Summary of Changes: ISM02.3 to ISM02.4

The following Summary of Changes highlights the major modifications implemented in SOW ISM02.4 compared to SOW ISM02.3.

This is a high-level summary and is not intended to be a complete or comprehensive listing of every modification. Interested parties are strongly encouraged to read the complete SOW ISM02.4 and familiarize themselves with all of the requirements.

Global

- References to "ISM02.3" have been updated to "ISM02.4".

Exhibit A

- **Section 5.4.4.6** – Requirements that the daily check of the infrared (IR) temperature detection device be documented and records maintained on file have been added.

Exhibit B

- **Section 2.2.1** – The instructions for data resubmission have been updated to indicate that corrected data submitted as "Additional Data" at the request of an EPA Regional data reviewer shall only include the affected pages and be accompanied by a revised Sample Delivery Group (SDG) Narrative documenting the reason(s) for the resubmittal.
- **New Section 2.4.5.9** – The following instructions for documenting the use of collision or reaction cell in the SDG Narrative have been provided: The Contractor shall document the use of collision or reaction cells for reducing ICP-MS interferences. The Contractor shall document: the type of cell and cell mode; the gas(es) used; any additional manufacturer-recommended instrument setup or QC applied to establish analytical conditions (e.g., oxide ratios); list the analysis conditions applied to each analyte and internal standard (e.g., mass), along with any changes in the course of the analytical sequence; and any deliberate use of molecular species to avoid isobaric interferences (e.g., $^{75}As^{16}O$ at mass 91 to avoid $^{40}Ar^{35}Cl$ at mass 75).
- **Section 3.3.7.1, Table 5** – Table 5 (Codes for Labeling Data) has been updated to clarify that laboratory QC samples not part of the SDG are to be reported as "ZZZZZZ"; Language for Footnote 1 has been added to clarify that instrument QC samples must not be reported as "ZZZZZZ".
- **Section 3.3.12** – The preparation methods for cyanide analysis have been updated from Midi-distillation and Micro-distillation to: Midi-distillation_Aqueous, Midi-distillation_Soil, Micro-distillation_Aqueous, and Micro-distillation_Soil.
- **Sections 3.4.2.2.4, 3.4.6.2.2, 3.4.6.2.4, 3.4.7.2.2, 3.4.7.2.4, 3.4.8.2.2, and 3.4.8.2.4** – Language in these sections has been updated to clarify that the value of the result is to be reported on Form 1-IN, Form 5A-IN, Form 5B-IN, and Form 6-IN if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; and that the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, is to be reported if the concentration is less than the adjusted MDL.

- **Section 3.4.10.2.1** – Form 8-IN reporting requirements have been clarified as follows: Under column "Initial Sample Result (I)", enter the measured value, corrected for any dilutions, for each analyte. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.
- **Section 3.4.10.2.3** – Form 8-IN reporting requirements have been clarified as follows: Under column "Serial Dilution Result (S)", enter the measured value for each analyte in the diluted sample. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.
- **Section 3.4.10.2.6** – The reporting requirements for the "*" flag under the "Q" column on Form 8-IN have been updated to indicate that the original sample concentration (reported on Form 1-IN) is to be compared to 50 times the adjusted MDL, and not to 50 times the unadjusted MDL reported on Form 9-IN.
- **Section 3.4.12.2.3** – The interelement correction factor for each ICP-AES analyte is to be reported, on Form 10A-IN and Form 10B-IN, to the number of decimal places stored by the instrument and used for correcting the analyte results, so that the adjustments made to sample results due to interference corrections can be accurately reproduced during data review.
- **Section 3.4.16.2.5** – The requirement to report the percent Relative Standard Deviation (%RSD) to one decimal place has been removed from the Form 13-IN instructions. The %RSD is to be reported to the number of significant figures specified in Section 3.2.4.
- **Section 3.4.18.2.4** – The Initial Calibration Percent Difference equation has been corrected to:

  $$\%D = \frac{\text{Found - True}}{\text{True}} \times 100$$

- **Section 3.4.19.2.6** – The requirements for the reporting of the calibration curve weighting factor have been updated as follows:

  The Inverse of the variance, inverse of the standard deviation, and inverse square of the standard deviation have been added to the list of weighting factors that may be used and reported on Form 16-IN to accommodate instruments that utilize these options.

  The reporting of "Inverse Square" for the weighting factor inverse square of concentration has been updated to "Inverse Square Conc".

Exhibit D – Introduction

- **Section 4.0** – Language has been added stating that stock solutions that are past the manufacturer's expiration date shall not be used to prepare analytical standards.

Exhibit D – General

- **Section 6.2.5** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Section 10.1.2.1** – The instructions for the pH determination of aqueous/water samples have been clarified as follows:

  The determination of pH is required for all aqueous/water samples at the time of receipt at the laboratory. For samples scheduled for Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma – Mass Spectrometry (ICP-MS), or Mercury analysis, if the pH is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative. For samples scheduled for Cyanide analysis, if the pH is <10, the Contractor shall immediately notify the Sample Management Office (SMO) of the affected sample(s) and pH value(s). SMO will contact the EPA Region. The EPA Region may require the Contractor to either proceed with the analysis or to not analyze the sample(s). The EPA resolution shall be documented in the SDG Narrative.

The Contractor shall follow the procedures for pH measurement based on the EPA SW-846 Method 9041A, Revision 1, July 1992 (pH paper) or the EPA SW-846 Method 9040C, Revision 3, November 2004 [electrometric method (i.e., pH meter and electronic hand-held pen)].

- **New Section 12.1.5** – Language has been added to clarify that the Leachate Extraction Blank (LEB) is not to be analyzed at a dilution under any circumstances.

Exhibit D – ICP-AES

- **Section 6.1.5** – Language has been added to clarify that manufacturer’s instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Sections 7.3, 7.3.3, and 10.4.3** – All references to "rinse blank" have been replaced with "rinse solution" to differentiate this sample from the calibration blank and preparation blank.
- **Section 8.3** – The contract required holding time requirements have been clarified as follows: The holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of TCLP or SPLP leachates is 180 days from the date of extraction.
- **Section 9.3.4.2** – Inverse of the variance, inverse of the standard deviation, and inverse square of the standard deviation have been added to the list of weighting factors that may be used to calculate the calibration curve to accommodate instruments that utilize these options.
- **Removed Section 9.3.5.4** – The requirement that the y-intercept of the calibration curve for each analyte shall be below the CRQL has been removed since the y-intercept and CRQL are not in the same units and, therefore, cannot be compared.
- **Section 9.5.1** – The requirement that the Contractor shall not analyze a blank immediately prior to the analysis of the Continuing Calibration Verification (CCV) has been added.
- **Section 10.1.1** – The instructions for the pH determination during the preparation of aqueous/water samples have been clarified as follows:

  If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

  If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.

- **Section 12.6.6.2** – Language has been updated to clarify that in instances where there is more than one serial dilution per matrix, per SDG, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix in the SDG.
- **Section 12.8.1** – Language has been added to clarify that the interelement corrections factors are to be determined following the procedures provided by the instrument manufacturer.

Exhibit D – ICP-MS

- **Section 4.1** – The following requirements have been added to address isobaric elemental interferences: Interferences from doubly charged ions may not be correctable. The Contractor shall monitor the intensities of the singly charged ions of those isotopes that can cause doubly charged interferences and note high readings in the Sample Delivery Group (SDG) Narrative.
- **Section 4.3** – The use of collision cell to reduce isobaric polyatomic ion interferences is permitted.
- **Sections 4.5, 7.3, 7.3.3, and 10.3.3** – All references to "rinse blank" have been replaced with "rinse solution" to differentiate this sample from the calibration blank and preparation blank.
- **Section 6.1.5** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Section 9.4.4.2** – Inverse of the variance, inverse of the standard deviation, and inverse square of the standard deviation have been added to the list of weighting factors that may be used to calculate the calibration curve to accommodate instruments that utilize these options.
- **Removed Section 9.4.5.4** – The requirement that the y-intercept of the calibration curve for each analyte shall be below the CRQL has been removed since the y-intercept and CRQL are not in the same units and, therefore, cannot be compared.
- **Section 9.6.1** – The requirement that the Contractor shall not analyze a blank immediately prior to the analysis of the CCV has been added.
- **Section 10.1.1** – The instructions for pH determination during the preparation of aqueous/water samples have been clarified as follows:

  If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

  If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.
- **Section 17.0, Table 2** – Nd^{++} and Sm^{++} have been added as "Other" interferents for ^{75}As; $Sm^{++}$and Gd^{++} for ^{78}Se; Gd^{++} for ^{80}Se; and $Dy^{++}$and Er^{++} for ^{82}Se because the doubly charged ions of these rare earth elements isotopes could erroneously be measured as isotopes of Arsenic or Selenium.

- **Section 17.0, Table 3** – Mass 150 is to be monitored, in addition to masses 77 and 82, for Arsenic; and masses 156, 160, and 164 are to be monitored, in addition to masses 78 and 82, for Selenium since isotopes of rare earth elements at these masses could cause interferences for these two analytes.
- **Section 17.0, Table 6** – Footnote 1 and Equation 16 regarding spiking level adjustments have been added under Table 6 (Spiking Levels for Matrix Spike Sample Analysis) for consistency with Table 2 in Exhibit-D/ICP-AES.

Exhibit D – Mercury

- **Section 6.1.2** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.
- **Section 8.3** – The contract required holding time requirements have been clarified as follows: The holding time for mercury is 26 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of TCLP or SPLP leachates is 26 days from the date of extraction.
- **Section 9.3.4.2** – Inverse of the variance, inverse of the standard deviation, and inverse square of the standard deviation have been added to the list of weighting factors that may be used to calculate the calibration curve to accommodate instruments that utilize these options.
- **Removed Section 9.3.5.4** – The requirement that the y-intercept of the calibration curve shall be below the CRQL has been removed since the y-intercept and CRQL are not in the same units and, therefore, cannot be compared.
- **Section 9.5.1** – The requirement that the Contractor shall not analyze a blank immediately prior to the analysis of the CCV has been added.
- **Section 10.1.1** – The instructions for pH determination during the preparation of aqueous/water samples has been clarified as follows:

  If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

  If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.
- **Section 17.0, Table 1** – The language for the frequency of the Continuing Calibration Blank (CCB) has been clarified as follows: Every hour and <u>at the beginning</u> and end of each analytical sequence. Performed immediately after the CCV.

Exhibit D – Cyanide

- **Section 6.1.1** – Language has been added to clarify that manufacturer's instructions are to be followed for the calibration and maintenance of adjustable pipettes.

- **Section 8.3** – The contract required holding time requirements have been clarified as follows: The holding time for cyanide is 12 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of SPLP leachates is 12 days from the date of extraction.
- **Section 9.3.4.2** – Inverse of the variance, inverse of the standard deviation, and inverse square of the standard deviation have been added to the list of weighting factors that may be used to calculate the calibration curve to accommodate instruments that utilize these options.
- **Removed Section 9.3.5.4** – The requirement that the y-intercept of the calibration curve shall be below the CRQL has been removed since the y-intercept and CRQL are not in the same units and, therefore, cannot be compared.
- **Section 9.5.1** – The requirement that the Contractor shall not analyze a blank immediately prior to the analysis of the CCV has been added.
- **Section 10.1.1** – The language regarding sample pH measurement has been clarified as follows: The Contractor shall measure the sample pH at the time of sample receipt to verify that the sample was properly preserved (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1).

Exhibit H

- **Section 2.2.2** – The rounding requirements have been clarified as follows: The values reported by the Contractor are used for data assessment. No raw data values in the SEDD files shall be rounded. The Contractor shall not use rounded intermediate values in calculating the final result, and no rounding shall be performed until reaching the final result.
- **Section 3.1.7** – The requirements for the Analysis node associated with an initial analysis, dilution, or reanalysis have been clarified as follows: Each SamplePlusMethod node must contain at least one Analysis node. A separate Analysis node is required for each dilution or reanalysis. Each InstrumentQC node (other than Initial Calibration) must contain one Analysis node. Any reanalysis must be preceded by an initial analysis. Any analysis reported as a dilution must also have a less-diluted analysis reported as initial. The initial analysis does not have to precede the diluted analysis.
- **Section 3.1.10** – The language has been updated, as follows, to include requirements for the reporting of collision or reaction cell internal standards data in the Peak node: Each Analyte node must contain at least one Peak node. For Level 2b, only the Analyte nodes under InstrumentQC must contain a Peak node. Within a RunBatch, a peak must be consistently identified. For an Inductively Coupled Plasma – Mass Spectrometer (ICP-MS) using collision or reaction cells on an analyte-by-analyte basis, internal standards reported from collision/reaction cell mode shall be reported with a "-Gas" suffix. If an internal standard applies to both the collision/reaction cell path and the normal path target analytes, report the internal standard results as separate peaks, using the "-Gas" suffix in PeakID to distinguish the collision/reaction cell results from the normal path results.
- **Section 7.0, Tables 1, 2, and 3** – The Instructions for the ClientMethodCode element under the SamplePlusMethod node have been updated from "Not required" to "Report "TCLP" or "SPLP" when applicable".
- **Section 7.0, Tables 1, 2, and 3** – "Report "None" if sample was not preserved" has been added to the Instructions for the Preservative element under the SamplePlusMethod node.

- **Section 7.0, Tables 1, 2, and 3** – "Tissue samples do not require "Percent_Solids" or "pH". Wipe samples do not require "Percent_Solids", "pH", or "Temperature"" has been added to the Instructions for the CharacteristicType element under the SamplePlusMethod/Characteristic node.
- **Section 7.0, Tables 1, 2, and 3** – The Instructions for the CharacteristicValue element under the SamplePlusMethod/Characteristic node have been updated to: For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
- **Section 7.0, Table 1** – The Instructions for the OriginalLabAnalysisID element under the SamplePlusMethod/Analysis node have been updated from "Not required" to "If a dilution or reanalysis of a previously analyzed sample is performed, report the Lab Analysis ID of the original sample that was used for the dilution or reanalysis".
- **Section 7.0, Tables 1, 2, and 3** – "Report "Wet" for tissue samples or for any other matrices (not aqueous/water) for which the results are not corrected for percent solids" has been added to the Instructions for the ResultBasis element under the SamplePlusMethod/Analysis node.
- **Section 7.0, Tables 1, 2, and 3** – "Report "Negative" for PB or LEB results less than the negative MDL (-MDL)" has been added to the Instructions for the ResultType element under the SamplePlusMethod/ReportedResult node.
- **Section 7.0, Tables 1, 2, and 3** – The Instructions for the DetectionLimit element under the SamplePlusMethod/Analysis/Analyte node have been updated from "Report the MDL" to "Report the MDL to at least two significant figures".
- **Section 7.0, Table 1** – In the Instructions for the IntermediateResult element under the SamplePlusMethod/Analysis/Analyte node, "uncorrected for dilution" has been replaced with "unadjusted for sample weight/volume, percent solids, or dilution factor".
- **Section 7.0, Tables 1, 2, and 3** – "Report "Negative" for PB or LEB results less than the negative MDL (-MDL)" has been added to the Instructions for the ResultType element under the SamplePlusMethod/Analysis/Analyte node.
- **Section 7.0, Table 1** – "For ICP-MS analysis using collision or reaction cell, a "-Gas" suffix must be applied to the PeakID" has been added to the Instructions for the PeakID element under the SamplePlusMethod/Analysis/Analyte/Peak node.
- **Section 7.0, Tables 1 and 2** – The Instructions for the ClientMethodCode element under the InstrumentQC node have been updated from "Not required" to "Report "TCLP" or "SPLP" when applicable".
- **Section 7.0, Tables 1 and 2** – The Instructions for the ClientMethodModificationID element under the InstrumentQC node have been updated from "Not required" to "Report the Modified Analysis Number, if applicable".

- **Section 7.0, Tables 1 and 2** – The Instructions for the DetectionLimit element under the InstrumentQC/Analysis/Analyte node have been updated from "Report the current MDL from the default aqueous preparation method or other appropriate method" to "Report the current MDL from the default aqueous preparation method or other appropriate method to at least two significant figures".
- **Section 7.0, Table 1** – In the Instructions for the IntermediateResult element under the InstrumentQC/Analysis/Analyte node, "uncorrected for dilution" has been replaced with "unadjusted for sample weight/volume, percent solids, or dilution factor".
- **Section 7.0, Tables 1 and 2** – "Report "Negative" for ICB or CCB results less than the negative MDL (-MDL)" has been added to the Instructions for the ResultType element under the InstrumentQC/Analysis/Analyte node.
- **Section 7.0, Tables 1 and 2** – "For ICP-MS analysis using collision or reaction cell, a "-Gas" suffix must be applied to the PeakID" has been added to the Instructions for the PeakID element under the InstrumentQC/Analysis/Analyte/Peak node.
- **Section 7.0, Table 1** – Instructions have been provided to report the ICP-AES interelement correction factors to the number of decimal places stored by the instrument and used for correcting the analytical data in the CorrectionFactor element under the InstrumentQC/AnalysisGroup/Analyte/AnalyteComparison node.
- **Section 7.0, Tables 1 and 2** – "Inverse_Of_Variance", "Inverse_Of_Standard_Deviation", and "Inverse_Square_Of_Standard_Deviation" have been added to the list of valid values in the Instructions for the WeightingFactor element under the InstrumentQC/AnalysisGroup/Analyte/Peak node.
- **Section 7.0, Table 1** – Instructions have been provided to report the analytes as they appear in the SOW in the ClientAnalyteName element under the InstrumentQC/AnalysisGroup/Analyte/Peak/PeakComparison node.
- **Appendix A, Table A-1** – "ReportedResult/DetectionLimit" has been replaced with "Analysis/Analyte/DetectionLimit" in the Instructions for the "DetectionLimit" column in the MDL study data deliverable table.
- **Appendix A, Table A-1** – "ReportedResult/DetectionLimitUnits" has been replaced with "Analysis/Analyte/DetectionLimitUnits" in the Instructions for the "DetectionLimitUnits" column in the MDL study data deliverable table.