

Spike Preparation Examples

Option 1 - If the spike solution volume is equal to 1% or less of the total sample volume, direct subtraction of the unspiked sample is allowed.

Option 1 Example:

0.5 mls of a 1000 mg/L standard spike added to 100 mls of sample has a theoretical value of 5 mg/L.

- (A) The spiked sample recovery is 5.1 mg/L
- (B) If the unspiked sample result is 0.5 mg/L
- (C) Theoretical value is 5.0 mg/L

The Percent Recovery = spiked sample recovery (A) – unspiked sample result (B) divided by theoretical value (C) X 100 or

$$\frac{5.1 - 0.5}{5.0} \times 100 = 92\% \text{ recovery}$$

$$\frac{A - B}{C} \times 100 = \text{Percent recovery}$$

Option 2 - Adjust spike solution to a known volume with sample. In this case the sample concentration must be adjusted.

Option 2 Example 1:

10 mls of spike (concentration 50 mg/L) brought to 100 mls with sample the theoretical MS value is 5 mg/L.

- (A) The spiked sample recovery is 5.1
- (B) If the unspiked sample result is 0.5 mg/L
- (C) % sample is 0.90 (sample volume used (90) divided by final volume (100))
- (D) Theoretical value is 5.0 mg/L

The Percent Recovery = spiked sample recovery (A) – (unspiked sample result (B) x % sample (C)) divided by theoretical value (D) X 100 or

$$\frac{5.1 - (0.5 \times 0.9)}{5.0} \times 100 = 93\% \text{ recovery}$$

$$\frac{A - (B \times C)}{D} \times 100 = \text{Percent recovery}$$

Option 2 Example 2: Larger spike volume

25 mls of spike (concentration 50 mg/L) brought to 250 mls with sample the theoretical MS value is 5 mg/L.

- (A) The spiked sample recovery is 5.6
- (B) If the unspiked sample result is 0.5 mg/L
- (C) % sample is 0.90 (sample volume used (225) divided by final volume (250))
- (D) Theoretical value is 5.0 mg/L

The Percent Recovery = spiked sample recovery (A) – (unspiked sample result (B) x % sample (C)) divided by theoretical value (D) X 100 or

$$\frac{5.6 - (0.5 \times 0.9)}{5.0} \times 100 = 103\% \text{ recovery}$$

$$\frac{A - (B \times C)}{D} \times 100 = \text{Percent recovery}$$

Corrective Action/Qualifications for MS

Spike accuracy is usually based on a range of percent recovery (e.g., 80-120%). Refer to the method of choice for specific acceptance criteria for the matrix spikes until the laboratory develops or adopts statistically valid, laboratory-specific performance criteria for accuracy. If a MS fails, and the LCS is acceptable, qualify the data for the MS sample. Repeated failures for a specific matrix may require use of an alternate method or method of standard addition. Base the sample batch acceptance on the results of the LCS analyses (and other quality control results) rather than the MS alone, because the matrix of the spiked sample may interfere with the method performance. If a MS and the associated LCS fail, re-prepare and reanalyze affected samples.

Post Digestion Spikes (PDS)

Post Digestion Spikes (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. (Exception: TCLP and SPLP samples are always spiked post digestion.)

Corrective Action/Qualifications for Post Digestion Spikes

In general, if the MS recovery for an analyte does not fall within the quality control acceptance range but the PDS recovery is acceptable, then a matrix affect (associated with the preparatory process) should be suspected and the unspiked sample results must be qualified on the basis of the matrix spike recovery. However, when historical data for the effect does not exist, the laboratory would normally be expected to perform a second digestion and reanalysis of the MS to confirm the result. The result would be confirmed if the MS recoveries and PDS recoveries for both sets of analyses were similar in magnitude and bias. When both the MS recovery and PDS recovery for a particular analyte falls outside of quality control acceptance range in the same manner (i.e., the PDS and MS failures are of similar magnitude and the direction of bias is the same), confirmatory analyses are unnecessary but the data must be qualified.

Parameters Excluded from MS Requirements

Acidity	Alkalinity
BOD/CBOD	Aquatic Humic Substances
Chlorophyll	All Bacteriological Parameters
Color – ADMI	Color - PtCo
Conductivity	Dissolved Oxygen
Ignitability	All Residues
Paint Filter Test	Turbidity
pH	Temperature
Salinity	Sulfite
Total Residual Chlorine	Vector Attraction Reduction (All Options)

(Field Laboratories and Field Setting analyses are exempt.)