

QSM 5.3 Change Sheet

1. Module 1 – The following section has been added:

“2.2.4 PT Samples Same as Regular Environmental Samples

PT samples must be analyzed and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples.”

2. Module 1 – The following section has been added:

“3.2.5 PT Samples Same as Regular Environmental Samples

PT samples must be analyzed and evaluated in the same manner as regular environmental samples. A laboratory shall employ the same quality control, sequence of preparation and analytical steps, and replicates as used when analyzing routine samples.”

3. Module 2, Section 5.4 – The following text has been removed:

*“The following shall be implemented **in lieu of** TNI sections 5.4 and 5.5.”*

“General ISO/IEC 17025:2005(E), Sections 5.4 and 5.5 are applicable to this standard.”

The following text has been added:

*“The following shall be implemented **in lieu of** TNI sections 5.4.4 and 5.4.5.”*

“General ISO/IEC 17025:2005(E), Sections 5.4.4 and 5.4.5 are applicable to this standard.”

4. Appendix B, Table B-3 (Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B)) comments for soil drying procedure – The following duplicated text has been removed:

“Commercial PT samples must reflect the grinding, extraction, and analysis steps as a minimum.”

5. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) – Minor formatting changes were made to ensure consistent spacing within the table.

6. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Acceptance Criteria for Biota Sample Preparation – The word “fileted” has been changed to “filleted”.

7. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) QC Check for AFFF and AFFF Mixture Samples, Preparation – An erroneous “,” has been removed.

8. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Acceptance criteria for Sample Cleanup Procedure – The following sentence has been removed:

“Removal of interferences from matrix.”

9. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Comments for Sample Cleanup Procedure – The following word has been replaced:

“background” was replaced with “interferences”.

10. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) minimum frequency for Mass Calibration – “and” has been removed from the second sentence and replaced with “,”.

11. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) acceptance criteria for Calibration, Calibration Verification, and Spiking Standards – the following text has been removed:

“in the standards” has been removed from the parenthetical statement of the third paragraph.

“If qualitative standards containing the branched and linear isomers cannot be purchased (i.e., only linear isomer is available), only the linear isomer shall be quantitated in samples because the identity of other peaks cannot be confirmed as PFAS without a standard.”

“All identified chromatographic peaks confirmed by qualitative standards must be integrated and the areas summed; other peaks that may be present but do not match a standard shall not be included.”

12. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Minimum Frequency for Sample PFAS Identification – The following text has been removed:

“Identify all positive sample detections per method.”

The following text has been added:

“All analytes detected in a sample.”

13. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) acceptance criteria for Sample PFAS Identification - The following text has been removed:

“Documentation of transitions and the ratio between them requires in-house acceptance criteria for comparison of the ratios of standards and field samples.”

“The ratio shall be within 50-150% of the midpoint standard of the ICAL.”

“On days ICAL is not performed, within 50-150% of the initial CCV standard.”

“S/N \geq 10:1 for all ions used for quantification and S/N \geq 3:1 for all ions used for confirmation.”

The following text has been added:

“Documentation of the primary and confirmation transitions and the ion ratio is required.”

“In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%.”

“Signal to Noise Ratio (S/N) must be \geq 10 for all ions used for quantification and must be \geq 3 for all ions used for confirmation.”

14. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) flagging criteria for Sample PFAS Identification - The following text has been added:

“Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as ‘estimated, biased high’.”

15. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Minimum Frequency for Ion Transitions (Precursor -> Product) – The following text has been removed:

“Identify all positive sample detections per method.”

The following text has been added:

“Every field sample, standard, blank, and QC sample.”

16. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) acceptance criteria for Initial Calibration (ICAL) - The following text has been removed:

“Option 3:”

The following text has been added:

“ICAL must meet one of the two options below.”

The following text has been moved from the last paragraph to be the 4th paragraph:

“Analytes must be within 70-130% of their true value for each calibration standard.”

17. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Comments for Initial Calibration (ICAL) – The following sentences have been removed:

“e.g., PFHxS and PFOS are not available as sulfonates but as their corresponding salts, such as Na⁺ and K⁺.”

“Isotope Dilution is required for all analytes.”

“External Calibration is not allowed.”

The following sentence has been added:

“External Calibration is not allowed for any analyte.”

18. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) QC Check for Retention Time Establishment – The following text has been removed:

“Retention Time establishment”

The following text has been added:

“Retention Time window position establishment”

19. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) QC Check for Retention Time – The following text has been added:

“(RT) window width”

20. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) QC Check for Extracted Internal Standard Analytes (EIS) – The following text has been removed.

“Extracted Internal Standard Analytes (EIS)”

The following text has been added:

“Extracted Internal Standard (EIS) Analytes”

21. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) acceptance criteria for Matrix Spike (MS) - The following text has been removed:

“Use in-house LCS limits if project limits are not specified.”

The following text has been added:

“A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.”

“If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.”

22. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) acceptance criteria for Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD) - The following text has been removed:

“For MSD: Use in-house LCS limits if project limits are not specified.”

The following text has been added:

“A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.”

“If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.”

23. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Minimum Frequency for Post Spike Sample – Erroneous quotation marks were removed.

24. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) with Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) comments for Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD) – The following spelling has been corrected:

“saple” has been replaced with “sample”.

25. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Corrective Action for Post Spike Sample – Erroneous quotation marks were removed.

The following text was removed:

“70-130%”

26. Appendix B, Table B-15 (Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water) Comments for Post Spike Sample – Erroneous quotation marks were removed.

27. Appendix B, Table B-23 (Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples Other than Explosives) acceptance criteria for Sample Preparation processing – The following text has been removed:

“Refer to method specific tables for LCS, surrogate, and blank acceptance criteria.”

The following text has been added:

“Refer to method specific tables for blank and other QC acceptance criteria.”

“Laboratories shall develop in-house surrogate acceptance criteria.”

28. Appendix B, Table B-23 (Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples other than Explosives) comments for Grinding – The following word has been replaced in the second sentence:

“Method Blank” has been replaced with “Grinding Blank”