

## APPENDIX K

---

### Analytical Quality Assurance/Quality Control Criteria and Instrument Calibration Tables



# Analytical Quality Assurance/Quality Control Criteria and Instrument Calibration Tables

---

This appendix presents the quality assurance/quality control (QA/QC) criteria and instrument calibration tables for each matrix and method combination to be collected. All analytes reported shall be present in the initial and continuing calibrations, laboratory control sample (LCS), and matrix spike/matrix spike duplicate (MS/MSD).

The calibrations and QA/QC shall meet the acceptance criteria specified in the tables provided in this appendix. For calibrations, the following must be met:

- All results reported shall be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and only give an estimate of the true concentration.
- Results shall be within the working range determined either by the daily initial calibration or by linear-range studies.
- Samples shall be diluted, if necessary, to bring analyte responses within the calibration range. Data that exceed the calibration range must be reported by the laboratory with the dilution results.
- Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in the calibration and quantitation of sample results.
- Calibration standards shall be traceable to standard materials.

Instrument calibration shall be achieved by beginning with the simplest approach first (the linear model through the origin), and then progressing through other options (nonlinear) until the acceptance criteria are met. When an analyte has more than one acceptable calibration model, results shall be reported from the simplest calibration model.

The initial calibration (ICAL) must be verified by a second-source standard. Multipoint calibrations shall contain the minimum number of calibration points specified in the applicable tables, with all points used for the calibration being contiguous. If more than the minimum number of standards are analyzed for the ICAL, all of the standards analyzed shall be included in the ICAL. The only exception to this rule is that a standard at either end of the calibration curve can be dropped from the calibration, if the requirement for the minimum number of standards is met and the low point of the calibration curve is at or below the LOQ for each analyte.

Analyte concentrations are determined with either calibration curves or response factors (RFs). Nonlinear calibration should be considered only when a linear approach cannot be

applied. It is not acceptable to use an alternative calibration procedure when a compound fails to perform in the usual manner. Nonlinear calibration may be necessary to achieve low detection limits or to address specific instrument techniques. However, it is not the intent of the U.S. Environmental Protection Agency (EPA) methods to allow nonlinear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance. When this type of nonlinear calibration occurs, it is indicative of instrument issues or operator error. When multipoint calibration is specified, the concentrations of the calibration standards shall bracket those expected in the samples.

For gas chromatography (GC) and GC/mass spectrometry (MS) methods, when using RFs to determine analyte concentrations, the average RF from the initial calibration shall be used. The continuing calibration shall not be used to update the RFs from the ICAL.

The continuing calibration verification (CCV) cannot be used as the LCS. A CCV is to be performed daily before sample analysis (unless an initial calibration and second-source-standard verification are performed immediately before sample analysis) and as required by the applicable method. In addition, the Department of Defense (DoD) QSM Version 4.1 (DoD, April 2009) requires that the concentration used for the CCV sample shall be between the low calibration standard and the midpoint of the calibration range. Finally, the lowest standard used must be the lowest concentration for which quantitative data are to be reported.

With the exception of the Alaska-specific, total petroleum hydrocarbon (TPH)-speciation and Soil/Sediment vapor methods, the following tables are derived from the DoD QSM Version 4.1 (DoD, April 2009):

- Table K1: LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Methods AK101 and AK102/ AK103
- Table K2: LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Methods NWEPH and NWVPH
- Table K3: LCS and MS/MSD Control Limits for Water Matrix, Method SW8021B
- Table K4: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8021B
- Table K5: LCS and MS/MSD Control Limits for Water Matrix, Method SW8081A
- Table K6: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8081A
- Table K7: LCS and MS/MSD Control Limits for Water Matrix, Method SW8082
- Table K8: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8082
- Table K9: LCS and MS/MSD Control Limits for Water Matrix, Methods SW8260B and SW8011
- Table K10: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8260B

- Table K11: LCS and MS/MSD Control Limits for Water Matrix, Methods SW8270C and SW8270C-SIM
- Table K12: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Methods SW8270C and SW8270C-SIM
- Table K13: LCS and MS/MSD Control Limits for Water Matrix, Methods SW6010B, SW6020 and SW7470A
- Table K14: LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Methods SW6010B, SW6020 and SW7471A
- Table K15: LCS/LCSD Control Limits for Air Matrix, Method TO-15
- Table K16: LCS/LCSD Control Limits for Air Matrix, Method TO-3
- Table K17: LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Method SW8290
- Table K18: Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH
- Table K19: Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082
- Table K20: Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)
- Table K21: Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A and SW7471A
- Table K22: Summary of Calibration and Quality Control Procedures for Method SW6020
- Table K23: Summary of Calibration and Quality Control Procedures for Method TO-15
- Table K24: Summary of Calibration and Quality Control Procedures for Method TO-3
- Table K25: Summary of Calibration and Quality Control Procedures for Method SW8290

Also included in this appendix are tables of the project laboratory variances to the DoD QSM Version 4.1 (DoD, April 2009) and/or the AFCEE QAPP Version 4.0.02 (AFCEE, May 2006). CH2M HILL's responses to the requested variances are included in the tables.

- Table K26: EMAX Laboratories Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009).
- Table K27: Applied Sciences Laboratory (field lab) Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009).
- Table K28: AirToxics Ltd. Requested Variances to the AFCEE QAPP Version 4.0.02 (AFCEE, May 2006).
- Table K29: TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009).

TABLE K1  
 LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Methods AK101 and AK102/AK103

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Gasoline Range Organics	60	120	≤20	NA	NA
Diesel Range Organics	75	125	≤20	NA	NA
Residual Range Organics	60	120	≤20	NA	NA
<b>Surrogate (choose 1)</b>					
Trifluorotoluene (GRO)	50	150	(for field samples)		
	60	120	(for laboratory control)		
Bromofluorobenzene (GRO)	50	150	(for field samples)		
	60	120	(for laboratory control)		
o-Terphenyl (DRO)	50	150	(for field samples)		
	60	120	(for laboratory control)		
n-Triacontane (RRO)	50	150	(for field samples)		
	60	120	(for laboratory control)		

Note:

Control limits are ADEC method specific.

TABLE K2  
 LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Method NWEPH/VPH

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
<b>VPH</b>					
C8-C10 Aromatics	70	130	≤20	NA	NA
C10-C12 Aromatics	70	130	≤20	NA	NA
C12-C13 Aromatics	70	130	≤20	NA	NA
C5-C6 Aliphatics	70	130	≤20	NA	NA
C6-C8 Aliphatics	70	130	≤20	NA	NA
C8-C10 Aliphatics	70	130	≤20	NA	NA
C8-C10 Aliphatics	70	130	≤20	NA	NA
<b>EPH</b>					
C8-C10 Aromatics	70	130	≤20	NA	NA
C10-C12 Aromatics	70	130	≤20	NA	NA
C12-C16 Aromatics	70	130	≤20	NA	NA
C16-C21 Aromatics	70	130	≤20	NA	NA
C21-C34 Aromatics	70	130	≤20	NA	NA
C8-C10 Aliphatics	70	130	≤20	NA	NA
C10-C12 Aliphatics	70	130	≤20	NA	NA
C12-C16 Aliphatics	70	130	≤20	NA	NA
C16-C21 Aliphatics	70	130	≤20	NA	NA
C21-C34 Aliphatics	70	130	≤20	NA	NA
<b>Surrogate</b>					
2,5-Dibromotoluene (VPH)	50	150	FID and PID		
o-Terphenyl (EPH)	50	150	FID and PID		

TABLE K2  
 LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Method NWEPH/VPH

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Chloro-octodecane	50	150	FID and PID		

Note:  
 Control limits are method specific.

TABLE K3  
 LCS and MS/MSD Control Limits for Water Matrix, Method SW8021B

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Benzene	75	125	≤20	NA	NA
Ethylbenzene	71	129	≤20	NA	NA
m,p-Xylenes	65	135	≤20	NA	NA
o-Xylene	65	135	≤20	NA	NA
Toluene	70	125	≤20	NA	NA
<b>Surrogates</b>					
Bromochlorobenzene	37	137			
1,4-Dichlorobutane	35	135			

Note:  
 Control limits are taken from AFCEE QAPP Version 4.0.02.

TABLE K4  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8021B

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Benzene	65	125	≤30	NA	NA
Ethylbenzene	61	129	≤30	NA	NA
m,p-Xylenes	55	145	≤30	NA	NA
o-Xylene	55	145	≤30	NA	NA
Toluene	60	125	≤30	NA	NA
<b>Surrogates</b>					
Bromochlorobenzene	37	137			
1,4-Dichlorobutane	35	135			

Note:  
 Control limits are taken from AFCEE QAPP Version 4.0.02

TABLE K5  
 LCS and MS/MSD Control Limits for Water Matrix, Method SW8081A

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
4,4-DDD	25	150	≤30	10	170
4,4-DDE	35	140	≤30	15	160
4,4-DDT	45	140	≤30	30	155
Aldrin	25	140	≤30	10	155
alpha-BHC	60	130	≤30	50	140
alpha-Chlordane	65	125	≤30	55	135
beta-BHC	65	125	≤30	55	135
delta-BHC	45	135	≤30	30	150
Dieldrin	60	130	≤30	50	140
Endosulfan I	50	110	≤30	40	120
Endosulfan II	30	130	≤30	10	150
Endosulfan Sulfate	55	135	≤30	40	150
Endrin	55	135	≤30	45	145
Endrin Aldehyde	55	135	≤30	40	150
Endrin Ketone	75	125	≤30	70	135
gamma-BHC	25	135	≤30	10	155
gamma-Chlordane	60	125	≤30	50	135
Heptachlor	40	130	≤30	30	145
Heptachlor Epoxide	60	130	≤30	50	140
Methoxychlor	55	150	≤30	40	165
Toxaphene	41	126	≤30	25	140
<b>Surrogates</b>					
DCBP	30	135			
TCMX	25	140			

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K6  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8081A

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
4,4-DDD	30	135	≤30	10	155
4,4-DDE	70	125	≤30	60	135
4,4-DDT	45	140	≤30	30	155
Aldrin	45	140	≤30	30	155
alpha-BHC	60	125	≤30	50	135
alpha-Chlordane	65	120	≤30	55	130
beta-BHC	60	125	≤30	50	135
delta-BHC	55	130	≤30	45	145
Dieldrin	65	125	≤30	55	135
Endosulfan I	15	135	≤30	10	155
Endosulfan II	35	140	≤30	20	160
Endosulfan Sulfate	60	135	≤30	50	145
Endrin	60	135	≤30	50	145
Endrin Aldehyde	35	145	≤30	20	165
Endrin Ketone	65	135	≤30	55	145
gamma-BHC	60	125	≤30	50	135
gamma-Chlordane	65	125	≤30	55	135
Heptachlor	50	140	≤30	35	155
Heptachlor Epoxide	65	130	≤30	55	140
Methoxychlor	55	145	≤30	45	155
Toxaphene	31	136	≤30	15	155
<b>Surrogates</b>					
DCBP	55	130			
TCMX	70	125			

Note:

Control limits are taken from DoD QSM Version 4.1.

**TABLE K7**  
 LCS and MS/MSD Control Limits for Water Matrix, Method SW8082

<b>Analyte</b>	<b>LCL</b>	<b>UCL</b>	<b>Precision</b>	<b>Lower ME Limit</b>	<b>Upper ME Limit</b>
Aroclor 1016	25	145	≤30	NA	NA
Aroclor 1260	30	145	≤30	NA	NA
Surrogate					
DCBP	40	135			

Note:

Control limits are taken from DoD QSM Version 4.1.

**TABLE K8**  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8082

<b>Analyte</b>	<b>LCL</b>	<b>UCL</b>	<b>Precision</b>	<b>Lower ME Limit</b>	<b>Upper ME Limit</b>
Aroclor 1016	40	140	≤30	NA	NA
Aroclor 1260	60	130	≤30	NA	NA
Surrogate					
DCBP	60	125			

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K9  
 LCS and MS/MSD Control Limits for Water Matrix, Methods SW8260B and SW8011\*

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	80	130	≤30	75	135
1,1,1-Trichloroethane	65	130	≤30	55	145
1,1,2,2-Tetrachloroethane	65	130	≤30	55	140
1,1,2-Trichloroethane	75	125	≤30	65	135
1,1-Dichloroethane	70	135	≤30	60	145
1,1-Dichloroethene	70	130	≤30	55	140
1,1-Dichloropropene	75	130	≤30	65	140
1,2,3-Trichlorobenzene	55	140	≤30	45	155
1,2,3-Trichloropropane	75	125	≤30	65	130
1,2,4-Trichlorobenzene	65	135	≤30	55	145
1,2,4-Trimethylbenzene	75	130	≤30	65	140
1,2-Dibromo-3-chloropropane	50	130	≤30	35	145
1,2-Dibromoethane (EDB)	80	120	≤30	75	125
1,2-Dichlorobenzene	70	120	≤30	60	130
1,2-Dichloroethane	70	130	≤30	60	140
1,2-Dichloropropane	75	125	≤30	65	135
1,3,5-Trimethylbenzene	75	130	≤30	65	140
1,3-Dichlorobenzene	75	125	≤30	65	130
1,3-Dichloropropane	75	125	≤30	65	135
1,4-Dichlorobenzene	75	125	≤30	65	130
2,2-Dichloropropane	70	135	≤30	60	150
2-Butanol	TBD	TBD	TBD	TBD	TBD
2-Butanone	30	150	≤30	10	170
2-Chlorotoluene	75	125	≤30	65	135
2-Hexanone	55	130	≤30	45	140
4-Chlorotoluene	75	130	≤30	65	135
4-Methyl-2-pentanone	60	135	≤30	45	145
Acetone	40	140	≤30	20	160
Benzene	80	120	≤30	75	130
Bromobenzene	75	125	≤30	70	130
Bromochloromethane	65	130	≤30	55	140
Bromodichloromethane	75	120	≤30	70	130
Bromoform	70	130	≤30	60	140
Bromomethane	30	145	≤30	10	165
Carbon disulfide	35	160	≤30	15	185
Carbon tetrachloride	65	140	≤30	55	150
Chlorobenzene	80	120	≤30	75	130
Chlorodibromomethane	60	135	≤30	45	145
Chloroethane	60	135	≤30	50	145

TABLE K9  
 LCS and MS/MSD Control Limits for Water Matrix, Methods SW8260B and SW8011\*

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Chloroform	65	135	≤30	50	150
Chloromethane	40	125	≤30	25	140
cis-1,2-Dichloroethene	70	125	≤30	60	135
cis-1,3-Dichloropropene	70	130	≤30	60	140
Dibromomethane	75	125	≤30	65	135
Dichlorodifluoromethane	30	155	≤30	10	175
Ethylbenzene	75	125	≤30	65	135
Hexachlorobutadiene	50	140	≤30	35	160
Isopropylbenzene	75	125	≤30	65	135
m,p-Xylenes	75	130	≤30	65	135
Methyl tert-butyl ether (MTBE)	65	125	≤30	55	135
Methylene chloride	55	140	≤30	40	155
Naphthalene	55	140	≤30	40	150
n-Butylbenzene	70	135	≤30	55	150
n-Propylbenzene	70	130	≤30	65	140
o-Xylene	80	120	≤30	75	130
p-Isopropyltoluene	75	130	≤30	65	140
sec-Butylbenzene	70	125	≤30	65	135
Styrene	65	135	≤30	55	145
tert-Butylbenzene	70	130	≤30	60	140
Tetrachloroethene	45	150	≤30	25	165
Toluene	75	120	≤30	70	130
trans-1,2-Dichloroethene	60	140	≤30	45	150
trans-1,3-Dichloropropene	55	140	≤30	40	155
Trichloroethene	70	125	≤30	60	135
Trichlorofluoromethane	60	145	≤30	45	160
Vinyl chloride	50	145	≤30	35	165
<b>Surrogates</b>					
4-Bromofluorobenzene	75	120			
Dibromofluoromethane	85	115			
1,2-Dichloroethane-d4	70	120			
Toluene-d8	85	120			

\*SW8011 for low-level 1,2-Dibromo-3-chloropropane and 1,2-Dibromoethane only

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K10  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8260B

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	75	125	≤30	65	135
1,1,1-Trichloroethane	70	135	≤30	55	145
1,1,2,2-Tetrachloroethane	55	130	≤30	40	145
1,1,2-Trichloroethane	60	125	≤30	50	140
1,1-Dichloroethane	75	125	≤30	65	135
1,1-Dichloroethene	65	135	≤30	55	150
1,1-Dichloropropene	70	135	≤30	60	145
1,2,3-Trichlorobenzene	60	135	≤30	50	145
1,2,3-Trichloropropane	65	130	≤30	50	140
1,2,4-Trichlorobenzene	65	130	≤30	55	140
1,2,4-Trimethylbenzene	65	135	≤30	55	145
1,2-Dibromo-3-chloropropane	40	135	≤30	25	150
1,2-Dibromoethane (EDB)	70	125	≤30	60	135
1,2-Dichlorobenzene	75	120	≤30	65	125
1,2-Dichloroethane	70	135	≤30	60	145
1,2-Dichloropropane	70	120	≤30	65	125
1,3,5-Trimethylbenzene	65	135	≤30	55	145
1,3-Dichlorobenzene	70	125	≤30	65	135
1,3-Dichloropropane	75	125	≤30	70	130
1,4-Dichlorobenzene	70	125	≤30	65	135
2,2-Dichloropropane	65	135	≤30	55	145
2-Butanol	TBD	TBD	TBD	TBD	TBD
2-Butanone	30	160	≤30	10	180
2-Chlorotoluene	70	130	≤30	60	140
2-Hexanone	45	145	≤30	30	160
4-Chlorotoluene	75	125	≤30	65	135
4-Methyl-2-pentanone	45	145	≤30	30	165
Acetone	20	160	≤30	10	180
Benzene	75	125	≤30	65	135
Bromobenzene	65	120	≤30	55	130
Bromochloromethane	70	125	≤30	60	135
Bromodichloromethane	70	130	≤30	60	135
Bromoform	55	135	≤30	45	150
Bromomethane	30	160	≤30	10	180
Carbon disulfide	45	160	≤30	30	180
Carbon tetrachloride	65	135	≤30	55	145
Chlorobenzene	75	125	≤30	65	130
Chlorodibromomethane	65	130	≤30	55	140
Chloroethane	40	155	≤30	20	175
Chloroform	70	125	≤30	65	135

TABLE K10  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Method SW8260B

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Chloromethane	50	130	≤30	40	140
cis-1,2-Dichloroethene	65	125	≤30	55	135
cis-1,3-Dichloropropene	70	125	≤30	65	135
Dibromomethane	75	130	≤30	65	135
Dichlorodifluoromethane	35	135	≤30	15	155
Ethylbenzene	75	125	≤30	65	135
Hexachlorobutadiene	55	140	≤30	40	155
Isopropylbenzene	75	130	≤30	70	140
m,p-Xylenes	80	125	≤30	70	135
Methyl tert-butyl ether (MTBE)*	65	125	≤30	55	135
Methylene chloride	55	140	≤30	40	155
Naphthalene	40	125	≤30	25	140
n-Butylbenzene	65	140	≤30	50	150
n-Propylbenzene	65	135	≤30	50	145
o-Xylene	75	125	≤30	70	135
p-Isopropyltoluene	75	135	≤30	65	140
sec-Butylbenzene	65	130	≤30	50	145
Styrene	75	125	≤30	65	135
tert-Butylbenzene	65	130	≤30	55	145
Tetrachloroethene	65	140	≤30	55	150
Toluene	70	125	≤30	60	135
trans-1,2-Dichloroethene	65	135	≤30	55	145
trans-1,3-Dichloropropene	65	135	≤30	55	140
Trichloroethene	75	125	≤30	70	130
Trichlorofluoromethane	25	185	≤30	10	215
Vinyl chloride	60	125	≤30	45	140
<b>Surrogates</b>					
4-Bromofluorobenzene	85	120			
Toluene-d8	85	115			

\*Criteria are for water matrix

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K11  
 LCS and MS/MSD Control Limits for Water Matrix, Methods SW8270C and SW8270C-SIM

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
<b>Polynuclear Aromatics (SW8270C-SIM)</b>					
1-Methylnaphthalene	45	105	≤30	35	115
2-Methylnaphthalene	45	105	≤30	35	115
Acenaphthene	45	110	≤30	35	120
Acenaphthylene	50	105	≤30	40	115
Anthracene	55	110	≤30	45	120
Benzo(a)anthracene	55	110	≤30	45	120
Benzo(a)pyrene	55	110	≤30	45	120
Benzo(b)fluoranthene	45	120	≤30	35	130
Benzo(k)fluoranthene	45	125	≤30	30	135
Benzo(g,h,i)perylene	40	125	≤30	25	135
Chrysene	55	110	≤30	45	120
Dibenzo(a,h)anthracene	40	125	≤30	30	140
Fluoranthene	55	115	≤30	45	125
Fluorene	50	110	≤30	40	120
Indeno(1,2,3-c,d)pyrene	45	125	≤30	30	140
Naphthalene	40	100	≤30	30	115
Phenanthrene	50	115	≤30	40	130
Pyrene	50	130	≤30	35	140
<b>Phenolic/Acidic (SW8270C)</b>					
2,4,5-Trichlorophenol	50	110	≤30	40	120
2,4,6-Trichlorophenol	50	115	≤30	40	125
2,4-Dichlorophenol	50	105	≤30	40	115
2,4-Dimethylphenol	30	110	≤30	15	125
2,4-Dinitrophenol	15	140	≤30	10	160
2-Chlorophenol	35	105	≤30	25	115
2-Methylphenol	40	110	≤30	25	120
2-Nitrophenol	40	115	≤30	25	125
3-Methylphenol/4-methylphenol	30	110	≤30	20	125
4,6-Dinitro-2-methylphenol	40	130	≤30	25	145
4-Chloro-3-methylphenol	45	110	≤30	35	120
4-Nitrophenol*	0	125	≤30	0	145
Benzoic Acid*	0	125	≤30	0	150
Pentachlorophenol	40	115	≤30	25	130
Phenol*	0	115	≤30	0	135
<b>Basic (SW8270C)</b>					
3,3-Dichlorobenzidine	20	110	≤30	10	125
4-Chloroaniline	15	110	≤30	10	125
<b>Phthalate Esters (SW8270C)</b>					
bis(2-Ethylhexyl)phthalate	40	125	≤30	30	140
Butylbenzyl phthalate	45	115	≤30	35	130
di-n-Butyl phthalate	55	115	≤30	45	125
di-n-Octyl phthalate	35	135	≤30	20	155
Diethylphthalate	40	120	≤30	30	130
Dimethylphthalate	25	125	≤30	10	145

TABLE K11  
 LCS and MS/MSD Control Limits for Water Matrix, Methods SW8270C and SW8270C-SIM

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
<b>Nitrosoamines (SW8270C)</b>					
n-Nitroso-di-n-propylamine	35	130	≤30	20	145
n-Nitroso-dimethylamine	25	110	≤30	10	125
n-Nitrosodiphenylamine	50	110	≤30	35	120
<b>Chlorinated Aliphatics (SW8270C)</b>					
bis(2-Chloroethoxy)methane	45	105	≤30	35	115
bis(2-Chloroethyl)ether	35	110	≤30	25	120
bis(2-Chloroisopropyl)ether	25	130	≤30	10	150
Hexachlorobutadiene	25	105	≤30	15	115
Hexachloroethane	30	100	≤30	15	105
<b>Halogenated Aromatics (SW8270C)</b>					
1,2,4-Trichlorobenzene	35	105	≤30	25	120
1,2-Dichlorobenzene	35	100	≤30	20	115
1,3-Dichlorobenzene	30	100	≤30	20	110
1,4-Dichlorobenzene	30	100	≤30	20	110
2-Chloronaphthalene	50	105	≤30	40	115
4-Bromophenyl phenyl ether	50	115	≤30	40	125
4-Chlorophenyl phenyl ether	50	110	≤30	40	120
Hexachlorobenzene	50	110	≤30	40	120
<b>Nitroaromatics (SW8270C)</b>					
2,4-Dinitrotoluene	50	120	≤30	40	130
2,6-Dinitrotoluene	50	115	≤30	35	130
2-Nitroaniline	50	115	≤30	35	125
3-Nitroaniline	20	125	≤30	10	145
4-Nitroaniline	35	120	≤30	20	130
Nitrobenzene	45	110	≤30	35	120
<b>Neutral Aromatics (SW8270C)</b>					
Carbazole	50	115	≤30	35	130
Dibenzofuran	55	105	≤30	45	115
<b>Others (SW8270C)</b>					
1,2-Diphenylhydrazine	55	115	≤30	45	120
Benzyl alcohol	30	110	≤30	15	125
Isophorone	50	110	≤30	40	125
<b>Surrogates</b>					
2-Fluorobiphenyl	50	110			
Terphenyl-d14	50	135			
2,4,6-Tribromophenol	40	125			
2-Fluorophenol	20	110			
Nitrobenzene-d5	40	100			

\*Poor performer

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K12

LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Methods SW8270C and SW8270C-SIM

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
<b>Polynuclear Aromatics (SW8270C-SIM)</b>					
1-Methylnaphthalene	45	105	≤30	35	115
2-Methylnaphthalene	45	105	≤30	35	115
Acenaphthene	45	110	≤30	35	120
Acenaphthylene	45	105	≤30	35	115
Anthracene	55	105	≤30	45	115
Benzo(a) anthracene	50	110	≤30	40	120
Benzo(a) pyrene	50	110	≤30	40	120
Benzo(b)fluoranthene	45	115	≤30	35	125
Benzo(k)fluoranthene	45	125	≤30	30	135
Benzo(g,h,i) perylene	40	125	≤30	25	140
Chrysene	55	110	≤30	45	120
Dibenzo(a,h) anthracene	40	125	≤30	25	140
Fluoranthene	55	115	≤30	45	125
Fluorene	50	110	≤30	40	115
Indeno(1,2,3-c,d)pyrene	40	120	≤30	25	135
Naphthalene	40	105	≤30	30	120
Phenanthrene	50	110	≤30	40	120
Pyrene	45	125	≤30	35	135
<b>Phenolic/Acidic (SW8270C)</b>					
2,4,5-Trichlorophenol	50	110	≤30	40	120
2,4,6-Trichlorophenol	45	110	≤30	30	120
2,4-Dichlorophenol	45	110	≤30	35	120
2,4-Dimethylphenol	30	105	≤30	20	115
2,4-Dinitrophenol	15	130	≤30	10	150
2-Chlorophenol	45	105	≤30	35	115
2-Methylphenol	40	105	≤30	30	115
2-Nitrophenol	40	110	≤30	30	120
3-Methylphenol/4-methylphenol	40	105	≤30	30	120
4,6-Dinitro-2-methylphenol	30	135	≤30	10	155
4-Chloro-3-methylphenol	45	115	≤30	35	125
4-Nitrophenol	15	140	≤30	10	160
Benzoic Acid*	0	110	≤30	0	130
Pentachlorophenol	25	120	≤30	10	135
Phenol	40	100	≤30	30	110
<b>Basic (SW8270C)</b>					
3,3-Dichlorobenzidine*	10	130	≤30	0	145
4-Chloroaniline*	10	95	≤30	0	110
<b>Phthalate Esters (SW8270C)</b>					
bis(2-Ethylhexyl)phthalate	45	125	≤30	35	140
Butylbenzyl phthalate	50	125	≤30	35	135
di-n-Butyl phthalate	55	110	≤30	45	120
di-n-Octyl phthalate	40	130	≤30	25	145
Diethylphthalate	50	115	≤30	40	125
Dimethylphthalate	50	110	≤30	40	120
<b>Nitrosoamines (SW8270C)</b>					
n-Nitroso-di-n-propylamine	40	115	≤30	30	125

TABLE K12

LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Methods SW8270C and SW8270C-SIM

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
n-Nitroso-dimethylamine	20	115	≤30	10	130
n-Nitrosodiphenylamine	50	115	≤30	40	125
<b>Chlorinated Aliphatics (SW8270C)</b>					
bis(2-Chloroethoxy)methane	45	110	≤30	30	120
bis(2-Chloroethyl)ether	40	105	≤30	25	115
bis(2-Chloroisopropyl)ether	20	115	≤30	10	130
Hexachlorobutadiene	40	115	≤30	25	130
Hexachloroethane	35	110	≤30	20	120
<b>Halogenated Aromatics (SW8270C)</b>					
1,2,4-Trichlorobenzene	45	110	≤30	30	120
1,2-Dichlorobenzene	45	100	≤30	35	105
1,3-Dichlorobenzene	40	100	≤30	30	110
1,4-Dichlorobenzene	35	105	≤30	25	115
2-Chloronaphthalene	45	105	≤30	35	115
4-Bromophenyl phenyl ether	45	115	≤30	35	130
4-Chlorophenyl phenyl ether	45	110	≤30	35	120
Hexachlorobenzene	45	120	≤30	35	130
<b>Nitroaromatics (SW8270C)</b>					
2,4-Dinitrotoluene	50	115	≤30	35	130
2,6-Dinitrotoluene	50	110	≤30	35	125
2-Nitroaniline	45	120	≤30	30	130
3-Nitroaniline	25	110	≤30	15	125
4-Nitroaniline	35	115	≤30	20	125
Nitrobenzene	40	115	≤30	30	125
<b>Neutral Aromatics (SW8270C)</b>					
Carbazole	45	115	≤30	30	130
Dibenzofuran	50	105	≤30	40	110
<b>Others (SW8270C)</b>					
1,2-Diphenylhydrazine	55	115	≤30	45	120
Benzyl alcohol	20	125	≤30	10	140
Isophorone	45	110	≤30	30	125
<b>Surrogates</b>					
2-Fluorobiphenyl	45	105			
Terphenyl-d14	30	125			
2,4,6-Tribromophenol	35	125			
2-Fluorophenol	35	105			
Phenol-d5/d6	40	100			
Nitrobenzene-d5	35	100			

\*Poor performer

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K13  
 LCS and MS/MSD Control Limits for Water Matrix, Methods SW6010B, SW6020, and SW7470A

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Aluminum	80	120	≤20	80	120
Antimony	80	120	≤20	80	120
Arsenic	80	120	≤20	80	120
Barium	80	120	≤20	80	120
Beryllium	80	120	≤20	80	120
Cadmium	80	120	≤20	80	120
Calcium	80	120	≤20	80	120
Chromium	80	120	≤20	80	120
Cobalt	80	120	≤20	80	120
Copper	80	120	≤20	80	120
Iron	80	120	≤20	80	120
Lead	80	120	≤20	80	120
Magnesium	80	120	≤20	80	120
Manganese	80	120	≤20	80	120
Mercury	80	120	≤20	No ME	No ME
Molybdenum	80	120	≤20	75	120
Nickel	80	120	≤20	80	120
Potassium	80	120	≤20	80	120
Selenium	80	120	≤20	75	120
Silver	80	120	≤20	75	120
Sodium	80	120	≤20	80	120
Thallium	80	120	≤20	80	120
Vanadium	80	120	≤20	80	120
Zinc	80	120	≤20	75	120

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K14  
 LCS and MS/MSD Control Limits for Soil/Sediment Matrix, Methods SW6010B, SW6020, and SW7471A

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
Aluminum	80	120	≤20	75	120
Antimony	80	120	≤20	75	120
Arsenic	80	120	≤20	80	120
Barium	80	120	≤20	80	120
Beryllium	80	120	≤20	80	120
Cadmium	80	120	≤20	80	120
Calcium	80	120	≤20	80	120
Chromium	80	120	≤20	80	120
Cobalt	80	120	≤20	80	120
Copper	80	120	≤20	80	120
Iron	80	120	≤20	80	120
Lead	80	120	≤20	80	120
Magnesium	80	120	≤20	80	120
Manganese	80	120	≤20	80	120
Mercury	80	120	≤20	No ME	No ME
Molybdenum	80	12	≤20	75	120
Nickel	80	120	≤20	80	120
Potassium	80	120	≤20	80	120
Selenium	80	120	≤20	75	120
Silver	75	120	≤20	75	125
Sodium	80	120	≤20	80	120
Thallium	80	120	≤20	80	120
Vanadium	80	120	≤20	80	120
Zinc	80	120	≤20	75	120

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K15  
 LCS/LCSD Control Limits for Air Matrix, Method TO-15

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
1,1,1-Trichloroethane	70	130	≤25	60	140
1,1,2,2-Tetrachloroethane	70	130	≤25	60	140
1,1,2-Trichloroethane	70	130	≤25	60	140
1,1-Dichloroethane	70	130	≤25	60	140
1,1-Dichloroethene	70	130	≤25	60	140
1,2-Dibromoethane (EDB)	70	130	≤25	60	140
1,2-Dichloroethane	70	130	≤25	60	140
1,2-Dichloropropane	70	130	≤25	60	140
Benzene	70	130	≤25	60	140
Carbon tetrachloride	70	130	≤25	60	140
Chloroethane	70	130	≤25	60	140
Chloroform	70	130	≤25	60	140
Chloromethane	70	130	≤25	60	140
cis-1,2-Dichloroethene	70	130	≤25	60	140
Ethylbenzene	70	130	≤25	60	140
Methylene chloride	70	130	≤25	60	140
m,p-Xylene	70	130	≤25	60	140
o-Xylene	70	130	≤25	60	140
Styrene	70	130	≤25	60	140
Tetrachloroethene	70	130	≤25	60	140
Toluene	70	130	≤25	60	140
trans-1,2-Dichloroethene	70	130	≤25	60	140
Trichloroethene	70	130	≤25	60	140
Vinyl chloride	70	130	≤25	60	140
Xylenes, Total	70	130	≤25	60	140
<b>Surrogates</b>					
4-Bromofluorobenzene	60	140			
1,2-Dichloroethane-d4	60	140			
Toluene-d8	60	140			

Note:

Control limits are taken from AFCEE QAPP Version 4.0.02

TABLE K16  
 LCS/LCSD Control Limits for Air Matrix, Method TO-3

Analyte	LCL	UCL	Precision
Gasoline Range Organics (C5-C10)	70	130	≤30
<b>Surrogate</b>			
4-Bromofluorobenzene	70	130	

TABLE K17  
 LCS and MS/MSD Control Limits for Water and Soil/Sediment Matrices, Method SW8290

Analyte	LCL	UCL	Precision	Lower ME Limit	Upper ME Limit
2,3,7,8-TCDD	NA	NA	≤20	NA	NA
1,2,3,7,8-PeCDD	NA	NA	≤20	NA	NA
1,2,3,4,7,8-HxCDD	NA	NA	≤20	NA	NA
1,2,3,6,7,8-HxCDD	NA	NA	≤20	NA	NA
1,2,3,7,8,9-HxCDD	NA	NA	≤20	NA	NA
1,2,3,4,6,7,8-HpCDD	NA	NA	≤20	NA	NA
OCDD	NA	NA	≤20	NA	NA
2,3,7,8-TCDF	NA	NA	≤20	NA	NA
1,2,3,7,8-PeCDF	NA	NA	≤20	NA	NA
2,3,4,7,8-PeCDF	NA	NA	≤20	NA	NA
1,2,3,4,7,8-HxCDF	NA	NA	≤20	NA	NA
1,2,3,6,7,8-HxCDF	NA	NA	≤20	NA	NA
2,3,4,6,7,8-HxCDF	NA	NA	≤20	NA	NA
1,2,3,7,8,9-HxCDF	NA	NA	≤20	NA	NA
1,2,3,4,6,7,8-HpCDF	NA	NA	≤20	NA	NA
1,2,3,4,7,8,9-HpCDF	NA	NA	≤20	NA	NA
OCDF	NA	NA	≤20	NA	NA

Note:

Control limits are taken from DoD QSM Version 4.1.

TABLE K18

Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Multipoint ICAL for all analytes (minimum five standards)	Before sample analysis.	AK101 and AK102/AK103: RSD ≤ 25%  NWEPH/NWVPH: RSD ≤ 20%	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.  Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin.
Continuing calibration verification	Daily after each ICAL and prior to sample analysis, after every 10 field samples, and at the end of the sequence.	AK101 and AK102/AK103: All analytes within ±25% of expected value.  NWEPH/NWVPH: All analytes within ±20% of expected value	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.
Retention time window position established for each analyte and surrogate	Each ICAL and after the initial daily CCV.	Position will be set using the midpoint standard of the ICAL curve. On days when an ICAL is not performed, the CCV is used.	NA.	NA.
Retention time window verification for each analyte and surrogate	Each calibration verification.	Analyte within established window.	Correct problem, then re-analyze all samples analyzed since the last acceptable retention time check.	Apply Q-flag to all results for the specific analyte(s) in the sample that are outside the established window.

TABLE K18

Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Method blank	One per analytical batch.	No analytes detected > ½ LOQ 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 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	<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> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.</p>
LCS (must contain all target analytes to be reported, including surrogates)	One per analytical batch.	Acceptance criteria in Tables K1 and K2.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>

TABLE K18

Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Surrogate spike	All field and QC samples.	Acceptance criteria in Tables K1 and K2.	<p>Correct problem, then re-extract and re-analyze all failed samples for failed surrogates in the associated preparatory batch.</p> <p>If obvious chromatographic interference with surrogate is present, , discuss in case narrative.</p>	<p>If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample.</p> <p>Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.</p>
Laboratory duplicate	One per analytical batch.	RPD $\leq$ 25%.	Correct problem, then re-extract and re-analyze affected samples.	For the specific analyte(s) in the parent sample, apply J-flag.
MS/MSD	One per 20 project samples per matrix as a minimum, and as defined on the chain-of-custody form.	Acceptance criteria in Tables K1 and K2.	<p>Assess data to determine whether there is a matrix effect or analytical error.</p> <p>Communicate potential matrix effects to CH2M HILL so an evaluation can be made regarding DQOs.</p>	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Retention time window width calculated for each analyte and surrogate	At method setup and after major maintenance (e.g., column change)	RT width is $\pm 3$ times standard deviation for each analyte RT from a 72-hour study.	NA	NA
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136, Appendix B). For the verification check to be valid, analytes must be detected and identified by method-specified criteria or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.

TABLE K18

Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If LOD verification fails, the laboratory must (1) repeat the MDL determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) LOQ and associated precision and bias must meet client requirements and be reported; or (2) in the absence of client requirements, must meet LCS control limits (see Box D-14 of the DoD QSM).	If the LOQ verification fails, laboratory must either establish a higher LOQ or modify method to meet client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of the DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by analyst until capability is demonstrated.

TABLE K18

Summary of Calibration and Quality Control Procedures for Methods AK101, AK102/AK103, NWEPH and NWVPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
<sup>a</sup> All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.				
<sup>b</sup> Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.				
Notes:				
B-flag	=	The analyte was found in an associated blank above one half the LOQ, as well as in the sample		
CFR	=	Code of Federal Regulations		
COD	=	coefficient of determination		
D	=	difference when using RFs or drift when using least square, regression, or nonlinear calibration		
DDT	=	dichlorodiphenyl-trichloroethane		
DoD	=	U.S. Department of Defense		
DQO	=	data quality objective		
J-flag	=	Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria		
GC	=	gas chromatography		
ICAL	=	initial calibration		
IS	=	internal standard		
LCS	=	laboratory control sample		
LOD	=	limit of detection		
LOQ	=	limit of quantitation		
MDL	=	method detection limit		
MS/MSD	=	matrix spike /matrix spike duplicate		
NA	=	not applicable		
NWEPH	=	North West Extractable Petroleum Hydrocarbons		
NWVPH	=	North West Volatile Petroleum Hydrocarbon		
PT	=	proficiency testing		
QC	=	quality control		
Q-flag	=	Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data		
QSM	=	Quality Systems Manual		
RF	=	response factor		
RPD	=	relative percent difference		
RRT	=	relative retention time		
RSD	=	relative standard deviation		

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Breakdown check (Endrin/DDT Method SW8081A only)	At the beginning of each 12-hour period, prior to the analysis of samples.	Degradation $\leq 15\%$ for both Endrin and DDT.	Correct problem, then repeat breakdown check.	Flagging criteria are not appropriate. No samples will be run until degradation is $\leq 15\%$ for both DDT and Endrin.
Multipoint ICAL for all analytes (minimum five standards)	Before sample analysis.	One of the following options (except for Method 8082, which may only use Option 1 or 2):  Option 1: Linear—RSD for each analyte $\leq 20\%$ .  Option 2: Linear—least squares regression $r \geq 0.995$ for each analyte.  Option 3: Nonlinear—COD $\geq 0.99$ (six points will be used for second order, seven points will be used for third order) (NA for Method SW8082).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.  Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin. All quantitation for multicomponent analytes, such as technical chlordane, toxaphene, and Aroclors, must be performed from a five-point calibration. Results may not be quantified from a single point.
Second-source calibration verification	Immediately after each ICAL.	All analytes within $\pm 20\%$ of expected value from the ICAL	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. Samples may not be analyzed until the calibration has been verified.

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
CCV	Daily, before sample analysis unless ICAL performed on same day, after every 10 samples, and at the end of the analysis sequence.	All analytes within $\pm 20\%$ of expected value from the ICAL.	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	<p>Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.</p>
Retention time window position established for each analyte and surrogate	Each ICAL and after the initial daily CCV.	Position will be set using the midpoint standard of the ICAL curve. On days when an ICAL is not performed, the CCV is used.	NA.	NA.
Retention time window verification for each analyte and surrogate	Each calibration verification.	Analyte within established window.	Correct problem, then re-analyze all samples analyzed since the last acceptable retention time check.	Apply Q-flag to all results for the specific analyte(s) in the sample that are outside the established window.
Method blank	One per analytical batch.	No analytes detected $> \frac{1}{2}$ LOQ 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. (See Box D-1 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	<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> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.</p>

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
LCS (must contain all target analytes to be reported, including surrogates)	One per analytical batch.	Acceptance criteria in Tables K3 through K8.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>
Surrogate spike	All field and QC samples.	Acceptance criteria in Tables K3 through K8.	<p>Correct problem, then re-extract and re-analyze all failed samples for failed surrogates in the associated preparatory batch.</p> <p>If obvious chromatographic interference with surrogate is present, , discuss in case narrative.</p>	<p>If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample.</p> <p>Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.</p>
MS/MSD	One per 20 project samples per matrix as a minimum and as defined on the chain-of-custody form.	Acceptance criteria in Tables K3 through K8.	<p>Assess data to determine whether there is a matrix effect or analytical error. Communicate potential matrix effects to CH2M HILL so an evaluation can be made regarding DQOs.</p>	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Second-column confirmation	All positive results.	Calibration and QC criteria same as for initial analysis or primary column analysis. Results between primary and confirmation result RPD $\leq$ 40%.	NA.	Apply J-flag to affected analyte(s) in associated sample. Discuss in case narrative. Report data from primary column.
Retention time window width established for each analyte and surrogate	At method setup and after major maintenance (for example, column change).	$\pm$ 3 times standard deviation for each analyte retention time from 72-hour study.	NA.	NA.
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136, Appendix B). For the verification check to be valid, analytes must be detected and identified by method-specified criteria or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If LOD verification fails, the laboratory must (1) repeat the MDL determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) LOQ and associated precision and bias must meet client requirements and be reported; or (2) in the absence of client requirements, must meet LCS control limits (see Box D-14 of the DoD QSM).	If the LOQ verification fails, laboratory must either establish a higher LOQ or modify method to meet client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of DoD QSM).	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for analytes not meeting criteria (see section C.1.f of DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision per the procedure in Appendix C of DoD QSM. No analysis will be allowed by an analyst until capability is demonstrated.

<sup>a</sup>All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

## Notes:

% RSD = percent relative standard deviation (QC and calibration statistic)

B-flag = The analyte was found in an associated blank above one half the LOQ, as well as in the sample

COD = coefficient of determination

D = difference when using RFs or drift when using least square, regression, or nonlinear calibration

DDT = dichlorodiphenyl-trichloroethane

DQO = data quality objective

GC = gas chromatography

ICAL = initial calibration

TABLE K19

Summary of Calibration and Quality Control Procedures for Methods SW8011, SW8021B, SW8081A, and SW8082

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
IS	=	internal standard		
J-flag	=	Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria		
LCS	=	laboratory control sample		
LOD	=	limit of detection		
LOQ	=	limit of quantitation		
MDL	=	method detection limit		
QC	=	quality control		
Q-flag	=	Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data		
QSM	=	Quality Systems Manual		
RF	=	response factor		
RPD	=	relative percent difference		
RRT	=	relative retention time		
RSD	=	relative standard deviation		
RSD	=	relative standard deviation		

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Mass spectrometer tuning check  Use bromofluorobenzene (SW8260B) or decafluorotriphenylphosphine (SW8270C)	Before ICAL and calibration verification.	Refer to criteria listed in method description.	Retune instrument and verify.	Not appropriate.
GC performance check (SW8270C only or when SW8270C is used for pesticide analyses)	Daily before analysis of sample or calibration standards.	Degradation $\leq 20\%$ for DDT. No visible peak tailing for benzidine or pentachlorophenol (as a default, tailing factors should be less than 2.0).	Correct problem, then repeat performance check.	Not appropriate.
Multipoint ICAL for all analytes (minimum five standards)	Before sample analysis.	<b>System performance compounds:</b> Average RF $\geq 0.30^c$ (SW8260B), $\geq 0.050$ (SW8270C), or as defined by method. <b>Calibration check compounds:</b> % RSD for RFs $\leq 30\%$ <b>and</b> one of the following options: Option 1: Linear—RSD for each analyte $< 15\%$ . Option 2: Linear—linear least squares regression $r > 0.995$ for each analyte. Option 3: Nonlinear—COD $\geq 0.99$ (six points will be used for second order, seven points will be used for third order). This is not a preferred option.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.  Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Second-source calibration verification	Once per ICAL.	All analytes within $\pm 20\%$ of expected 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. Samples may not be analyzed until the calibration has been verified.
CCV	Daily, before sample analysis (unless ICAL performed on same day), and after every 12 hours of analysis time.	<b>System performance compounds:</b> Average RF $\geq 0.30^c$ (SW8260B). Average RF $\geq 0.050$ (SW8270C). <b>Or</b> as defined by the method. <b>Calibration check compounds:</b> $\leq 20\%$ D. All analytes within $\pm 20\%$ D of expected value from ICAL.	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position will be set using the midpoint standard of the ICAL curve. On days when an ICAL is not performed, the CCV is used.	NA.	NA.
Retention time window verification for each analyte	Each sample.	RRT of the analyte within $\pm 0.06$ RRT units of ICAL. Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (for example, column clipping.)  With each sample, the RRT will	Correct problem then re-analyze all samples analyzed since the last retention time check.	Apply Q-flag to all results for the specific analyte(s) in the sample which are outside the established window.

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
		be compared with the most recently updated RRT. If the RRT has changed by more than $\pm 0.06$ RRT units since the last update, there has been a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to re-establish the retention times.		
Method blank	One per analytical batch.	No analytes detected $> \frac{1}{2}$ LOQ (for common lab contaminants, no analytes detected $>$ LOQ) 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. (See Box D-1 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	<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> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.</p>
LCS (must contain all target analytes to be reported, including surrogates)	One LCS per analytical batch.	Acceptance criteria Tables K9 through K12.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p>

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Surrogate spike	All field and QC samples.	Acceptance criteria Tables K9 through K12.	Correct problem, then re-extract and re-analyze all failed samples for failed surrogates in the associated preparatory batch.  If obvious chromatographic interference with surrogate is present, , discuss in case narrative.	Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.  If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample.  Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.
IS	Each sample, standard, and QC sample.	Retention time $\pm$ 30 seconds from retention time of the IS in the ICAL midpoint standard. Extracted ion current profile area within -50 to +100% of area from IS in ICAL mid-point standard.	Inspect mass spectrometer and GC for malfunctions and make corrections as appropriate. Re-analysis of samples analyzed while the system was malfunctioning is mandatory.	Problem must be corrected. Results may not be reported without a valid IS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If re-analysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to the specific analyte(s) associated with the non-compliant IS.
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form.	Acceptance criteria Tables K9 through K12.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to CH2M HILL so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM <sup>c</sup> )	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (See 40 CFR, Part 136, Appendix B). All analytes must be detected and identified by method-specified criteria for the for the verification check to be valid, or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must (1) repeat the detection limit determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup: (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if a laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of DoD QSM.	(1) The LOQ and associated precision and bias must meet client requirements and must be reported or (2) in the absence of client requirements, must meet LCS control limits. See Box D-14 of the DoD QSM.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by an analyst until capability is demonstrated.

<sup>a</sup>All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

<sup>c</sup>SW8260B:RF,  $\geq 0.1$  for chloromethane, bromoform, and 1,1-dichloroethane.

## Notes:

% R = percent recovery (QC statistic)

% RSD = percent relative standard deviation (QC and calibration statistic)

B-flag = The analyte was found in an associated blank above one half the LOQ, as well as in the sample

COD = coefficient of determination

D = difference when using RFs or drift when using least square, regression, or nonlinear calibration

DDT = dichlorodiphenyl-trichloroethane

DQO = data quality objective

GC = gas chromatography

ICAL = initial calibration

IS = internal standard

J-flag = Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria

LCS = laboratory control sample

LOD = limit of detection

LOQ = limit of quantitation

MDL = method detection limit

QC = quality control

TABLE K20

Summary of Calibration and Quality Control Procedures for Methods SW8260B and SW8270C (Full Scan and SIM)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Q-flag	=	Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data		
QSM	=	Quality Systems Manual		
RF	=	response factor		
RPD	=	relative percent difference		
RRT	=	relative retention time		
RSD	=	relative standard deviation		
SIM	=	selective ion monitoring		
RSD	=	relative standard deviation		

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Instrument detection limit study	At initial setup and after significant change in instrument type, personnel, test method, or sample matrix.	Instrument detection limits will be < LOD.	NA.	NA.
Linear dynamic range (SW6010B only)	Every 6 months.	Within $\pm 10\%$ of true value.	NA.	NA.
ICAL for all analytes (minimum one standard and a blank three- to five-point standard preferred), five points required for SW7470A/SW7471A	Daily before sample analysis.	If more than one standard is used, correlation coefficient must be $\geq 0.995$ .	If applicable, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
ICV (second source)	Daily after ICAL.	All analytes within $\pm 10\%$ of expected value.	Correct problem and verify second-source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
CCV	After every 10 samples and at the end of the analysis sequence.	SW6010B: All analyte(s) within $\pm 10\%$ of expected value. SW7470A/SW7471A: analyte(s) within $\pm 20\%$ of expected value.	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Calibration blank	Before beginning a sample run, after every calibration verification, and at the end of the analytical sequence.	No analytes detected > LOD.	Correct problem, re-prepare and re-analyzed calibration blank. All samples following the last acceptable calibration blank must be re-analyzed.	Apply B-flag to all associated positive results for in all samples associated with the blank.
Low-level calibration check standard (at or below LOQ) (SW6010B only)	Daily, after ICAL; not required for multipoint calibration (three or more points) with low standard at or below LOQ.	Analyte(s) with $\pm 20\%$ of expected value.	Correct problem, then re-analyze.	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 LOQ.
Method blank	One per analytical batch.	No analytes detected > $\frac{1}{2}$ LOQ (for common lab contaminants, no analytes detected >LOQ) 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. (See Box D-1 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	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 re-analyzed.  If re-analysis cannot be performed, data must be qualified and explained in the case narrative.  Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.
ICS (SW6010B only)	At the beginning of an analytical run.	ICS A: Absolute value of all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes).  ICS AB: Within $\pm 20\%$ of expected value.	Terminate analysis; locate and correct problem; re-analyze ICS and all associated samples.	If corrective action fails, apply Q-flag to the specific analyte(s) in all samples associated with the ICS.

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
LCS (must contain all analytes to be reported)	One per analytical batch.	Acceptance criteria in Tables K13 and K14.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>
Dilution test (SW6010B only)	Each new sample matrix, at least once per analytical batch (only applicable for analytes with concentrations > 50 times LOQ).	Five fold dilution must agree within $\pm 10\%$ of the original determination.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.
Post-digestion spike addition	When dilution test fails or if an analyte concentration for all samples in a batch is less than 50 times LOQ.	Recovery within 75 to 125% of expected result.	Run all associated samples in the preparatory batch by method of standard additions or see flagging criteria.	<p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.</p> <p>Spike concentration should produce a concentration of 10-100 times the LOQ.</p>
Method of standard additions	When matrix interference is confirmed.	NA.	NA.	Document use of method of standard additions in case narrative.
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form.	Acceptance criteria Tables K9 through K12.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to CH2M HILL so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification checks.	Detection limits established (see 40 CFR, Part 136, Appendix B). All analytes must be detected and identified by method-specified criteria for the for the verification check to be valid, or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.
LOD determination and verification (reference Section D.1.2.1 and Box D-13 Of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must (1) repeat the detection limit determination and LOD verification at a higher concentration; or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup: (1) verify LOQ and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if a laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) The LOQ and associated precision and bias must meet client requirements and must be reported; or (2) in the absence of client requirements, must meet control limits of the LCS.  See Box D-14 of the DoD QSM.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of the DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by analyst until capability is demonstrated.

<sup>a</sup>All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

## Notes:

% R	=	percent recovery (QC statistic)
% RSD	=	percent relative standard deviation (QC and calibration statistic)
B-flag	=	The analyte was found in an associated blank above one half the LOQ, as well as in the sample
DoD	=	U.S. Department of Defense
DQO	=	data quality objective
ICAL	=	initial calibration
ICS	=	Interference check solution
ICV	=	initial calibration verification
IS	=	internal standard
ICS	=	Interference check solution
J-flag	=	Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria
LCS	=	laboratory control sample
LOD	=	limit of detection
LOQ	=	limit of quantitation
NA	=	not applicable

TABLE K21

Summary of Calibration and Quality Control Procedures for Methods SW6010B, SW7470A, and SW7471A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
MDL	=	method detection limit		
MS/MSD	=	matrix spike/matrix spike duplicate		
PT	=	proficiency testing		
QC	=	quality control		
Q-flag	=	Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data		
QSM	=	Quality Systems Manual		
RPD	=	relative percent difference		
RSD	=	relative standard deviation		

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Instrument detection limit study	At initial setup.	Detection limits established will be $\leq$ LOD.	None.	NA; samples cannot be analyzed without a valid instrument detection limit.
Linear dynamic range	Every 6 months.	Within $\pm$ 10% of true value.	NA.	NA.
Mass spectrometer tuning sample	Before ICAL.	Mass calibration $\leq$ 0.1 amu from the true value.  Resolution < 0.9 amu full width at 10% peak height.  Stability: RSD $\leq$ 5% for at least four replicate analyses.	Retune instrument then re-analyze tuning solution.	Not appropriate.
ICAL for all analytes (minimum one standard and a blank)	Daily before sample analysis.	If more than one standard is used, correlation coefficient must be $\geq$ 0.995.	If applicable, correct problem and repeat ICAL.	Problem must be corrected.  Samples may not be analyzed until there is a valid ICAL.
ICV (second source)	After ICAL, before beginning a sample run at a concentration other than used for calibration.	All analytes within $\pm$ 10% of expected value.	Correct problem and verify second-source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Problem must be corrected.  Samples may not be analyzed until the calibration has been verified.
CCV	After every 10 samples and at the end of the analysis sequence.	All analytes within $\pm$ 10% of expected value.	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Calibration blank	Before beginning a sample run, after every calibration verification, and at the end of the analytical sequence.	No analytes detected > LOD.	Correct problem, re-prepare and re-analyzed calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all associated positive results in all samples associated with the blank.
Low-level calibration check standard (at or below LOQ)	Daily, after ICAL  Not required if multipoint calibration (three or more points) with low standard at or below LOQ is performed.	All analyte(s) with $\pm 20\%$ of expected value.	Correct problem then re-analyze.	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 LOQ.
Method blank	One per analytical batch.	No analytes detected > $\frac{1}{2}$ LOQ (for common lab contaminants, no analytes detected >LOQ) 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. (See Box D-1 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	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.  If re-analysis cannot be performed, data must be qualified and explained in the case narrative.  Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run or once during a 12-hour period, whichever is more frequent.	<b>ICS-A:</b> Absolute value of all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes).  <b>ICS-AB:</b> Within $\pm 20\%$ of expected value.	Terminate analysis; locate and correct problem; re-analyze ICS; re-analyze all affected samples.	If corrective action fails, apply Q-flag to the specific analyte(s).

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
LCS for all analytes	One LCS per analytical batch.	Acceptance criteria Tables K9 through K12.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>
Dilution test	Each matrix in a analytical batch (only applicable for analytes with concentrations $\geq 50$ times LOQ).	Five-fold dilution must agree within $\pm 10\%$ of the original determination.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.
Post-digestion spike addition	When dilution test fails or if an analyte concentration for all samples in a batch is less than 50 times LOQ.	Recovery within 75 to 125% of expected results.	Run all associated samples in the preparatory batch by method of standard additions or see flagging criteria.	<p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.</p> <p>Spike concentration should produce a concentration of 10-100 times the LOQ.</p>
Method of standard additions	When matrix interference is confirmed.	NA.	NA.	Document use of method of standard additions in case narrative.

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
MS/MSD	One per 20 samples per matrix.	Acceptance criteria Tables K9 through K12.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to CH2M HILL so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Internal standards	Every sample.	IS intensity within 30 to 120% of intensity of the IS in the ICAL.	Re-analyze sample at a five-fold dilution with addition of appropriate amounts if internal standards.	Flagging criteria are not appropriate.
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM <sup>c</sup> )	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136 Appendix B). Analytes must be detected and identified by method-specified criteria for the verification check to be valid, or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue to run the MDL study until all criteria are passing.	NA.

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: (1) Repeat the detection limit determination and LOD verification at a higher concentration; or (2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup: (1) verify LOQ and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if a laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) The LOQ and associated precision and bias must meet client requirements and must be reported; or (2) In the absence of client requirements, must meet control limits of the LCS.  See Box D-14 of the DoD QSM.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of the DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by an analyst until capability is demonstrated.

<sup>a</sup>All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

TABLE K22

Summary of Calibration and Quality Control Procedures for Method SW6020

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Notes:				
% R	=	percent recovery (QC statistic)		
% RSD	=	percent relative standard deviation (QC and calibration statistic)		
B-flag	=	The analyte was found in an associated blank above one half the LOQ, as well as in the sample		
DoD	=	U.S. Department of Defense		
DQO	=	data quality objective		
ICAL	=	initial calibration		
ICS	=	Interference check solution		
ICV	=	initial calibration verification		
IS	=	internal standard		
J-flag	=	Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria		
LCS	=	laboratory control sample		
LOD	=	limit of detection		
LOQ	=	limit of quantitation		
NA	=	not applicable		
MDL	=	method detection limit		
MS/MSD	=	matrix spike/matrix spike duplicate		
PT	=	proficiency testing		
QC	=	quality control		
Q-flag	=	Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data		
QSM	=	Quality Systems Manual		
RPD	=	relative percent difference		
RSD	=	relative standard deviation		

TABLE K23  
Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Mass spectrometer tuning check	Before ICAL and calibration verification.	Refer to criteria listed in method description.	Retune instrument and verify.	Not appropriate.
Use 4-bromofluorobenzene				
Initial multipoint calibration for all analytes (ICAL) (minimum of five standards)	Initially; as needed.	% RSD of $\leq 30\%$ , with up to two analytes $\leq 40\%$ .	Correct the problem and repeat the initial calibration.	Flagging criteria are not appropriate.  Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin
Second-source calibration verification (ICV)	After every ICAL.	All analytes within $\pm 30\%$ of expected value.	Correct problem and verify second-source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Problem must be corrected.  Samples may not be analyzed until the calibration has been verified.
CCV	Daily, before sample analysis (unless ICAL performed on same day), and after every 24 hours of analysis time.	All analytes within $\pm 30\%$ of expected value	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

TABLE K23

Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Method blank	Once per preparation batch.	No analytes detected > ½ LOQ (for common lab contaminants, no analytes detected >LOQ) 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 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	<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> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.</p>
LCS (must contain all target analytes to be reported)	One per sample preparation batch.	Acceptance criteria in Table K15.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>

TABLE K23  
Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Laboratory duplicate	At least one per analytical batch.	RPD $\leq$ 25%.	Correct the problem, then reprepare and re-analyze the original sample and laboratory duplicate.	If corrective action fails, apply J-flag to the specific analyte(s) in the sample.
Surrogate	All field and QC samples.	Acceptance criteria in Table K15.	Correct problem, then re-extract and re-analyze all failed samples for failed surrogates in the associated preparatory batch.  If obvious chromatographic interference with surrogate is present, discuss in case narrative.	If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample.  Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.
IS	Each sample.	Retention time within $\pm$ 30 seconds from retention time of the midpoint standard in the ICAL.  Extracted ion current profile area within -50 to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and make corrections as appropriate. Re-analysis of samples analyzed while the system was malfunctioning is mandatory.	Problem must be corrected. Results may not be reported without a valid IS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If re-analysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to the specific analyte(s) associated with the non-compliant IS.

TABLE K23  
Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136, Appendix B). For the verification check to be valid, analytes must be detected and identified by method-specified criteria or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If LOD verification fails, the laboratory must (1) repeat the MDL determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) LOQ and associated precision and bias must meet client requirements and be reported; or (2) in the absence of client requirements, must meet LCS control limits (see Box D-14 of the DoD QSM).	If the LOQ verification fails, laboratory must either establish a higher LOQ or modify method to meet client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.

TABLE K23  
Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of the DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by analyst until capability is demonstrated.

TABLE K23  
Summary of Calibration and Quality Control Procedures for Method TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
----------	-------------------	---------------------	--------------------------------	--------------------------------

<sup>a</sup>All corrective actions associated with project work shall be documented, and all records shall be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Notes:

% RSD = percent relative standard deviation (QC and calibration statistic)

B-flag = The analyte was found in an associated blank above one half the LOQ, as well as in the sample

CCV = continuing calibration verification

DoD = U.S. Department of Energy

ICAL = initial calibration

ICV = initial calibration verification

IS = internal standard

J-flag = Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria

LCS = laboratory control sample

LOD = limit of detection

LOQ = limit of quantitation

MDL = method detection limit

NA = Not Applicable

Q-flag = Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data

QSM = Quality Systems Manual

RPD = relative percent difference

RSD = relative standard deviation

TABLE K24

Summary of Calibration and Quality Control Procedures for Method TO-3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Initial multipoint calibration for all analytes (ICAL) (minimum of five standards)	Initially; as needed.	% RSD of $\leq 30\%$	Correct the problem and repeat the initial calibration.	Flagging criteria are not appropriate.  Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin
Second-source calibration verification (ICV)	After every ICAL.	All analytes within $\pm 30\%$ of expected value.	Correct problem and verify second-source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Problem must be corrected.  Samples may not be analyzed until the calibration has been verified.
CCV	Daily, before sample analysis (unless ICAL performed on same day), and after every 24 hours of analysis time.	All analytes within $\pm 30\%$ of expected value.	Correct problem and verify CCV source standard. Rerun CCV. If that fails, correct problem and repeat ICAL. Reanalyze all samples since the last successful CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.  If reanalysis cannot be performed, data must be qualified and explained in the case narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

TABLE K24  
Summary of Calibration and Quality Control Procedures for Method TO-3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Method blank	Once per preparation batch.	No analytes detected > ½ LOQ 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 of DoD QSM)	Assess data. Correct problem. If necessary, reprepare and re-analyze method blank and all samples processed with the contaminated blank.	<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> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.</p>
LCS (must contain all target analytes to be reported)	One per sample preparation batch.	Acceptance criteria in Table K16.	Correct problem, then re-analyze the LCS and all samples in the associated preparatory batch for the failed analytes.	<p>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.</p> <p>If re-analysis cannot be performed, data must be qualified and explained in the case narrative.</p> <p>Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.</p>

TABLE K24  
Summary of Calibration and Quality Control Procedures for Method TO-3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
Laboratory duplicate	At least one per analytical batch.	RPD $\leq$ 25%.	Correct the problem, then reprepare and re-analyze the original sample and laboratory duplicate.	If corrective action fails, apply J-flag to the specific analyte(s) in the sample.
Surrogate	All field and QC samples.	Acceptance criteria in Table K16.	Correct problem, then re-extract and re-analyze all failed samples for failed surrogates in the associated preparatory batch.  If obvious chromatographic interference with surrogate is present, discuss in case narrative.	If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample.  Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136, Appendix B). For the verification check to be valid, analytes must be detected and identified by method-specified criteria or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If LOD verification fails, the laboratory must (1) repeat the MDL determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.

TABLE K24

Summary of Calibration and Quality Control Procedures for Method TO-3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of the DoD QSM).	(1) LOQ and associated precision and bias must meet client requirements and be reported; or (2) in the absence of client requirements, must meet LCS control limits (see Box D-14 of the DoD QSM).	If the LOQ verification fails, laboratory must either establish a higher LOQ or modify method to meet client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of the DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by analyst until capability is demonstrated.

TABLE K24  
 Summary of Calibration and Quality Control Procedures for Method TO-3

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
----------	-------------------	---------------------	--------------------------------	--------------------------------

<sup>a</sup>All corrective actions associated with project work shall be documented, and all records shall be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Notes:

% RSD = percent relative standard deviation (QC and calibration statistic)

B-flag = The analyte was found in an associated blank above one half the LOQ, as well as in the sample

CCV = continuing calibration verification

DoD = U.S. Department of Defense

ICAL = initial calibration

ICV = initial calibration verification

IS = internal standard

J-flag = Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria

LCS = laboratory control sample

LOD = limit of detection

LOQ = limit of quantitation

MDL = method detection limit

NA = not applicable

PT = proficiency testing

Q-flag = Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data

RPD = relative percent difference

RSD = relative standard deviation

TABLE K25

Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Tuning	Before ICAL and calibration verification.	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. Re-run affected samples.	Flagging criteria are not appropriate. No samples may be accepted without a valid tune.
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 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 of labeling on the chromatogram;  <u>And:</u> Absolute retention times for switching from one homologue to the next $\geq 10$ seconds for all components of the mixture.	Correct problem, then repeat column performance check.	Flagging criteria are not appropriate.
Initial calibration 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; and S/N ratio $\geq 10$ for all target analyte ions; and RSD $\leq 20\%$ for the RF for all 17 unlabeled standards and 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. Samples may not be analyzed until there is a valid ICAL.  Calibration may not be forced through the origin

TABLE K25  
Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
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; and For unlabeled standards, RF within $\pm 20\%$ D of RF established in ICAL; and 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 re-analyze all samples analyzed since the last successful CCV. End-of-run CCV: 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, re-analyze samples with positive detections if necessary.	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.  If re-analysis 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.
Method blank	One per preparatory batch, run after calibration standards and before samples.	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. If required, reprep and re-analyze method blank and all samples processed with the contaminated blank.	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.  If re-analysis cannot be performed, data must be qualified and explained in the

TABLE K25  
Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
				case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS (or fortified field blank)	One per preparatory batch.	Acceptance criteria: Table K17.	Correct problem, then reprep and re-analyze the LCS and all samples in the associated preparatory batch for failed analytes.	<p>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.</p> <p>If re-analysis 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.</p>
Sample duplicate	One per preparatory batch per matrix.	RPD $\leq$ 25% (between sample and sample duplicate), per method.	Examine the project-specific DQOs. Contact CH2M Hill 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
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form	Acceptance criteria: Table K17.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to CH2M HILL so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Internal standards (IS)	Every field sample, standard, and QC sample.	Percent recovery for each IS in the original sample (prior to dilutions) must be within 40-135%, per	Correct problem, then reprep and re-analyze the samples with failed IS.	Apply Q-flag to results of all affected samples

TABLE K25  
Summary of Calibration and Quality Control Procedures for Method SW8290

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

TABLE K25  
Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
Sample specific EDL/EQL	Calculated for each 2,3,7,8-substituted isomer that is not identified.	Per method.	NA.	Flagging criteria are not appropriate.
Sample 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 TE concentration	All positive detections, as required.	Per method.	NA.	Flagging criteria are not appropriate.
MDL study (as part of the LOD process; see Section D.1.2.1 of the DoD QSM)	At initial setup and then once per 12-month period or quarterly MDL verification.	Detection limits established (see 40 CFR, Part 136, Appendix B.) All analytes must be detected and identified by method-specified criteria for the for the verification check to be valid, or the verification check must produce a response that is at least 3 times the instrument noise level and greater than the response in the blanks associated with the MDL study.	Continue the MDL study until all criteria are met.	NA.

TABLE K25

Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
LOD determination and verification (see Section D.1.2.1 and Box D-13 of the DoD QSM)	At initial setup and verified quarterly (if a laboratory uses multiple instruments for a given method, the LOD must be verified on each).	The apparent signal-to-noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must (1) repeat the detection limit determination and LOD verification at a higher concentration or (2) perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	NA; samples may not be analyzed without a valid LOD.
LOQ establishment and verification (see Section D.1.2.2 and Box D-14 of the DoD QSM)	At initial setup: (1) verify LOQ; and (2) determine precision and bias at the LOQ; then verify LOQ quarterly (if a laboratory uses multiple instruments for a given method, the LOQ must be verified on each; see Box D-14 of DoD QSM).	(1) The LOQ and associated precision and bias must meet client requirements and must be reported or (2) in the absence of client requirements, must meet LCS control limits.  See Box D-14 of the DoD QSM.	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	NA; samples may not be analyzed without a valid LOQ.
Results reported between the MDL and LOQ	None.	None.	None.	Apply J-flag to all results between MDL and LOQ. If no result below the MDL, report to the MDL, apply U-flag.
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C of DoD QSM).	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 of the DoD QSM).	NA. This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (for example, LCS or PT sample). No analysis will be allowed by an analyst until capability is demonstrated.

TABLE K25  
 Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
----------	-------------------	---------------------	--------------------------------	---

<sup>a</sup>All corrective actions associated with project work will be documented, and all records will be maintained by the laboratory.

<sup>b</sup>Flagging criteria will be applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed..

Notes:

- % R = percent recovery (QC statistic)
- % RSD = percent relative standard deviation (QC and calibration statistic)
- B-flag = The analyte was found in an associated blank above one half the LOQ, as well as in the sample
- CCV = continuing calibration verification
- D = difference when using RFs or drift when using least square, regression, or nonlinear calibration
- DQO = data quality objective
- EDL = Estimated Detection Limit
- EMPC = estimated maximum possible concentration
- EQL = Estimated Quantitation Limit
- GC = gas chromatography
- ICAL = initial calibration
- IS = internal standard
- J-flag = Estimated: The analyte was positively identified, but the associated concentration is estimated above the MDL and below the LOQ or the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria
- LCS = laboratory control sample
- LOD = limit of detection
- LOQ = limit of quantitation
- MDL = method detection limit
- MS/MSD = matrix spike/matrix spike duplicate
- QC = quality control
- Q-flag = Estimated: This indicates that one or more QC criteria fail. Data must be carefully assessed by CH2M HILL (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data
- QSM = Quality Systems Manual
- RF = response factor
- RPD = relative percent difference
- RRT = relative retention time

TABLE K25  
 Summary of Calibration and Quality Control Procedures for Method SW8290

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Laboratory Flagging Criteria <sup>b</sup>
RSD	= relative standard deviation			
RT	= retention time			
S/N	= signal-to-noise			
TCDD	= tetrachlorodibenzo-p-dioxin			
TE	= toxicity equivalents			

TABLE K26

EMAX Laboratories Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
SW8260B/SW8270C/SW8270C-SIM	Second Source Calibration Verification for GCMS (8260/8270): Value of second source for all analytes within 20% of expected value.	EMAX proposes using an acceptance criteria of 25%. This variance is consistent with Method, DOD QSM v3 and AFCEE QAPP that has been in effect for over 5 years and recognizes multi-component second source variability.	Accepted for second source calibration check. CCV must be within $\pm 20\%$ of True Value.
SW8260B/SW8270C/SW8270C-SIM	Continuing Calibration Verification for GCMS (8260/8270): % Difference for all analytes within 20% of expected value.	EMAX proposes using an acceptance criteria of 25% for bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, cis-1,3-dichloropropene, trans-1,3-dichloropropene, and naphthalene.	Accepted for the listed analytes only. All other analytes must be within $\pm 20\%$ of True Value.

TABLE K27

Applied Sciences Laboratory (field lab) Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
No variances requested.			

TABLE K28

AirToxics Ltd. Requested Variances to the AFCEE QAPP Version 4.0.02 (AFCEE, May 2006)

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
Table 7.2.2.3-1	Chloromethane RL 0.5 ppbv (1.03 µg/m <sup>3</sup> )	Chloromethane RL 2 ppbv (4.13 µg/m <sup>3</sup> )	Accepted. The raised RL for chloromethane meets project screening criteria of 14 µg/m <sup>3</sup> .

Notes:

µg/m<sup>3</sup> = microgram per cubic meter

AFCEE = Air Force Center for Engineering and the Environment

QAPP = Quality Assurance Project Plan

RL = reporting limit

LCS = laboratory control sample

QSM = Quality Systems Manual

RSD = relative standard deviation

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

### 1.0 Tuning & Breakdown Check for Method 8270C-SIM

Some QSM and method requirements for full-scan Method 8270 analysis are not compatible with SIM protocols. As a result, the following variances are requested:

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
Breakdown check (DDT Method 8270 only)	Perform at the beginning of each 12-hour period, prior to analysis of samples. Evaluate DDT breakdown and tailing of Pentachlorophenol and Benzidine.	Do not perform. As the analysis is for PAH only, the performance of DDT, pentachlorophenol, and benzidine is irrelevant to the analysis.	Accepted. Breakdown check not required for Method SW8270C-SIM.
Tuning	Prior to ICAL and at the beginning of each 12-hour period. Check using DFTPP; verify relative abundance of ions as specified in the method.	Perform a mass tuning check (autotune) using PFTBA, evaluate masses 69, 219 and 264 and verify that each is within $\pm 0.50$ amu of the target mass. SIM analysis does not evaluate the entire spectrum for each analyte, therefore, the relative abundance check is irrelevant.	Accepted. Breakdown check not required for Method SW8270C-SIM.

## Notes:

DDT = dichlorodiphenyl-trichlorethane

DFTPP = decafluorotriphenylphosphine

ICAL = initial calibration

PFTBA = perfluorotributylamine

QSM = Quality Systems Manual

SIM = Selective ion monitoring

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

## 2.0 Method 8260 Poor Behaving Analytes

The following compounds may behave poorly with respect to calibration and LCS recovery.

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
Acetone, Carbon Disulfide, 2-Butanone, 4-Methyl-2-pentanone, 2-Hexanone, Bromoform, 1,2-Dibromo-3-chloropropane, Naphthalene, 1,2,3-Trichlorobenzene, Dichlorodifluoromethane  <b>Appendix IX Analytes:</b> <i>Acrolein,</i> <i>Trichlorotrifluoroethane,</i> <i>t-Butanol,</i> <i>Vinyl Acetate,</i> <i>Isobutanol,</i> <i>1,4-Dioxane,</i> <i>2-Chloroethyl vinyl ether,</i> <i>T-1,4-Dichloro-2-Butene</i>	<b>ICAL</b> RSD $\leq$ 15%, or linear regression correlation coefficient $\geq$ 0.995, or non-linear curve fitting coefficient of determination $\geq$ 0.99	<b>ICAL:</b> Allow the use of 'force through zero' for the listed compounds. This is a change from Method 8000B requirements.	Not accepted for the listed analytes that are not Appendix IX. Force-through-zero not allowed.  Not applicable for Appendix IX analytes which are not target analytes for Galena
	<b>Second Source</b> within $\pm$ 20% of True Value	<b>Second Source:</b> Allow $\pm$ 30% of True Value for Appendix IX (Italicized) compounds. These are generally difficult compounds which are rarely requested.	Not Applicable: Appendix IX analytes are not target analytes for Galena.
	<b>CCV:</b> %Difference/Drift $\leq$ 20%	<b>CCV:</b> Allow 30%D for Appendix IX (italicized) compounds. These are generally difficult compounds which are rarely requested	Not Applicable: Appendix IX analytes are not target analytes for Galena.
	<b>LCS:</b> Spike all analytes to be performed.	<b>LCS:</b> Do not spike appendix IX (italicized) compounds. As these are rarely requested, historical limits may not accurately reflect current performance.	Not Applicable: Appendix IX analytes are not target analytes for Galena.
	<b>LCS:</b> All results must be within QSM or historical control limits	<b>LCS:</b> If sporadic marginal exceedances are not permitted, permit acceptance of high recoveries and no detections in the samples. With long analyte lists, some exceedances are to be expected..	Accepted.

Notes:

- CCV = continuing calibration verification  
 ICAL = initial calibration  
 LCS = laboratory control sample  
 QSM = Quality Systems Manual  
 RSD = relative standard deviation

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

### 3.0 Method 8270 Poor Behaving Analytes

The following compounds may behave poorly with respect to calibration and LCS recovery.

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
<b>Poor Performer (QSM Table G-2)</b> 3,3'-Dichlorobenzidine, Benzoic Acid, 4-Nitrophenol <b>Difficult Analytes</b> Benzidine, 2,4-Dinitrophenol, 4,6-Dintro-2-methylphenol, Pentachlorophenol <b>Additional Analytes</b> Aniline, 3,3'-Dimethylbenzidine, p-Phenylenediamine, 1,2-Dinitrobenzene, 1,3-Dintrobenzene, 1,4-Dinitrobenzene, 2,3,4,6-Tetrachlorophenol, 1,3,5-Trinitrobenzene, Dimethoate, 2-Sec-Butyl-4,6-Dinitrophenol, Methyl Parathion, 4-Nitroquinoline, Methapyrilene, Kepone, Famphur.	<b>ICAL</b> RSD $\leq$ 15%, or linear regression correlation coefficient $\geq$ 0.995, or non-linear curve fitting coefficient of determination $\geq$ 0.99	<b>ICAL:</b> Allow the use of 'force through zero' for the listed compounds. This is a change from Method 8000B requirements.	Not applicable: Benzidine, and additional analytes are not target analytes for Galena.
	<b>Second Source</b> within $\pm$ 20% of True Value	<b>Second Source:</b> Allow 30%D for Benzidine, 3,3-dimethylbenzidine & additional analytes. These are generally difficult compounds which degrade easily.	Not applicable: Benzidine, and additional analytes are not target analytes for Galena.
	<b>CCV:</b> %Difference/Drift $\leq$ 20%	<b>CCV:</b> Allow 30%D for Benzidine, 3,3-dimethylbenzidine & additional analytes. These are generally difficult compounds which degrade easily.	Not applicable: Benzidine, and additional analytes are not target analytes for Galena.
	<b>LCS:</b> Spike all analytes to be performed	<b>LCS:</b> Do not spike additional analytes, benzidine, 3,3-dimethylbenzidine. Some of these are not present in the standard spiking solution in use in the laboratory, and may degrade easily.	Not applicable: Benzidine, and additional analytes are not target analytes for Galena.
	<b>LCS:</b> All results must be within QSM or historical control limits	<b>LCS:</b> If sporadic marginal exceedances are not permitted, permit acceptance of high recoveries and no detections in the samples. With long analyte lists, some exceedances are to be expected	Accepted.

Notes:

CCV = continuing calibration verification  
 ICAL = initial calibration  
 LCS = laboratory control sample  
 QSM = Quality Systems Manual  
 RSD = relative standard deviation

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

#### 4.0 Method 8270 (SIM) Poor Behaving Analytes

The following compounds may behave poorly with respect to calibration and LCS recovery.

Parameter	Requirement	Proposed Change & Justification	CH2M HILL Response
Naphthalene, Indeno(123-cd)pyrene, Dibenz(ah)anthracene, Benzo(ghi)perylene, d5-Nitrobenzene	<b>Second Source</b> within $\pm 20\%$ of True Value	<b>Second Source:</b> Allow 25%D for the listed compounds by this method. SIM analysis at the sensitivity required may have a little more variability.	Accepted for second source calibration check. CCV must be within $\pm 20\%$ of True Value.

#### 5.0 PAH-SIM Analysis by Isotope Dilution (WS-MS-0006)

Proposed Change & Justification	CH2M HILL Response
This method is an in-house method employing low-resolution mass spectrometry and isotope dilution techniques. It therefore does not fall within the purview of the Tables in Appendix F of the QSM, and the calibration and QC parameter requirements are available in UFP-format tables for inclusion in specific QAPPs.	PAH-SIM analysis will not be allowed for Galena. PAH analysis will be performed by SW8270C-SIM only.

#### 6.0 Chemical Warfare Degradate Analysis by GC/MS-SIM (WS-MS-0003)

Proposed Change & Justification	CH2M HILL Response
This method is an in-house method employing low-resolution mass spectrometry. It therefore does not fall within the purview of the Tables in Appendix F of the QSM, and the calibration and QC parameter requirements are available in UFP-format tables for inclusion in specific QAPPs	Chemical warfare degradate analysis will not be required for Galena.

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

### 7.0 Common Laboratory Contaminants

Proposed Change & Justification	CH2M HILL Response
The concentrations of common laboratory contaminants in the method blank shall not exceed the reporting limit. When determining whether a blank is contaminated the LOQ will be used rather than ½ the LOQ.	Accepted for Ca, Fe, K, Na, Si and Zn only.  NOT accepted for Pb; Pb must be evaluated in blanks at ½ the LOQ.

The common laboratory contaminants for TestAmerica West Sacramento comprise the following:

Method	Analytes
Volatile Organics (SW8260B and SW8021B)	acetone, 2-butanone, and methylene chloride
GC/MS Semivolatiles (SW8270C)	Phthalate esters
Metals (SW6010B and SW6020)	Ca, Fe, K, Na, Pb, Si, Zn
Dioxins SW8290	OCDD
PCBs 1668	PCB-118

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

### 8.0 Dilutions for Organic Analytes

Proposed Change & Justification	CH2M HILL Response
<p>TestAmerica West Sacramento often uses screening techniques to determine appropriate dilution levels. Screening can include visual observation of the samples or sample extracts in instances where high concentrations of organic compounds are evident (e.g., non-aqueous liquid phase).</p> <p>If the dilution is overestimated by the screening technique (i.e., peak/signal is too low), the lab will conduct additional analyses to produce lower detection limits.</p> <p>The laboratory may not be able to analyze less dilute samples if significant levels of non-target and/or target compounds are present at concentrations high enough to prevent further dilutions.</p> <p>In order to protect our instruments from failure and contamination, initial analysis of sample dilutions based on screening data will be used to document any samples containing concentrations high enough to prevent further dilutions. Screening data will be included in the data package.</p> <p>TestAmerica West Sacramento will make every attempt to minimize dilutions and to analyze extracts without dilution if it can be done without damaging the analytical equipment.</p>	<p>Accepted.</p>

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

### 9.0 Explosives by EPA Method 8330B

Proposed Change & Justification	CH2M HILL Response
<p>For samples that require grinding by puck mill, the method specifies that a commercially purchased LCS be run through the grinding process. It is accepted that the QSM recovery limits cannot be met for all compounds. Recovery limits used for the LCS will be those provided by the manufacturer. TestAmerica West Sacramento will also produce a second LCS that is composed of sand spiked with the target compounds that does not go through grinding. The results for both LCSs will be provided to the client in the data package.</p>	<p>Explosives by SW8330 will not be required for Galena.</p>

### 10.0 High Resolution MS Isotope Dilution Methods

Proposed Change & Justification	CH2M HILL Response
<p>When reporting data below the LOQ for methods 8290, 1668 , 1698 and 1699, results will be "J" flagged down to the estimated detection limit (EDL) rather than the LOD. High resolution MS instruments have the capability and method requirement that the EDLs be calculated. These EDLs are analyte and sample specific estimates of detection sensitivity produced during the actual instrumental analysis. As such the EDL is a better estimate of analyte sensitivity in a specific sample than are the statistically derived LOD or MDL.</p>	<p>Accepted.</p>

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

11.0 PCB congeners by Isotope Dilution – Method 1668A

Proposed Change & Justification	CH2M HILL Response
<p>PCB congeners will be reported only to the LOQ. PCBs are common trace laboratory contaminants at levels &lt; ½ the LOQ. TestAmerica will analyze and report to the estimated detection limit (EDL) if requested but this will often result in many trace “J” value hits in samples and method blanks that are well below the LOQ and are not necessarily characteristic of the site being studied.</p> <p>The 5-point Initial Calibration for the PCB congeners will be carried out only for the congeners required by EPA Method 1668. As per the method, all other congeners will be calculated off a 1-point calibration. Second source samples will only be controlled off the congeners that have a 5-point calibration.</p> <p>The Laboratory Control Sample (LCS) will be required to be spiked and controlled only with the congeners that have a 5-point calibration.</p>	<p>PCB Congeners by 1668A will not be required for Galena.</p>

12.0 Total Petroleum Hydrocarbon (TPH) Analyses

Proposed Change & Justification	CH2M HILL Response
<p>Tests reporting petroleum hydrocarbon results will be reported as the concentration found in the sample for the specified carbon range. Unless specified in the site specific QAPP, “unknown” will not be reported for results not matching standard peak profiles.</p> <p>TPH diesel analyses requiring scanning for several possible diesel range products will have QC samples spiked with and controlled off a single diesel range standard. If the QAPP identifies a project specific product (motor oil, jet fuel, etc) as an analyte of concern then the QC will include spikes of that compound.</p>	<p>Accepted.</p> <p>The Galena project requires that TPH analysis be conducted by the Alaska-specific methods AK101 and AK102/AK103. These methods do not allow for the reporting of “unknown hydrocarbons”. Carbon ranges are specified in the method and QC must include spikes of those compounds.</p>

TABLE K29

TestAmerica West Sacramento Requested Variances to the DoD QSM Version 4.1 (DoD, April 2009)

## 13.0 Pesticides by Method 8081A

Proposed Change & Justification	CH2M HILL Response
<p>Compounds not in the TestAmerica West Sacramento standard analyte list below will not be spiked into QC samples and will not be used as control compounds in the analysis. The multi-component analyte toxaphene will only be spiked into the MS/MSD if it is identified in the project specific QAPP as an analyte of concern. Individual chlordane isomers (<math>\alpha</math> and <math>\gamma</math>) are spiked into the LCS and MS/MSD and are used as a measure of chlordane recovery. For this reason the multi-component analyte technical chlordane will not be spiked in the LCS or MS/MSD.</p>	<p>Accepted: TestAmerica's standard target analyte list matches the target analytes list for Galena. Toxaphene is a target compound and must be spiked in the LCS and MS/MSD.</p>

TestAmerica West Sacramento Standard Analyte List for SW8081A:

Aldrin	Endosulfan I
$\alpha$ -BHC	Endosulfan II
$\beta$ -BHC	Endosulfan Sulfate
$\delta$ -BHC	Endrin
$\gamma$ -BHC (Lindane)	Endrin Aldehyde
$\alpha$ -Chlordane	Endrin Ketone
$\gamma$ -Chlordane	Heptachlor
4,4'-DDD	Heptachlor Epoxide
4,4'-DDE	Methoxychlor
4,4'-DDT	Toxaphene
Dieldrin	

