Definitions: Quality Control (QC) Checks

Introduction

This document serves as a starting point for understanding the definitions of various quality control (QC) Checks and the associated calculations.

Purpose

To familiarize new TRACES users with the various quality control (QC) and quality assurance (QA) definitions and calculations. To ensure that the quality of the sample data meets the minimum standards based on testable criteria, e.g., EPA 6020B, 200.8, SW-846.

Scope

Though the QC document is based on various ICP-MS QC criteria, it does have applications to various LC-MS and GC-MS methodologies.

Referenced Documents

- EPA Methods SW-846
- EPA Methods 6020B
- EPA Methods 200.8
  Trace Metals Analysis by ICP-MS – PBM. (n.d.). https://www2.gov.bc.ca/assets/gov/environment/research-monitoring-and-reporting/monitoring/emre/methods/sept2017/bc_moe_icpms_metals_15sept2017.pdf

Responsibilities

1. **Users**
   1.1. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols and the QC Checks and their definitions and roles.
   1.2. It is the Users responsibility to ensure the QC data is maintained and available for easy reference and inspection.
   1.3. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.

Definitions

1.1 Initial Calibration Verification (ICV)

The calibration curve must be verified by the analysis of an ICV standard (at or near mid-range) from an independent source. The ICV result must be within 10% of the true value for the calibration to be considered valid. If the ICV is outside QC limits, the instrument must be re-calibrated.

Definitions

1.2 Interference Check Standards (ICS)
ICP interelement corrections are verified by analyzing ICSs at the beginning and each time the analytical run exceeds an 8-hour shift. Results must be within ±20% of the true value for each element.

1.3 Continuing Calibration Verification (CCV)
The working standard curve must be verified by analyzing the CCV standard (at or near the mid-range) after every 10 samples and the ENDCCV at the end of the sequence. CCV/ENDCCV results must be within ±10% of the true value, or the previous ten samples must be reanalyzed.

1.4 Initial/Continuing Calibration Blanks (ICB/CCB)
The ICB/CCB results must be less than the reporting limit (RL) for the sequence to continue.

1.5 Method Blank
A method blank must be prepared for each analytical batch of samples (not to exceed 20 samples) of the same matrix. The method blank results must be less than the RL. A method blank containing an analyte concentration >RL may be used in instances when the sample concentrations are at least 10 times the method blank concentration.

1.6 Laboratory Control Sample (LCS)
Aqueous and solid LCSs must be obtained from an independent source and must be prepared with each analytical batch of samples using the same preparation method as that employed for the samples with the frequency of 1 in 20 samples per matrix. The LCS sample may be either a certified reference material or a blank matrix spiked with the target analytes from an independent source at or near mid-range of the calibration. LCS results for each analyte must be within the specifications supplied by the vendor or within 75 - 125% of the true value and are calculated as in Section 2.1.

1.7 Matrix Spike/Matrix Spike Duplicate (Spikes)
At least one MS and one MSD sample must be digested with every 10 samples of the same matrix, or with each project type to verify the accuracy of the method. In the event there is not sufficient sample available in the batch to run a MS/MSD, an LCS/laboratory control sample duplicate (LCSD) must be run. Recoveries are calculated as in Section 2.2.
The Relative Percent Difference (RPD) of MS/MSD samples must be within ±20% and calculated as in Section 2.2.

1.8 Linear Analytical Range (LAR)
One or more linear analytical range (LAR) standards must be analyzed to determine the maximum linear range of the calibration for each element. Recovery must be within 90-110 %. If the recovery is outside these limits, the maximum calibration standard concentration defines the linear range. LAR standards must be analyzed and reported on a quarterly basis.

1.9 Serial Dilution
A sample (typically the sample chosen for the MS/MSD) from each project in an analytical batch is analyzed at a 5x dilution in conjunction with the samples. The concentration in the undiluted sample must be greater than or equal to 50x the IDL to obtain a meaningful comparison. The results of the serial dilution are multiplied by the dilution factor and compared to the original determination (undiluted

sample). Agreement within + 10% between the concentrations for the undiluted sample and the diluted sample indicates the absence of matrix interferences for undiluted samples meeting the 50x IDL criteria. If the concentration of all analytes in all samples is less than 50x the IDL, serial dilution is not performed. Samples may also be successively diluted and analyzed to eliminate interferences. These samples will be identified as dilution samples and not as serial dilutions.

1.10 Dilution Analysis

If the concentration of any analyte in any sample exceeds the linear range, the sample must be diluted and re-analyzed. An appropriate dilution or series of dilutions (for example, 5x, 10x, 20x) may be required depending on the concentration in the undiluted sample. Results are reported from the lowest dilution that falls within the linear range.

If chemical/physical matrix effects are suspected or for analytes that saturate the detector, samples must be diluted and re-analyzed. An appropriate dilution or series of dilutions may be required depending on the concentrations in the undiluted sample. Comparisons are first made with respect to the undiluted sample and then, within the series. Based on the analyst's professional judgement, results are reported from the diluted sample that has the smallest dilution factor and indicates the absence of interferences.

An optional approach to determine if chemical/physical matrix effects are present is to use post digestion spike (PDS) analysis.

1.11 Post Digestion Spikes (PDS)

PDS are used for some analyses (e.g., metals) to assess the ability of a method to successfully recover target analytes from an actual sample matrix after the digestion process has been performed. The PDS results are used with MS results to evaluate matrix interferences. The MS and PDS should be prepared from the same environmental sample. A PDS is not to be analyzed in place of a MS.

Post Digestion Spikes must be reported as post-digested and must not be misrepresented as pre-digested spikes.

1.12 Initial Demonstration of Capability

Initial proficiency in ICP analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicates of a well-mixed reference standard are analyzed using the procedures outlined in this SOP. Calculate the average mean and standard deviation (s) in mg/L for waters and mg/kg for soils. The QAO (Quality Assurance Officer/TRACES Staff) will tabulate the results from all the analysts per matrix per parameter and calculate control limits.

1.13 Method Detection Limit Studies (MDL)

Method detection limit (MDL) studies will be run on an annual basis for each ICP instrument for the water and soil matrix to verify the minimum concentration that can be measured and reported with 99% confidence. A minimum of seven replicates will be used for the study (EPA 1984).

1.14 Reporting Limit Standards (RL)

The RLW standard is run each time water samples are analyzed by the ICP. The RLS standard is run each time soil samples are run on the ICP. The RLS and RLW upper and lower control limits are established based on the soil and water MDL (method detection limit) study results.

1.15 Instrument Detection Limits (IDL)
IDLs are run on a quarterly basis on each ICP and are used as the basis for the serial dilution analysis.

1.16 Nonconformance Memo
A nonconformance memo will be issued/generated any time a user notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the Quality Assurance Officer/TRACES Staff for verification of corrective action.

Calculations of QC Checks

2.1 Laboratory Control Sample Calculations

Percent recovery (%R) must be within 75-125% and calculated as:

$$\%R = \frac{(LCS - B)}{SA} \times 100$$

LCS = LCS result, ug/L or mg/kg

B = Method blank result, ug/L or mg/kg

SA = Spike added, ug/L or mg/kg

2.2 Matrix Spike/Matrix Spike Duplicate Sample Calculations

Spike sample percent recovery (%R) must be within 75-125% and calculated as follows:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

SSR = Spiked sample result

SR = Sample result

SA = Spike added

The Relative Percent Difference (RPD) of matrix spike and matrix spike duplicate samples should be within ±20% and calculated as follows:

$$RPD = \frac{(S - D)}{(S - D)/2} \times 100$$

S = %R for matrix spike sample

D = %R for matrix spike duplicate sample

2.3 Sample Concentration (Liquid samples)

For the determination of metal concentration in solution (**Liquid Sample**), the value in milligrams per liter (mg/L) or micrograms per liter (ug/L) can be selected via the ICP-MS instrument. For aqueous samples, report concentrations as µg/L, where µg/L = mg/L x 1000.

If a dilution of the sample is required:

concentration of metal in sample = $A \times \frac{C+B}{C}$

A = mg/L (or ug/L) of metal in diluted aliquot from the reported value

B = Acid blank matrix used for dilution in mL

C = Sample aliquot in mL

2.4 Sample Concentration (Solid samples)

For solid samples, all concentrations are calculated as mg/kg based on wet weight; thus:

mass of metal in mg/kg of sample = $\frac{A \times V}{F \times W} \times DF$

A = mg/L (or ug/L) of metal in processed sample from reported value

F = concentration unit factor (1 for mg/L, 1000 for ug/L)

V = Final volume of the processed sample, mL

W = Weight of sample, grams

DF = Dilution factor for diluted samples (no sample dilution DF=1)

IF DILUTION of the solid sample is required, DF is as follows:

$$DF = \frac{C+B}{C}$$

B = mL of acid blank matrix used for dilution

C = mL of sample aliquot

Definitions

For solid samples, reported concentration as mg/kg is based on dry weight as follows:

mg metal/kg sample (dry sample) = $\frac{mg/kg\ (wet)}{(S/1000)}$

S = percent of total solids in the sample

*The TRACES Manager will provide full details during hands-on training.