Supplemental information sheets provide minor corrections or clarifications to requirements outlined in the Department of Defense (DoD) and the Department of Energy (DOE) Quality Systems Manual (QSM). The supplemental information is version specific, and changes will be incorporated in the next revision of the DoD/DOE QSM. Supplemental Information used along with the QSM provides requirements for laboratory accreditation. Updated Appendix B Tables are included in the Appendix of the supplemental information only where changes have been made, reflecting all applicable updates described in this document.

Supplemental Information

QSM 6.0 Requirement	Supplemental Information: 02/08/2024
Table B-24 Matrix Duplicate Minimum Frequency	The matrix duplicate requirement identified in Table B-24 of QSM 6.0 may be omitted for matrices other than AFFF.
QSM 6.0 Requirement	**Supplemental Information: 03/11/2024**
Module 1 Clause 4.1.1	The laboratory shall perform proficiency testing (PT) for individual isomers if the isomers are listed individually on the laboratory's Certificate of Accreditation. For example, if the laboratory lists m and p-xylene and o-xylene separately on the Certificate, the analytes shall be reported separately during PT, but if the laboratory only lists total xylene on the Certificate, only total xylenes shall be reported.
Module 6 Clause 7.1.5.c.ii.c	Background subtraction measurements for gas-proportional and semiconductor alpha/beta detectors shall be performed monthly. Changed from "quarterly."
Module 6 Clause 7.3.3.a.x.b	The Duplicate Error Ratio (DER) between the sample and the Matrix Duplicate is ≤ 3. Changed from "< 3."
Module 6 Clause 7.3.3.a.x.c	The relative percent difference (RPD) is less than or ≤ 25%. Changed from "< 25%."

Module 6 Clause 8.5.1.c.ii	Each Cell/Detector pair efficiency shall be verified at least annually. The continuing efficiency for each Cell/Detector pair shall be within 25% of the initially determined efficiency. Changed from "+ 25%."
Module 6 Clause 8.5.3.a.v	The acceptance criteria for the method blank shall be |ZBlank| ≤ 3 or within laboratory-developed criteria of ±3 standard deviations of the mean. Changed from "|ZBlank| < 3 and + 3 standard deviations."
Module 6 Clause 8.5.3.b.iii	The LCS shall meet customer specified requirements, acceptance criteria of |ZLCS| ≤ 3, or laboratory-developed acceptance criteria of ± 3 standard deviations of the mean that are within 25% of the known LCS value. Changed from "|ZLCS| < 3 and + 3 standard deviations."
QSM 6.0 Requirement	**Supplemental Information: 08/07/2024**
Table B-3 Sample Preparation and Processing	Note: Drying/grinding may not be appropriate for all analytes.
Table B-3 Matrix Spike (MS)	Reported analytes may be spiked into the MS after analytical subsampling.
Table B-3 Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Reported analytes may be spiked into the MSD after analytical subsampling.

Table B-8 Laboratory Control Sample Duplicate (LCSD)	The LCSD QC Check row shall be added to Table B-8. The Minimum Frequency: If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Acceptance Criteria: Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD Corrective Action and Qualification Criteria: Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Table B-22 Instrument Sensitivity Check Acceptance Criteria	All reported analytes for the ISC shall be within ± 35% of true value. Changed from “All reported analytes and surrogates within ± 20% of true value.”
Table B-22 Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE) Acceptance Criteria	If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 35%, respectively. Changed from “… %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively.”
Table B-25 Internal Standard (IS)	The IS requirement identified in Table B-25 may be omitted when IS are not used.
Table B-30 Surrogate Spike QC Check	A surrogate fortification standard shall be added prior to any processing (e.g. prior to drying/grinding or extraction). Changed from “a solid surrogate fortification standard.”
QSM 6.0 Requirement	**Supplemental Information: 12/06/2024**

Table B-4	The acceptance criteria for common contaminants in a method blank shall be included in the Method Blank row and omitted from the Internal Standard row.
Table B-13 Confirmation of positive results Acceptance Criteria	Peak area counts ratio within 2.1 – 3.9. Changed from "Peak area counts ratio within ± 30% of the average peak area count ratio of the mid-range calibration standard, if the calibration is performed on the same day as the analysis, or otherwise, within the average peak area count ratios of all the CCV runs of the analytical batch."
Table B-13 Confirmation of positive results Corrective Action and Qualification Criteria	If Isotope Ratio is not within acceptance criteria and the measured concentration of the sample is above the LOQ, the sample shall be reanalyzed. If the sample was not cleaned (i.e., pretreatment), the sample shall be reprepared using a cleanup procedure and analyzed. Dilution may be an appropriate alternative to a cleanup procedure if perchlorate concentrations are sufficient to allow quantitation after dilution. If the Isotope Ratio remains outside acceptance criteria after cleanup, apply qualifier to result and explain in the case narrative. Changed from "If Isotope Ratio is not within acceptance criteria, the sample shall be reanalyzed. If the sample was not pretreated, the sample shall be reprepared using cleanup procedures and analyzed. If the Isotope Ratio remains outside acceptance criteria after cleanup, use alternative techniques to confirm presence of perchlorate, e.g., a post spike sample or dilution to reduce any interference, and apply qualifier to result and explain in the case narrative. The use of cleanup procedures, post spike samples, and dilutions, and the disposition of results of alternate techniques used to confirm presence of perchlorate shall be discussed in the case narrative."
QSM 6.0 Requirement	**Supplemental Information: 02/10/2025**

Table B-9 Interference Check Solutions (ICS) or Multi-Element Spectral Interference Checks (SIC) Acceptance Criteria	ICS-AB: Within ± 20% of true value. ICS-AB is not required if the instrument is able to read negative responses. Changed from "ICS-AB: Within ± 20% of true value."
Module 6 Section 3.0 Terms and Definitions	**Reporting Limit:** A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix. Added definition for reporting limit.
Module 6 Clause 5.2.5.e	MDAs are determined based on factors and conditions such as instrument settings and matrix type, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required reporting limit (RL). Sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the RLs. If RLs are not achieved, then the cause shall be discussed comprehensively in the case narrative. Changed from "MDAs are determined based on factors and conditions such as instrument settings and matrix type, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required Decision Level. Sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the Decision Levels. If Decision Levels are not achieved, then the cause shall be discussed comprehensively in the case narrative."
QSM 6.0 Requirement	**Supplemental Information: 04/22/2025**
Tables B-1, B-2, B-3, B-4, B-14, B-22, B-25, and B-31 Internal Standards (IS) Acceptance Criteria	Acceptance Criteria for IS can be established using the midpoint standard in the ICAL or average of the ICAL standards. Changed from "using the midpoint standard in the ICAL."

Tables B-1, B-2, B-3, B-4, B-12, B-14, B-22, B-25, B-31 Retention Time (RT) or Relative Retention Time (RRT) establishment Acceptance Criteria	Acceptance Criteria for RT or RRT can be established using the midpoint standard of the ICAL or average of the ICAL standards. Changed from "using the midpoint of the ICAL."
QSM 6.0 Requirement	**Supplemental Information: 08/06/2025**
Table B-24 AFFF samples	QC Check should be titled Samples – Preparation (AFFF and Fluorine Free Foam (F3) only). Changed from "AFFF samples"
Table B-24 AFFF samples Minimum Frequency	Each F3 sample. Note: This includes F3 formulation samples that are to be evaluated for MIL-PRF-32725 compliance.
Table B-24 AFFF samples Acceptance Criteria	AFFF and F3 samples shall be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9. Note: The LCSD listed in Section 11.2.6 of DoD AFFF01 is not required. All AFFF and F3 samples shall be prepared and analyzed in duplicate in the same manner as aqueous samples (e.g., solid phase extraction, extracted internal standards, carbon cleanup, etc.) Changed from "AFFF samples shall be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9. All AFFF samples shall be prepared and analyzed in duplicate in the same manner as aqueous samples (e.g., solid phase extraction, extracted internal standards, carbon cleanup, etc.)."
Table B-24 Matrix Duplicate Minimum Frequency	The matrix duplicate requirement identified in Table B-24 of QSM 6.0 may be omitted for matrices other than AFFF and Fluorine Free Foam.
Table B-24 Instrument Blank Acceptance Criteria	As recommended or required in the reference method. Changed from "No analytes detected > ½ LOQ."

Table B-24 Method Blank Acceptance Criteria	No analytes detected > ½ LOQ, >1/3 the regulatory compliance limit, or greater than 1/10 the concentration in a sample in the extraction batch, whichever is greatest. Changed from "No analytes detected > ½ LOQ."
Table B-24 Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLLCS) Acceptance Criteria	Note: AFFF and F3 samples are to use the acceptance criteria for aqueous samples.
Table B-24	EPA Method 1633 required QC Checks shall be included in Table B-24 for completeness, the rows include: Mass Calibration Mass Accuracy (Calibration) Verification Initial Calibration (ICAL) for all analytes Retention Time (RT) and Relative Retention Time (RRT) establishment Retention Time (RT) window width Instrument Sensitivity Check (ISC) Continuing Calibration Verification (CCV) Qualitative Identification Standard Bile Salt Interference Check Non-extracted Internal Standard (NIS) Qualitative Peak Identification
QSM 6.0 Requirement	**Supplemental Information: 09/02/2025**
Module 2 Clause 6.2.10	"Radioactive samples" are samples identified by the customer and/or screened by the laboratory as radioactive and are being submitted to the laboratory for analytical testing (radiochemistry, inorganic, etc.).
Table C-37	The control limits for 4-Amino-2,6-dinitrotoluene, Tetryl, 2,4,6-Trinitrotoluene, and 3,5-Dinitroaniline may be omitted.
QSM 6.0 Requirement	**Supplemental Information: 04/09/2026**

Module 6 Clause 7.1.3.a	Prior to use of an initial calibration for analysis of samples, the laboratory shall verify the initial instrument calibration with a reference standard. The laboratory may obtain the standard from a source or a lot independent of the reference standard used in the initial calibration, if available. Changed from “The laboratory shall obtain the standard from a source or a lot independent of the reference standard used in the initial calibration, if available.”
Module 6 Clause 7.1.4.b.i.a	Semiconductors: At least twice weekly, but not on consecutive days, for a continuously operating detector and using an autosampler; day of use for a non-continuously operating detector. Changed from “Semiconductors: At least twice weekly, but not on consecutive days, for a continuously operating detector;”
Module 6 Clause 7.3.3.a.v	If the activity of the sample is greater than or equal to five times the spiking level, |ZMS| ≤ 3 shall be used. Changed from “If the activity of the sample is greater than five times the spiking level, |ZMS| ≤ 3 shall be used.”
Module 6 Clause 7.3.3.a.x.a	|ZDup| ≤ 3, which is equivalent to MARLAP defined |ZRep|, if using MARLAP (equation 18.2); Changed from “|ZDup| ≤ 3 if using MARLAP (equation 18.2);”
Table B-16	Table B-16 has been revised, and the revisions are applicable to the entire table.
Table B-17	Table B-17 should be titled Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation). Changed from “Gamma Isotopes by Gamma Spectrometry” Table B-17 updated to this version of supplemental information. Revisions are applicable to the entire table.

Table B-18	Table B-18 should be titled Alpha and/or Beta Particles by Gas Flow Proportional Counting. Changed from “Alpha and Beta Particles by Gas Flow Proportional Counting.” Table B-18 updated to this version of supplemental information. Revisions are applicable to the entire table.
Table B-19	Table B-19 should be titled Radioactive Nuclides by Liquid Scintillation Counter Analysis Changed from “Tritium in Water by Liquid Scintillation Counter Analysis” Table B-19 updated to this version of supplemental information. Revisions are applicable to the entire table.
Table B-20	Table B-20 should be titled Radon Scintillation (Ra-226 by Lucas Cell). Changed from “Radium-226 (Radon) by Radon Scintillation (Ra-226 by Lucas Cell)” Table B-20 updated to this version of supplemental information. Revisions are applicable to the entire table.

Appendix B Quality Control Requirements

Table B-3.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Sample Preparation and Processing	Each field sample, PT, LCS, and blank when ISM is required.	Before drying and grinding, sample shall be sieved to 2 mm. Note: Drying/grinding may not be appropriate for all analytes. Following 2 mm sieve, entire sample shall be dried and ground following the laboratory's grinding procedure for ISM.	NA.
Grinding Procedure for ISM	Initial method validation and any time major equipment is changed or when a reduction in the number or time of grinding cycles occur.	The laboratory shall demonstrate that grinding procedure is capable of reducing the particle size to <75 microns by passing representative portions of ground demonstration samples through a 200-mesh sieve.	NA.
Grinding for ISM	Each field sample, PT, LCS, and blank when ISM is required.	Grinding periods shall not exceed 60 seconds and shall be followed by a 2-minute or longer cool down period between the grind cycles.	NA.
Grinding Blank for ISM For batch preparation, the Grinding Blank may serve as the Method Blank.	One per batch using Ottawa sand or a verified clean soil. Grinding blank shall be processed immediately after a customer-identified sample with suspected high target analyte concentration or after the LCS.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample whichever is greater.	The laboratory may reprepare and analyze the ground grinding blank to confirm results outside of acceptance criteria. If the grinding blank is not reprepared and analyzed, or if contamination is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
ISM Laboratory Replicates	At the analytical subsampling step, ISM laboratory replicates shall be performed on one ISM sample in each preparation batch. ISM replicates shall not be performed on any sample identified as a QC or blank (e.g., LCS, Grinding Blank or other QC). The laboratory shall prepare and analyze three or more replicates, each consisting of a minimum of 30 increments of the same mass and depth.	RSD shall not exceed 20%. RSD does not apply if all results are below the LOQ.	The laboratory may reprepare and analyze laboratory replicates to confirm the RSD result outside of acceptance criteria. If the laboratory replicates are not reprepared and analyzed, or if the RSD outside of acceptance criteria is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Initial calibration (ICAL) for all analytes	At instrument set-up and when needed based on reference method requirements or QC results, before sample analysis.	Minimum 5 levels. Each analyte shall meet one of the two options below: Option 1: %RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve or average of the ICAL standards when ICAL is performed or on days when ICAL is not performed, the initial CCV for the sequence is used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Laboratory Control Sample (LCS) If a laboratory utilizes a self-spiked solid LCS, the fortification of ISM samples shall be performed before any preparation steps performed, such as drying, grinding, and sieving. For ISM, a solid reference material containing all reported analytes shall be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample.	One per preparatory batch. Shall contain all surrogates and all analytes to be reported. A Standard Reference Material (SRM) that is used for a solid LCS can be ground as a single batch and subsampled repeatedly as long as the SRM is within expiration date.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria (beginning at the subsampling step for ISM samples) if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample	If sufficient sample is not available for either a MSD or MD,	Recovery: Same as LCS acceptance criteria.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Duplicate (LCSD)	one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes. Reported analytes may be spiked into the MS after analytical subsampling.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes. Reported analytes may be spiked into the MS after analytical subsampling.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Surrogate Spike Surrogates shall be added to each field sample, blank, and LCS before any processing (e.g., before drying, grinding or extraction)	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (HPLC) (Method 8330B) Including Incremental Sampling Methodology (ISM) for Solid Samples			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Confirmation of positive results (second column) The laboratory may also use mass spectrometry for confirmation.	All results > the DL shall be confirmed. Samples without detected target analytes do not require second-column analysis.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%. RPD evaluation between primary and secondary column does not apply if both results are below LOQ.	Apply qualifier to affected results and explain in the case narrative. The laboratory shall identify the primary column for each target analyte. If results are reported from the secondary column due to interference or QC outside acceptance criteria, the laboratory shall apply qualifier to affected analyte results and explain in the case narrative.

Table B-4.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) **Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.**			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Tune Check	Before ICAL or as stated in the reference method, whichever is most frequent.	Specific ion abundance criteria of BFB or DFTPP from the reference method revision used for analysis and reporting.	Retune instrument and verify. If the method requires an ICAL after a change in tuning parameters, follow the reference method as written (e.g., 8260D, 8270E). Qualification of data is not appropriate.
Performance Check (SVOC, except when analyzing only PAHs or PCBs)	EPA Method 8270E: prior to initial calibration. EPA Methods 8270C, 8270D and any other SVOC GC/MS method: at the beginning of each 12-hour period, before analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol present at their normal responses and tailing factor ≤ 2.	Correct problem, then repeat performance check. Qualification of data is not appropriate.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$; If the specific version of a reference method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements shall also be met.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater. Common contaminants (Methylene chloride, Acetone, 2-Butanone, and Phthalates) shall not be detected > LOQ.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-4. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)
Note: This table is for GC/MS Analysis in full scan mode. Requirements for GC/MS in selected ion monitoring mode are in Table B-22.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Surrogate Spike **If Used**	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified prior to reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-8.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check	If results are reported above the calibration range, the recommendations or requirements for linear range determination and checks listed in the reference method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.
Other Performance Checks	As recommended or required in the reference method.	If the reference method includes additional performance checks such as ICV, the checks shall be performed and evaluated as described in the reference method.	Correct problem, then repeat performance checks. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum: one high standard and a Calibration Blank. If a multi-level calibration is used: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Single Point Calibration: The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value. Multi-level calibration: Option 1: RSE for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 20%, respectively. The maximum allowable %RSE shall be 20%. Acceptable %RE at the low level also meets the requirement for the LLCCV.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	After ICAL, before sample analysis, every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Lower Limit of Quantitation (LLOQ) Verification	Daily. The LLOQ verification shall be less than or equal to the LOQ.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	The absolute values of all analytes < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Interference Check Solutions (ICS) or Multi-Element Spectral Interference Check (SIC)	After ICAL and before sample analysis. The laboratory shall perform an ICS or a multi-element SIC at the concentrations and frequency as recommended or required in the reference method.	ICS-A or SIC: Absolute value of concentration for all non-spiked project analytes < 1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ± 20% of true value. ICS-AB is not required if the instrument is able to read negative responses.	Correct problem, repeat ICS or SIC. Qualification of data is not appropriate.
Additional Spectral Interference Check (SIC)	Any additional SICs listed in the reference method shall be followed.	As recommended or required in the reference method.	Correct problem, repeat SIC. Qualification of data is not appropriate.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch.	Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-8. Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Dilution Test	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is within the linear range and > 25x the LOQ before dilution or as in referenced method.	Five-fold dilution shall agree within the percentage recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is not sufficiently high to perform the dilution test.	As recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-9.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Linear Range Check	If results are reported above the calibration range, the recommendations or requirements for linear range determination and checks listed in the reference method shall be followed.	Within ± 10% of true value.	Data shall not be reported above the calibration range without a passing Linear Range check. Dilute samples until they are within the calibration range or verify the Linear Range according to the referenced method. If the samples cannot be reanalyzed, apply qualifier to analyte results exceeding the calibration range and explain in the case narrative.
Tune Check	Before ICAL.	Mass calibration < 0.1 Da from the true value; Resolution < 0.9 Da full width at 10% peak height. Follow instrument manufacturer's recommendations for additional tuning criteria.	Retune instrument and verify. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum: one high standard and a Calibration Blank. If a multi-level calibration is used: Minimum of 5 standards and a calibration blank when evaluating with a linear regression, 6 standards and a calibration blank when evaluating with a quadratic regression. Single Point Calibration: The curve shall be verified with a low level (at or below the LOQ) and a mid-level calibration verification standard. Low level calibration verification: Within 80-120% of the true value. This also meets the requirement for the LLCCV. Mid-level calibration verification: Within 90-110% of the true value. Multi-level calibration: Option 1: RSE for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at the mid-range and low level of the calibration shall be 10% and 20%, respectively. The maximum allowable %RSE shall be 20%. Acceptable %RE at the low level also meets the requirement for the LLCCV.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	After ICAL, before sample analysis, every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Lower Limit of Quantitation (LLOQ) Verification	Daily. The LLOQ verification shall be less than or equal to the LOQ.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS)	Every field sample, standard and QC sample.	IS intensity in the samples is evaluated as recommended or required in the reference method.	Perform a 5X dilution, reanalyze sample. Repeat dilutions until IS intensities are acceptable. Qualification of data is not appropriate.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after every CCV.	The absolute values of all analytes < ½ LOQ or < 1/10th the amount measured in any sample, whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Interference Check Solutions (ICS) or Multi-Element Spectral Interference Checks (SIC)	Daily, after ICAL and before sample analysis or every 12 hours of continuing sample analysis, whichever is more frequent.	ICS-A or SIC: Absolute value of concentration for all non-spiked project analytes < 2X LOQ (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of true value. ICS-AB is not required if the instrument is able to read negative responses.	Correct problem, repeat ICS. Correct problem; reanalyze ICS or SIC, reanalyze all samples following the last SIC within acceptance criteria. Qualification of data is not appropriate.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-9. Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Dilution Test	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is within the linear range and > 25x the LOQ before dilution or as in referenced method.	Five-fold dilution shall agree within the percentage recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Post-Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails and if target analyte concentration of the parent sample is not sufficiently high to perform the dilution test.	As recommended or required in the reference method.	None, unless required by the project. Apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-12.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 15%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 10% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) establishment	Once per ICAL and at the beginning of the analytical sequence.	RT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV}	Daily before sample analysis; after every 10 samples; and at the end of the analytical run.	All reported analytes within ± 10% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria. Hexavalent Chromium: Laboratory-developed acceptance criteria no wider than ± 10%.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 10% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-12. Common Anions and Hexavalent Chromium Analysis by Ion Chromatography (IC)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all reported analytes. MD: Shall be analyzed for all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 15% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-13.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up, at least annually or as specified by the manufacturer, whichever is more frequent, after major maintenance, and when needed based on method requirements or QC results, prior to sample analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As per instrument manufacturer	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification	Following each mass calibration Mass calibration shall be verified with the mid-level perchlorate standard.	Perchlorate ions shall be within ± 0.3 Da of mass 99, 101, and 107 or their respective daughter ion masses (83, 85, and 89), depending on which ions are quantitated.	Correct problem, then repeat verification or mass calibration as needed. Qualification of data is not appropriate.
Conductivity Limit Study	At initial setup and when major changes occur in the method's operating procedures (e.g., addition of cleanup procedures, column changes, mobile phase changes).	Reported perchlorate concentration within ± 20% of true value and the internal recovery and calibration standard (IRCS) recovery within ± 50% of the mid-level standard.	Decrease anion concentrations in the dissolved salt solution and repeat Conductivity Limit Study. Qualification of data is not appropriate.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Conductivity Limit Check Sample (CLCS)	Daily before sample analysis and after every 20th field sample. The CLCS shall be prepared with the dissolved salt solution at the conductivity limit and perchlorate concentration at the LOQ and undergo the same filtration as aqueous field samples.	Reported perchlorate concentration within ± 20% of true value, and the internal recovery and calibration standard (IRCS) recovery within ± 50% of the CCV.	Correct problem. Reanalyze all samples and QC samples since last passing CLCS. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed. If poor recovery from the cleanup filters is suspected, a different lot of filters shall be used to reprepare and analyze all samples since last passing CLCS.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum of 6 calibration levels shall be used. The calibration shall meet one of the options below: Option 1: linear least squares regression: $r^2 \geq 0.99$; Option 2: non-linear least squares regression (quadratic): $r^2 \geq 0.995$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 15% and 50%, respectively. The maximum allowable %RSE shall be 15%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Retention Time (RT)	Every field sample, standard, and QC sample.	Retention times of the native perchlorate peak and the IRCS perchlorate peak shall be within ± 0.2 minutes.	Correct problem, then rerun ICAL. Qualification of data is not appropriate.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10th field sample; and at the end of the analytical batch run.	Reported perchlorate concentration within ± 15% of true value.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Recovery and Calibration Standard (IRCS) **^{18}O-labeled perchlorate**	Every field sample, standard, and QC sample.	Measured IRCS area within ± 50% of the value from the average of the IRCS area counts of the ICAL if the calibration is performed on the same day as the analysis, or otherwise, the first CCV of the analytical batch. Measured IRCS area in the first CCV of the analytical batch within ± 50% of the value from the average of the IRCS area counts of the ICAL.	Inspect instrument for malfunctions, verify conductivity is below the conductivity limit, and correct problem. All affected samples shall be reanalyzed. If samples cannot be reanalyzed, apply qualifier to results associated with IRCS outside acceptance criteria and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria. If QSM Appendix C Limits are not available, then laboratory-developed acceptance criteria shall be no wider than ± 20%.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 15% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-13. Perchlorate Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS and LC/MS/MS) and Ion Chromatography/Mass Spectrometry (IC/MS and IC/MS/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Confirmation of positive results **Isotope Ratio $^{35}Cl/^{37}Cl$**	Every field sample, standard, and QC sample.	Evaluate the relative abundances of the m/z 83/85 or m/z 99/101 ions in the chromatogram depending on which ions are quantitated. Peak area counts ratio within 2.1 – 3.9	If Isotope Ratio is not within acceptance criteria and the measured concentration of the sample is above the LOQ, the sample shall be reanalyzed. If the sample was not cleaned (i.e., pretreatment), the sample shall be reprepared using a cleanup procedure and analyzed. Dilution may be an appropriate alternative to a cleanup procedure if perchlorate concentrations are sufficient to allow quantitation after dilution. If the Isotope Ratio remains outside acceptance criteria after cleanup, apply qualifier to result and explain in the case narrative.

Table B-14.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	DFTPP mass range from 51-443 m/z using acceptance criteria from Method 8271.	Correct problem, then repeat tune check. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 15%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at the mid-level and low level of the calibration shall be 20% and 50%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICAL not performed; after every 10 samples or 12 hours of analysis time, whichever is sooner; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-14. Chemical Warfare Agents by Gas Chromatography/Mass Spectrometry (GC/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-16.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (IC or ICAL) **(Energy, efficiency, and FWHM peak resolution)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits indicating a change in performance since the previous calibration. Shall use traceable calibration source (CS) that matches sample test source (STS) configuration (type, size, and position relative to the detector).	Verify manufacturer's specifications for point source efficiency; At least three isotopes within the energy range of 3 to 6 MeV, bracketing the energy range region of interest (ROI) for commonly used isotopes, when possible. (Energy) Energy vs. channel slope equation < 15 keV per channel. (Energy) Full Width – Half Maximum (FWHM) < 100 keV for each peak used for calibration. (Resolution) Minimum of 3,000 net counts in each peak to achieve a relative count uncertainty less than 2%. (Efficiency) Peak energy positions of all calibration isotopes are within 40 keV of reference peak energies. (Energy)	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration Verification (ICV) **(If CRM and/or SRM are not used)**	After initial calibration for energy/efficiency and prior to analysis of samples.	Determine peak location, resolution, and ROI/alpha peak efficiency (where counting efficiency is an analytical requirement) using at least two alpha peaks. (MARLAP 18.5.6.3) FWHM ≤ 100 keV and within ± 40 keV of corresponding calibration peaks in initial energy calibration.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate. Qualification of data is not appropriate.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV) **(source check or pulser check)**	Weekly (not to exceed seven days) source check or pulser check verification before analysis of samples. Pulser check may only be used to verify energy calibration when using radiotracers during analysis. Source check or tracer may be used to verify energy, FWHM and efficiency.	CCV energy response shall be monitored by one of the following: Option 1: Source checks shall have a tolerance limit or control chart set at ± 3% or ± 3σ (MARLAP 18.5.6.3) or FWHM ≤ 100 keV and within 40 keV of corresponding calibration peaks in initial energy calibration. Option 2: Pulser check observed peak centroid falls within ± 40 keV from reference energy or shall have a tolerance limit or control chart set at ± 3% or ± 3σ (MARLAP 18.5.6.3) at least 5 daily checks.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed if no samples were analyzed. If the reanalyzed CCV is acceptable, proceed with analysis. Otherwise, correct problem and analyze passing CCV or recalibrate. All associated samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all associated samples and explain in the case narrative.
Background Subtraction Count (BSC) Measurement	At least monthly, before initial use or after initial calibration. BSC test source shall match STS configuration (e.g., type, size, pressure, and position relative to the detector).	The counting interval for the long count shall equal to or longer than the associated sample counting time and be representative of the background rate. Each target and tracer isotope region of interest (ROI) activity shall be sufficiently low enough to ensure required detection limits are met. Background shall: Use a statistical test to determine a significant change in the background count rate value. (MARLAP 18.5.6.3) Be within ± 3σ of mean activity of recent BSCs for total ROI for all isotopes of interest (minimum of 10 BSC values).	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. All associated samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Contamination Check (ICC)	Performed weekly and after counting laboratory defined high-level samples.	Instrument contamination shall: Use a statistical test to determine a significant change in the background count rate value. MARLAP 18.5.6.3) Be within ± 3σ of mean activity of recent ICCs for total ROI for each target and tracer isotopes of interest. (minimum of 10 ICC values).	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish BSC or ICC. All associated samples since last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples, including the process of handling, preparation, and analysis (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$ (MARLAP 18.4.1, Equation 18.1).	If not within the control limit, recount the MB to confirm results, unless all associated sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all associated QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples and contain representative nuclides within the energy ranges of those nuclides to be reported.	LCS should meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the known value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$ (MARLAP 18.4.3, Equation 18.3).	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all associated QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to associated samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when chemical yield tracers or carriers are employed).	MS shall be monitored by one of the following: Option 1: If activity of the sample < 5X the spiking level, recoveries shall be within 60-140% if customer or reference method requirements are not specified. Option 2: If activity of the sample ≥ 5X the spiking level, then $\|Z_{MS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.4).	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; Option 2: RPD is ≤ 25%. Option 3: Control limit for $\|Z_{Dup}\| \leq 3$ (MARLAP 18.4.2, Equation 18.2).	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Isotopic yield within 30-110% if customer or reference method requirements are not specified. FWHM < 100 keV and peak energy within ± 50 keV of known peak energy.	The data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the peaks are fully resolved and within their ROIs, the data can be qualified and reported as is. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-16. Isotopic Determinations by Alpha Spectrometry			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Carriers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	If carrier yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-17.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Manufacture Detector Characterization (Detector characterization replaces the ICAL efficiency) Energy and FWHM calibration still required. **(Energy, efficiency and FWHM peak resolution)**	Before initial use. Follow instrument manufacture guidelines to return instrument into control after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration. Detector characterization replaces the ICAL efficiency. Energy and FWHM calibration still required.	Option 1: Peak energy difference is within 0.1 keV of reference energy for all points. (Energy) Peak Full Width at Half Maximum (FWHM) < 2.5 keV at 1332 keV. (Resolution) Energy vs. channel slope equation shall be accurate to 0.5 keV. (Energy) Option 2: Verify manufacturer's specifications for gamma peak resolution. (Energy) The following applies to both options 1 & 2: Efficiency at each reference peak covering the energy range for each defined geometry, matrix, 95% confidence limit of the fitted function: ≤ 8% over energy range. Volume differences associated with geometry may be handled by the software. In this case, only one common volume for each geometry/matrix needs to be verified.	Correct problem, follow instrument manufacture guidelines. May require an updated instrument characterization. Qualification of data is not appropriate.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (IC or ICAL) **(Energy, efficiency and FWHM peak resolution)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits indicating a change in performance since the previous calibration. Use a traceable calibration source (CS) that matches sample test source (STS) configuration (type, size, geometry and position relative to the detector), when appropriate.	Minimum of 10,000 net counts (to achieve a 1% count uncertainty) in each reference peak for at least six calibration peaks that bracket the range of use. For standard broad spectrum detectors, follow one of the two options: Option 1: Peak energy difference is within 0.1 keV of reference energy for all points. (Energy) Peak Full Width at Half Maximum (FWHM) < 2.5 keV at 1332 keV. (Resolution) Energy vs. channel slope equation shall be accurate to 0.5 keV. (Energy) Option 2: Verify manufacturer's specifications for gamma peak resolution. (Energy) The following applies to options 1 & 2: Efficiency at each reference peak covering the energy range for each defined geometry/matrix. 95% confidence limit of the fitted function: ≤ 8% over energy range. (MARLAP 18.5.6.2) Low energy or thin-window gamma systems shall be calibrated to an energy range in accordance with manufacturer's specifications or data sheets.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration Verification (ICV) **(If CRM and/or SRM are not used)**	After initial calibration for energy/efficiency and prior to analysis of samples.	Observed peaks of second source standard fall within ± 10% of initial calibration value relative to energy, FWHM, and efficiency. Minimum of 5,000 net counts in each peak in at least four calibration verification peaks that bracket the range of use.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Semiconductors for a continuously operating detector and using an autosampler: at least twice weekly, but not on consecutive days. Semiconductors for a non-continuously operating detector and Scintillation Detectors: daily or before analysis of samples. When working with long sample count times or batch sequences that run more than a day, CCV shall be performed at the beginning and end of each analytical batch. The elapsed time between CCVs shall not exceed seven days.	Source checks shall meet one of the following: Option 1: Source Check shall have a tolerance limit or control chart set at ± 3% or ± 3σ. (MARLAP 18.5.6.3) Option 2: Peak Energy check and Efficiency check: low, mid, and high energies shall be within 10% of the initial calibration value; and The following applies to options 1 & 2: FWHM: Low, mid, and high energies shall be within 10% of initial FWHM value.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed if no samples were analyzed. If the reanalyzed CCV is acceptable, proceed with analysis. Otherwise, correct problem and analyze passing CCV or recalibrate. All associated samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all associated samples and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Background Subtraction Count (BSC) Measurement	At least monthly, before initial use or after initial calibration.	The counting interval for the long count shall equal to or longer than the associated sample counting time and be representative of the background rate. Activity shall be low enough to meet customer-provided requirements and shall: Use a statistical test to determine a significant change in the background count rate value. Be within 3σ of the mean activity of the BSC for identified background peaks, so long as the total count time is at least equal to the longest Test Source count time. (MARLAP 18.5.6.2)	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. All associated samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Contamination Check (ICC) **(Short Term Background Check)**	Daily or when working with long count times, before and after each analytical batch and after counting high-activity samples.	No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); Integrate spectrum to check for contamination across instrument operating range differences such as normal range (50 keV to 2000 keV) and/or low energy gamma detectors (10 keV to 200 keV) or detectors that have a range of 10 keV to well above 2000 keV. Activity shall be low enough to meet customer-provided requirements, and the result shall be within the tolerance limit or control chart limits of ± 3% or ± 3σ of the mean activity.	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish ICC or BSC. All associated samples since the last passing ICC shall be evaluated and re-analysis performed as necessary. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples, including the process of handling, preparation, and analysis (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	Count time shall be equal to or longer than the longest associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$ (MARLAP 18.4.1, Equation 18.1).	If not within the control limit, recount the MB to confirm results, unless all associated sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all associated QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-17. Gamma Isotopes by Gamma Spectrometry (Not applicable to Scintillation)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples including geometry and contain representative nuclides within the energy ranges of those nuclides to be reported.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the known value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.3)	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all associated QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to associated samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; Option 2: RPD is ≤ 25% Option 3: Control limit for $\|Z_{Dup}\| \leq 3$ (MARLAP 18.4.2, Equation 18.2).	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-18.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICALV) – <u>Voltage Plateau</u> **(Separate plateaus determined for alpha and beta activity)**	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits indicating a change in performance since the previous calibration.	Verify manufacturers' specifications. Plot voltage vs. count rate to determine proper operation voltages. Perform a series of counts in ≤ 50V steps from approximately 300-1500V. Determine a usage range where slope of the plateau is < 5% over 100V change.	Correct problem, then repeat ICAL-Voltage Plateau determination. Qualification of data is not appropriate.
Initial Calibration (ICALE) – <u>Efficiency</u>	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits indicating a change in performance since the previous calibration. ICALE shall match STS configuration.	Detector counting efficiency, using appropriate traceable CS, shall be determined for each radionuclide used to analyze test sources. Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using known sources. A 1σ counting uncertainty of ≤ 1% shall be achieved for all detector efficiency determinations. (MARLAP 18.5.6.1)	Correct problem, then repeat ICAL-Efficiency determination. Qualification of data is not appropriate.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICALT) – Cross-Talk Factors	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits indicating a change in performance since the previous calibration.	Determine cross-talk factors (alpha into beta and beta into alpha) for each nuclide using a traceable CS per matrix and method. Verify manufacturer's specifications for cross-talk in alpha and beta channels.	Correct problem, then repeat ICAL-Cross Talk Factors determination. Qualification of data is not appropriate.
Initial Calibration (ICALSA) – Self-Absorption Curve	Before initial use; after significant adjustment, maintenance, repair, or refurbishment, including replacement of key components; when there is a change in performance following any instrument repair; after modification of operating parameters that affect the instrument response; or when instrument performance checks exceed predetermined limits, indicating a change in performance since the previous calibration.	Using traceable CS, establish a mass attenuation curve with a minimum of seven mass-attenuated standards. For each radionuclide of interest, establish mathematical function (curve) of detector efficiency vs. source mass loading. 95% confidence limit of the fitted function (curve) over the calibration range to ≤ 10% uncertainty for alpha and ≤ 5% uncertainty for beta. (MARLAP 18.5.6.1)	Correct problem, then repeat ICAL-Self Absorption Curve determination. Qualification of data is not appropriate.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration Verification (ICV) **(If CRM and/or SRM are not used)**	After ICALE and prior to analysis of samples.	Establish a tolerance limit immediately after the initial counting efficiency calibration and after instrument loss of control. Option 1: A tolerance limit or control chart shall be set at ± 3% or 3σ of the mean (MARLAP 18.5.6.1). Option 2: Value of second source calibration for each isotope within ± 10% of initial calibration value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICALE and ICV, as appropriate. Qualification of data is not appropriate.
Continuing Calibration Verification (CCV)	Daily or before use and after a counting gas change. When working with long sample count times or batch sequences that run more than a day, CCV shall be performed at the beginning and end of each analytical batch. The elapsed time between CCVs shall not exceed seven days.	Minimum of 2,000 net counts for each energy type (Alpha, Beta). Response checks shall be within a tolerance limit or control chart limits ± 3% or ± 3σ of the mean. (MARLAP 18.5.6.1)	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed if no samples were analyzed. If the reanalyzed CCV is acceptable, proceed with analysis. Otherwise, correct problem and analyze passing CCV or recalibrate. All associated samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all associated samples and explain in the case narrative.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Background Subtraction Count (BSC) Measurement	At least monthly, before initial use or after initial calibration. Determine alpha and beta background after efficiency calibration for each detector using a contamination-free source mount.	The counting interval for the long count shall equal to or longer than the associated sample counting time and be representative of the background rate. Use a statistical test to determine a significant change in the background count rate value. (MARLAP 18.5.6.1) Establish a background count rate value based on measurement uncertainty or count a long background for a time interval that is 1 to 4 times the typical test-source counting time. Use statistical testing to determine a change in the long background count rate value.	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. All associated samples since last passing BSC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.
Instrument Contamination Check (ICC) **(Short count for controlling contamination)**	Daily or when working with long count times, before and after each analytical batch and after counting high-activity samples.	Use a statistical test to determine a significant change in the background count rate value. (MARLAP 18.5.6.1) Within ± 3σ of mean activity of recent ICCs for total ROI for all isotopes of interest (minimum of 10 ICC values).	Check control chart for trends and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish ICC or BCS. All associated samples since the last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples, including the process of handling, preparation, and analysis (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$. (MARLAP 18.4.1, Equation 18.1)	If not within the control limit, recount the MB to confirm results, unless all associated sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all associated QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples and contain representative nuclides within the energy ranges of those nuclides to be reported	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Laboratory-developed control limits shall be within 25% of the known value. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.3)	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all associated QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to associated samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when chemical yield tracers or carriers are employed, or when performing a direct/non-destructive measurement).	MS shall be monitored by one of the following: Option 1: If activity of the sample < 5X the spiking level, recoveries shall be within 60-140% recovery if customer or reference method requirements are not specified. Option 2: If activity of the sample ≥ 5X the spiking level, then $\|Z_{MS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.4)	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; Option 2: The relative percent difference (RPD) is < 25%. Option 3: Control limit for $\|Z_{Dup}\| \leq 3$ (MARLAP 18.4.2, Equation18.2).	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Isotopic yield within 30-110% if customer or reference method requirements are not specified.	If Isotopic yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-18. Alpha and/or Beta Particles by Gas Flow Proportional Counting			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Carriers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	If carrier yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-19.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) **(Efficiency, ROI)**	Before initial use, following repair or loss of control, and upon incorporation of new or changed instrument settings. Use a traceable calibration source (CS), typically unquenched Liquid Scintillation cocktails tagged with 3H or 14C.	Verify manufacturer's specifications for counting efficiency and establish energy regions of interest (ROI) window settings for nuclides of interest (MARLAP 18.5.6.4 Table 18.8).	Correct problem, then repeat ICAL for Efficiency and ROI. Qualification of data is not appropriate
Method Calibration (QCAL) **(Quench Curve)**	After ICAL, following repair or loss of control, upon incorporation of new or changed instrument settings, matrix, or cocktail type. Use a traceable calibration source.	Analyze a minimum of five different quench factor sources. Obtain a minimum of 10,000 counts for isotope of calibration (MARLAP 18A.5). The quench curve shall meet one of the following: Option 1: Correlation coefficient for quench curve ≥ 0.995. Option 2: Count individual calibration source to achieve ROI (1σ) measurement uncertainty of ≤1%. 95% confidence limit of the fitted function < 5% over expected quench range. (MARLAP 18.5.6.4 Table 18.8).	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Calibration (QCAL) **(Standard Addition)** Internal standard or standard addition - radionuclide of interest.	Once after each ICAL. Use a traceable calibration source. Add a spike to a duplicate processed sample or add a spike to a sample that has been counted and then recount.	Activity of spike should be at least four times the anticipated maximum radionuclide activity in a test source. (MARLAP 18.5.6.4) ROI relative counting uncertainty <1% (MARLAP 18.5.6.4) QCAL (Standard Addition) shall be monitored by one of the following: Option 1: Statistically evaluate replicate test-source analyses (MARLAP 18.5.6.4) Option 2: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; or the relative percent difference (RPD) is ≤ 25%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration Verification (ICV) **(If CRM and/or SRM are not used)**	After initial calibration.	Value of each second source nuclide ± 10% of initial calibration value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate. Qualification of data is not appropriate.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis for short counting intervals. When working with long sample count times or batch sequences that run more than a day, CCV shall be performed at the beginning and end of each analytical batch if the elapsed time between CCVs is not longer than seven days. ROI for unquenched traceable reference standards (typically 3H or 14C).	Response acceptance criteria shall be ± 3% or 3σ of the mean for each standard counted.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed if no samples were analyzed. If the reanalyzed CCV is acceptable, proceed with analysis. Otherwise, correct problem and analyze passing CCV or recalibrate. All associated samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all associated samples and explain in the case narrative.
Instrument Contamination Check (ICC) **(Unquenched Blank)**	Daily before sample analysis for short counting intervals. When working with long count times or batch sequences that run more than a day, the ICC shall be performed at the beginning and end of each analytical batch.	Instrument contamination shall be monitored by one of the following: Option 1: Use a statistical test to determine a significant change in the unquenched background ROI count rate value. Option 2: Within ± 3σ of mean activity of recent ICCs for total ROI for all isotopes of interest (minimum of 10 ICC values or use range statistics low number of data points).	Check control chart for trend and recount. If still out of control, determine cause and correct problem. If background activity has changed, re-establish ICC. All associated samples since last passing ICC shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to associated sample results and explain in the case narrative.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples, including the process of handling, preparation, and analysis (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	MB shall be counted for at least the same amount of time as samples. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$. (MARLAP 18.4.1, 18.1) Option 3: Evaluate background against a Background Quench Curve to correct the background to the quench of the sample.	If not within the control limit, recount the MB to confirm results, unless all associated sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all associated QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. LCS matrices shall be consistent with the associated samples and contain representative nuclides within the energy ranges of those nuclides to be reported	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. (MARLAP 18.4.1) Option 2: Control limit for $\|Z_{LCS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.3) Laboratory-developed control limits shall be within 25% of the known value.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all associated QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to associated samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when chemical yield tracers or carriers are employed).	MS shall be monitored by one of the following: Option 1: If activity of the sample < 5X the spiking level, within 60-140% recovery if customer or reference method requirements are not specified. Option 2: If activity of the sample ≥ 5X the spiking level, then $\|Z_{MS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.4.)	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-19. Radioactive Nuclides by Liquid Scintillation Counter Analysis			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; Option 2: The relative percent difference (RPD) is ≤25%. Option 3: Control limit for $\|Z_{Dup}\| \leq 3$ (MARLAP 18.4.2, Equation 18.2).	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Tracers (if used)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Isotopic yield within 30-110% if acceptance criteria are not specified by the reference method or customer.	If Isotopic yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.
Carriers (if used)	Every field sample and any associated batch QC samples as chemical yield monitor.	Chemical yield within 30-110% if customer or reference method requirements are not specified.	If carrier yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-20.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Initial Calibration (ICAL) **Instrument Operational Voltage and Cell/Detector Efficiency**	Before initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. Establish the operating voltage plateau for each detector. Use traceable Ra-226 calibration source (CS) that matches sample test source (STS) configuration (type, size and position relative to the detector).	Verify manufacturer's specifications. Plot voltage vs. count rate to determine proper operating voltages.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration Verification (ICV) **(If CRM and/or SRM are not used)**	After initial calibration.	Within ± 10% of initial calibration value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate. Qualification of data is not appropriate.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Detector response check before use, using an appropriate STS. Each Cell/Detector pair efficiency shall be checked at least annually.	The results of this detector response check shall fall within established laboratory-developed control limits. Continuing efficiency for each Cell/Detector pair shall be within ± 25% of the initial average.	Check control chart for trends. Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed if no samples were analyzed. If the reanalyzed CCV is acceptable, proceed with analysis. Otherwise, correct problem and analyze passing CCV or recalibrate. All associated samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to specific nuclides in all associated samples and explain in the case narrative.
Background Subtraction Count (BSC) Measurement, Cell Contamination Check	Before use for each Cell/Detector pair.	The counting interval for the long count shall equal to or longer than the associated sample counting time and be representative of the background rate. Results shall be within ±3σ of mean activity of recent BSCs (minimum of 10 BSC values). Use a statistical test to determine a change in the background count rate value.	If background activity has changed, re-establish BSC. All associated samples since last passing BSC shall be reanalyzed. Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If the samples cannot be reanalyzed, apply qualifier to sample results and explain in the case narrative.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch. Blank matrices shall be consistent with the associated samples, including the process of handling, preparation, and analysis (e.g., radon-free distilled or deionized water, representative solid material, or physically and chemically identical filter media).	Count time shall be equal to or longer than associated sample count time. MB shall be monitored by one of the following: Option 1: Laboratory-developed control limits of ± 3σ of the mean. Option 2: Control limit for $\lvert Z_{Blank} \rvert \leq 3$. (MARLAP 18.4.1, Equation 18.1).	If not within the control limit, recount the MB to confirm results, unless all associated sample results are > 5X the MB activity. Inspect MB control chart for indication of significant bias. Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all associated QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch.	LCS shall meet customer-specified limits, if provided. Otherwise, the LCS shall be monitored by one of the following: Option 1: Use laboratory-developed control limits of LCS ± 3σ of the mean. Option 2: Control limit for $\|Z_{LCS}\| \leq 3$. (MARLAP 18.4.3, Equation 18.3) Laboratory-developed control limits shall be within 25% of the known value.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the nuclides not within acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected samples results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch. (MS not required when chemical yield tracers or carriers are employed).	MS shall be monitored by one of the following: Option 1: If activity of the sample < 5X the spiking level, within 60-140% recovery if customer or reference method requirements are not specified. Option 2: If activity of the sample ≥ 5X the spiking level, then |ZMS| ≤ 3 (MARLAP 18.4.3 Equation 18.4).	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.

Table B-20. Radon Scintillation (Ra-226 by Lucas Cell)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Duplicate (MD)	One per preparatory batch per matrix.	MD shall be monitored by one of the following: Option 1: The duplicate error ratio (DER) between the sample and the duplicate is ≤ 3; Option 2: RPD is ≤25%. Option 3: Control limit for $\|Z_{Dup}\| \leq 3$. (MARLAP 18.4.2, Equation 18.2)	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, qualify specific nuclides in the parent and explain in the case narrative.
Carriers (if used quantitatively)	Every field sample and any associated batch QC samples as isotopic yield monitor.	Chemical yield within 30-110% if not specified by the customer or the reference method.	If carrier yield is outside the control limit, the data shall be evaluated to determine the sources of difference. Reprepare and analyze sample if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to specific nuclides in all associated samples in the associated preparatory batch and explain in the case narrative.

Table B-22.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification (according to manufacturer instructions)	Before each ICAL.	Masses within the mass range to be monitored shall be within ± 0.2 Da of the target mass.	Correct problem, then repeat verification. Qualification of data is not appropriate.
Instrument Sensitivity Check (ISC)	Daily. The ISC standard shall be less than or equal to the LOQ.	S/N ≥ 10:1 for all quantitation ions and S/N ≥ 3:1 for all other characteristic ions. All reported analytes and surrogates within ± 35% of true value.	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Characteristic Ions	Minimum of 3 structurally significant ions that are not isotopic clusters, when possible. IS and surrogates may use fewer ions.	The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities shall be > 0.	Qualification of data is not appropriate.
Chromatography Performance Checks (e.g., DDT Tailing factor in 8270)	As recommended or required by the full-scan reference method.	As recommended or required by the full-scan reference method	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, prior to sample analysis.	Average response factor ≥ 0.01 for each reported analyte. Minimum 5 levels when calibrating by %RSD or linear regression and 6 levels when calibrating by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the reference method has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 35%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) or Relative Retention Time (RRT) Width	With each sample.	Characteristic ions shall maximize in the same scan or within one scan of each other. Within each SIM descriptor window, characteristic ions for the earliest eluting target analyte shall begin at baseline and latest eluting target analyte shall end at baseline. Option 1: The RT shall be within ±10 seconds of the established RT. Option 2: RRT window width is ± 0.06 from the established RRT. Note: After maintenance is performed which may affect RT, RRTs may be updated based on the daily CCV providing that each peak or peak cluster starts and ends at baseline, within the SIM descriptor window.	Correct problem, then rerun ICAL and any affected samples. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	Response factor ≥ 0.01 for each reported analyte All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method recommends or requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 20% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-22. Organics Analysis by Gas Chromatography/Mass Spectrometry (GC/MS) in Selected Ion Monitoring (SIM) Mode			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	Every field sample and QC sample, spiked at a concentration no greater than the mid-range of the calibration. PAH analysis: Surrogates required for polycyclic aromatic hydrocarbon (PAH) target analytes shall include: fluoranthene-d_{10} and 2-methylnaphthalene-d_{10}. If samples are prepared by ISM the surrogate requirements for PAH analysis are identified in Table B-30.	Same as the LCS acceptance criteria. RF for PAH surrogates ≥ 0.40.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-24.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Samples – Preparation (AFFF and Fluorine Free Foam (F3) only)	Each AFFF sample. Note: This does not include AFFF samples that are to be evaluated for MIL-PRF-14385 compliance. Those AFFF samples shall be evaluated in compliance with DoD AFFF01, not Method 1633. A copy of the latest version of DoD AFFF01 can be found at https://denix.osd.mil/edqw/ Each F3 sample. Note: This includes F3 formulation samples that are to be evaluated for MIL-PRF-32725 compliance.	AFFF and F3 samples shall be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9. Note: The LCSD listed in Section 11.2.6 of DoD AFFF01 is not required. All AFFF and F3 samples shall be prepared and analyzed in duplicate in the same manner as aqueous samples (e.g., solid phase extraction, extracted internal standards, carbon cleanup, etc.)	NA.
Matrix Duplicate (MD) (AFFF and F3 only)	Each AFFF and F3 sample prepared using an aliquot of the field sample shall be prepared in duplicate.	RPD of all analytes ≤ 30% between sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MD is identified, reanalyze the MD or reprepare and analyze the MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Mass Calibration	As recommended or required in the reference method.	As recommended or required in the reference method.	As recommended or required in the reference method. Qualification of data is not appropriate.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Accuracy (Calibration) Verification	As recommended or required in the reference method.	As recommended or required in the reference method.	As recommended or required in the reference method. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	As recommended or required in the reference method.	As recommended or required in the reference method. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and isotopically labeled analog.	As recommended or required in the reference method.	NA
Retention Time (RT) window width	As recommended or required in the reference method. The retention time window must include all isomers present in the isomeric standard mixtures (qualitative or quantitative).	As recommended or required in the reference method.	NA

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Blank (IB)	At the beginning of the analytical sequence, immediately following the highest standard analyzed, and after each bracketing CCV and ISC.	As recommended or required in the reference method.	If acceptance criteria are not met after the highest calibration standard, calibration shall be performed using a lower concentration for the highest standard until acceptance criteria is met. If field sample analyte concentrations exceed the highest calibration standard and the same analytes in the following field sample or in consecutive following field samples also exceed the IB acceptance criteria, the affected samples shall be reanalyzed using a fresh aliquot of the sample extract. If the extract cannot be reanalyzed and re-extraction is not possible, apply qualifier to affected results and explain in the case narrative.
Instrument Sensitivity Check (ISC)	At least once per analytical sequence. An ISC or CCV must be analyzed at the beginning and end of the analytical sequence and after every 10 sample extracts. The ISC and CCV may be alternated.	As recommended or required in the reference method.	As recommended or required in the reference method. Qualification of data is not appropriate.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	At least once per analytical sequence. An ISC or CCV must be analyzed at the beginning and end of the analytical sequence and after every 10 sample extracts. The ISC and CCV may be alternated.	As recommended or required in the reference method.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Qualitative Identification Standard	As recommended or required in the reference method to follow the definition of analytical sequence.	As recommended or required in the reference method.	NA
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ, >1/3 the regulatory compliance limit, or greater than 1/10 the concentration in a sample in the extraction batch, whichever is greatest.	Correct problem. If required, reprepare and analyze MB and all QC samples and affected field samples processed with the contaminated blank if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLLCS) Method 1633 equivalent to the LCS is the Ongoing Precision and Recovery Standard (OPR). Method 1633 equivalent to the LLLCS is Low-Level Ongoing Precision and Recovery Standard (LLOPR).	One set per preparatory batch Shall contain all EIS, NIS, and all analytes to be reported.	Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix or target analyte under evaluation, a laboratory shall use laboratory-developed recovery acceptance criteria no wider than any acceptance criteria provided by the customer. Preliminary acceptance criteria of 40-150% shall be used until acceptance criteria are developed by the laboratory in accordance with Method 1633. Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix or target analyte under evaluation, the laboratory-developed acceptance criteria shall not be < 40%. Note: AFFF and F3 samples are to use the acceptance criteria for aqueous samples.	Correct problem. If required, reprepare and analyze the LCS and/or LLLCS and all affected QC samples and field samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Bile Salt Interference Check	Once per analytical sequence.	As recommended or required in the reference method.	As recommended or required in the reference method. Qualification of data is not appropriate.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Non-extracted Internal Standard (NIS)	As recommended or required in the reference method.	As recommended or required in the reference method.	As recommended or required in the reference method
Extracted Internal Standard (EIS)	Every field sample and QC sample	Isotopically labeled analogs of analytes shall be used when they are commercially available. Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix or EIS under evaluation, a laboratory shall use laboratory-developed recovery acceptance criteria no wider than any acceptance criteria provided by the customer. Preliminary laboratory-developed acceptance criteria of 20-150% shall be used until laboratory acceptance criteria are developed in accordance with Method 1633. Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix or EIS under evaluation, the lower limit of the laboratory-developed acceptance criteria cannot be < 20%.	Notify the customer for additional measures to be taken. Apply qualifier to affected analyte results and explain in the case narrative.

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (Method 1633)			
The laboratory shall meet all the requirements contained in Method 1633*. This table also contains additional requirements that the laboratory shall meet. *The version the laboratory is accredited for or newer version.			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Qualitative Peak Identification **Signal to Noise (S/N)** **Ion Abundance Ratio (IAR)** **Retention Time (RT)**	As recommended or required in the reference method.	S/N, IAR, and RT: As recommended or required in the reference method.	As recommended or required in the reference method. Apply qualifier to affected analyte results and explain in the case narrative.

Table B-25.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: <u>Option 1:</u> %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3:</u> non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 40%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Instrument Sensitivity Check (ISC)	Before analysis and at least once every 12 hours. The ISC standard shall be less than or equal to the LOQ.	All reported analytes and surrogates within ± 40% of true value	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater. For analytes reported across a RT range (e.g., TPH variations, chlordane), the RT window of the method-defined marker compounds are established as stated above. The RT range is calculated based on the lower limit of the RT window for the first marker compound and the upper limit of the RT window for the last marker compound.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action and Qualification Criteria
Continuing Calibration Verification (CCV)	Daily before sample analysis if ICAL not performed; after every 10 samples or 12 hours of analysis time, whichever is sooner; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 25% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Internal Standard (IS) **If Used**	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and LC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 30% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-25. Chemical Warfare Agents and Agent Breakdown Products by Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike	All field and QC samples.	The laboratory shall use the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-30.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Grinding	Each field sample.	The entire field sample shall be ground. Drying is typically performed to aid in grinding and is therefore optional for dry soils. The need for grinding is a project decision. Removing a portion of the field sample, not to exceed 10 g, is only allowed to determine the percent moisture for surrogate spikes. The field sample shall be well mixed before removing a portion.	The laboratory shall explain in the case narrative whether grinding was performed.
Grinding Procedure for Incremental Sampling Methodology (ISM)	When sample grinding is performed, initial method validation and any time major equipment is changed or when a reduction in the number or time of grinding cycles occur.	The laboratory shall demonstrate that the grinding procedure reduces the particle size to < 75 microns by passing representative portions of ground demonstration samples through a 200-mesh sieve.	Correct problem. Qualification of data is not appropriate.
Grinding Cycle	When sample grinding is performed using a puck mill, each field sample.	When a puck mill is used to grind the samples, grinding cycles shall not exceed 60 seconds and shall be followed by a 2-minute or longer cool down period between the grind cycles.	Correct problem and reprepare samples. Qualification of data is not appropriate.
Grinding Blank For batch preparation, the Grinding Blank may serve as the Method Blank.	When sample grinding is performed, a grinding blank is required. One per preparatory batch using Ottawa sand or a verified clean soil. A Grinding Blank shall be performed immediately after a customer-identified sample with suspected high target analyte concentration or after the LCS.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	The laboratory may reprepare and analyze the ground grinding blank to confirm results outside of acceptance criteria. If the grinding blank is not reprepared and analyzed, or if contamination is confirmed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
ISM Laboratory subsampling Replicates The laboratory shall prepare and analyze three or more replicates, each consisting of a minimum of 30 increments of the same mass and depth.	At the analytical subsampling step prior to extraction. When grinding is not performed, each ISM field sample. When grinding is performed, one ISM sample in each preparation batch.	RSD shall not exceed 30%.	RSD failure indicates the sample is not representative. If available, reprepare an additional field sample utilizing grinding procedures. Otherwise, apply qualifier and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS) A laboratory-prepared solid LCS may be used. The fortification shall be performed prior to any preparation steps. The LCS shall be prepared and analyzed in exactly the same manner as the field samples, including all drying and grinding steps.	One per preparatory batch. Shall contain all surrogates and all analytes to be reported. A Standard Reference Material (SRM) that is used for a LCS can be ground as a single batch and subsampled repeatedly as long as the SRM is within expiration date.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Matrix Spike (MS) Spiking is performed after ISM preparation prior to extraction.	One per preparatory batch if sufficient material is provided.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.

Table B-30. Polycyclic Aromatic Hydrocarbon (PAH) Sample Processing by Incremental Sampling Methodology (ISM)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Surrogate Spike Surrogates shall be added to each field sample, blank, and LCS. A surrogate fortification standard shall be added prior to any processing (e.g. prior to drying/grinding or extraction). If drying/grinding is not performed, surrogates may be added after analytical subsampling.	All field samples, blanks and LCS.	The laboratory shall use the following PAH surrogates and respective recovery criteria if the corresponding native analytes are reported unless customer-provided limits are specified. Recovery: Acenaphthylene-d8 within 10% to 120%. Benz[a]anthracene-d12 within 36% to 120%. Benzo[a]pyrene-d12 within 14% to 120%. Fluorene-d10 within 29% to 120%.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.

Table B-31.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Ion Transitions (Parent-> Product)	Before method implementation.	The chemical derivation of the ion transitions shall be documented.	NA.
Mass Calibration	At instrument set-up; at least annually or as specified by the manufacturer, whichever is more frequent; after major maintenance; and when needed based on method requirements or QC results, before analysis. Mass calibration shall be performed using the calibration compounds and procedures prescribed by the manufacturer.	As recommended or required by the instrument manufacturer.	Correct problem, then repeat mass calibration. Qualification of data is not appropriate.
Mass Accuracy Verification (as recommended or required by the method; otherwise according to manufacturer instructions)	Before each ICAL.	Masses within the mass range to be monitored shall be within 0.2 Da of the target mass.	Correct problem, then repeat verification. Qualification of data is not appropriate.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Instrument Sensitivity Check (ISC)	Before analysis and at least once every 12 hours. The ISC standard shall be less than or equal to the LOQ.	S/N ≥10:1 for all quantitation ions and S/N ≥3:1 for all other characteristic ions in the lowest calibration standard used to define the quantitative range. All reported analytes and surrogates within ± 30% of true value.	Correct problem, then repeat ISC. If problem persists, repeat ICAL, raising LOQ to a concentration where criteria are met. Qualification of data is not appropriate.
Initial Calibration (ICAL) for all analytes	At instrument set-up and when needed based on method requirements or QC results, before sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the reference method has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.
Evaluation of Relative Error (%RE) or Relative Standard Error (%RSE)	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range of the ICAL and at less than or equal to the LOQ, or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 20% and 30%, respectively. The maximum allowable %RSE shall be 30%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Retention Time (RT) and Relative Retention Time (RRT) establishment	Once per ICAL and at the beginning of the analytical sequence. Established for each analyte and surrogate.	RT or RRT shall be set using the midpoint standard of the ICAL or average of the ICAL standards when ICAL is performed, or on days when ICAL is not performed, the initial CCV for the sequence shall be used.	NA.
Retention Time (RT) window width **No Internal Standard Used**	At method set-up and after major maintenance (e.g., column change). Calculated for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA.
Relative Retention Time (RRT) window width **Internal Standard Used**	With each sample.	RRT window width is ± 0.06 from the established RRT.	NA.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 10 field samples; and at the end of the analytical batch run.	All reported analytes and surrogates within their respective retention time window and within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. If the specific version of a reference method requires additional evaluation (e.g., average RFs) these additional requirements shall also be met.	Where an assignable cause isolated to only the CCV is identified, one CCV may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCV is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCV or recalibrate. All affected samples since last passing CCV shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.
Continuing Calibration Blank (CCB)	Immediately after the ICAL and immediately after the CCV.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated sample(s) whichever is greater.	Where an assignable cause isolated to only the CCB is identified, one CCB may be reanalyzed immediately (i.e., within one hour and no samples analyzed). If the immediate CCB is acceptable, proceed with analysis. Sample reanalysis is not required. Otherwise, correct problem and analyze passing CCB or recalibrate. All affected samples since last passing CCB shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to affected analyte results and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Internal Standard (IS)	Every field sample, standard, and QC sample.	RT within ± 10 seconds from RT of the midpoint standard in the ICAL or average of the ICAL standards; EICP area within -30% to +30% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV may be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. All affected samples shall be reanalyzed. If the samples cannot be reanalyzed, apply qualifier to analyte results associated with the IS outside acceptance criteria and explain in the case narrative.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in the associated samples whichever is greater.	Where an assignable cause isolated to only the MB is identified, the MB may be reanalyzed. Otherwise, reprepare and analyze MB and all affected QC and field samples in the associated preparatory batch if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Laboratory Control Sample (LCS)	One per preparatory batch. Shall contain all surrogates and all reported analytes.	Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use Appendix C limits. If Appendix C limits are not available, use laboratory-developed acceptance criteria.	Where an assignable cause isolated to only the LCS is identified, the LCS may be reanalyzed. Otherwise, reprepare and analyze the LCS and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.
Laboratory Control Sample Duplicate (LCSD)	If sufficient sample is not available for either a MSD or MD, one LCSD shall be included in the preparatory batch. Shall contain all surrogates and all reported analytes.	Recovery: Same as LCS acceptance criteria. Precision: RPD of all analytes ≤ 20% between LCS and LCSD.	Where an assignable cause isolated to only the LCSD is identified, the LCSD may be reanalyzed. Otherwise, reprepare and analyze the LCSD and all affected QC and field samples in the associated preparatory batch for the analytes outside acceptance criteria if sufficient sample material is available. If the samples cannot be reprepared and analyzed, apply qualifier to affected analyte results of all samples in the associated preparatory batch and explain in the case narrative.

Table B-31. Organics Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS) and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) (e.g., Method 8321, not to include Method 1633)			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action and Qualification Criteria**
Matrix Spike (MS)	One per preparatory batch if sufficient material is provided. Shall contain all surrogates and all reported analytes.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the MS is identified, reanalyze the MS or reprepare and analyze the MS if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch if sufficient material is provided. MSD: Shall contain all surrogates and all reported analytes. MD: Shall be analyzed for all surrogates and all reported analytes.	Recovery: Same as the LCS recovery acceptance criteria. Precision: RPD of all analytes ≤ 30% between MS and MSD or sample and MD. RPD does not apply if both results are below the LOQ.	If an assignable cause isolated to only the MSD or MD is identified, reanalyze the MSD or MD or reprepare and analyze the MSD or MD if sufficient sample material is available, as indicated by the cause. Otherwise, apply qualifier to affected analyte results in the parent sample and explain in the case narrative.
Surrogate Spike	All QC and field samples.	Same as the LCS acceptance criteria.	If an assignable cause isolated to only the surrogates is identified in a field sample, reprepare and analyze the field sample if sufficient sample material is available. If obvious chromatographic interference is present, repreparation and analysis may not be necessary, but the customer shall be notified before reporting data. If samples with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte results associated with the surrogates outside acceptance criteria and explain in the case narrative.