

Excerpts from the MEL Handbook

<http://aww.ecology.ecy.wa.gov/programs/eap/forms/labmanual.pdf>

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Quality Control Samples

The primary types of quality control samples used to evaluate and control the accuracy of laboratory analyses are check standards, duplicates, spikes, and blanks. The most important of these in evaluating analytical precision and bias is the check standard; commonly referred to as a laboratory control sample. Duplicate samples and spikes assist in evaluating the effects of matrix. Field replicates also give an estimate of both matrix and sample collection on data quality. Blanks are a special category in that they are almost always done, and assume a very high priority when performing trace analyses near the limit of detection.

p. 78-80. Types of Blanks used for Quality Control

Method Blanks

Blanks are an important type of quality control for trace level analyses. Method blanks are prepared and analyzed along with the samples to measure the response of the analytical system to the analyte at a theoretical concentration of zero.

Field Blanks

Field blanks may detect bias due to contamination. Field blanks are to be handled, transported, and analyzed in the same manner as the samples collected the same day. In order to ensure minimal contamination obtain Deionized Reagent Water for field blanks.

Contamination may be due to:

- Sample containers
- Sampling equipment
- Filtration equipment
- Surroundings
- Preservatives
- Transportation or storage practices
- Other samples
- Laboratory analysis

Use field blanks to detect specific problems or to meet legal or regulatory requirements.

Three common types of field blanks are described below.

1. **Transport (or Trip) blanks** are carried to and from the field along with the other sample containers from the site. They may indicate contamination from the sample containers, cross-contamination during shipment, storage, or laboratory contamination.

2. **Transfer blanks** are prepared in the field while splitting or transferring samples. They may indicate contamination from sampling equipment, the surroundings, sample containers, or cross-contamination during shipment. Each transfer blank requires the use of two pre-cleaned sample containers. One container is filled at the laboratory with reagent grade water and serves as the source for the transfer.

The second container is sent empty and serves as the receiving container; this container is the transfer blank to be analyzed. Transfer blank water must be from the same source as the method blank water used by the laboratory performing the analysis. Transfer blanks are prepared in the most contaminated sampling area to attempt to simulate a worst-case scenario regarding environmental contributions to sample contamination. They should be prepared at a rate of one per day per sample matrix regardless of whether the samples are to be shipped that day. Transfer blanks must be transported with the same set of sample bottles they accompanied to the field, and must be packaged with the associated matrix.

3. **Sampling (Equipment or Rinsate) blanks** are prepared by exposing reagent grade water to the equipment and containers used to collect or transfer the samples. These blanks may indicate contamination from sampling equipment, sample containers, cross-contamination during shipment, storage, or laboratory contamination.

Data Review/Verification

A review of the data is performed for all analyses ordered from the Manchester Laboratory. If the analysis is performed at the Manchester Laboratory, final data review is normally performed by the unit supervisor or an analyst experienced with the method. Chemists on the Manchester Laboratory personnel perform the data review for analytical work sub-contracted to commercial laboratories.

The data review process is an integral part of result generation at Manchester Laboratory.

The review begins with an inspection of the final results for completeness to determine if all analytes have been reported and proper extraction, digestion, and analysis has been performed. After inspection of the data summaries, the raw data is carefully reviewed. If target compounds have been detected in the matrix, we determine the quality of the chromatography or instrument output.

If surrogates are used, then surrogate recoveries are carefully evaluated to evaluate analyte recoveries, and to ascertain that non-detected analytes would have been detected had they been present.

Calibration

Calibration or standardization is the procedure by which solutions of known concentration are used to determine the relationship between analytical response and concentration.

The linearity of the calibration and the sensitivity of the analysis are evaluated for the samples analyzed.

At this point the data reviewer evaluates:

- Calibrations are within specifications.
- Check standards are within specifications.
- Reported detection limits are supported by the data reported.
- Necessity and appropriateness of dilutions.

Error Checking in Data

Known errors in the data can be corrected. Typical of these kinds of problems are:

- Mistakes in units, calculations, dilutions, or dates.
- Transcription errors.
- Completeness of the data package coming from the laboratory.

Evaluating Potential Problems with Analytical Data

- Interferences are present in the sample
- Calibration data is out of limits
- A mistake has been made in units or dilutions

- The data package is incomplete
- Spike recoveries outside of QC limits
- Significance of blank contamination, if any. Are similar levels found in the samples?
- Other contamination documented by the laboratory.

In these instances the data may indicate the samples need to be reanalyzed. If reanalysis provides similar results, indicating possible matrix effects, or if reanalysis is not possible, the data may be qualified.

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Data Qualifiers

Code Definition

E Reported result is an estimate because it exceeds the calibration range.

G Value is likely greater than result reported; result is an estimated minimum value (used primarily in microbiology.)

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N The analysis indicates the present of an analyte for which there is presumptive evidence to make a “tentative identification”.

NJ The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.

NAF Not analyzed for.

NC Not calculated.

REJ The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

U The analyte was not detected. The value preceding the "U" represents the sample quantitation limit.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.