Quality Control Definitions and Analysis Procedures

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Samples for Initial Demonstration of Performance or Capability

Initial Demonstration of Performance or Capability (IDC): A set of samples analyzed to determine the performance of a new analytical method that is conducted before an analyst runs the first analytical batch. The IDC typically includes MDL and IPR studies (discussed below).

Method Detection Limit (MDL): Used to establish the ability to detect the analyte, the analyst determines the MDL using the instrument, reagents and standards that are used with the analysis of the method. The method detection limit is a statistically determined value that has a 99% probability that it is different from the reagent water blank.

To determine the MDL, analyze at least seven replicates of a standard solution that is one- to five-times the estimated detection limit. Use the appropriate *t* value for a 99% confidence interval (n -1) for the number of replicates analyzed. For example, the *t* value for seven replicates is 3.143. Analyze the seven replicates, take the standard deviation of this analysis, and multiply this value by 3.143.

Initial Precision and Recovery (IPR): To establish the ability to generate precision results and determine accuracy of the method, analyze four or 10 replicates of a mid-range standard. Most laboratories find four replicates sufficient. Calculate the average percent recovery of the replicates and standard deviation of the analyte. Ensure that the recovery is within 10%[1] of the nominal value. Calculate the percentage of relative standard deviation (%RSD) by dividing the standard deviation by the average.

Samples for Ongoing Quality Assurance

Analytical Batch: A set of environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents. The number of samples in a batch must not exceed 20[1] environmental samples. If a day's analysis (one contiguous eight-hour period) is comprised of only eight samples, this also constitutes an analytical batch.

Matrix Spikes (MS and MSD): The analyst must spike in duplicate a minimum of 5% of all samples run in an analytical batch (one spike duplicate per maximum of 20 samples [one batch]). Spike two separate sample aliquots with a stock standard one- to five-times higher than the background concentration of the sample. Calculate the percent recovery of the analyte (P) in each aliquot using the following equation:

$$P = \frac{(A - B)100}{T}$$

Where, A = measured concentration of the spiked sample, B = measured concentration of unspiked sample, T = true concentration of the spike.

The percent recovery of the analyte should meet the current laboratory acceptance criteria. For

wastewater spikes, the percent recovery should be ± 20%[1], and drinking and surface water spikes should be ± 10%[1].

Compute the relative percent difference (RPD) between the spiked sample results using the following equations:

$$RPD = \frac{(D_1 - D_2)}{(D_1 + D_2)/2} \text{ x } 100$$

Where, D1 = concentration of analyte in the sample, D2 = concentration of the analyte in the second (duplicate) sample. The RPD for duplicates should be ± 20%[1], and drinking and surface water spikes should be ± 10%[1].

Duplicate Sample: If there was not sufficient sample collected to analyze a MSD, analyze a duplicate sample to collect precision data. This duplicate sample must be analyzed at a minimum of 5% of samples run in an analytical batch (i.e., one sample duplicate for every 20 samples). Calculate the relative percent difference (RPD) between the duplicate samples from the equation above. Some regulatory agencies require a sample duplicate for each analytical batch, in addition to a matrix spike and matrix spike duplicate. Consult your local agency for these quality control guidelines.

Laboratory Reagent Blanks (LRB): Laboratory reagent water blanks are analyzed to ensure that there is no contamination in the reagent water. Analyze laboratory blanks during method startup and with each analytical batch of no more than 20 samples. The blank must be subjected to the same procedural steps as a sample. If the blank concentration is higher than the minimum concentration level (MDL, Practical Quantitation Limit [PQL] or lowest standard in calibration curve[1]), stop the analysis of the batch. All samples within a batch must be associated with an uncontaminated blank before the results may be reported for regulatory purposes.

Ongoing Precision and Recovery (OPR): With every analytical batch, a midrange standard must be prepared from a stock standard and analyzed with the same procedural steps as a sample. The results of OPR should be ± 10%[1] of the nominal value. If the criteria are not met, the batch analysis is judged to be out of control, and the problem must be identified and corrected and the analytical batch reanalyzed.

Ongoing Quality Assurance: Quality Control Samples Commonly Included in an Analytical Batch

The following quality assurance (QA) samples are often run with each analytical batch. Each laboratory should follow established standard operating procedures (SOPs), protocols, and regulatory guidelines set forth by local regulatory and certification bodies to ensure quality assurance procedures are in place.

1. **Laboratory Reagent Blank (LRB)**: Analyze a reagent blank. Following the normal method procedure; pipet the reagent water that was used to prepare the IPR and MDL solutions. The reagent water takes the place of sample matrix in this quality control (QC) sample.

2. **Ongoing Precision and Recovery (OPR)**: Analyze a mid-range standard and calculate the percentage of recovery when compared to the true concentration of the standard.

3. **Duplicate Sample**: Analyze a sample in duplicate (two analyses of the same sample) to determine precision and calculate relative percentage difference between the duplicate set.

4. **Matrix Spikes and Spike Duplicates (MSD)**: Analyze one un-spiked unknown as well as two duplicate spikes of the sample and determine the percentage of recovery from the un-spiked and spiked matrix and the relative percentage difference between the two spiked samples.

[1]These are commonly used values for reporting. All quality control values must meet the current laboratory acceptance criteria. If your laboratory has not set these specifications, please contact your local regulatory agency for further instruction.

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