Matrix Spiking Policy
(NC WW/GW LC Branch 05/07/2020)

NC Administrative Code 15A NCAC 02H .0805 (a) (7) (D) states: “Unless the referenced method states a greater frequency or the parameter is not amenable to spiking, laboratories shall spike five percent of samples monthly. Laboratories analyzing fewer than 20 samples per month shall analyze one Matrix Spike during each month that samples are analyzed”.

If MS results are out of control, the unspiked sample result must be qualified or the laboratory must take corrective action to rectify the effect, use another method, or employ the method of standard additions. When the method of choice specifies MS performance acceptance criteria for accuracy, and the laboratory chooses to develop statistically valid, laboratory-specific limits, the laboratory-generated limits cannot be less stringent than the criteria stated in the approved method. In addition, if a MS duplicate is analyzed, the laboratory may choose to follow the method defined acceptance criteria or develop statistically valid acceptance criteria that are not less stringent than the criteria stated in the approved method.

When spiking with multi-component standards, if the method does not specify the spiking components, the following will apply.

No. of Target Analytes	Requirement
1-10	Spike All
11-20	Minimum of 10 or 80%, whichever is greater
>20	Minimum of 16

Add a concentration that yields results within the operational range of the, or method specified level to selected samples. Use the same concentration as for the Laboratory Fortified Blank (LFB) to allow analysts to separate the matrix’s effect from the laboratory’s performance.

The concentration of the spiked samples must be bracketed by the calibration range. If the spiked sample result is over the calibration range, the spiked sample must be diluted and re-analyzed. It is not acceptable to dilute the sample first and then add the spike solution so as not to affect bias attributed to matrix.

The volume of spike solution used in MS preparation must in all cases be ≤ 5% of the total MS volume. It is preferable that the spike solution constitutes ≤ 1% of the total MS volume so that the MS can be considered a whole volume sample with no adjustment (i.e., volume correction) by calculation necessary. If the spike solution volume constitutes >1% of the total sample volume, the sample concentration must be adjusted by calculation.

If the sample concentration is below the reporting limit, the laboratory may to choose to use the actual value detected or zero for the amount of target in the unspiked sample. The choice must be described in the Quality Assurance Manual and/or Standard Operating Procedure and must not include both options. If the sample concentration is determined to be a negative number, zero must be used for the amount of target in the unspiked sample.

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.)

Parameters Not Amenable to Spiking

Acidity	** EPA 1680 and 1681 both	Chlorophyll *a*
Alkalinity	require a Matrix Spike.	Color – ADMI
Bacteriological Parameters**	BOD/CBOD	Color - PtCo

Conductivity
Dissolved Oxygen
Free Available Chlorine
Hardness – Titration
Ignitability
Paint Filter Test
Residues – All
pH
Salinity
Sulfite
Temperature
Total Residual Chlorine
Turbidity
Vector Attraction Reduction - (All Options)