

Table F-2. Organic Analysis by Gas Chromatography					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be $\leq 15\%$.	Correct problem, then repeat breakdown check.	Flagging is not appropriate.	No samples shall be run until degradation of DDT and Endrin is each $\leq 15\%$.
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$; 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. Calibration may not be forced through the origin.
Retention time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes and surrogates within established RT windows. All target analytes and surrogates within $\pm 15\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table F-2. Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes within established RT windows. All target analytes and surrogates within $\pm 15\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater. Common contaminants must not be detected $> LOQ$.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of the difference, i.e., matrix effect or analytical error.

Table F-2. Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD \leq 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits.	Correct problem, then re-prep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed (except for single column methods such as TPH by Method 8015 where confirmation is not an option or requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD \leq 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, report the result from the primary column.

Table – 2A. Organic Analysis by High-Performance Liquid Chromatography (HPLC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes(including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 20\%$; 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.	Flagging is not appropriate.	Minimum 5 levels for linear, and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. Calibration may not be forced through the origin.
Retention time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	NA.	Calculated for each analyte and surrogate.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes and surrogates within established RT windows. All target analytes and surrogates within $\pm 15\%$ of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

Table – 2A. Organic Analysis by High-Performance Liquid Chromatography (HPLC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes and surrogates within established RT windows. All target analytes and surrogates within $\pm 15\%$ true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification..	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for the failed target analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

Table – 2A. Organic Analysis by High-Performance Liquid Chromatography (HPLC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD \leq 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits.	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column/detector RPD \leq 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Confirmation of a UV detector with a UV diode array detector or vice versa is not allowed. Use project-specific reporting requirements if available; otherwise, report the result from the primary column.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B and 8330C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil drying procedure	Each sample and batch LCS.	Laboratory must have a procedure to determine when the sample is dry to constant weight. Record date, time, and ambient temperature on a daily basis while drying samples.	NA.	Flagging is not appropriate.	
Soil sieving procedure	Each sample and batch LCS.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging is not appropriate.	
Soil grinding procedure	Initial demonstration.	The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging is not appropriate.	
Soil grinding blank	Between each sample.	A grinding blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as a field sample. No target analytes must be detected > 1/2 LOQ.	All blank results must be reported and the affected samples must be flagged accordingly if blank criteria is not met.	If any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the sample following that blank.	The laboratory must prepare a grinding blank between each sample, however, all grinding blanks are not required to be analyzed. Only grinding blanks ground following a sample with target analytes detected >= LOD must be analyzed.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B and 8330C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil subsampling process	Each sample, duplicate, and batch LCS.	Entire ground sample is mixed, spread out on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth to sum a ~10 g subsample.	NA.	Flagging is not appropriate.	
Soil sample triplicate	At the subsampling step, one sample per batch. Cannot be performed on any sample identified as a blank (e.g., trip blank, field blank, method blank).	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the Quantitation Range of the method. The RSD for results above the LOQ must not exceed 20%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reported per the client, apply J-flag if acceptance criteria are not met and explain in the case narrative.	
Aqueous sample preparation	Each sample and associated batch QC samples.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required.	NA.	Flagging is not appropriate.	The salting-out procedure is not permitted.
Initial calibration (ICAL)	At instrument setup and after ICV or CCV failure, prior to sample analysis.	The apparent signal-to-noise ratio at the LOQ must be at least 5:1. ICAL must meet one of the three options below: Option 1: RSD for each analyte $\leq 15\%$; 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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B and 8330C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analyte(s) and surrogates within $\pm 20\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes and surrogates within $\pm 20\%$ of the true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater. Common contaminants must not be detected $> LOQ$.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem. If required, re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	A solid reference material containing all reported analytes must be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B and 8330C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. MSD or MD: RPD of all analytes $\leq 20\%$ (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits.	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria are the same for the confirmation analysis as for initial or primary column analysis. Results between primary and second column RPD $\leq 40\%$.	Report from both columns.	Apply J-flag if RPD $> 40\%$. Discuss in the case narrative.	Confirmation of a UV detector with a UV diode array detector or vice versa is not allowed. Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis.

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by HPLC, LC/MS, or LC/MS/MS (Method 8330B and 8330C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
					<p>Secondary column – Must be capable of resolving (separating) all of the analytes of interest and must have a different retention time order relative to the primary column.</p> <p>Use project specific reporting requirements if available; otherwise report from the primary column.</p>

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Table - 4. Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance check.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the target analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: <u>Option 1:</u> RSD for each analyte \leq 15%; <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.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. Calibration may not be forced through the origin. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of	With each sample.	RRT of each target analyte	Correct problem, then	NA	RRTs may be updated based

Table - 4. Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Relative Retention Times (RRT)		within ± 0.06 RRT units.	rerun ICAL.		on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, perform analysis of a second source standard prior to sample analysis.	All target analytes and surrogates within $\pm 20\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis and after every 12 hours of analysis time.	All target analytes and surrogates within $\pm 15\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	

Table - 4. Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.

Table - 4. Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits.	Correct problem, then re- prep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL.	Verify mass calibration per method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Retention time window defining mix	At method set-up and prior to analyzing calibration standards.	Verify descriptor switching times per method.	Correct problem, then repeat retention time window defining mix.	Flagging is not appropriate.	
GC column performance check (for SP-2331 column or equivalent)	At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed.	<p><u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers:</u> Resolved with a valley of $\leq 25\%$.</p> <p><u>For calibration verification standard only:</u> Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley.</p>	Correct problem, then repeat column performance check.	Flagging is not appropriate.	Needed only if using a column other than DB-5 or equivalent.
GC Column performance check (for DB-5 column or equivalent)	<p>At the beginning and end of each 12-hr period during which samples or calibration solutions are analyzed.</p> <p>Included with the ICAL standard (CC3) and the calibration verification standard.</p>	<p><u>Peak separation of standard CC3:</u> Peak between the ^{13}C-2,3,7,8-TCDD and ^{13}C-1,2,3,4-TCDD must be resolved with a valley of $\leq 25\%$;</p> <p><u>For calibration verification standard only:</u> Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of $\leq 50\%$.</p>	Correct problem, then repeat column performance check.	Flagging is not appropriate.	No samples shall be analyzed until GC column performance check is within criteria.

Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes identified in method	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	<p>Ion abundance ratios must be in accordance with the method.</p> <p>RSD of the RFs \leq 15% for labeled IS and unlabeled PCDD/PCDF.</p>	Correct problem then repeat ICAL.	Flagging is not appropriate.	<p>No samples shall be analyzed until ICAL has passed.</p> <p>Calibration may not be forced through the origin.</p>
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	<p>Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards.</p> <p>Sensitivity criteria of an S/N ratio $>$ 2.5 for unlabeled PCDD/PCDF ions and $>$ 10 for labeled internal and recovery standards.</p> <p>All target analytes and IS within \pm 20% of true value.</p>	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Calibration verification	At the beginning of each 12-hr period of sample analysis, after successful GC and MS resolution checks.	<p>Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards.</p> <p>Sensitivity criteria of an S/N ratio $>$ 2.5 for unlabeled PCDD/PCDF ions and $>$ 10 for labeled internal and recovery standards.</p> <p>All target analytes and IS within \pm 20% of true value.</p>	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	<p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.</p>	<p>Results may not be reported without a valid calibration verification.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>

Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards (IS)	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to any dilutions) must be within 25-150% of the CCV.	Correct problem, then re-prep and reanalyze the sample(s) with failed IS.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Sensitivity check	At the end of 12-hr sample analysis period or at the end of analysis (whichever comes first) (Injection must be done within the 12-hr period.).	See calibration verification for criteria on ion abundances, and S/N ratios. See retention time window defining mix for retention time criteria.	Correct problem, then repeat calibration and reanalyze samples indicating a presence of PCDD/PCDF less than LOQ or when maximum possible concentration is reported.	Flagging is not appropriate.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if criteria are not met and explain in the case narrative..	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-5. DioxIn/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits.	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	

Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PCDD/PCDF Identification	Identify all positive sample detections per method.	<p>Verify that absolute RT at maximum height is within -1 to +3 seconds of that for corresponding labeled standard, or the RRT of analytes is within 0.05 RRT units of that for unlabeled standard in the calibration verification standard, or RT for non-2,3,7,8-substituted isomers within the RT window established by the window defining mix for the corresponding homologue per method.</p> <p>Absolute RTs of the recovery standards must be within ± 10 seconds of those in the calibration verification standard.</p> <p>All ions listed in Table 8 of the method must be present in the SICP, must maximize simultaneously (± 2 sec.), and must have not saturated the detector.</p> <p>S/N ratio of ISs ≥ 10 times background noise. Remaining ions in Table 8 of the method must have an S/N ratio ≥ 2.5 times the background noise</p> <p>Ion abundance in Table 9 of the method must be met for all analytes, internal, and recovery standards.</p>	<p>Correct problem, then re-prepare and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards. If PCDF is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to the method.</p>	Flagging is not appropriate.	Positive identification of 2,3,7,8-TCDF on the DB-5 or equivalent column must be reanalyzed on a column capable of isomer specificity (DB-225) (see method).

Table F-5. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Estimated Maximum Possible Concentration (EMPC)	Determined for each 2,3,7,8-substituted isomer that did not meet ion abundance ratio criteria (Table 9 of the method) or PCDFs where peak representing a corresponding PCDF was detected.	Response for both quantitation ions must have an S/N ≥ 2.5 ; all other criteria from sample PCDD/PCDF identification above. For PCDF, no signal with S/N > 2.5 is detected at the same retention time (± 2 seconds) as the corresponding PCDF channel.	NA.	Flagging as appropriate.	
Sample 2,3,7,8-TCDD toxicity equivalents concentration (TEQ)	All positive detections.	If the TEQ is greater than 0.7 ppb for soil/sediment or fly ash, 7 ppb for chemical waste, or 7 ppt for an aqueous sample; and 2,3,7,8-TCDF is either detected or reported as an EMPC, then better isomer specificity may be required than can be achieved on the DB-5 column or equivalent.	NA.	Flagging is not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.

Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Resolving Power	Prior to ICAL and at the beginning and the end of each 12-hour period of analysis.	Static resolving power $\geq 10,000$ (10% valley) for identified masses and most intense lock-mass ion of reference compound $\leq 10\%$ full-scale deflection.	Retune instrument and verify. Rerun affected samples.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check	Prior to ICAL or calibration verification. At the beginning of each 12-hr period during which samples or calibration solutions are analyzed.	<u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers:</u> Resolved with a valley of $\leq 25\%$. Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram. Absolute retention times for switching from one homologous series to the next ≥ 10 sec. for all components of the mixture.	Correct problem then repeat column performance check.	Flagging is not appropriate.	Use GC column performance check solution If the laboratory operates during consecutive 12-hr periods. No samples shall be analyzed until performance check is within criteria.
Initial calibration (ICAL) for all analytes identified in method	At instrument setup and after ICV or CCV failure, prior to sample analysis, and when a new lot is used as standard source for HRCC-3, sample fortification (IS), or recovery solutions.	Ion abundance ratios in accordance with the method. S/N ratio ≥ 10 for all target analyte ions. RSD $\leq 20\%$ for the response factors (RF) for all 17 unlabeled standards. RSD $\leq 20\%$ for the RFs for the 9 labeled IS.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed run until ICAL has passed. Calibration may not be forced through origin.

Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	Ion abundance specified in the method must be met;. For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ D of the mean of RF established in ICAL.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification	At the beginning of each 12-hour period, and at the end of each analytical sequence.	<p>Ion abundance specified in the method must be met.</p> <p>For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; <u>and</u> For labeled standards, RF within $\pm 30\%$ D of RF established in ICAL.</p>	<p>Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.</p> <p><u>End-of-run CCV:</u> If the RF for unlabeled standards $\leq 25\%$ RPD and the RF for labeled standards $\leq 35\%$ RPD (relative to the RF established in the ICAL), the mean RF from the two daily CCVs must be used for quantitation of impacted samples instead of the ICAL mean RF value. If the starting and ending CCVRFs differ by more than 25% RPD for unlabeled compounds or 35% RPD for labeled compounds, the sample may be quantitated against a new initial calibration if it is analyzed within two hours. Otherwise reanalyze samples with positive detections if</p>	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards (IS)	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to dilutions) must be within 40 – 135% of the ICAL average RF.	Correct problem, then re-prep and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples and explain in the case narrative.	

Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch, run after calibration standards and before samples.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD \leq 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Internal Standards (IS)	Every field sample, standard, and QC sample.	% Recovery for each IS in the original sample (prior to dilutions) must be within 40 – 135%.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples.	
Sample Estimated Maximum Possible Concentration (EMPC)	Every sample with a response S/N \geq 2.5 for both quantitation ions.	Identification criteria per method must be met, and the S/N of response for both quantitation ions must be \geq 2.5	NA.	Flagging as appropriate.	
Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration	All positive detections.	Per method.	NA.	Flagging is not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.

Table F-6. Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PCDD/PCDF identification	Identify all positive sample detections per method.	<p><u>2,3,7,8-substituted isomers with labeled standards:</u> Absolute RT at maximum height within -1 to +3 seconds of that for corresponding labeled standard; <u>2,3,7,8-substituted isomers with unlabeled standards:</u> RRT within 0.005 RRT units of that in calibration verification standard; <u>Non-2,3,7,8-substituted isomers:</u> RT within RT window established by column performance check solution for corresponding homologue, per method; <u>and</u> Ions for quantitation must maximize simultaneously (± 2 sec.); <u>and</u> Ion abundance ratios in accordance with criteria in Table 8 of the method; <u>and</u> S/N ratio of ISs ≥ 10 times background noise; <u>and</u> S/N ratio of all remaining ions for unlabeled analytes ≥ 2.5 times background noise; <u>and For PCDF:</u> No signal present having an S/N ratio ≥ 2.5 for the corresponding ether (PCDPE) detected at the same retention time (± 2 sec).</p>	Correct problem, then re-prepare and reanalyze the samples with failed criteria for any of the internal, recovery, or cleanup standards. If PCDPE is detected or if sample peaks present do not meet ion abundance ratio criteria, calculate the EMPC (estimated maximum possible concentration) according to method.	Flagging is not appropriate.	Positive identification of 2,3,7,8-TCDF on the DB-5 or equivalent column must be reanalyzed on a column capable of isomer specificity (DB-225) (see method).

Table- F-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA) (7000 Series Methods)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	FLAA and GFAA: minimum three standards and a calibration blank; CVAA/Mercury: minimum 5 standards and a calibration blank No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes within $\pm 10\%$ of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All target analytes within $\pm 10\%$ of the true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reprep or reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected $> LOD$.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank.

Table- F-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA) (7000 Series Methods)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD < 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.

Table- F-7. Inorganic Analysis by Atomic Absorption Spectrophotometry (AA) (7000 Series Methods)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-Digestion Spike (PDS) Addition (Flame AA and GFAA only)	One per preparatory batch if MS or MSD fails.	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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Table F-7A. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry (Method 6010)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All target analytes within $\pm 10\%$ of the true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-7A. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry (Method 6010)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-level Calibration Check Standard (Low-level ICV)	Daily, after one-point ICAL.	All target analytes within $\pm 20\%$ of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected $> \text{LOD}$.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank.

Table F-7A. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry (Method 6010)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	<p><u>ICS-A:</u> Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes);</p> <p><u>ICS-AB:</u> Within \pm 20% of true value.</p>	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	<p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p>	<p>Must contain all target analytes. Results may not be reported without a valid LCS.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.

Table F-7A. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry (Method 6010)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD < 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fail. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

Table – F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range.	Within $\pm 10\%$ of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes, within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All target analytes within $\pm 10\%$ of the true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low Level ICV)	Daily, after one-point ICAL.	All target analytes within $\pm 20\%$ of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.

Table – F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	<u>ICS-A</u> : Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); <u>ICS-AB</u> : Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

Table - F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all target analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD < 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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Table F-1. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	<p>Minimum three standards and a reagent blank.</p> <p>No samples shall be analyzed until ICAL has passed.</p> <p>Calibration must not be forced through the origin.</p>
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis, after every 15 field samples and at the end of the analysis sequence.	All target analytes within $\pm 10\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for hexavalent chromium in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for hexavalent chromium in all samples in the associated preparatory batch.	<p>Results may not be reported without a valid method blank.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>

Table F-1. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re- prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for hexavalent chromium in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	Once per preparatory batch.	Spike recovery within 85–115%.	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error. Verification check ensures lack of reducing conditions or interference from matrix.
Matrix spike Duplicate (MSD) or Matrix Duplicate (MD)	<u>Aqueous matrix:</u> One per every 10 project samples. <u>Solid matrix:</u> One per preparatory batch.	Spike recovery within 85–115%. RPD ≤ 20%.	Dilute and reanalyze sample; persistent interference indicates the need to use the method of standard addition, alternative analytical conditions, or an alternative method. Re- prep and reanalyze all samples in the prep batch.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. Results may not be reported without a valid pair. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Soluble and Insoluble Pre-Digestion Matrix Spikes (solid matrix samples only)	One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.	MS recoveries within 75 – 125%.	Correct problem and re-homogenize, re-digest, and reanalyze samples. If that fails, evaluate against LCS results.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.	

Table F-1. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-digestion Matrix Spike (solid matrix samples)	One per preparatory batch.	Recovery within 85 - 115%.	No specific CA, unless required by the project.	Apply J-flag to all results for hexavalent chromium if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations > 50 X LOQ prior to dilution.

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Table F-1. Cyanide Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed.
Distillation Verification	Once after each ICAL, with two distilled ICAL standards prior to sample analysis. Not required if all ICAL standards are distilled.	Within $\pm 10\%$ of true value.	Correct problem, rerun distilled standards or repeat ICAL.	Flagging is not appropriate.	One high and one low distilled ICAL standard. No samples shall be analyzed until distillation technique has been verified.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	Within $\pm 10\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for cyanide in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, re-rep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all cyanide results in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence. After ICV and each CCV.	No analytes ≥ 0.03 mg/L.	Correct problem and reanalyze all samples analyzed since the last acceptable calibration blank.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank.

Table F-1. Cyanide Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-rep and reanalyze the LCS and all samples in the associated preparatory batch, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the LCS limits, the data shall be evaluated to the source of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) and Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. RPD \leq 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

Table F-1. Common Anions Analysis (Method 9056)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for All Analytes	ICAL prior to sample analysis.	$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum 3 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Retention Time Window Position Establishment	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Established for each analyte.
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 over a 24-hour period.	NA.	NA.	Calculated for each analyte.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes within established RT windows. All target analytes within $\pm 10\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Freshly prepared ICV. No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes within established retention time windows. All target analytes within $\pm 10\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-1. Common Anions Analysis (Method 9056)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for all target analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all target analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all target analytes. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise, default to acceptance criteria in the DoD QSM, if available; otherwise use in-house LCS control limits. RPD < 15% (between MS and MSD).	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all target analytes. The data shall be evaluated to determine the source of difference.

Table F-1. Perchlorate Analysis by Mass Spectrophotometry Methods

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference Threshold 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).	Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 80-120% window.	NA.	Flagging criteria are not appropriate.	This study and site history will determine the concentration at which the ICS suppressors should be set.
Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses show large deviations from known masses, major instrument maintenance is performed, or the instrument is moved).	Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for an analytical run, and the same mass calibration must be used for all data files in an analytical run. Mass calibration must be verified by acquiring a full scan continuum mass spectrum of a perchlorate stock standard. Perchlorate ions should be within $\pm 0.3 m/z$ of mass 99, 101, and 107 or their respective daughter ion masses (83, 85, and 89), depending on which ions are quantitated.	If the mass calibration fails, recalibrate. If it still fails, consult manufacturer instructions on corrective maintenance.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration.
Tune Check	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standards must contain the analytes of interest and meet acceptance criteria outlined in the laboratory SOP.	Retune instrument and verify. If the tune check will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging is not appropriate.	No samples shall be analyzed without an acceptable tune check.

Initial Calibration (ICAL)	At instrument setup or after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the two options below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least r \geq squares regression for each analyte: $r^2 \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum of 5 calibration levels must be used. The calibration is linear and shall not be forced through the origin. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL.	Perchlorate concentration must be within $\pm 15\%$ of its true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	ICV shall be a second source standard with its concentration at the midpoint of the calibration. No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	On days an ICAL is performed, after every 10 field samples and at the end of the analytical sequence. On days an ICAL is not performed, at the beginning of the sequence, after every 10 field samples and at the end of the analytical sequence.	Perchlorate concentration must be within $\pm 15\%$ of its true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Detection Limit Verification (DLV)	One per batch. Prior to sample analysis and at the end of the analysis sequence. It can be analyzed after every 10 samples in order to reduce the reanalysis rate.	Perchlorate concentration must be within $\pm 30\%$ of its true value.	Correct problem. Rerun DLV and all samples analyzed since last successful DLV. If that fails, then repeat ICAL. If a sample with perchlorate concentration at or between the LOD and LOQ is bracketed by a failing DLV, it must be reanalyzed. A sample with concentration above the LOQ can be reported.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable DLV.	Results less than the LOQ may not be reported without a valid DLV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Perchlorate spike concentration is approximately 2 times the limit of detection.

Table F-12. Perchlorate Analysis (Methods 6850 and 6860) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Isotope Ratio ³⁵ Cl/ ³⁷ Cl	Every sample, batch QC sample, and standard.	Monitor for either the parent ion at masses 99/101 or the daughter ion at masses 83/85 depending on which ions are quantitated. Must fall within 2.3 to 3.8.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample must be re-extracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate, i.e., a post spike sample or dilution to reduce any interference, etc.	If reanalysis after cleanup fails to meet acceptance criteria, data must be qualified with a Q-flag and explained in the case narrative. The disposition of results of alternate techniques used to confirm presence of perchlorate must be discussed in the case narrative.	Decision to report data failing ratio check should be thoroughly documented in case narrative. The use of cleanup procedures, post spike samples, and dilutions must be identified in the case narrative.
Internal Standard (IS)	Addition of ¹⁸ O-labeled perchlorate to every sample, batch QC sample, standard, instrument blank, and method blank.	Measured ¹⁸ O IS area within $\pm 50\%$ of the value from the average of the IS area counts of the ICAL. RRT of the perchlorate ion must be $1.0 \pm 2\%$ (0.98 - 1.02).	Rerun the sample at increasing dilutions until the $\pm 50\%$ acceptance criteria are met. If criteria cannot be met with dilution, the interference are suspected and the sample must be re-prepped using additional pretreatment steps.	If reanalysis after pretreatment steps fails to meet acceptance criteria, data must be qualified with a Q-flag and explained in the case narrative.	If peak is not within retention time window, presence is not confirmed. Failing internal standard must be thoroughly documented in the case narrative.
Interference Check Sample (ICS)	One ICS is prepared with every batch of 20 samples and must undergo the same preparation and pretreatment steps as the samples in the batch. It verifies the method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily.	Perchlorate concentration must be within $\pm 20\%$ of its true value.	Correct problem. Reanalyze all samples and QC samples in the batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed.	Flagging criteria are not appropriate.	Analysis of a standard containing perchlorate at the LOQ and interfering anions at the concentration determined by the interference threshold study. No samples may be reported that are associated with a failing ICS.

Table F-12. Perchlorate Analysis (Methods 6850 and 6860) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Reagent Blank (LRB)	Prior to calibration, after samples with over range concentration of perchlorate, and at the end of the analytical sequence.	No perchlorate detected > 1/2 RL.	Reanalyze reagent blank (until no carryover is observed) and all samples processed since the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated batch.	Problem must be corrected. Results may not be reported without a valid reagent blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. Re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits, or 80-120% (whichever is more stringent) to verify calibration and to check method performance.	Correct problem. Re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	LCS must be spiked at the LOQ. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. LCS must undergo the same preparation and pretreatment steps as the samples in the batch.

Table F-12. Perchlorate Analysis (Methods 6850 and 6860) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch per matrix. The MS must be spiked at the LOQ.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. Recovery within 80-120% or within laboratory generated limits, whichever is more stringent.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	<p>The MS must be spiked at the LOQ.</p> <p>If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error.</p> <p>MS must undergo the same preparation and pretreatment steps as the samples in the batch.</p>
Matrix Spike Duplicate (MSD) or Laboratory Duplicate (LD)	One per preparatory batch per matrix. The MSD must be spiked at the LOQ.	<p>QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits.</p> <p>MSD: Recovery within 80-120% or within laboratory generated limits, whichever is more stringent.</p> <p>MSD or LD: RPD < 15%.</p>	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	<p>The MSD must be spiked at the LOQ.</p> <p>The data shall be evaluated to determine the source of difference.</p>

Table F13-CWA (Chemical Warfare Agents by GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	DFTPP Mass range from 51-443 m/z	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analyte(s) and surrogates within $\pm 25\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All target analytes and surrogates within $\pm 25\%$ of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Retention Time Window Position Establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
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.	NA.	NA.	Calculated for each analyte and surrogate.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes within established RT windows. All target analytes and surrogates within $\pm 55\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F13-CWA (Chemical Warfare Agents by GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected $> 1/2$ LOQ or $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit, whichever is greater. Common contaminants must not be detected $> LOQ$.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F13-CWA (Chemical Warfare Agents by GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. If LCS limits are not available use 50 – 150%.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise, use in-house control limits. If in-house limits are not available, use 50 -150%.	Correct problem, then re-prepare and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table F-14. Perfluorinated Compounds by LC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	Minimum of 5 calibration standards to establish linearity at method set-up and after major maintenance.	Each calibration point for each analyte must calculate to be within 75-125%, except the lowest cal point which must calculate to within 70-130%	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples may be run until ICAL has passed. Calibration must not be forced through the origin. Calibration can be linear (5 standards) or quadratic (6 standards); weighting is allowed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Within $\pm 25\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging is not appropriate.	No samples may be run until calibration has been verified.
Continuing Calibration Verification (CCV)	Analysis of mid-level standard after every 10 field samples. All samples must be bracketed by the analysis of a standard demonstrating that the system was capable of accurately detecting and quantifying perfluorinated compounds.	Within $\pm 25\%$ of true value.	Immediately analyze two consecutive CCVs. If both pass, samples can be reported without reanalysis. If either of these two CCVs fail, take corrective action(s) until an acceptable CCV is obtained. All affected samples since the last acceptable CCV must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table – F14. Perfluorinated Compounds by LC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standard (IS)	Addition of isotopically labeled analytes to every sample, batch QC sample, standard, instrument blank, and method blank.	Determine that the absolute areas of the quantitation ions of the IS(s) are within 50-150% from the average areas measured during initial calibration.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. For failed QC samples, correct problem, and rerun all associated failed field samples.	Apply Q-flag and discuss in the case narrative.	Failing internal standard should be thoroughly documented in the case narrative.
Tune Check	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 amu of true value.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging is not appropriate.	Problem must be corrected. Sample analysis shall not proceed without acceptable tuning.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prepare and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use its in-house statistically established LCS control limits for the purpose of batch control, and its in-house statistically established LCS control limits must be within project-specified LCS control limits. If in-house limits do not exist use 70-130%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table – F14. Perfluorinated Compounds by LC/MS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch per matrix.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. If in-house limits do not exist use 70-130%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch per matrix.	QC acceptance criteria specified by the project, if available; otherwise use in-house LCS control limits. If in-house limits do not exist use 70-130%. MSD or MD: RPD of all analytes ≤ 30% (between MS and MSD or sample and MD)	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of error. Analyze MS/MSD for low concentration samples and sample/MD for high concentration samples.
Surrogate	All field and QC samples.	Within ± 30% of true value.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. For failed QC samples, correct problem, and rerun all failed samples.	Apply Q-flag and discuss in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.