HRSM01.2 Clarifications

The following clarifications highlight the technical modifications, known as of this date, that will be necessary to SOW HRSM01.2 in addition to the updates listed in the "Summary of Changes: HRSM01.1 to HRSM01.2" document on EPA's Superfund Analytical Services and Contract Laboratory Program website. The purpose of these clarifications is to respond to questions raised by a number of stakeholders and to make the SOW easier and less confusing to implement. It is anticipated that additional questions may lead to further clarifications during the base period. Responses or changes to address those questions will be assessed, along with these clarifications, for inclusion into a new revision of the SOW prior to moving into the first option period of the contracts.

Global

• The following EPA staff title has been updated:

EPA Regional Laboratory Contracting Officer Representative (EPA Regional Laboratory COR) has been replaced with EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR).

Exhibit B

- Section 1.1, Table 1, Item E Deliverables for Proficiency Testing (PT) Audits are no longer to be
 distributed to QATS. All deliverables are to be delivered only to the Sample Management Office
 (SMO).
- **Section 3.4.2.2.11/Last sentence** The requirement to list the labeled compounds and labeled cleanup standards on Form 1A-HR has been removed.
- Section 3.4.2.2.12 The reporting instructions for the "Concentration" column on Form 1A-HR have been updated as follows: "For detected CDD/CDF target analytes and WHO Toxic Congeners (including co-eluting WHO Toxic Congeners) meeting all identification criteria, enter the concentration (if the result is greater than or equal to the adjusted MDL) in the appropriate units in the "Concentration" column. Leave the field blank if the analyte is not identified. Enter the concentration for detected non-WHO Toxic Congeners in the appropriate units in the "Concentration" column. If the non-WHO Toxic Congener is not detected, report the adjusted CRQL, calculated from the CRQL listed in Exhibit C Chlorinated Dibenzo-p-Dioxins and Chlorinated Dibenzofurans and Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits, in this "Concentration" column. All tissue results must be reported on a wet weight basis. For positively identified target analytes, the Contractor shall report the concentrations as uncorrected for blank contaminants. Report all analytical results to two (2) significant figures."
- Section 3.4.2.2.13.2 The instructions for reporting the "J" flag on Form 1A-HR have been updated as follows: "Indicates an estimated value. This flag is used for any detected CDD/CDF analyte or WHO Toxic Congener meeting all the identification criteria in Exhibit D Analytical Methods, when the result is greater than or equal to the adjusted MDL and less than the adjusted CRQL calculated from the CRQL listed in Exhibit C Chlorinated Dibenzo-p-Dioxins and Chlorinated Dibenzofurans and Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits. This flag is also used for any detected non-WHO Toxic Congener meeting all the identification criteria in Exhibit D Analytical Methods, when the result is less than the adjusted CRQL, as well as for detected homologues and total homologues as applicable."

- Section 3.4.2.2.14 The reporting instructions for the "EMPC/EDL/MDL" column on Form 1A-HR have been updated as follows and the NOTE has been removed: "Under column "EMPC/EDL/MDL", enter the calculated EMPC, EDL, or MDL as indicated in Exhibit D Analytical Methods, Section 11.0, for CDD/CDF target analytes and WHO Toxic Congeners (including co-eluting WHO Toxic Congeners) that are not identified. Report the MDL value for the CDD/CDF target analytes and the WHO Toxic Congeners in this column when the analyte was not detected and the calculated EDL is less than the adjusted MDL. Leave the field blank for non-WHO Toxic Congeners."
- New Section 3.4.2.2.17 The following instructions for Form 1A-HR have been added: "A separate Form 1A-HR is required for the confirmation analysis for TCDF or PCB-77, PCB-126, or PCB-169, if applicable. Report the confirmation analysis column information in the "GC column" field. Enter the concentration or EDL/MDL/EMPC and the applicable laboratory qualifier for the confirmed analytes in the appropriate fields."
- Sections 3.4.3.1, 3.4.4.1, and 3.4.5.1 The last sentence in each section has been updated to indicate that Form 1B-HR, Form 1C-HR, and Form 1D-HR are not required for instrument blank and LCS/LCSD analyses.
- **Sections 3.4.7.2 and 3.4.9.2** The reporting instructions in each section have been updated to indicate that the "Date Received" field on Form 3A-HR and Form 4-HR is to be left blank.
- Sections 3.4.12 and 3.4.13 The reporting instructions for Form 6A-HR, Form 6B-HR, Form 6C-HR, Form 7A-HR, and Form 7B-HR have been updated to indicate that the internal standards are to be listed on the forms along with the other labeled analogs where necessary. Contractors may leave blank any fields on the forms where a response is expected for %RSD, RR, RRF, or %D for the internal standards, and should enter a value of 1.0 for RRT for these analytes on Form 6C-HR and Form 7B-HR. In addition, a response is required, for ion abundance ratio of the internal standards, on Form 6B-HR.

Exhibit B - Forms

• Form 5B-HR – The "Quality Control (QC) Limits:" header field has been updated to "Quality Control (QC) Limits: ≤ 25%".

Exhibit D – CDD/CDF

• Section 9.3.2.2 – The procedure for documenting the HRSM system tune has been updated to the following requirements: "Documentation of the instrument resolving power shall be completed by recording the peak profiles of the reference peaks chosen for each descriptor. While generating the peak profiles, the detector zero shall be adjusted to allow presentation of the profile shoulders on-scale (Method 8290A, Figure 5 – Peak Profiles Representing Two PFK Reference Ions at m/z 305 and 381) so the resolution can be manually evaluated. The format of the peak profiles shall show a horizontal axis calibrated in u or ppm, and a vertical scale in percent maximum signal. The result of the peak width measurement (perform at 5% of the maximum, which corresponds to the 10% valley definition) must appear on the profile, and must not exceed 100 ppm (i.e., 0.038 u for a peak at m/z 380.9760). Both the low and the high exact masses must be displayed to demonstrate the accuracy of the mass calibration. The mass resolution and the accelerating voltages of each mass profile must be present on the profile. This documentation shall be provided for a minimum of one descriptor during each check of the

- static resolving power of each instrument used, and shall contain identifying information, including instrument ID, time, and date."
- Section 9.5.7.2 The entire section ("The isomer specificity shall be resolved with a valley of ≤25% in all calibration standards") has been removed.
- **Section 17/Table 5** The "LCS/LCSD %R" values for the labeled compounds and the cleanup standard have been updated. (An updated Table 5 is attached.)
- Section 17/Table 11 Analyte "1,2,3,6,7,8-HxCDD" on row 7 in the Table has been updated to "1,2,3,6,7,8-HxCDF".

Exhibit D - CBC

- Section 9.3.2.2/Second sentence The procedure for documenting the HRSM system tune has been updated to the following requirements: "While generating the peak profiles, the detector zero shall be adjusted to allow presentation of the profile shoulders on-scale (Method 8290A, Figure 5 Peak Profiles Representing Two PFK Reference lons at m/z 305 and 381) so the resolution can be manually evaluated. The format of the peak profiles shall show a horizontal axis calibrated in u or ppm, and a vertical scale in percent maximum signal. The result of the peak width measurement (perform at 5% of the maximum, which corresponds to the 10% valley definition) must appear on the profile, and must not exceed 100 ppm (i.e., 0.038 u for a peak at m/z 380.9760). Both the low and the high exact masses must be displayed to demonstrate the accuracy of the mass calibration. The mass resolution and the accelerating voltages of each mass profile must be present on the profile. This documentation shall be provided for a minimum of one descriptor during each check of the static resolving power of each instrument used, and shall contain identifying information, including instrument ID, time, and date."
- Section 17/Table 2 The m/z formula for exact m/z 359.8415 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_5$ 37 Cl" to " 12 C $_{12}$ H $_4$ 35 Cl $_5$ 37 Cl"; the m/z formula for exact m/z 361.8385 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_4$ 37 Cl $_2$ " to " 12 C $_{12}$ H $_4$ 35 Cl $_4$ 37 Cl $_2$ "; the m/z formula for exact m/z 363.8356 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_3$ 37 Cl $_2$ "; the m/z formula for exact m/z 495.6856 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_9$ 37 Cl" to " 12 C $_{12}$ H $_4$ 35 Cl $_9$ 37 Cl"; the m/z formula for exact m/z 507.7258 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_9$ 37 Cl" to " 13 C $_{12}$ 35 Cl $_9$ 37 Cl"; the m/z formula for exact m/z 509.7229 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_8$ 37 Cl $_2$ " to " 13 C $_{12}$ 35 Cl $_8$ 37 Cl $_2$ "; and the m/z formula for exact m/z 511.7199 has been updated from " 13 C $_{12}$ H $_4$ 35 Cl $_8$ 37 Cl $_4$ " to " 13 C $_{12}$ 35 Cl $_7$ 37 Cl $_3$ ".
- Section 17/Table 5 The congener name for analyte name PCB-208 has been updated from "2,2',3,3,'4,5,5',6,6'-NoCB" to "2,2',3,3',4,5,5',6,6'-NoCB".
- Section 17/Table 6 The CB congener name for analyte name PCB-19 has been updated from "2,2',6'-TrCB" to "2,2',6-TrCB"; the CB congener name for analyte name PCB-208 has been updated from "2,2',3,3',4',5,5',6,6'-NoCB" to "2,2',3,3',4,5,5',6,6'-NoCB"; the CB congener name for analyte name PCB-19L has been updated from "¹³C₁₂-2,2',6'-TrCB" to "¹³C₁₂-2,2',6-TrCB"; and the CB congener name for analyte name PCB-208L has been updated from "¹³C₁₂-2,2',3,3',4',5,5',6,6'-NoCB".

Exhibit H

• **Section 2.1** – The web address for information on the Staged Electronic Data Deliverable (SEDD) has been updated to http://www.epa.gov/clp/staged-electronic-data-deliverable-sedd.

- Section 7/Page 25 The instructions for the AnalyteType element associated with the ReportedResult node have been updated to include "Derived" for Homologues, TEQ, or Total (except for LCS/LCSD analysis).
- Section 7/Page 27 The instructions for the ClientMethodID element associated with the PreparationPlusCleanup node have been updated from "Not required." to "Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements." for sample, LCS/LCSD, and MB/IB analyses."
- **Section 7/Page 28** The AnalyteGroupID element associated with the Analyte node is no longer required for the LCS/LCSD analysis.
- Section 7/Page 30 The instructions for the QuantitationLimitType element associated with the
 Analyte node have been updated from "Report "CRQL_sa"" to "Report "CRQL"", and the
 instructions for the QuantitationLimit element associated with the Analyte node have been
 updated from "For target analytes with SOW specified CRQLs, report the adjusted CRQL to at
 least two significant figures" to "For target analytes, report the CRQL to at least two significant
 figures."
- Section 7/Page 30 The instructions for the ReportingLimitType element associated with the
 Analyte node have been updated from "Report "EDL_sa"" to "Report "EDL"", and the
 instructions for the ReportingLimit element associated with the Analyte node have been
 updated from "Report the adjusted EDL as applicable to at least two significant figures for target
 Dioxins, Furans, and WHO Toxic Congeners" to "For target Dioxins, Furans, and WHO Toxic
 Congeners, report the EDLs as applicable to at least two significant figures."
- Section 7/Pages 30-31 The AnalyteGroup node and associated elements (AnalyteGroupID, AnalyteName, AnalyteNameContext, AnalyteType, CASRegistryNumber, ClientAnalyteID, ClientAnalyteName, and Result) are no longer required for the LCS/LCSD analysis.
- Appendix A, Section 1.0 The format requirement for the Microsoft® Excel file name of the
 Method Detection Limit (MDL) study data deliverable has been updated. The file name format
 is now required to be "MDL_#.xls", where "#" can be any naming convention selected by the
 Contractor. (An updated Appendix A is attached.)
- Appendix A, Table A-1 The MDL study data deliverable table has been updated to include a
 "Required" field that identifies the columns that are always required to be populated in the
 deliverable spreadsheet. (An updated Appendix A is attached.)
- Appendix A, Table A-1 The "SOW" and "ClientMethodCode" column names have been changed to "MethodSource" and "PreparationMethod", respectively. In addition, the "ClientMethodType" column name has been changed to "Method" and the associated Instructions have been updated. (An updated Appendix A is attached.)
- Appendix A, Table A-1 The Instructions for the "ClientMethodCategory", "Level", and "ColumnID" columns have been updated. (An updated Appendix A is attached.)
- Appendix A, Table A-1 The Instructions for the "AnalyzedDate##" column have been updated to include the required Date format. (An updated Appendix A is attached.)

TABLE 5. QUALITY CONTROL ACCEPTANCE CRITERIA FOR CHLORINATED DIBENZO-p-DIOXINS AND CHLORINATED DIBENZOFURANS IN LABORATORY CONTROL SAMPLE/LABORATORY CONTROL SAMPLE DUPLICATE AND SAMPLES

| CDD/CDF | Test Conc (ng/mL) | LCS/LCSD %R | Labeled Compound %R in Sample | |
|--|----------------------|-------------|-------------------------------|--|
| 2,3,7,8-TCDD | 10 | 67-158 | | |
| 2,3,7,8-TCDF | 10 | 75-158 | | |
| 1,2,3,7,8-PeCDD | 50 | 70-142 | | |
| 1,2,3,7,8-PeCDF | 50 | 80-134 | | |
| 2,3,4,7,8-PeCDF | 50 | 68-160 | | |
| 1,2,3,4,7,8-HxCDD | 50 | 70-164 | | |
| 1,2,3,6,7,8-HxCDD | 50 | 76-134 | | |
| 1,2,3,7,8,9-HxCDD | 50 | 64-162 | | |
| 1,2,3,4,7,8-HxCDF | 50 | 72-134 | N/A | |
| 1,2,3,6,7,8-HxCDF | 50 | 84-130 | | |
| 1,2,3,7,8,9-HxCDF | 50 | 78-130 | | |
| 2,3,4,6,7,8-HxCDF | 50 | 70-156 | | |
| 1,2,3,4,6,7,8-HpCDD | 50 | 70-140 | | |
| 1,2,3,4,6,7,8-HpCDF | 50 | 82-132 | | |
| 1,2,3,4,7,8,9-HpCDF | 50 | 78-138 | | |
| OCDD | 100 | 78-144 | | |
| OCDF | 100 | 63-170 | | |
| Labeled Compound | | | | |
| ¹³ C ₁₂ -2,3,7,8-TCDD | 100 | 20-175 | 25-164 | |
| ¹³ C ₁₂ -2,3,7,8-TCDF | 100 | 22-152 | 24-169 | |
| ¹³ C ₁₂ -1,2,3,7,8-PeCDD | 100 | 21-227 | 25-181 | |
| ¹³ C ₁₂ -1,2,3,7,8-PeCDF | 100 | 21-192 | 24-185 | |
| ¹³ C ₁₂ -2,3,4,7,8-PeCDF | 100 | 13-328 | 21-178 | |
| ¹³ C ₁₂ -1,2,3,4,7,8-HxCDD | 100 | 21-193 | 32-141 | |
| ¹³ C ₁₂ -1,2,3,6,7,8,-HxCDD | 100 | 25-163 | 28-130 | |
| ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF | 100 | 19-202 | 26-152 | |
| ¹³ C ₁₂ -1,2,3,6,7,8-HxCDF | 100 | 21-159 | 26-123 | |
| ¹³ C ₁₂ -1,2,3,7,8,9-HxCDF | 100 | 17-205 | 29-147 | |
| ¹³ C ₁₂ -2,3,4,6,7,8,-HxCDF | 100 | 22-176 | 28-136 | |
| ¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD | 100 | 26-166 | 23-140 | |
| ¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF | 100 | 21-158 | 28-143 | |
| ¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF | 100 | 20-186 | 26-138 | |
| ¹³ C ₁₂ -OCDD | 200 | 13-198 | 17-157 | |
| Cleanup Standard | | | | |
| ³⁷ Cl ₄ -2,3,7,8-TCDD | 10 | 31-191 | 35-197 | |

APPENDIX A - FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

1.0 FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

The Method Detection Limit (MDL) study data deliverable consists of a Microsoft® Excel spreadsheet containing the following columns (Table A-1) in the order specified.

The "Required" field in Table A-1 identifies the columns that are always required to be populated.

The Contractor shall provide one spreadsheet for each combination of instrument ID, analytical method, and preparation method used to report results under this contract.

The Contractor shall deliver the spreadsheets to the recipients specified in Table 1 of Exhibit B - Reporting and Deliverables Requirements.

The format for the Microsoft® Excel file name shall be MDL_#.xls, where # can be any naming convention selected by the Contractor.

TABLE A-1. MDL STUDY DATA DELIVERABLE

| Column | Required | Instruction |
|----------------------------|----------|---|
| LabID | Х | Report the agency assigned Lab Code. |
| LabContract | Х | Report the Lab Contract Number per the instructions for Header/LabContract. |
| MethodSource | X | Report the SOW per the instructions for SamplePlusMethod/ClientMethodID. |
| Method | X | Report the analytical method per the instructions for Header/LabDataPackageName. |
| PreparationMethod | | Report the preparation method per the instructions for PreparationPlusCleanup/ClientMethodID. |
| ClientMethodCategory | | Report the method category per the instructions for SamplePlusMethod/ClientMethodCategory if applicable. |
| ClientMethodModificationID | | Report the MA number per the instructions for SamplePlusMethod/ClientMethodModification ID if applicable. Otherwise leave null. |
| Level | | Leave null. |
| Matrix | X | Report the sample matrix per the instructions for SamplePlusMethod/MatrixID. |
| InstrumentID | X | Report the instrument ID per the instructions for Analysis/InstrumentID. |
| ColumnID | X | Report the GC column ID per the instructions for Analysis/Column if applicable. |
| ClientAnalyteID | X | Report the analyte per the instructions for ReportedResult/ClientAnalyteID. |
| Peak | | Leave null. |
| ResultUnits | Х | Report the units for the replicate concentrations reported per the instructions for ReportedResult/ResultUnits. |

Exhibit H - Appendix A

| Column | Required | Instruction |
|---------------------|----------|--|
| Replicate## | X | The Laboratory shall include as many columns as there are replicates reported. Usually this would be seven, but more than seven replicates can be reported. The Laboratory shall report the results of the analysis of each replicate for each analyte. Each column shall be labeled "Replicate##", where the ## shall be replaced with the numeric designation of the replicate (e.g., Replicate01 for the first, Replicate02 for the second, Replicate03 for the third, etc.). |
| LabAnalysisID## | X | Following each Replicate## column, the Laboratory shall report a LabAnalysisID## column, reporting the LabAnalysisID of that replicate for that analyte per the instructions for Analysis/LabAnalysisID. The LabAnalysisID## columns shall be labeled in the same manner as the Replicate## columns. |
| AnalyzedDate## | X | Following each LabAnalysisID## column, the Laboratory shall report a AnalyzedDate## column, reporting the analysis date and time for that replicate for that analyte per the instructions for the Analysis/AnalyzedDate data element. The AnalyzedDate## columns shall be labeled in the same manner as the Replicate## columns. (MMDDYYYYThh:mm:ss) |
| StandardDeviation | Х | Report the calculated standard deviation of the replicates for each analyte to at least three significant figures. |
| StudentsTValue | Х | Report the appropriate Student's T value for the degrees of freedom based on the number of replicates and 99% for the onesided test. |
| DetectionLimit | X | Report the calculated Detection Limit for each analyte per the instructions for ReportedResult/DetectionLimit. |
| DetectionLimitUnits | Х | Report the appropriate units for the preparation method per the instructions for ReportedResult/DetectionLimitUnits. |
| MDLAcceptable | Х | Enter "Y" if the calculated MDL is less than one-half the CRQL for the analyte and matrix. Otherwise enter "N". |
| ExpectedResult | Х | Report the concentration for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResult. |
| ExpectedResultUnits | Х | Report the concentration units for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResultUnits. |

Exhibit H - Appendix A

| Column | Required | Instruction |
|-------------------------|----------|---|
| ConcentrationAcceptable | Х | Enter "Y" if the concentration of the analyte in the MDL standards was less than or equal to 10 times the calculated MDL for that analyte. Otherwise enter "N". |
| EffectiveDate | X | Report the date on which the Laboratory began to use the calculated MDL for reporting sample results for that analyte, instrument, and method formatted per the instructions for Header/DateFormat. This date cannot precede the analysis date of the MDL replicates. |