



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 1 of 73

Laboratory Name:
Location:

A2LA Master Code:
Assmnt ID:
Cert No.

Assessor Instructions: Review a representative sample of laboratory quality control procedures and data packages to adequately cover the methods on the Scope of Accreditation, to determine compliance with the following hierarchy of requirements: project specific requirements, current DoD ELAP QSM Appendix F table requirements (attached), or method requirements. Record the results in the table below and enter relevant information in the A2LA test method review matrix (Form A312).

Appendix F Tables	Compliant		Comment
	Y	N	
F-1			
F-2			
F-3			
F-4			
F-5			
F-6			
F-7			
F-8			
F-9			
F-10			
F-11			
F-12			



Appendix F – SW-846 Quality Control Requirements

In many cases, SW-846 methods are ambiguous or provide insufficient detail in regards to Quality Control (QC) requirements. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for SW-846 analytical methods commonly used when investigating DoD sites. The tables specify the minimum DoD requirements, as well as additional clarification. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the method is made. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating minimum DoD requirements when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed unless project-specific or regulatory approval is required.

SW-846 Methods

This appendix is based on all method versions available at the time of publication, regardless of status (promulgated, draft, or proposed). The requirements in this appendix represent the minimum requirements for DoD regardless of method version. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed unless project-specific or regulatory approval is required.

Table F-1 below presents a summary of the definition, purpose, and evaluation of the major SW-846 QC checks required in the subsequent QC tables (F-2 through F-12) for the various methods. The definition column describes what the QC check is and how it is performed. The purpose column describes why the check is important for assessing and measuring the quality of the data being generated. The evaluation column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms relevant to this appendix follows Table F-12.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 3 of 73

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The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 4 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin and DDT – Method 8081, DDT – Method 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	All positive results must be confirmed.
Continuing calibration verification (CCV)	The verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging.
Demonstrate acceptable analytical capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Dilution test (metals only)	Analysis of a positive sample, which has been diluted to a concentration one-fifth of the original, to confirm that there is no interference in the original sample analysis. (Modified COE)	To assess matrix interference.	Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the method of standard additions. Results outside acceptance limits indicate a possible matrix effect. For ICP, a post-digestion spike must be run; for GFAA, a recovery test must be run.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 5 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation (continued)

QC Check	Definition	Purpose	Evaluation
Duplicate sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (Modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix.	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
GC column performance check (Methods 8280 and 8290 only)	Analysis of method-specified compounds to verify chromatographic separation of dioxin isomers. (Method)	To evaluate the performance of the analytical system and establish retention time window markers for dioxin isomers.	Sample analysis may not begin until method-specified criteria are met.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method.	To establish a calibration curve for the quantification of the analytes of interest.	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Instrument detection limit (IDL) study (Methods 6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide an evaluation of instrument sensitivity.	IDLs must be established before samples can be analyzed.
Interference check solutions (ICP and ICP/MS only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence.	No samples can be run if this check does not pass acceptance criteria.
Internal standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any samples associated with out-of-control results must be reanalyzed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 6 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation (continued)

QC Check	Definition	Purpose	Evaluation
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC check. The inability to achieve acceptable recoveries in the LCS indicates problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a “clean” matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
Linear dynamic range or high-level check standards (ICP and ICP/MS only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high-level standard.	This QC check establishes the upper linear range of the calibration.
Low-level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte equal to or less than the reporting limit.	To confirm the accuracy of measurements at or near the RL.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix spike (MS)	A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method’s recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The laboratory should not correct for recovery; only report the results of the analyses and the associated matrix spike results and indicate that the results from these analyses have increased uncertainty.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 7 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation (continued)

QC Check	Definition	Purpose	Evaluation
Matrix spike duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte.	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared with the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
Matrix verification sample (hexavalent chromium only)	A pH-adjusted filtrate that has been spiked with hexavalent chromium to ensure that the sample matrix does not have a reducing condition or other interferences that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferences that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
Method blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interference or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure laboratory accuracy/bias. This sample could indicate whether contamination is occurring during sample preparation and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analyte(s) in all samples in the associated preparatory batch, as appropriate. For common laboratory contaminants, no analytes detected > the RL. See Section D.1.1.1 and Box D-1.
Method of standard additions (ICP/GFAA only)	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is also called spiking the sample.)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 8 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation (continued)

QC Check	Definition	Purpose	Evaluation
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects.	To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures.	To verify the absence of an interference, the spike recovery must be between 75% and 125%. Results outside the acceptance limits require a method of standard additions (MSA) for all samples within the batch.
Recovery test (GFAA only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects.	To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. Results outside the acceptance limits require a MSA for all samples within the batch.
Retention time window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the retention time window (i.e., start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To identify analytes of interest.	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
Retention time window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or group of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight retention time windows may result in false negatives or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the second-source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.



The American Association for Laboratory Accreditation


F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 9 of 73

Table F-1. Summary of Quality Control Check Definitions, Purpose, and Evaluation (continued)

QC Check	Definition	Purpose	Evaluation
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the matrix spike is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS, matrix spike), the bias in the analytical system can be determined.
Tuning (mass spectrometer methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer.	Proper tuning of the mass spectrometer must be verified prior to sample analysis.

	<i>The American Association for Laboratory Accreditation</i>	
	F223 – A2LA Current DoD ELAP QSM – Appendix F Compliance Report	Document Issued: May 13, 2009
	Page 10 of 73	

As always, project-specific requirements identified by the client supersede any requirements listed in the following tables. The requirements are meant to be the default, to be used when project-specific direction based on DQOs is not included.

Tables F-2 through F-10 are organized in most cases by instrument type. The applicable methods are specified in the table title. When there are exceptions (i.e., the QC check does not apply to all methods or instrument types in the table), they are noted in the first column of the table (“QC Check”). Each table contains the following fields (or columns):

QC Check: The name of the QC measure that is required. If the check is only applicable to certain methods from the table, they will be noted in parentheses in this field.

Minimum Frequency: Describes how often the QC check must be performed and, if relevant, at what point in the process (for example, prior to sample analysis). Some QC checks are only performed when another QC check fails. This will be noted in the minimum frequency field.

Acceptance Criteria: The standard that the QC check must satisfy in order to proceed without performing corrective action. In some cases there are multiple options, all equivalently acceptable by DoD, for acceptance of a single QC check. These options will be listed and the appropriate option should be applied. There may be references to acceptance criteria published by DoD. The LCS control limits for certain methods can be found in Appendix G.


Corrective Action: If a QC check does not meet the acceptance criteria specified in the preceding field, the corrective action field identifies what steps must be taken to ensure that the results will be valid. Requirements usually include finding the cause of failure of the acceptance criteria and rerunning the QC check. The corrective action field will also specify which other QC checks must be rerun to ensure valid data.

Flagging Criteria: Where flagging is appropriate, the qualifier flag is listed in this field along with the criteria for using the flag. Flagging should only be used as a last resort. Data should only be flagged once corrective action has been performed. In many cases the field states “Flagging criteria is not appropriate.” This means that corrective action must continue until the problem is solved and the QC check satisfies its acceptance criteria. Samples will not be accepted without successful completion of this QC check. Flagging is only appropriate in cases where the samples cannot be reanalyzed. This field will also specify when additional information should be detailed in the case narrative.

Comments: This field contains further clarification of any of the previous five fields.

The following tables detail DoD-specific QC requirements for SW-846 methods, organized by instrument type:

- Table F-2: Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A)
- Table F-3: Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B)
- Table F-4: Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)
- Table F-5: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (Method 8280)
- Table F-6: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (Method 8290)

	<i>The American Association for Laboratory Accreditation</i>	
	F223 – A2LA Current DoD ELAP QSM – Appendix F Compliance Report	Document Issued: May 13, 2009
	Page 11 of 73	

- Table F-7: Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 series)
- Table F-8: Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)
- Table F-9: Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)
- Table F-10: Cyanide Analysis (Methods 9010, 9012, and 9014)
- Table F-11: Common Anions Analysis (Method 9056)
- Table F-12: Perchlorate Analysis (Methods 6850 and 6860)



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 12 of 73

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 15\%$ for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq 15\%$ for both DDT and Endrin.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 13 of 73

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$; Option 2: linear least squares regression: $r \geq 0.995$; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	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.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL; <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 14 of 73

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL; <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. 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	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. 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) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 15 of 73

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. 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.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. 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 difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed 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.	Alternative surrogates are recommended when there is obvious chromatographic interference.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 16 of 73

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 17 of 73

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Flagging criteria are not appropriate.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
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 criteria are 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 criteria are not appropriate.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 18 of 73

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 criteria are 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. Grinding blanks can be analyzed individually or composited. No target analytes detected greater than 1/2 Reporting Limit (RL).	All blank results must be reported and the affected samples must be flagged accordingly if blank criteria is not met.	If the composite grinding blank exceeds the acceptance criteria, apply B-flag to all samples associated with the grinding composite. If any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the sample following that blank.	
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 criteria are not appropriate.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 19 of 73

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil sample triplicate	At the subsampling step, one sample per batch. Cannot be performed on any type of blank sample.	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 RL must not exceed 20%.	Corrective action must be taken if this criterion is not met (e.g., the grinding process should be investigated to ensure that the samples are being reduced to a sufficiently small particle size).	Apply J-flag if corrective action does not solve problem and no sample available.	
Aqueous sample preparation	Each sample.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required. The salting-out procedure is not permitted.	NA.	Flagging criteria are not appropriate.	
Initial calibration (ICAL)	Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. Once calibration curve or line is generated, the lowest calibration standard must be re-analyzed.	The apparent signal-to-noise ratio at the RL must be at least 5:1. If linear regression is used, $r \geq 0.995$. If using Internal Standardization, $RSD \leq 15\%$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	No samples can be run without a valid ICAL. Analysis by HPLC UV, LC/MS, or LC/MS/MS is allowed.
Second source calibration verification (ICV)	Immediately following ICAL.	All analyte(s) and surrogates within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to 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 expected value from the ICAL.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 20 of 73

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per 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. In-house laboratory control limits for the LCS must demonstrate the laboratory's ability to meet the project's MQOs.	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 (see full explanation in Appendix G).	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.	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.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery must meet LCS limits.	Examine the project-specific DQOs. 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.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery must meet LCS limits and relative percent difference (RPD) < 20%.	Examine the project-specific DQOs. 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 difference.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 21 of 73

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation analysis	When target analytes are detected on the primary column using the UV Detector (HPLC) at concentrations exceeding the Limit of Detection (LOD).	Calibration and QC criteria are the same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	Report from both columns.	If there is a > 40% RPD between the two column results, data must be J-flagged accordingly.	Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis. 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. Any HPLC column used for confirmation analysis must be able to resolve and quantify all project analytes. Detection by HPLC UV, LC/MS or LC/MS/MS. Calibration and calibration verification acceptance criteria is the same as for the primary analysis.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 22 of 73

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The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 23 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation \leq 20%.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 24 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<p><u>1. Average response factor (RF) for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>SVOCs ≥ 0.050.</p> <p><u>2. RSD for RFs for CCCs:</u> VOCs and SVOCs $\leq 30\%$ and one option below:</p> <p><u>Option 1:</u> RSD for each analyte $\leq 15\%$;</p> <p><u>Option 2:</u> linear least squares regression $r \geq 0.995$;</p> <p><u>Option 3:</u> non-linear regression—coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 25 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	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.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	<p>Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).</p> <p>With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.</p>



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 26 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	<p><u>1. Average RF for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>SVOCs ≥ 0.050.</p> <p><u>2. %Difference/Drift for all target compounds and surrogates:</u> VOCs and SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).</p>	<p>DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken.</p> <p>Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.</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 last acceptable CCV.	<p>Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 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. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $> RL$ (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	<p>Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 27 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. 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.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: $RPD \leq 30\%$ (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. 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 difference.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 28 of 73

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed 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.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 29 of 73

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The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 30 of 73

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
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to analyzing calibration standards.	Verify MS calibration per the method.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted 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 criteria are not appropriate.	
GC column performance check (for SP-2331 column or equivalent)	Prior to ICAL or calibration verification standards and for each 12-hour period of sample analysis.	<u>Peak separation between 2,3,7,8-TCDD and other TCDD isomers:</u> Resolved with a valley of $\leq 25\%$, per method; <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 $\leq 50\%$, per method.	Correct problem then repeat column performance check.	Flagging criteria are not appropriate.	Needed only if using a column other than DB-5 or equivalent.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 31 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
GC Column performance check (for DB-5 column or equivalent)	Included with the ICAL standard (CC3) or the calibration verification standard.	<u>Peak separation of standard CC3</u> : Peak between the 2,3,7,8-TCDD and 1,2,3,4-TCDD must be resolved with a valley of $\leq 25\%$, per method; <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 $\leq 50\%$, per method.	Correct problem then repeat column performance check.	Flagging criteria are not appropriate.	
Initial calibration (ICAL) for all analytes identified in method	ICAL prior to sample analysis and as needed by the failure of calibration verification standard.	Ion abundance ratios in accordance with the method; <u>and</u> RSD of the RFs $\leq 15\%$ for labeled IS and unlabeled PCDD/PCDF per method.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Calibration verification	At the beginning of each 12-hour period of sample analysis, after successful GC and MS resolution checks.	Ion abundance specified in the method must be met for all PCDD/PCDF peaks, including labeled internal and recovery standards; <u>and</u> Sensitivity criteria of an S/N ratio > 2.5 for unlabeled PCDD/PCDF ions and > 10 for labeled internal and recovery standards per method; <u>and</u> RF for each analyte and IS within $\pm 20\%$ (% difference) of RF established in ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples analyzed since the last successful calibration verification.	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 successful calibration verification.	Problem must be corrected. Results may not be reported without a valid calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 32 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sensitivity check	At the end of 12-hour sample analysis period or at the end of analysis (whichever comes first) (Injection must be done within the 12-hour period.).	See criteria for retention time check, ion abundances, and S/N ratios noted above for calibration and response verification standard per method.	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 criteria are not appropriate.	
Method blank	One per preparatory batch.	Use project-specific criteria, if available. Otherwise, no analytes detected \geq LOD for the analyte or \geq 5% of the associated regulatory limit for the analyte or \geq 5% of the sample result for the analyte, whichever is greater, per method.	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. 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.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than \pm 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. 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.	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.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 33 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. 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.	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 any dilutions) must be within 25-150%, per method.	Correct problem, then reprep and reanalyze the sample(s) with failed IS.	Apply Q-flag to results of all affected samples.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 34 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PCDD/PCDF identification	Identify all positive sample detections per method.	Verify that absolute RT at maximum height is within -1 to +3 secs. 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; <u>and</u> Absolute RTs of the recovery standards must be within ±10 sec. of those in the calibration verification standard; <u>and</u> 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; <u>and</u> 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 <u>and</u> Ion abundance in Table 9 of the method must be met for all analytes, internal, and recovery standards.	Correct problem, then reprep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards. If PCDPE is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to the method.	Flagging criteria are 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).



The American Association for Laboratory Accreditation


F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 35 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample specific estimated detection limit / estimated quantitation limit (EDL / EQL)	Calculated for each 2,3,7,8-substituted isomer that was not identified.	Per method.	NA.	Flagging criteria are not appropriate.	
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 PCDPE was detected.	Response for both quantitation ions must be ≥ 2.5 times S/N ratio of background; all other criteria from sample PCDD/PCDF identification above; PCDE peak at the same RT (± 2 sec.) must have S/N < 2.5 .	NA.	Flag as appropriate.	
Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration	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 criteria are not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.
Results reported between DL and LOQ	Positive detections calculated per method.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

	<i>The American Association for Laboratory Accreditation</i>	
	F223 – A2LA Current DoD ELAP QSM – Appendix F Compliance Report	Document Issued: May 13, 2009
		Page 36 of 73

Page intentionally left blank.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 37 of 73

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
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	At the beginning and the end of each 12-hour period of analysis.	Static resolving power \geq 10,000 (10% valley) for identified masses per method, <u>and</u> lock-mass ion between lowest and highest masses for each descriptor and level of reference compound \leq 10% full-scale deflection, per method.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 38 of 73

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
GC column performance check	Prior to ICAL or calibration verification. Use GC performance check solution per method.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of $\leq 25\%$, per method; <u>and</u> Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; <u>and</u> 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 criteria are not appropriate.	
Initial calibration (ICAL) for all analytes identified in method	ICAL prior to sample analysis, as needed by the failure of calibration verification standard, 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 criteria in Table 8 of the method; <u>and</u> S/N ratio ≥ 10 for all target analyte ions; <u>and</u> RSD $\leq 20\%$ for the response factors (RF) for all 17 unlabeled standards <u>and</u> RSD $\leq 20\%$ for the RFs for the 9 labeled IS.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through origin.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 39 of 73

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
Calibration verification	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios in accordance with criteria in Table 8 of the method; <u>and</u> 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.	Correct problem, repeat calibration verification standard. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV. <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 CCV RFs 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 necessary.	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 successful calibration verification.	Problem must be corrected. Results may not be reported without a valid calibration verification. Flagging is only appropriate in cases where the samples cannot be reanalyzed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 40 of 73

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	One per preparatory batch, run after calibration standards and before samples.	Use project-specific criteria, if available. Otherwise, no analytes detected \geq LOD for the analyte or \geq 5% of the associated regulatory limit for the analyte or \geq 5% of the sample result for the analyte, whichever is greater, per method.	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS (or fortified field blank)	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than \pm 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.
Sample duplicate	One per preparatory batch per matrix (see Box D-7).	$RPD \leq 25\%$ (between sample and sample duplicate), per method.	Examine the project-specific DQOs. 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.	
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. 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.	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.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 41 of 73

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)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. RPD ≤ 20% (between MS and MSD) per method.	Examine the project-specific DQOs. 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 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%, per method.	Correct problem, then reprep and reanalyze the samples with failed IS.	Apply Q-flag to results of all affected samples.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 42 of 73

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</u> For PCDF: No signal present having a S/N ratio ≥ 2.5 for the</p>	Correct problem, then reprep and reanalyze the samples with failed criteria for any of the internal, recovery, or cleanup standards. If PCDFE 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 criteria are 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).
L:\Forms\F223 – A2LA Current	DoD ELAP QSM	<p>corresponding other (PCDFE) detected at the same retention time (± 2 sec).</p>	Appendix F Compliance Report		



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 43 of 73

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 specific estimated detection limit / estimated quantitation limit (EDL / EQL)	Calculated for each 2,3,7,8-substituted isomer that is not identified.	Per method.	NA.	Flagging criteria are not appropriate.	
Sample estimated maximum possible concentration (EMPC)	Every sample that indicates a detection \geq 2.5 times S/N response.	Identification criteria per method must be met, and response for both quantitation ions must be \geq 2.5 times S/N ratio for background.	NA.	Flag as appropriate.	
Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration	All positive detections, as required.	Per method.	NA.	Flagging criteria are not appropriate.	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.
Results reported between DL and LOQ	Positive detections calculated per method.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 44 of 73

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The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 45 of 73

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be \leq LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within \pm 10% of true value.	NA.	NA.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 46 of 73

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within $\pm 10\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	<u>ICP</u> : within $\pm 10\%$ of true value; <u>GFAA</u> : within $\pm 20\%$ of true value; <u>CVAA</u> : within $\pm 20\%$ of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. 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 (ICP only)	Daily, after one-point ICAL.	Within $\pm 20\%$ of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 47 of 73

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	<u>ICS-A:</u> Absolute value of concentration for all non-spiked 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 ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 48 of 73

Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA) (Methods 6010 and 7000 Series) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. 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 difference.
Dilution test (ICP and GFAA only)	One per preparatory batch.	Five-fold dilution must agree within ± 10% of the original measurement.	<u>ICP</u> : Perform post-digestion spike (PDS) addition; <u>GFAA</u> : Perform recovery test.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x LOD.	Recovery within 85-115%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 49 of 73

**Table F-7. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry and Atomic Absorption Spectrophotometry (AA)
(Methods 6010 and 7000 Series) (continued)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 50 of 73

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
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be \leq LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Tuning	Prior to ICAL.	Mass calibration \leq 0.1 amu from the true value; Resolution $<$ 0.9 amu full width at 10% peak height; For stability, RSD \leq 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) for all analytes (minimum one high standard and a calibration blank)	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 51 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within $\pm 10\%$ of true value.	Verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All analytes within $\pm 10\%$ of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. 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	Daily, after one-point ICAL.	Within $\pm 20\%$ of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Linear dynamic range or high-level check standard	Every 6 months.	Within $\pm 10\%$ of true value.	NA.	NA.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 52 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > 1/2 RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run and every 12 hours.	<u>ICS-A</u> : Absolute value of concentration for all non-spiked 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 ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 53 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests (dilution test and post-digestion spike addition) are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	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.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD < 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. 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 difference.
Dilution test	One per preparatory batch.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples < 50 x LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Internal standards (IS)	Every sample.	IS intensity within 30-120% of intensity of the IS in the ICAL.	Reanalyze sample at 5-fold dilution with addition of appropriate amounts of internal standards.	Flagging criteria are not appropriate.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 54 of 73

Table F-9. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for the analyte that did not meet criteria (see Section C.1 f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Reference blank (reagent water)	Before beginning standards or sample analysis.	NA.	NA.	NA.	Used for blank subtraction of standards, field and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with Method 7196A (Section 7.1)).
Initial calibration (ICAL) (minimum three standards and a calibration blank)	Daily ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV) (also known as independently prepared check standard)	Before beginning a sample run.	Value of second source within $\pm 10\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 55 of 73

Table F-9. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 15 field samples and at the end of the analysis sequence.	Value of CCV within \pm 10% of true value.	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification.	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.	Problem must be corrected. No samples may be run until calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method blank	One per preparatory batch.	No analyte detected $>$ 1/2 the reporting limit and $>$ 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per preparatory batch.	QC acceptance criteria specified by DoD; see Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G).	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.	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.
Sample matrix verification (also known as matrix spike)	Once for every sample matrix analyzed.	Spike recovery within 85–115%.	If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use method of standard additions.	Flagging criteria are not appropriate.	Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in Method 7196A (Sections 7.4 and 7.5).



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 56 of 73

Table F-9. Inorganic Analysis by Colorimetric Hexavalent Chromium (Method 7196) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	<u>Aqueous matrix</u> : One per every 10 project samples per matrix. <u>Solid matrix</u> : One per preparatory batch per matrix.	<u>Aqueous matrix</u> : RPD ≤ 20% (between MS and MSD or sample and sample duplicate). <u>Solid matrix</u> : RPD ≤ 30%.	Examine project-specific DQOs. Contact the client as to additional measures to be taken.	Flagging criteria are not appropriate.	Refer to sample matrix verification sample for MS data evaluation.
Pre-digestion matrix spikes (solid matrix samples only, Method 3060)	One soluble and insoluble pre-digestion MS analyzed per preparatory batch prior to analysis.	MS recoveries within 75–125%.	Correct problem and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results.	If corrective action fails, apply J-flag to the analyte in all samples in the associated preparatory batch.	
Post-digestion matrix spike	One per preparatory batch.	Recovery between 85–115%.	Correct problem and rehomogenize, redigest, and reanalyze samples. Persistent interference indicates the need to use an alternative method or analytical conditions, or to use method of standard additions.	NA.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 57 of 73

Page intentionally left blank.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 58 of 73

Table F-10. Cyanide Analysis (Methods 9010, 9012, and 9014)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Initial calibration (ICAL) (six standards and a calibration blank)	Daily ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has passed. All calibration standards must be distilled if samples are expected to contain sulfides.
Distilled standards (one high and one low)	Once per multipoint calibration.	Within $\pm 15\%$ of true value.	Correct problem, then repeat distilled standards.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until distilled standards have passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Within $\pm 15\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 59 of 73

Table F-10. Cyanide Analysis (Methods 9010, 9012, and 9014) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > 1/2 RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	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 (see full explanation in Appendix G).	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.	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.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, the method of standard additions shall be used for the analysis.	For the specific analyte in the parent sample, apply J-flag if acceptance criteria are not met.	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.
Matrix spike duplicate (MSD) or sample duplicate (replicate)	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 60 of 73

Table F-10. Cyanide Analysis (Methods 9010, 9012, and 9014) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 61 of 73

Table F-11. Common Anions Analysis (Method 9056)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Retention time (RT) window width calculated for each analyte	After 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.	
Initial calibration (ICAL) for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis.	$r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Initial calibration verification (ICV) (second source)	Once after each ICAL, prior to beginning a sample run.	All analytes within $\pm 10\%$ of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 62 of 73

Retention time window position establishment for each analyte	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.	
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The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 63 of 73

Table F-11. Common Anions Analysis (Method 9056) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within $\pm 10\%$ of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. 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	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per preparatory batch.	Laboratory in-house limits not to exceed $\pm 20\%$. Control limits may be not greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3.	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 (see full explanation in Appendix G).	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.	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.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed $\pm 20\%$).	Examine the project-specific DQOs. 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.	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.



The American Association for Laboratory Accreditation


F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 64 of 73

Table F-11. Common Anions Analysis (Method 9056) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed $\pm 20\%$). RPD $\leq 15\%$ (between MS and MSD).	Examine the project-specific DQOs. 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 difference.
Sample duplicate (replicate)	One per every 10 samples.	%D $\leq 10\%$ (between sample and sample duplicate).	Correct problem and reanalyze sample and duplicate.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	The data shall be evaluated to determine the source of difference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

	<i>The American Association for Laboratory Accreditation</i>	
	F223 – A2LA Current DoD ELAP QSM – Appendix F Compliance Report	Document Issued: May 13, 2009
		Page 65 of 73

Page intentionally left blank.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 66 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for the analyte that did not meet criteria (see Section C.1.f).	Flagging criteria are not appropriate.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Initial calibration (ICAL)	Minimum of 5 calibration standards to establish linearity at method set-up and after major maintenance.	$r \geq 0.995$ or $RSD \leq 20\%$. The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be \leq LOD.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. The calibration is linear and shall not be forced through the origin.
Initial calibration verification (ICV)	Once after each ICAL, analysis of a second source standard at the midpoint of the calibration.	Within $\pm 15\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. 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 perchlorate.	Within $\pm 15\%$ of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	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.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 67 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Limit of detection verification (LODV) (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.	Within $\pm 30\%$ of true value.	Correct problem and rerun LODV and all samples analyzed since last successful LODV. If a sample with perchlorate concentration at or between the LOD and RL is bracketed by a failing LODV, it must be reanalyzed. A sample with concentration above the RL 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 LODV.	Problem must be corrected. Results may not be reported without a valid LODV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Perchlorate spike concentration is approximately 2 times the limit of detection.
Isotope ratio $^{35}\text{Cl}/^{37}\text{Cl}$	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. Theoretical ratio ~ 3.06 . 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 should be reextracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate (i.e., a post spike sample, dilution to reduce any interference, etc.).	Apply J-flag if acceptance criteria are not met.	Decision to report data failing ratio check should be thoroughly documented in 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 reprepiped using additional pretreatment steps.	Apply Q-flag and discuss in the case narrative.	If peak is not within retention time window, presence is not confirmed. Use for quantitation and to ensure identification. Failing internal standard should be thoroughly documented in the case narrative.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 68 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.	Within $\pm 30\%$ of true value.	Correct problem and then reanalyze all samples in that batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to reextract 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 RL and interfering anions at the concentration determined by the interference threshold study. Monitor recovery of perchlorate and retention time. No samples may be reported that are associated with a failing ICS.
Laboratory reagent blank	Prior to calibration, after samples with overrange concentration of perchlorate, and at the end of the analytical sequence.	No perchlorate detected $> \frac{1}{2}$ 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.
Tuning	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. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging criteria are not appropriate.	Problem must be corrected. Sample analysis shall not proceed without acceptable tuning.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 69 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 without greatly exceeding the range. 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.
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 85-115% window.	NA.	Flagging criteria are not appropriate.	This study and site history will determine the concentration at which the ICS suppressors should be set.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 70 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank (MB)	One per preparatory batch.	No perchlorate detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, then see criteria in Box D-1. 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.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Method blank must undergo the same preparation and pretreatment steps as the samples in the batch.
Laboratory control sample (LCS)	One per preparatory batch. LCS must be spiked at the RL.	Recovery within method requirements, laboratory-generated limits, or 80-120% (whichever is more stringent) to verify calibration and to check method performance.	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 (see full explanation in Appendix G).	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.	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.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7). The MS must be spiked at the RL.	Recovery within 80-120% or within laboratory generated limits, whichever is more stringent.	Examine the project-specific DQOs. 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.	For matrix evaluation only. 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. MS must undergo the same preparation and pretreatment steps as the samples in the batch.



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 71 of 73

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

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or laboratory duplicate (LD)	One per preparatory batch per matrix (see Box D-7). The MSD must be spiked at the RL.	MSD: Recovery within 80-120% or within laboratory generated limits, whichever is more stringent. MSD or laboratory duplicate: RPD < 15%.	Examine the project-specific DQOs. 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 difference.
Results reported between DL and LOQ	Positive detections calculated per method.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	



List of Acronyms for Appendix F

C

CC3	The third of five solutions for instrument calibration used in Method 8280
CCC	Calibration check compounds
CCV	Continuing calibration verification
CFR	Code of Federal Regulations
COD	Coefficient of determination
CV	Calibration verification
CV-IS	Calibration verification of internal standards

D

D	Difference or drift
DDT	2,2-bis(p-chlorophenyl)-1,1,1-trichloroethane/dichlorodiphenyl-trichloroethane/p,p'-DDT
DoD	Department of Defense
DQO	Data quality objective
DRO	Diesel range organics

E

EDL	Estimated detection limit
EQL	Estimated quantitation limit
EMPC	Estimated maximum possible concentration
EICP	Extracted ion current profile

G

GC	Gas chromatography
GC/MS	Gas chromatography/mass spectrometry
GFAA	Graphite furnace atomic absorption spectrophotometry
GRO	Gasoline range organics

H

HPLC	High performance liquid chromatography
HxCDD	Hexachlorodibenzo-p-dioxin (solution used for calibration verification)

I

ICAL	Initial calibration
ICP	Inductively coupled plasma atomic emission spectrometry
ICP/MS	Inductively coupled plasma/mass spectrometry
ICS	Interference check solution
ICV	Initial calibration verification
IS	Internal standard
IDL	Instrument detection limit

L



The American Association for Laboratory Accreditation

F223 – A2LA Current DoD ELAP QSM – Appendix F
Compliance Report

Document Issued:
May 13, 2009

Page 73 of 73

LCS	Laboratory control sample
LOD	Limit of detection
LOQ	Limit of quantitation
M	
MS	Mass spectrometry
MS	Matrix spike
MSA	Method of standard additions
MSD	Matrix spike duplicate
P	
PCB	Polychlorinated biphenyl
PCDD	Polychlorinated dibenzodioxin
PCDF	Polychlorinated dibenzofuran
PDS	Post-digestion spike
PE	Performance evaluation
PT	Proficiency testing
Q	
QC	Quality control
QSM	DoD Quality Systems Manual for Environmental Laboratories
R	
RF	Response factor
RL	Reporting limit
RPD	Relative percent difference
RRO	Residual range organics
RRT	Relative retention time
RSD	Relative standard deviation
RT	Retention time
S	
SICP	Selected ion current profile
S/N	Signal to noise ratio
SPCC	System performance check compound
SVOC	Semivolatile organic compound
T	
TCDD	Tetrachlorodibenzo-p-dioxin
TCDF	Tetrachlorodibenzofuran
V	
VOC	Volatile organic compound