| **10.0 Cyanide Data Validation** |
| --- |
| **10.1** **Cyanide Data Review – Blank Data Analysis** |
| **A method blank is used to assess contamination from the laboratory environment, equipment, and/or reagents, so a method blank must be carried throughout the entire sample preparation and analytical process for each batch of samples analyzed. This includes exposure to all glassware, equipment, solvents, filtration, and reagents that are used with field samples. Any free cyanide measured in the method blank that exceeds the quantitation limit indicates that contamination is present. The source of the contamination should be determined and corrected before performing any sample analysis. Any sample included in an analysis batch that has an unacceptable method blank concentration should be reanalyzed in a subsequent batch after the contamination problem is resolved. One reagent blank per analytical batch or one in every 20 samples should be used to determine if contamination or any memory effects are occurring.** |
| 10.1.1 Is the method blank data present for each batch of approximately 20 samples (matrix and sample number dependent), including TCLP?*Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results may be qualified as estimated (“J” for positive results and “UJ” for non-detected compounds) based upon the Data Validator’s judgment.*  | **Indicate yes or no:** |
| 10.1.2 Is there an indication that samples in the batch associated with the blank were diluted? **Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).***Acton: If yes, identify dilution factors.* | **Indicate yes or no:****If yes, list the dilution factor(s):** |
| 10.1.3 Was cyanide detected in any blanks? Was cyanide found in the samples associated with the blank? **Note: A list of samples associated with each of the contaminated blanks should be prepared.** *Action: If blank contamination is identified, follow the directions in the Table 10-1 below for qualifying data based on blank results.* | **Indicate yes or no:****If yes, the sample IDs associated with the blank and summarize any actions taken:** |

|  |
| --- |
| **Table 10-1: Blank Actions for Cyanide** |
| **Blank Result** | **Sample Result** | **Action** |
| Detect ≤ QL | Non-detect | No qualification |
| Detect ≤ QL | Report at QL and qualify as U |
| > QL | J+ or no qualification |
| > QL | Non-detect | No qualification |
| Detect ≤ QL | Report at QL and qualify as U |
| > QL but < 10x the Blank Result | Report at Blank Result and use professional judgment to qualify results as J+ or R |
| ≥ 10x the Blank Result | No qualification |

|  |
| --- |
| **10.2 Cyanide Data Review - Duplicates** |
| **Duplicate samples are used to demonstrate acceptable method precision by the laboratory at the time of analysis. Field samples should be used for duplicate sample analysis. At least one duplicate sample should be prepared and analyzed from each batch of samples of a similar matrix type (*e.g.*, aqueous/water or soil/sediment/waste).** |
| 10.2.1 Did the project SAP, QAPP, or DQOs include collecting collocated or duplicate samples? If so, were an appropriate amount duplicates collected? | **Indicate yes, no, or N/A:**  |
| 10.2.2 Was the duplicate analysis performed on a field sample? | **Indicate yes, no, or N/A:**  |
| 10.2.3 Were RPDs within the established control limits (≤20%)??*Action: Determine whether RPD values exceed control limits by using Table 10-2 below. If duplicate sample results are outside of the criteria, samples with detected results should be qualified as estimated and flagged with “J”. Non-detected results should be qualified as estimated and flagged with “UJ”.* | **Record the recovery data out of criteria and control limits.** |
| 10.4.4 Verify the calculations for at least one RPD.$$RPD= \frac{\left|S -D\right|}{{\left(S +D\right)}/{2}} ×100$$Where:S = Sample result (original)D = Duplicate result | **Show results of verified RPD calculation:** |

|  |
| --- |
| **Table 10-2: Duplicate Sample Actions for Cyanide**  |
| **Criteria** | **Action** |
| **Detect** | **Non-detect** |
| Both original sample and duplicate sample results are ≥ 5x the QL and RPD > 20%\* | J | UJ |
| RPD > 100% | Use professional judgment | Use professional judgment |
| Both original sample and duplicate sample results are ≥ 5x the QL and RPD ≤ 20% | No qualification | No qualification |
| Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate > QL\* | J | UJ |
| Original sample or duplicate sample result < 5x the QL (including non-detects) and absolute difference between sample and duplicate ≤ QL | No qualification | No qualification |

\* Project DQOs may allow the use of less restrictive criteria (*e.g.*, 35% RPD, 2x the QL) to be assessed against duplicate soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.

|  |
| --- |
| **10.3 Cyanide Data Review – Laboratory Control Samples** |
| **Laboratory Control Samples (LCSs) are analyte-free water or solid, clean control matrixes similar to the sample matrix, spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received. The LCS should be spiked at the same levels and using the same spiking materials as the corresponding MS and MSD. When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can acceptably perform unbiased analysis in a clean matrix.**  |
| 10.3.1 Was an LCS prepared, extracted, analyzed, and reported once per batch of 20 samples?**Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted. This information should be found in the injection log.***Action: If LCS information cannot be found, contact the applicable party or laboratory for re-submittal of the data package. If LCS information is not present, qualify all detected results as “J” and all non-detect results as “UJ” or reject all results based on best professional judgment. If matrix spikes were not performed either, reject all results.* | **Indicate yes or no:****Summarize any actions taken:** |
| 10.3.2 Were LCS results within suggested QC limits (85% - 115%) or limits provided by the lab? **Note: Use 85% - 115% unless appropriate lab-specific LCS limits have been developed. The results for solid and aqueous LCSs should always be within the control limits. If out of limits, the laboratory should terminate the analysis, correct the problem, and the samples should be re-digested and re-analyzed. If still unacceptable, then all samples after the last acceptable method blank must be re-prepared and reanalyzed, along with all other appropriate analysis batch QC samples.** *Action: Refer to Table 10-3 below to determine whether data needs to be qualified. If >115%, qualify all detect data as “J+”. If <85%, qualify detect data as “J-“ and non-detect data as “UJ”.* | **Indicate yes or no:****Summarize any actions taken:** |
| 10.3.3 Verify the calculations for at least one %R.$$\%R = \left(\frac{Measured Concentration }{Spiked Amount}\right) ×100$$*Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.* | **Show results of verified %R calculation:** |

|  |
| --- |
| **Table 10-3: Lab Control Sample Actions** |
| **Criteria** | **Action** |
| **Detect** | **Non-detect** |
| LCS not prepared with samples  | J or R | UJ or R |
| LCS not prepared at specified concentrations | J | UJ |
| Aqueous/water and soil/sediment %R < 50% | J- | R |
| Aqueous/water and soil/sediment %R 50-84% | J- | UJ |
| Aqueous/water and soil/sediment %R 85-115% | No qualification | No qualification |
| Aqueous/water and soil/sediment %R 116-140% | J+ | No qualification |
| Aqueous/water and soil/sediment %R > 140% | R | No qualification |

|  |
| --- |
| **10.4 Cyanide Data Review – Matrix Spikes** |
| **Matrix spikes are performed to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology. At least one spiked sample (pre-distillation) should be prepared and analyzed for each batch of samples with a similar matrix type (*e.g.*, aqueous/water or soil/sediment/waste). The data user may also require that a specific sample be used for the matrix spike sample analysis.**  |
| 10.4.1 Is matrix spike data present for each batch of approximately 20 samples (matrix and sample number dependent)?*Action: If not present, request information from the applicable party or laboratory. If the required method blank(s) was not analyzed, sample results should be qualified as estimated (“J” for detected results and “UJ” for non-detected results).*  | **Indicate yes or no:** |
| 10.4.2 Was each matrix spike prepared from a field sample?*Action: If not, sample results should be qualified as estimated (“J” for detected results and “UJ” for non-detected results).*  | **Indicate yes or no:** |
| 10.4.3 Do all pre-distillation matrix spike sample results fall with the established control limits?*Action: If not, verify a post-distillation spike was prepared and analyzed.* | **Indicate yes or no:** |
| 10.4.4 If a post-distillation spike was analyzed, were matrix spike sample results within the established control limits?*Action: Use Table 10-4 below to qualify results that are not within the established control limits.* | **Indicate yes or no:** |
| 10.4.5 Verify the calculations for at least one %R.$$\%R = \frac{SSR -SR}{SA} ×100$$Where:SSR = Spike sample ResultSR = Sample ResultSA = Spike Added**Note: When the sample result is reported as non-detect, the sample result should be set at 0 for calculating the %R.** | **Show results of verified %R calculation:** |
| 10.4.6 If a matrix spike duplicate was performed, were any relative percent differences (RPDs) for matrix spike and matrix spike duplicate recoveries are outside the QC limits (RPD > 20%)?**Note:  The MS/MSD results may be used to determine the need for data qualification.  Outliers should be identified.** | **Record the recovery data out of criteria and control limits.** |
| 10.4.7 Verify the calculations for at least one RPD.$$RPD = \frac{\left|MSR -MSDR\right|}{\left(\frac{MSR +MSDR}{2}\right)} ×100$$Where: MSR= Matrix spike result for the spiking analyte in the MS sample MSDR= Matrix spike result for the spiking analyte in the MSD sample  | **Show results of verified RPD calculation:** |

|  |
| --- |
| **Table 10-4: Matrix Spike Actions for Cyanide**  |
| **Criteria** | **Action** |
| **Detect** | **Non-detect** |
| Matrix Spike not performed at the specified frequency | J | UJ |
| Matrix Spike not prepared from a field sample | J | UJ |
| Matrix Spike %R < 30%Post-distillation spike %R < 75% | J- | R |
| Matrix Spike %R < 30%Post-distillation spike %R ≥ 75% | J | UJ |
| Matrix Spike %R 30-74%Post-distillation spike %R < 75% | J- | UJ |
| Matrix Spike %R 30-74%Post-distillation spike %R ≥ 75% | J | UJ |
| Matrix Spike %R > 125%Post-distillation spike %R > 125% | J+ | No qualification |
| Matrix Spike %R > 125%Post-distillation spike %R ≤ 125% | J | No qualification |
| Matrix Spike %R < 30%No post-distillation spike performed | J- | R |
| Matrix Spike %R 30-74%No post-distillation spike performed | J- | UJ |
| Matrix Spike %R 75-125%No post-distillation is required | No qualification | No qualification |
| Matrix Spike %R > 125%No post-distillation spike performed | J+ | No qualification |

NOTE: Project DQOs may allow the use of less restrictive criteria (*e.g.*, 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples due to laboratory variability arising from the sub-sampling of non-homogenous soil samples.