Traceability

Definition

Traceability of measurement means the ability of relating a result or measurement to appropriate state, national, or international standards through an unbroken chain of documented comparisons.

Historical NR 149 Traceability Requirements

Records to be retained include but are not limited to records of

- ❑ **Samples processed so that any sample may be traced back to the analyst, date collected, date analyzed, and method used including raw data, intermediate calculations, results, and the final report.** *[NR 149.06(1)(a)]*
- ❑ **Quality control results shall be traceable to all of the associated sample results.** *[NR 149.06(1)(b)]*

2008 Revisions to NR 149

149.39(3)(a)
(3) ANALYTICAL AND TECHNICAL RECORDS. (a) The laboratory shall maintain all analytical and technical records containing raw and derived data, or original observations, necessary to allow historical reconstruction of all laboratory activities that contributed to generating reported results. 149.39(3)

149.45(1)(a) Measurement traceability. (1) STANDARDS, REAGENTS AND REFERENCE MATERIALS. (a) The laboratory shall ensure that results of analyses can be linked to all the standards and reagents used to derive results.

149.45(2)(a) 3. The laboratory shall maintain records that detail the preparation of intermediate and working standards and reagents. These records shall link the intermediate and working standards and reagents to their respective originating stocks or neat

compounds and shall indicate their date of preparation, expiration and the identity of the preparer.

149.45(2)(b) The laboratory shall retain records and certificates that trace reference materials used to calibrate or verify analytical support equipment to the source of the corresponding reference materials.

Traceability- Standards

Whenever a standard is analyzed with a set of samples, documentation needs to be established that will answer critical questions much later, after results have been generated. In the simplest example, consider a set of samples analyzed for total phosphorus three (3) years ago. Can you produce documentation that calibration standards used with that analysis were prepared correctly and from stock standard materials that had not exceeded their expiration dates? How about similar documentation for other critical reagents used in the testing? If you can answer, "yes", then your documentation system provides adequate traceability.

For a given set of results, a lab must be able to provide documentation that:

- standards used were not used beyond their expiration date (*unless there is documentation to support their continued validity*)
- standards contain required elements.
- standards are prepared properly and at appropriate concentrations.
- second source standards are used at the appropriate times and situations.

Examples:

- Calibration standards (including CCV)
- Second source standards (ICV)
- Spiking standards (MS, LCS, Surrogate, Internal)
- Quality Control standards (QCS)
- Proficiency standards (PT)
- Stock standards
- Intermediate or working standards
- Instrument performance check (IPC) solutions.
- Tuning solutions (*generally for MS analyses*)
- Interference Check Standards (ICS)

The easiest thing to do is to give each of the standards a unique identification code. Then on your benchsheets, wherever a standard is listed, this code is linked to it so a data reviewer can trace back to that standard and find all of the required standard information. The simplest application of this concept derives a "identifier code" from the Standard Logbook #, Page (of the logbook)#, and Line # (of the logbook page). For example, a standard code of 12-27-19 would correspond to information located in logbook #12, on page 27, line 19.

Required Documentation

The information you want to have documented for each standard is

- Parameter or analyte(s) *(identity)*
- Manufacturer (*source*)
- Concentration
- Purity (*for "neat" standards used in trace organic analyses*)
- Preparation date
- Expiration date
- Date received (*if the standard is the original source received from the manufacturer*)
- Certificates of analyses from the manufacturer (*just keep*)
- Lot number
- Preparation records (for standards or mixed standards/reagents created in the lab)
    - Source Standard ID
    - Source volume used
    - New Standard ID
    - New Standard final volume
    - Preparation date
    - Expiration date
    - Chemical Name (*identity*)
    - Preparer
    - Solvent ID

Traceability- Individual Reagents/Chemicals

Examples of Reagents that need to be linked to all results:

- Solid Chemicals
- Acids
- Bases
- Chemical Preservatives
- Solvents

The easiest thing to do is to give each of the reagents a unique identification code. Then on your benchsheets, wherever a reagent is listed, this code is linked to it so a data reviewer can trace back to that reagent and find all of the required reagent information.

The information you want to have documented for each reagent is

- Chemical Name (identity)
- Manufacturer (source)
- Concentration
- Purity

- Date made
- Date expires
- Date received if the reagent is the original source received from the manufacturer
- Certificates of analyses from the manufacturer (just keep)
- Lot number
- Preparation records
    - Source Reagent ID
    - Source volume used
    - New Reagent ID
    - New Reagent final volume
    - Date prepared
    - Date expires
    - Chemical Name (identity)
    - Preparer
    - Solvent ID

Traceability- Mixed Reagents/Chemicals

Examples of Mixed Reagents/Solutions that need to be linked to all results:

- Solid Chemicals
- Acids
- Bases
- Chemical Preservatives
- Solvents

The easiest thing to do is to give each of the reagents a unique identification code. Then on your benchsheets, wherever a reagent is listed, this code is linked to it so a data reviewer can trace back to that reagent and find all of the required reagent information.

The information you want to have documented for each reagent is

- Chemical Name (identity)
- Manufacturer (source)
- Concentration
- Purity
- Date made
- Date expires
- Date received if the reagent is the original source received from the manufacturer
- Certificates of analyses from the manufacturer (just keep)
- Lot number
- Preparation records
    - Source Reagent ID

- Source volume used
- New Reagent ID
- New Reagent final volume
- Date prepared
- Date expires
- Chemical Name (identity)
- Preparer
- Solvent ID

Traceability- Reference Materials

Examples of Reference Materials

- Analytical balance
- Weights
- Thermometers (liquid, IR, digital)
- Volumetric Auto-Pipettes
- Spectrophotometer wavelength check

The easiest thing to do is to give each of the reference materials a unique identification code. Then on your benchsheets, wherever a reference material is used, this code is linked to it so a data reviewer can trace back to that reference material and find all of the required reference material information.

The information you want to

record for each reference material in order to certify their accuracy is (must be traceable to primary NIST standards)

- Type of material (identity)
- Manufacturer (source)
- Date of last calibration
- Correction factors if any
- Date expires
- Who calibrated the reference material
- Material ID number
- Certificates from manufacturer that demonstrate the materials accuracy

Traceability- Sample Results

Examples of data

that must be maintained and/or recorded in order to ensure that historical reconstruction of all lab activities that contributed to the generation of lab results can be linked:

[How you do this, the format used, is up to you, as long as the data can stand on its own and be reconstructed.]

- Sample ID's
- Container ID's when used (such as BOD bottles, TSS boats, …)
- Initial sample volumes
- Final sample volumes
- Prep Date/Time/Analyst/Method
- Analysis Date/Time/Analyst/Method
- Units associated to all results and entries
- Dilution factors
- Curve concentrations
- Final concentrations
- Spike volumes added to samples
- Calibration curve variables (correlation coefficient, slope, y-intercept)
- Clear identification of parent samples for matrix spikes and sample replicates
- Raw data readings (absorbance, mV, area, …)
- Percent recoveries for QC samples (known standards, LCS, MS)
- Precision results for replicates *(if analysis is required)*
- Method blank results in concentration
- Raw data

Traceability- Sample Handling

(5) SAMPLE RECEIPT DOCUMENTATION. The laboratory shall document the receipt and condition of all samples in chronological hard copy or electronic records. The records may be maintained in any format that retains the following information:

- Client or project associated with the samples.
- Dates of sample collection
- Dates of laboratory receipt.
- Times of sample collection and laboratory receipt for samples to be analyzed for tests with holding times equal to or less than 48 hours.
- The unique sample identification code assigned by the lab.
- An unequivocal link between the lab ID and any field ID assigned by the collector.
- Documentation of sample preservation status and condition on receipt.
- The requested analyses *(unless the lab collects and analyzes its own samples and analyses are directed by permit).*
- Requested test methods, when specified
- Comments from inspection associated with the lab's Sample Acceptance Policy.
- Documentation that samples are stored at the appropriate temperature.
- Documentation that sample extracts or digestates are stored at the appropriate temperature.