these procedures. If the standards used for a calibration and/or calibration check are not NIST traceable, documentation of standard preparation must be referenced and uniquely traceable (e.g. to all materials used to make standard, date of fabrication, expiration date, manufacturer, person creating standard, and where documented, etc.). The calibration or calibration check shall be performed daily or upon each use, whichever is less frequent or as stated in the instrument specific procedure. If upon analysis a sample exceeds the expected range of measurement (e.g. exceeds the highest calibration point), either a dilution will be performed on the sample to obtain a result within the calibration range or a new standard will be prepared to extend the range of measurement.

2.5 Samples and Standards Storage/Archiving

When samples and standards are not in the possession of the individual designated with their custody, they shall be stored in a secure area with associated documentation. A secure area is defined as an area where access to the samples and standards is limited and controlled (e.g., secure facility). Samples and standards shall be controlled to preclude the mixing of like samples and

standards. Samples and standards for which analyses or tests have been performed shall be identified and maintained in a separate part of the storage area. When chemicals are not in use, they shall be stored in the appropriate storage place.

Samples, standards, and chemicals shall be stored in areas where the environment is controlled to prevent their degradation in accordance with section 2.4. Upon expiration of samples, standards, or chemicals, the samples, standards, or chemicals will be properly discarded (if hazardous, contact Center 6200 ES&H coordinator for guidance). If expired samples, standards, or chemicals are not discarded (for legal or other reasons) they shall be segregated or suitably identified (tags, markings) to prevent their use.

Archiving of samples and standards is done to preserve them for future investigation or review. The organization responsible for archiving samples and standards shall have a documented and approved process for accomplishing the archiving task. This process shall allow for the identifying, tracing, and retrieving of archived samples and standard materials, and shall provide for controlled environmental conditions commensurate with the intended use of the samples and standards. Once sample(s) are terminated and archived for future investigation or review, each sample will be documented in the SN or SN Supplement. When it is determined that no further investigation is required, sample(s) will be disposed and documented per Section 2.6. If it is determined that further investigation is required, all pertinent information concerning the sample and investigation will be documented in the SN or SN Supplement in accordance with NP 20-2, and the sample(s) will be removed from archive and placed in a secure storage area.

2.6 Samples and Standards Disposition

Upon sample(s) or standard(s) disposal, the final disposition shall be documented in the SN or SN Supplement. Standards that are purchased (e.g. JIT) are excluded from this requirement as these are maintained in the Chemical Inventory System (CIS).

3.0 Records

Note: Implementation of NP 13-1 generates uniquely labeled samples and standards and associated records pertaining to their use, collection, tracking, analysis, and disposition. Applicable sampling procedures (TPs, SPs, or other implementing procedures) are controlled by other NPs (for example, NP 20-1 or NP 20-2).

The following records, which may be generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

QA Record

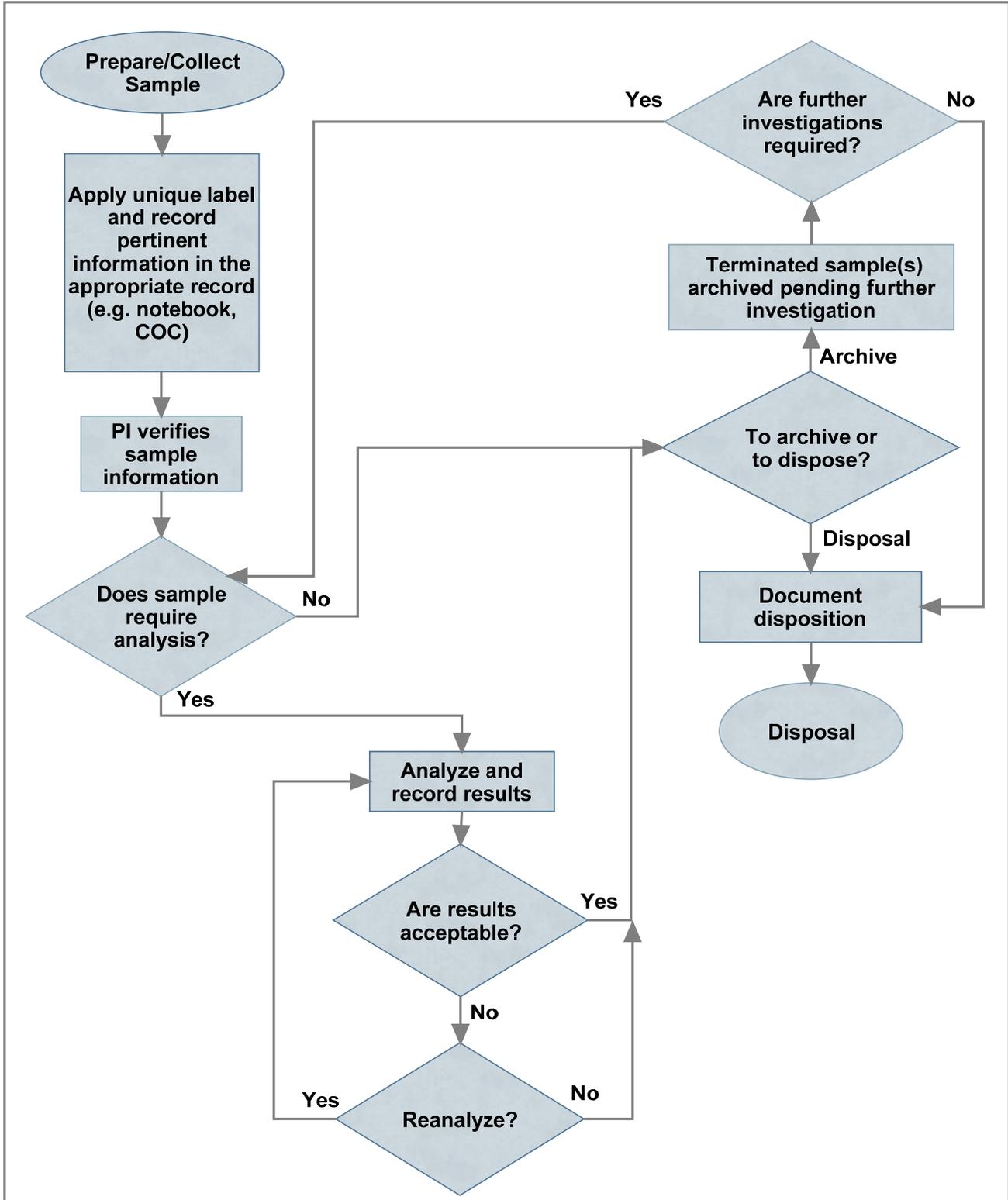
- Drilling Logs
- Field Sampling Notebooks
- Scientific Notebooks
- Scientific Notebook Supplements

4.0 Appendices

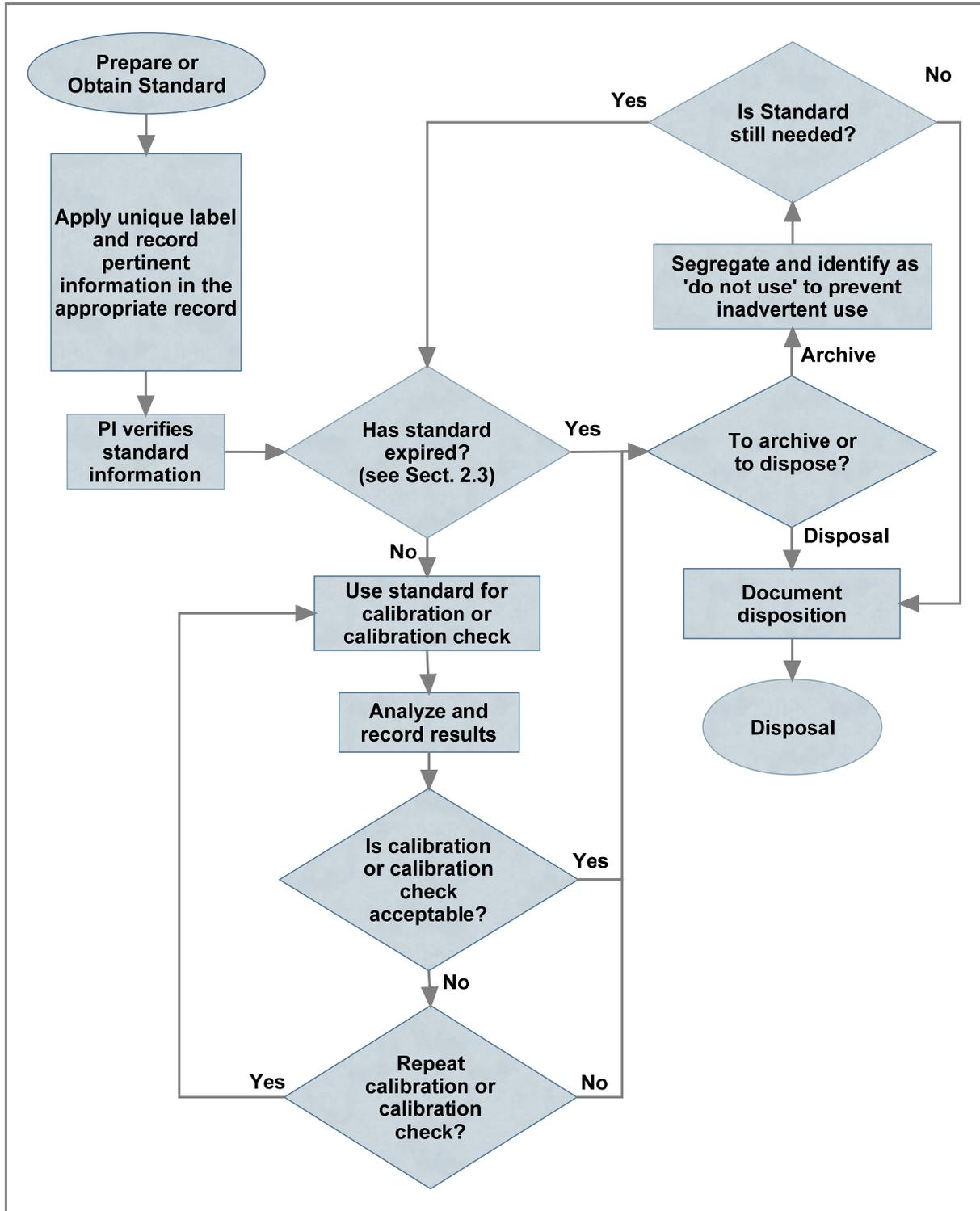
Appendix A: NP 13-1 Sample Control Process Flowchart

Appendix B: NP 13-1 Standard Control Process Flowchart

Appendix A NP 13-1 Sample Control Process Flowchart



Appendix B NP 13-1 Standard Control Process Flowchart



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