

# U. S. Department of Energy



## Consolidated Audit Program

### Checklist 3

## Data Quality for Inorganic Analyses

Revision 5.4

November 2012

Audit ID:

Date:

Approved by:  DOECAP Manager, January 2012

Audit ID: \_\_\_\_\_ Laboratory: \_\_\_\_\_ Auditor: \_\_\_\_\_

## Areas of Review during Audit

1.0 Standard Operating Procedures (SOPs)	2.0 Balances
3.0 Thermometers and Pipettes	4.0 Sample Preparation
5.0 Metals Digestion	6.0 Method Detection Limits (MDLs)
7.0 Analytical Standards & Reagents	8.0 Instrument Operations & Maintenance
9.0 Method Blanks	10.0 Laboratory Control Samples (LCS)
11.0 Matrix Spike/Matrix Spike Duplicates (MS/MSD)	12.0 Multipoint Calibration Procedures – GFAA
13.0 Inductively Coupled Plasma Mass Spectrometry (ICP/MS)	14.0 Multipoint Calibration Procedures – ICP-ABS
15.0 Multipoint Calibration Procedures – CVAA	16.0 Quality Control (QC) of Instrument Gases
17.0 Wet Chemistry Procedures – Cyanide Distillation	18.0 Reactivity Determinations – Colorimetric Cyanide
19.0 Reactivity Determinations – Titrimetric Sulfide	20.0 Toxicity Characteristic Leaching Procedure (TCLP) Extractions
21.0 Ignitability Tests	22.0 Corrosivity Determination
23.0 Corrosivity Towards Steel	24.0 Total Organic Carbon (TOC)
25.0 Total Organic Halogens (TOX)	26.0 Biological Oxygen Demand (BOD) Determinations
27.0 Chemical Oxygen Demand (COD) Determinations	28.0 Total Phosphorous, Nitrate/Nitrite, & Anions Determination
29.0 Kinetic phosphorescence Analysis	30.0 Sample Dilutions
31.0 Data Review	

A = Acceptable  
F = Finding

U = Unsatisfactory  
O = Observation

NA = Not Applicable

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### Referenced regulations are accessible at the following URLs:

- <https://doecap.oro.doe.gov/>
- <http://www.epa.gov/epawaste/hazard/testmethods/index.htm>

### NOTE:

- When audit findings are written against site-specific documents (i.e., SOPs, QA Plans, licenses, permits, etc.), a copy of the pertinent requirement text from that document must be attached to this checklist for retention in DOECAP files.
- Fully document any deviation from the LOI or the requirements of QSAS Revision 2.7
- Refer to Page 58 for the record of revision.

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
1.0	<b>Standard Operating Procedures (SOPs)</b>		
1.1	Analytical procedures followed by the analyst agree with the written SOPs.  <i>QSAS, Section 5.4.1.1</i>		
1.2	All observations and results recorded by the laboratory are on pre-printed forms, electronic media, or entered into permanent laboratory logbooks.  <i>QSAS, Section 4.12.2.1</i>		
1.3	Hardcopy laboratory notebooks (logbooks) comply with the following: <ul style="list-style-type: none"> <li>• Permanent, bound logbooks are required and loose leaf binders shall not be used;</li> <li>• entries are made in a permanent fashion and corrections are made without obliterating the original data;</li> <li>• entries are dated and signed by the person responsible for performing the activity at the time the activity is performed;</li> <li>• entries are in chronological order; and</li> <li>• notebooks are protected against damage, deterioration, or loss.</li> <li>•</li> </ul> Electronic logbooks are permitted and must be protected against change and are controlled, protected as primary records.  <i>QSAS, Section 4.12.1.5 and 4.12 DOE-6</i>		

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1.4	<p>All logbook pages must be closed when the activities documented are completed or carried over to another logbook page. The person responsible for performing the closure shall be the one who performed the last activity documented. Closure shall occur at the end of the last activity performed or as soon as practicable thereafter.</p> <p><i>QSAS, Section 4.12 DOE-6</i></p>		

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2.0	<b>Balances</b>		
2.1	Balances are located in an area where the environment has little or no effect on measurement accuracy.  <i>QSAS, Section 5.3.1</i>		
2.2	Balances are calibrated at least annually by an independent company or source, not associated with the laboratory daily operation.  <i>QSAS, Section 5.5 DOE-4</i>		
2.3	Balances are checked prior to use that they are used using NIST-traceable weights. The daily balance check brackets the range of measurements to be made and the check is recorded.  <i>QSAS, Sections 5.5.2.1 d</i>		
2.4	Class 1 (formerly referred to as Class S) certified check weights shall be verified every five years. Alternatively, Class 1 check weights may be reverified using controlled check weight standards that are used exclusively for this purpose.  Alternatively, Class 1 check weights may be reverified using controlled check weight standards that are used exclusively for this purpose (the weights must be traceable to National Metrology Institute traceable references)  <i>QSAS, Section 5.5 DOE-4</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>3.0</b>	<b>Thermometers and Pipettes</b>		
3.1	Liquid-in-glass thermometers are calibrated against a NIST-traceable standard.  <i>QSAS, Section 5.5.2.1 b</i>		
3.2	Thermometers, pipettes, and automatic sample dispensers are uniquely identified by an identification protocol.  <i>QSAS, Section 5.5 DOE-3</i>		
3.3	Accuracy of all non-Class A pipettes and automatic sample dispensers used for quantitative measurement is checked for accuracy at least quarterly and is verified daily or prior to use.  <i>QSAS, Section 5.5 DOE-5</i>		

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<b>4.0</b>	<b>Sample Preparation</b>		
4.1	Sample preparation areas are kept clean to avoid contamination.  <i>QSAS, Sections 5.3.3 and 5.3.5</i>		
4.2	All sample preparations are conducted in a hood.  <i>EPA SW-846, Methods 3031 Section 7.2, 3050B Section 7.1, 3051 Section 7.3 and 3052 Section 7.3052</i>		

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<b>5.0</b>	<b>Metals Digestion</b>		
5.1	<p>The temperature of metals digestion equipment (hotplate, hot block, water baths, etc.) is monitored and recorded on a regular basis. Corrective action is taken if the temperature of the metals digestion equipment falls outside the range established by the laboratory.</p> <p><i>QSAS, Section 5.5 DOE-4</i></p>		
5.2	<p>For digestion procedures, the laboratory uses water meeting ASTM specifications (or equivalent) for “Type II” water (ASTM D1193).</p> <p><i>EPA SW-846, Chapter One, Section 5.0, Definitions – Reagent Water</i></p>		
5.3	<p>The laboratory uses reagent or trace metal grade acid for digestion procedures.</p> <p><i>EPA SW-846, Method 6010B, Section 5.1; QSAS, Appendix D.1.4</i></p>		
5.4	<p>The drying oven:</p> <ul style="list-style-type: none"> <li>• has a temperature measurement device; and</li> <li>• the temperature is monitored and documented.</li> </ul> <p><i>QSAS, Section 5.5 DOE-4</i></p>		
5.5	<p>Method blanks, spiked samples, and Laboratory Control Samples (LCSs) are carried through the same digestion process.</p> <p><i>QSAS, Appendix D.1.1.1 a, and D.1.1.2.1.a</i></p>		

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<b>6.0</b>	<b>Method Detection Limits (MDL)</b>		
6.1	MDLs have been established by analytical method for each instrument configuration and matrix. MDLs are determined by the protocol in the mandated test method.  <i>QSAS, Appendix C.3.1</i>		

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<b>7.0</b>	<b>Analytical Standards and Reagents</b>		
7.1	Standards and reference materials are traceable to EPA or NIST-certified standards, including: <ul style="list-style-type: none"> <li>• initial calibration standards;</li> <li>• continuing calibration standards; and</li> <li>• spiking standards.</li> </ul> <i>QSAS, Section 5.6.3.2</i>		
7.2	Standards are assigned a unique identification number traceable to the original standard. The unique identification number and the expiration date are placed on the standards container. <i>QSAS, Sections 5.6.4 c) and d)</i>		
7.3	Labels for purchased stock mixtures and reagents contain the following information: <ul style="list-style-type: none"> <li>• date opened; and</li> <li>• expiration date.</li> </ul> <i>QSAS, Sections 5.6 DOE-1</i>		

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7.4	<p>Secondary standard solutions are traceable to a standards preparation log and labeled with the following information:</p> <ul style="list-style-type: none"> <li>• secondary standard tracking identification number;</li> <li>• preparer's initials;</li> <li>• preparation date; and</li> <li>• secondary standard expiration date.</li> </ul> <p><i>QSAS, Sections 5.6.4 c) and d)</i></p>		
7.5	<p>Standards and reference materials logbook of standards preparation contains the following information:</p> <ul style="list-style-type: none"> <li>• standards identification number;</li> <li>• standards prepared;</li> <li>• matrix noted;</li> <li>• spiking standards;</li> <li>• pretreatment;</li> <li>• volume/weight of standards;</li> <li>• final volume; and</li> <li>• preparation methods.</li> </ul> <p><i>QSAS, Section 5.6.4 c)</i></p>		
7.6	<p>Reagent are checked for purity prior to use and the supporting documentation of the checks is filled in a manner that is easily retrievable.</p> <p><i>QSAS Section 5.6 DOE-1</i></p>		

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<b>Item Number</b>	<b>Line of Inquiry</b>	<b>Status</b>	<b>Summary of Observations/Objective Evidence Reviewed Audit Notes</b>
7.7	If the initial calibration is not verified using an independent source, the LCS is prepared from an independent source.  <i>QSAS, Section 3.1, "LCS"</i>		

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<b>8.0</b>	<b>Instrument Operations and Maintenance</b>		
8.1	Out-of-calibration equipment is tagged or segregated and not used until it has been recalibrated. Equipment consistently found to be out-of-calibration is repaired or replaced.  <i>QSAS, Sections 5.5.2.1 b) and 5.5.7</i>		
8.2	Calibration results are maintained as permanent laboratory records and contain sufficient information to allow monitoring of instrument performance over time.  <i>QSAS, Section 5.5.10 d)</i>		

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<b>9.0</b>	<b>Method Blanks</b>		
9.1	Method blanks are prepared and analyzed with each batch of 20 samples or less.  <i>QSAS, Appendix D.1.1.1 b)</i>		
9.2	When a method blank fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.  <i>QSAS, Appendix D.1.1.1 d)</i>		

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<b>10.0</b>	<b>Laboratory Control Sample (LCS)</b>		
10.1	An LCS is prepared and analyzed with each batch of 20 samples or less.  <i>QSAS, Appendix D.1.1.2.1 b)</i>		
10.2	<ul style="list-style-type: none"> <li>• The results of the LCS are calculated in percent recovery and the calculation is documented.</li> <li>• The laboratory defines acceptance limits.</li> <li>• When an LCS fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.</li> </ul> <i>QSAS, Appendix D.1.1.2.1 d)</i>		

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<b>11.0</b>	<b>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</b>		
11.1	One MS/MSD or matrix spike/duplicate is prepared and analyzed as part of the DQO process or as specified by the test method.  <i>QSAS, Appendix D.1.1.3.1 b) and D.1.1.3.2 b)</i>		
11.2	When an MS/MSD or matrix spike/duplicate fails to meet the laboratory or method acceptance criteria, a corrective action process is initiated.  <i>QSAS, Appendix D.1.1.3.1 d) and D.1.1.3.2 d)</i>		

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<b>12.0</b>	<b>Multipoint Calibration Procedures - Graphite Furnace Atomic Absorption (GFAA) EPA SW-846, Method 7000A</b>		
12.1	Reagent grade water will be at least 16 megohms-cm quality  <i>EPA SW-846, Method 7000A, Section 5.2</i>		
12.2	For Nitric Acid (concentrated):  <ul style="list-style-type: none"> <li>• use spectrograde certified acid for AA;</li> <li>• prepare a 1:1 dilution with water; and</li> <li>• ensure that acid reagent blank is &lt; the Instrument Detection Limit (IDL) prior to using.</li> </ul> <i>EPA SW-846, Method 7000A, Section 5.3</i>		
12.3	For Hydrochloric Acid (1:1):  <ul style="list-style-type: none"> <li>• use spectrograde certified acid for AA;</li> <li>• prepare a 1:1 dilution with water; and</li> <li>• ensure acid reagent blank is &lt; IDL prior to using.</li> </ul> <i>EPA SW-846, Method 7000A, Section 5.4</i>		
12.4	A calibration curve is prepared each day with a minimum of a calibration blank and three standards.  <i>EPA SW-846, Method 7000A, Section 8.2</i>		
12.5	The calibration standards are prepared fresh at the time of analysis.  <i>EPA SW-846, Method 7000A, Section 5.7</i>		

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12.6	<p>After calibration, the calibration curve is verified using a calibration blank and a calibration check standard derived from a source independent of that used to prepare the initial calibration standards.</p> <p><i>EPA SW-846, Method 7000A, Section 8.2</i></p>		
12.7	<p>The independent calibration check standard is at or near mid-range of the calibration and is within 10% of its true value for the curve to be considered valid.</p> <p><i>EPA SW-846, Method 7000A, Section 8.2</i></p>		
12.8	<p>When more than 10 samples are analyzed on a particular instrument per day, the multipoint calibration is verified after every 10 samples by analyzing a standard at the mid-range of the curve. The standard value is within 20% of the true value, or the samples associated with it are repeated.</p> <p><i>EPA SW-846, Method 7000A, Section 8.3</i></p>		
12.9	<p>Dilution Test:</p> <ul style="list-style-type: none"> <li>• One diluted sample is analyzed per batch.</li> <li>• The concentration of the analyte is at least 25 times the estimated detection limit.</li> <li>• Dilute the sample by at least 5 fold and reanalyze.</li> <li>• Concentration agreement within 10% between the undiluted and diluted sample indicates the absence of interferences and the sample may be analyzed without using the method of standard additions.</li> </ul> <p><i>EPA SW-846, Method 7000A Section 8.6.1</i></p>		

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12.10	<p>Recovery Test:</p> <ul style="list-style-type: none"> <li>Add a known amount of the analyte to bring the concentration of the analyte to 2 to 5 times the original concentration. (If all of the samples in the batch have analyte concentrations below the MDL, spike the selected sample at 20 times the detection limit.)</li> <li>The spike recovery is 85-115%. If the % recovery criteria are not met, the method of standard additions is used for all samples in the batch.</li> </ul> <p><i>EPA SW-846, Method 7000A, Section 8.6.2</i></p>		
12.11	<p>Method of Standard Addition (MSA):</p> <ul style="list-style-type: none"> <li>The laboratory employs the MSA when interference is suspected or a new matrix is encountered.</li> </ul> <p><i>EPA SW-846, Method 7000A, Section 8.7</i></p>		

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<b>13.0</b>	<b>Inductively Coupled Plasma – Mass Spectrometry (ICP-MS), EPA SW-846, Method 6020</b>		
13.1	<p>Instrument Detection Limits (IDLs):</p> <ul style="list-style-type: none"> <li>• IDLs are established by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.</li> <li>• Each measurement is performed as a separate analytical sample.</li> <li>• IDLs are determined at least every three months and kept with the instrument log book.</li> </ul> <p><i>EPA SW-846, Method 6020, Section 8.2</i></p>		
13.2	<p>The ICP-MS is capable of providing resolution better than or equal to atomic mass unit (amu) at 10% peak height. The system has a mass range from at least 6 to 240 amu and a data system that allows for corrections for isobaric interferences and the application of the internal standard technique.</p> <p><i>EPA SW-846, Method 6020, Section 4.1.1</i></p>		
13.3	<p>Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. Instrument equilibration is verified by analyzing a tuning solution at least four times with a relative standard deviation of <math>\leq 5\%</math> for the analytes in the tuning solution.</p> <p><i>EPA SW-846, Method 6020, Section 7.4</i></p>		

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13.4	<p>Mass calibration and resolutions checks are conducted in the mass regions of interest prior to analysis. If the mass calibration differs by more than 0.1 amu from the true value, the mass calibration is adjusted to the correct value. The resolution is also verified to be less than 0.9 amu full width at 10% peak height prior to analysis.</p> <p><i>EPA SW-846, Method 6020, Section 7.5</i></p>		
13.5	<p>Mass spectrometer tuning solutions for verifying instrument resolution, mass calibration and thermal stability contain elements representing all of the mass regions of interest and the tuning solution is within the required specifications.</p> <p><i>EPA SW-846, Method 6020, Section 5.8</i></p>		
13.6	<p>For each instrument in use, a calibration procedure is performed for the analytes of interest (at a minimum) using a calibration blank and a single calibration point standard. The average of at least three integrations is used for calibration.</p> <p><i>EPA SW-846, Method 6020, Section 7.6</i></p>		
13.7	<p>Immediately after the calibration has been established, the calibration is verified for every analyte by the analysis of a calibration verification standard.</p> <p><i>EPA SW-846, Method 6020, Section 7.8.</i></p>		
13.8	<p>Flush the system with the rinse blank solution until the signal levels return to the method's level of quantitation before the analysis of each sample.</p> <p><i>EPA SW-846, Method 6020, Section 7.9</i></p>		

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13.9	<p>A calibration verification blank [Continuing Calibration Blank (CCB)] and either a Continuing Calibration Verification (CCV) or an Initial Calibration Verification (ICV) are analyzed after every tenth sample and at the beginning and end of the sample run.</p> <p><i>EPA SW-846, Method 6020, Section 8.8.2</i></p>		
13.10	<p>A corrective action process is implemented when the calibration check standards are not within 10% of the accepted value. If this limit is not met, the analysis is terminated and the problem corrected. Any samples associated with the out-of-control calibration are reanalyzed.</p> <p><i>EPA SW-846, Method 6020, Section 7.8</i></p>		
13.11	<p>If the instrument is re-sloped (to correct for instrument drift) or recalibrated, a new analysis of a CCV and CCB is performed prior to further sample analysis.</p> <p><i>EPA SW-846, Method 6020, Section 7.8</i></p>		
13.12	<p>The intensities of all internal standards are monitored for all analyses. When the intensity of the internal standards fails to fall between 30% – 120% of the intensity of the initial calibration standard, the sample is diluted fivefold (1+4) and reanalyzed using the proper amount of internal standards until the standard intensity falls within the prescribed window.</p> <p><i>EPA SW-846, Method 6020, Section 8.3</i></p>		

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13.13	<p>The intensity levels of the internal standards for calibration blank and instrument check standard are within 20% of the intensity level of the original calibration solution. If not, analysis is terminated, the problem corrected, recalibration performed and verified, and affected samples are reanalyzed. (<i>Continuation of LOI 13.12</i>)</p> <p><i>EPA SW-846, Method 6020, Section 8.3</i></p>		
13.14	<p>Interference levels are corrected by the instrument data system and are accounted for in the data report.</p> <p><i>EPA SW-846, Method 6020, Sections 5.6 and 8.4</i></p>		
13.15	<p>The Interference Check Solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The ICS solution is prepared from ultra-pure reagents.</p> <p><i>EPA SW-846, Method 6020, Sections 5.6 and 5.6.1</i></p>		
13.16	<p>Both ICSs A and AB are analyzed at the beginning of an analytical run, or once every 12 hours, whichever is more frequent.</p> <p><i>EPA SW-846, Method 6020, Section 8.9</i></p>		
13.17	<p>If samples that are more concentrated than the linear range for an analyte (or species needed for a correction) are not diluted to bring them within range, a less abundant isotope may be measured. The linearity of the alternate mass is confirmed by the appropriate calibration.</p> <p><i>EPA SW-846, Method 6020, Section 7.10</i></p>		

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13.18	<p>Post Digestion Spike:</p> <ul style="list-style-type: none"> <li>• An analyte spike is added to a portion of a prepared sample or its dilution.</li> <li>• If the recovery is not within 75-125%, or within the laboratory defined acceptance criteria, the dilution test or MSA is performed.</li> <li>• Results of the dilution are <math>\pm 10\%</math> of the original sample concentration.</li> </ul> <p><i>EPA SW-846, Method 6020, Section 8.6</i></p>		
13.19	<p>The post spike addition is based on the indigenous concentration of each element in the sample. If the spike recovery is outside the specified limits, the sample is diluted and reanalyzed to compensate for the matrix effect. The results agree to within 10% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect.</p> <p><i>EPA SW-846, Method 6020, Section 8.6</i></p>		
13.20	<p>For each new matrix, a dilution test is performed that:</p> <ul style="list-style-type: none"> <li>• analyzes a five-fold dilution on a sample containing analytes <math>\geq 100</math> times the reagent blank;</li> <li>• has results that agree with <math>\pm 10\%</math> of the original determination; and</li> <li>• if the <math>\pm 10\%</math> criteria are not met, an interference effect is suspected. One dilution test is included for each batch of 20 samples or less.</li> </ul> <p><i>EPA SW-846, Method 6020, Section 8.5</i></p>		

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<b>14.0</b>	<b>Multipoint Calibration Procedures – Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) Method 6010B</b>		
14.1	<p>Linear Dynamic Range (LDR):</p> <ul style="list-style-type: none"> <li>• Determine the signal responses from a minimum of three different concentration standards across the range.</li> <li>• The upper range limit is an observed signal no more than 10% below the level extrapolated from lower standards.</li> <li>• Determine new dynamic ranges whenever there is a significant change in instrument response.</li> <li>• The ranges are determined every six months for those analytes that periodically approach the upper limit.</li> </ul> <p><i>EPA SW-846, Method 6010B, Section 7.2.5.4</i></p>		

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14.2	<p>Interelement Correction (IEC) Factor:</p> <ul style="list-style-type: none"> <li>• When IEC are applied, their accuracy should be verified daily, by analyzing spectral interference check solutions</li> <li>• If the multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis</li> <li>• If the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at <math>\pm</math> one reporting limit from zero, daily verification is not required.</li> <li>• All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs</li> <li>• Data for the initial and periodic verification of the IEC factors is maintained in a manner that is readily accessible to the analyst.</li> </ul> <p><i>EPA SW-846, Method 6010B, Sections 3.1.9 and 7.2.3.6</i></p>		
14.3	<p>For each instrument in use, a calibration procedure is performed daily consisting of (at a minimum) a blank and a standard.</p> <p><i>EPA SW-846, Method 6010B, Section 7.3</i></p>		
14.4	<p>Following daily calibration, an ICV, calibration blank, and CCV are analyzed.</p> <p><i>EPA SW-846, Method 6010B, Section 7.4</i></p>		

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14.5	<p>A calibration blank and either a CCV or an ICV are analyzed after every tenth sample and at the end of the sample run.</p> <p><i>EPA SW-846, Method 6010B, Section 7.4</i></p>		
14.6	<p>The check standard and calibration verification standard verifies that the instrument is within <math>\pm 10\%</math> of the calibration value with the relative standard deviation <math>&lt; 5\%</math> from replicate integrations.</p> <p><i>EPA SW-846, Method 6010B, Sections 7.4 and 8.6.1.2</i></p>		
14.7	<p>When the calibration verification falls outside the method or laboratory established criteria, the sample analysis is discontinued, the cause determined, and the instrument recalibrated. All samples following the last acceptable ICV, CCV, or check standard are reanalyzed.</p> <p><i>EPA SW-846, Method 6010B, Sections 7.4 and 8.6.1.2</i></p>		
14.8	<p>The analysis data of the calibration blank, check standard, ICV, and CCV are kept on file with the sample analysis data.</p> <p><i>EPA SW-846, Method 6010B, Section 7.4</i></p>		

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14.9	<p>The results of the calibration blank are within three times the IDL. If not, the analysis is repeated two more times and the results averaged. If the average is not within three standard deviations of the background mean, the analysis is terminated, the problem corrected, and the previous 10 samples are recalibrated and reanalyzed.</p> <p>(NOTE: If the blank is &lt; 1/10<sup>th</sup> the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration need not be performed before continuation of the run.)</p> <p><i>EPA SW-846, Method 6010B, Section 8.6.1.3</i></p>		
14.10	<p>Interference Check Solutions (ICS):</p> <p>ICSs are analyzed at the beginning of an analytical run. Results are within 20% of the true value.</p> <p><i>EPA SW-846, Method 6010B, Section 8.6.2</i></p>		
14.11	<p>Dilution Test:</p> <p>For each new matrix, a dilution test is performed and a five-fold dilution is analyzed on a sample containing analytes <math>\geq 100</math> times the IDL after dilution.. Results of the dilution agree within <math>\pm 10\%</math> of the original determination.</p> <p><i>EPA SW-846, Method 6010B, Sections 8.5 and 8.5.1</i></p>		

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14.12	<p>Post Digestion Spike:</p> <p>For each new matrix, an analyte spike is added to a portion of a prepared sample. If the recovery is not within 75-125%, the dilution test or MSA is performed. Results of the dilution are <math>\pm 10\%</math> of the original sample concentration. The spike is prepared at a concentration to yield a result between 10 and 100 times the IDL.</p> <p><i>EPA SW-846, Method 6010B, Sections 7.7 and 8.5</i></p>		

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<b>15.0</b>	<b>Multipoint Calibration Procedures - Cold Vapor Atomic Absorption Spectrometry (CVAA) Methods: Hg Analysis by EPA SW-846 Methods 7470A and 7471A</b>		
15.1	The multipoint calibration is performed using five standards and a blank.  <i>EPA SW-846, Method 7470A, Section 7.2; and EPA SW-846, Method 7471A, Section 7.3</i>		
15.2	The working standards are prepared fresh before the analysis.  <i>EPA SW-846, Method 7470A, Section 5.10; and EPA SW-846, Method 7471A, Section 5.8</i>		
15.3	If the laboratory performs EPA SW-846, Method 7471A, aqua regia is used for sample and standards preparation.  <i>EPA SW-846, Method 7471A, Sections 7.1 and 7.3</i>		
15.4	If the laboratory performs EPA SW-846, Method 7471A, triplicate 0.2 g portions of the untreated sample are used during sample preparation.  <i>EPA SW-846, Method 7471A, Section 7.1</i>		
15.5	Hot plates that are used are adjustable and capable of maintaining a temperature of 90° - 95° C.  <i>EPA SW-846, Method 7471A, Section 4.10</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>16.0</b>	<b>Quality Control (QC) of Instrument Gases</b>		
16.1	For Atomic Absorption methods, fuel and oxidant gases of the appropriate quality to ensure proper instrument performance are used, including, but not limited to, the following: <ul style="list-style-type: none"> <li>• acetylene (high purity);</li> <li>• compressed air (“clean and dry”);</li> <li>• nitrous oxide;</li> <li>• argon (commercial grade); and</li> <li>• nitrogen (commercial grade).</li> </ul> <i>EPA SW-846, Method 7000A, Section 5.5</i>		
16.2	For ICP/MS analyses, argon gas supply is high purity grade (99.99%)  <i>EPA SW-846, Method 6020, Section 4.1.2</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>17.0</b>	<b>Wet Chemistry Procedures Cyanide Distillation by EPA SW-846, Method 9010, and Analysis by EPA SW-846, Method 9014</b>		
17.1	The laboratory performs pretreatment on all samples suspected of containing cyanide amenable to chlorination.  <i>EPA SW-846, Method 9010B, Section 7.1</i>		
17.2	During the distillation process, if the sample is suspected to contain sulfide, bismuth nitrate is added to the sample.  <i>EPA SW-846, Method 9010B, Section 7.2.3 or Method 9010C, Section 7.2.3</i>		
17.3	During the distillation process, if bismuth nitrate has been added to the sample, or if the sample is suspected to contain nitrate or nitrite, sulfamic acid is added to the sample.  <i>EPA SW-846, Method 9010B, Section 7.2.4 or Method 9010C, Section 7.2.4</i>		
17.4	The laboratory uses six standards and a blank for the preparation of the multipoint calibration curve.  <i>EPA SW-846, Method 9014, Sections 7.3 1</i>		
17.5	At least one matrix spike sample is analyzed with each batch of 20 samples or less through the entire sample preparation and analytical process.  <i>EPA SW-846, Method 9010B, Section 8.5 or Method 9010C, Section 8.5; EPA SW-846, Method 9014, Section 8.5</i>		

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17.6	One replicate sample is analyzed with each batch of 20 samples or less.  <i>EPA SW-846, Method 9010B, Section 8.4 or Method 9010 C, Section 8.4</i>		
17.7	One reagent blank is analyzed with each analytical batch or per every 20 samples.  <i>EPA SW-846, Method 9010B, Section 8.2. or Method 9010C, Section 8.2</i>		
17.8	One check standard is analyzed with every analytical batch.  <i>EPA SW-846, Method 9010B, Section 8.3 or Method 9010C, Section 8.3; EPA SW-846, Method 9014, Section 8.3</i>		
17.9	The method of standard additions is used for analysis of all samples that suffer from matrix interference.  <i>EPA SW-846, Method 9010B, Section 8.7 or Method 9010C, Section 8.7; EPA SW-846, Method 9014, Section 8.6</i>		

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<b>18.0</b>	<b>Reactivity Determinations - Colorimetric Cyanide by EPA SW-846, Method 9012A and 9012B</b>		
18.1	Pre-treatment is performed on all samples suspected to be amenable to chlorination.  <i>EPA SW-846, Method 9012A, Section 7.1 or Method 9012B, Section 7.1</i>		
18.2	During distillation, bismuth nitrate is added to samples suspected to contain sulfides  <i>EPA SW-846, Method 9012A, Section 7.1.6 or Method 9012B, Section 7.1.6</i>		
18.3	During distillation, sulfamic acid is added to samples suspected to contain nitrites or nitrates, or samples to which bismuth sulfate was previously added.  <i>EPA SW-846, Method 9012A, Section 7.2.3 or Method 9012B, Section 7.2.3</i>		
18.4	A standard curve is prepared by plotting absorbance versus concentration.  <i>EPA SW-846, Method 9012A, Sections 7.4.4 and 7.5.2 or Method 9012B, Sections 7.4.4 and 7.5.2</i>		
18.5	The calibration curve is verified with every sample batch by analyzing a mid-range standard.  <i>EPA SW-846, Method 9012A, Section 8.2 or Method 9012B, Section 8.2</i>		

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<b>Item Number</b>	<b>Line of Inquiry</b>	<b>Status</b>	<b>Summary of Observations/Objective Evidence Reviewed Audit Notes</b>
18.6	A matrix spike sample is run for every 10 samples.  <i>EPA SW-846, Method 9012A, Section 8.3 or Method 9012B, Section 8.3</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>19.0</b>	<b>Reactivity Determinations - Titrimetric Sulfide – EPA SW-846, Method 9034</b>		
19.1	A reagent blank is analyzed once every 20 samples, or with each analytical batch, whichever is more frequent.  <i>EPA SW-846, Method 9034, Section 8.2</i>		
19.2	A check standard is analyzed once every 20 samples, or with each analytical batch, whichever is more frequent.  <i>EPA SW-846, Method 9034, Section 8.3</i>		
19.3	A matrix spike sample is analyzed once every 20 samples, or with each analytical batch, whichever is more frequent.  <i>EPA SW-846, Method 9034, Section 8.4</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>20.0</b>	<b>TCLP Extractions – EPA SW-846, Method 1311, Toxicity Characteristic Leaching Procedure (TCLP)</b>		
20.1	Agitation apparatus rotates at $30 \pm 2$ rpm. Verification of the rotation is documented and a frequency of the verification has been defined.  <i>EPA SW-846, Method 1311 Section 4; QSAS, Section 5.5.2.1</i>		
20.2	Zero Headspace Extraction (ZHE) units are leak checked after every extraction by monitoring the pressure gauge or by pressurizing the unit, submerging in water, and checking for bubbles. The laboratory has a corrective action procedure for handling leaking ZHE vessels.  <i>EPA SW-846, Method 1311, Section 4.2.1</i>		
20.3	ZHE Extract Collection Devices:  The final extract for ZHE is collected in Tedlar bags or glass, stainless steel, or polytetrafluoroethylene (PTFE) gas-tight syringes.  <i>EPA SW-846, Method 1311, Section 4.6</i>		
20.4	Extraction Fluid Reagents: <ul style="list-style-type: none"> <li>• Extraction fluid reagents are prepared using ASTM Type II water and reagent grade chemicals or equivalent.</li> <li>• The pH of extraction fluid 1 is <math>4.93 \pm 0.05</math> (4.88-4.98).</li> <li>• The pH of extraction fluid 2 is <math>2.88 \pm 0.05</math> (2.83-2.93).</li> <li>• The pH is checked prior to use.</li> </ul> <i>EPA SW-846, Method 1311, Section 5.7</i>		

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20.5	<p>Determination of Percent Solids:</p> <ul style="list-style-type: none"> <li>• If the percent solid is determined to be &lt; 0.5%, the sample is filtered through 0.6-0.8µm filter paper, solids are discarded, and the liquid is analyzed.</li> <li>• If the percent solid is determined to be ≥ 0.5% the liquid, if any, is separated from the solid phase and stored for later analysis</li> <li>• The solid phase is extracted using the amount of extraction fluid equal to 20 times the weight of the solid phase</li> <li>• Following extraction the liquid extract is filtered through 0.6-0.8µm glass fiber filter.</li> </ul> <p><i>EPA SW-846, Method 1311, Sections 2.1 and 2.2</i></p>		
20.6	<p>In determination of particle size reduction, particle size is &lt; 9.5mm.</p> <p><i>EPA SW-846, Method 1311, Section 7.1.3</i></p>		
20.7	<p>Determination of Appropriate Extraction Fluid:</p> <ul style="list-style-type: none"> <li>• For ZHE extraction, fluid #1 is used.</li> <li>• For TCLP extraction, fluid determination is based on pH. <ul style="list-style-type: none"> <li>• if pH &lt; 5, fluid #1 is used;</li> <li>• if pH &gt; 5, even after addition of HCl, fluid #2 is used.</li> </ul> </li> </ul> <p><i>EPA SW-846, Method 1311, Section 7.1.4</i></p>		
20.8	<p>The room temperature is maintained at 23 ± 2°C during agitation.</p> <p><i>EPA SW-846, Method 1311, Sections 7.2.11 and 7.3.12.3</i></p>		

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20.9	<p>TCLP and ZHE extraction devices are rotated at <math>30 \pm 2</math> rpm for <math>18 \pm 2</math> hours.</p> <p><i>EPA SW-846, Method 1311, Sections 7.2.11 and 7.3.12.3</i></p>		
20.10	<p>Immediately following TCLP extraction collection, the pH of the extract is recorded and the extract is preserved for analysis. Metals aliquots are acidified with nitric acid to <math>\text{pH} &lt; 2</math> and organic aliquots are refrigerated.</p> <p><i>EPA SW-846, Method 1311, Section 7.2.14</i></p>		
20.11	<p>The method blank for TCLP/ZHE is of the same extraction fluid as the sample(s), tumbled in an extraction vessel, and filtered in the same manner as the sample(s).</p> <p><i>EPA SW-846, Method 1311, Section 8.1</i></p>		
20.12	<p>A minimum of one matrix spike is performed for each batch. Matrix spikes are added after filtration of the TCLP/ZHE extract and prior to preservation.</p> <p><i>EPA SW-846, Method 1311, Sections 8.2 and 8.2.1</i></p>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
21.0	<b>Ignitability Test: EPA SW-846, Methods 1010, Pensky-Martin Closed Cup, and 1020A (Seta-Flash Closed Cup), ASTM D93-06 (Pensky-Martens), or ASTM D3278-96 (Seta-Flash)</b>		
21.1	The stirring and heat-up rates are monitored and documented.  <i>ASTM Methods (D93-06 and D3278-96)</i>		
21.2	The apparatus employed by the laboratory meets the requirements of either ASTM D93-94 (Pensky-Martens) or ASTM D3278-82 (Seta-Flash).  <i>ASTM Methods (D93-06 and D3278-96)</i>		
21.3	When samples contain non-filterable suspended solids or liquids that form surface films, the Pensky-Martens method is used.  <i>ASTM Methods (D93-06 and D3278-96)</i>		
21.4	Calibrations are verified using a fluid with a known flash point.  <i>QSAS, Section 5.6.3.2</i>		
21.5	The calibration verification is performed immediately after calibration, at the beginning of each analytical batch, and after the last site sample has been analyzed.  <i>QSAS, Section 5.5.10 c)</i>		
21.6	If the Seta Flash method is used, two measurements are obtained for each sample. A corrective action process is initiated if duplicate measurement results are not within established limits.( This requirement does not apply for the Pensky-Martens method.)  <i>ASTM Methods (D93-06)</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
22.0	<b>Corrosivity Determination: pH Electrometric Measurement by EPA SW-846, Method 9040B, and Method 9040C</b>		
22.1	<p>pH meters in operation at the time of the audit have the following characteristics:</p> <ul style="list-style-type: none"> <li>• temperature compensating, and</li> <li>• capable of measuring sample and reference standards temperatures</li> </ul> <p><i>EPA SW-846, Method 9040B, Sections 4.5 and 7.0 or EPA SW-846 Method 9040C, Sections 4.5 and 7.0</i></p>		
22.2	<p>Apparatus includes a magnetic stirrer with stirring bar made of non-reactive material, such as Teflon.</p> <p><i>EPA SW-846, Method 9040B, Section 4.4 or 9040C, Section 4.4</i></p>		
22.3	<p>The following calibration criteria are met:</p> <ul style="list-style-type: none"> <li>• calibrations are performed daily and each time the instrument is set up;</li> <li>• curve contains a minimum of two points that are at ~ 3 pH units apart;</li> <li>• verification is performed immediately after calibration and after the last site sample is analyzed.</li> </ul> <p><i>EPA SW-846, Method 9040B, Section 7.1 and Method 9040C, Section 7.1; QSAS, Section 5.5.10 c)</i></p>		

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23.0	<b>Corrosivity Towards Steel by EPA SW-846, Method 1110</b>		
23.1	Test apparatus includes: <ul style="list-style-type: none"> <li>• kettle or flask;</li> <li>• reflux condenser;</li> <li>• thermowell;</li> <li>• temperature regulating device;</li> <li>• heating device; and</li> <li>• specimen support system.</li> </ul> <i>EPA SW-846, Method 1110, Section 4.1 or Method 1110A, Section 4.1</i>		
23.2	The minimum ratio of volume of waste to area of the metal coupon to be used is 40 ml/cm <sup>2</sup> .  <i>EPA SW-846, Method 1110, Section 4.5.3 or Method 1110A, Section 4.5.3</i>		
23.3	Coupons are cleaned after immersion and prior to weighing.  <i>EPA SW-846, Method 1110, Section 7.6 or Method 1110A, Section 7.6</i>		
23.4	A blank is cleaned with the test coupons and its weight loss subtracted from that calculated for the test coupons.  <i>EPA SW-846, Method 1110, Section 7.7 or Method 1110A, Section 7.7</i>		
23.5	Duplicate samples are analyzed on a routine basis.  <i>EPA SW-846, Method 1110, Section 8.2 or Method 1110A, Section 8.2</i>		

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<b>24.0</b>	<b>Total Organic Carbon (TOC) by EPA SW-846, Method 9060A</b>		
24.1	Samples are stored at $4 \pm 2^{\circ}\text{C}$ . <i>EPA SW-846, Method 9060A, Section 6.2</i>		
24.2	Samples are homogenized. <i>EPA SW-846, Method 9060A, Section 7.1</i>		
24.3	The calibration of the instrument is verified by an independently prepared check standard every 15 samples. <i>EPA SW-846, Method 9060A, Section 8.3</i>		
24.4	Blanks are processed and analyzed with each batch. <i>EPA SW-846, Method 9060A, Section 8.2</i>		
24.5	One spike duplicate sample is analyzed for every 10 samples. <i>EPA SW-846, Method 9060A, Section 8.4</i>		
24.6	Samples are analyzed in quadruplicate as required by the method. Both the average and the range are reported. <i>EPA SW-846, Method 9060A, Section 7.6</i>		

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<b>25.0</b>	<b>Total Organic Halogens (TOX) by EPA SW-846, Methods 9020B and 9022</b>		
25.1	Purity of the activated carbon is verified before use.  <i>EPA SW-846, Method 9020B, Section 3.2;</i> <i>EPA SW-846, Method 9022, Section 3.2</i>		
25.2	For calibration, the repeatability of the method background is established each day by analysis of nitrite wash blanks.  <i>EPA SW-846, Method 9020B, Section 7.2.2;</i> <i>EPA SW-846 Method 9022, Section 7.2.2</i>		
25.3	For calibration, duplicate calibration standards and the blank standard are pyrolyzed each day before analysis.  <i>EPA SW-846, Method 9020B, Section 7.2.3</i>		
25.4	An independent calibration check standard is run after calibration.  <i>EPA SW-846, Method 9020B, Section 8.4</i>		
25.5	For neutron activation, calibration is performed daily using radioactive standards.  <i>EPA SW-846, Method 9022, Section 7.2.3</i>		
25.6	For neutron activation, an independent calibration check standard is run every 15 samples.  <i>EPA SW-846, Method 9022, Section 8.5</i>		
25.7	Samples are analyzed in duplicate.  <i>EPA SW-846, Method 9020B, Section 8.2</i>		

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25.8	At least two blanks are analyzed to establish the repeatability of the method background prior to sample analysis. Method blanks are analyzed between every eight analytical determinations.  <i>EPA SW-846, Method 9020B, Section 8.3</i>		
25.9	For neutron activation, at minimum, one blank per sample batch is run.  <i>EPA SW-846, Method 9022, Section 8.4</i>		
25.10	A matrix spike sample is run every 10 samples  <i>EPA SW-846, Method 9020B, Section 8.5; EPA SW-846, Method 9022, Section 8.6</i>		

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Item Number	Line of Inquiry	Status	Summary of Observations/Objective Evidence Reviewed Audit Notes
<b>26.0</b>	<b>Biological Oxygen Demand (BOD) Determinations - EPA Method 405.1 and Standard Methods (SM) 5210B</b>		
26.1	The dissolved oxygen meter calibration is verified before and after use. <i>QSAS, Section 5.5.10.c</i>		
26.2	The incubator or water bath is thermostatically controlled at 20° ± 1° C <i>SM 5210B, Section 2b</i>		
26.3	The dilution water is checked for purity. <i>SM 5210B, Section 4b</i>		
26.4	Seed control is performed to determine seed Dissolved Oxygen (DO) uptake. <i>SM 5210B, Section 4d.2</i>		
26.5	A dilution water blank is performed and the acceptance limit for DO uptake is not more than 0.2 mg/l. <i>SM 5210B, Section 4b</i>		
26.6	The samples are incubated for five days at 20° C in the dark. <i>EPA Method 405.1</i>		

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<b>27.0</b>	<b>Chemical Oxygen Demand (COD) Determinations – EPA Method 410.1; 410.2; 410.3 or 410.4 and Standard Methods (SM) 5220</b>		
27.1	The proper COD method has been selected.  <i>SM 5220A, Section 2</i>		
27.2	Interferences and limitations been adequately addressed.  <i>SM 5220A, Section 2; EPA Methods 410.1, 410.2, and 410.4, Section 4.0</i>		
27.3	If the samples require preservation, the sample is preserved with H <sub>2</sub> SO <sub>4</sub> to a pH of < 2.  <i>EPA Methods 410.1, 410.2, and 410.3, Section 3.0</i>		
27.4	If EPA Method 5220D is employed, the calibration curve contains at least five standards with COD equivalents to cover each concentration range.  <i>SM 5220D, Section 4c</i>		
27.5	A new calibration curve is prepared for each new lot of tubes or ampules, or when standards prepared differ by 5% or more from the standard calibration curve.  <i>SM 5220D, Section 4c</i>		

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28.0	<b>Total Phosphorus, Nitrate/Nitrite and Anions Determinations: EPA SW-846, Method 9056, Determination of Inorganic Anions by Ion Chromatography (chloride, fluoride, bromide, nitrate, nitrite, phosphate, and sulfate)</b>		
28.1	All reported results are within the working range of the instrument.  <i>QSAS, Section 5.5.2.2.1 h); EPA SW-846, Method 9056, Section 7.2.2.10</i>		
28.2	The calibration curve consists of, at a minimum, three concentration levels for each analyte of interest and a blank.  <i>EPA SW-846, Method 9056, Section 7.1.2; SM 1020B, Section 10.b</i>		
28.3	A midrange calibration standard is analyzed after every 10 injections. The instrument is recalibrated if the response changes more than 5%.  <i>EPA SW-846, Method 9056, Section 8.2; SM 1020B, Section 10 c</i>		
28.4	A duplicate sample is analyzed with every 10 samples.  <i>EPA SW-846, Method 9056, Section 8.3; SM 4500-PH, Section 4, as applies to Total Phosphorous</i>		
28.5	A linear calibration plot with an acceptable correlation coefficient, intercept, and slope is calculated and included with the raw data package.  <i>EPA SW-846, Method 9056, Section 7.3.3</i>		

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28.6	<p>The samples are filtered or allowed to settle prior to analysis. Samples that contain particles larger than 0.45µm and reagent solutions that contain particles larger than 0.20 µm require filtration to prevent damage to instrument columns and flow systems.</p> <p><i>EPA SW-846, Method 9056, Section 3.4; SM 4500-P B, Section 1</i></p> <p><i>(NOTE: This does not apply to reagent filtration.)</i></p>		
28.7	<p>The retention time for each analyte is within 10% of the calibration standard's average retention time (<i>anion analysis only</i>). Retention times are documented.</p> <p><i>EPA SW-846, Method 9056, Section 7.1.4</i></p> <p><i>(NOTE: This does not apply to Total Phosphorous.)</i></p>		

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<b>29.0</b>	<b>Kinetic Phosphorescence Analysis</b>		
29.1	Water samples are at least evaporated to dryness and wet-ashed.  <i>QSAS, Appendix D.1 DOE-11; ASTM Method D5174 – 02, Section 4.2</i>		
29.2	Reagent grade water shall be used to prepare samples, reagents, standards, and for the rinsing of glassware for the determination of low-level uranium by KPA.  <i>QSAS, Appendix D.1.7.a.4; D.1 DOE-11</i>		
29.3	For all low-level uranium analysis, prior to initial use, all new glassware with the exception of cuvettes used in KPA measurements, shall be soaked in hot 8M nitric acid for at least two hours and then in room temperature 8M nitric acid overnight.  <i>QSAS, Appendix D.1.7.a.3; D.1 DOE-11</i>		
29.4	For each sample, both the sample and sample-plus-spike shall be measured to demonstrate that there are no quenching interferences.  <i>QSAS, Appendix D.1.7.a.2; D.1 DOE-11</i>		

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29.5	<p>A new aliquot of the sample is prepared when the following documented criteria are not met:</p> <ul style="list-style-type: none"> <li>• phosphorescence lifetimes are &lt; 150<math>\mu</math>s or &gt; 350<math>\mu</math>s;</li> <li>• linear regression coefficient of the decay plot is &gt; 0.96 for samples where the measured concentration is greater than the RDL;</li> <li>• standard addition recovery is &lt; 90%</li> <li>• analyte concentration is not in the range of the calibration curve used;</li> <li>• reference ratio is &lt; 0.9 or &gt; 1.1; and</li> <li>• continuing calibration check standard is not within 10% of the known value.</li> </ul> <p><i>QSAS, Appendix D.1.7.b; D.1 DOE-11b</i></p>		
29.6	<p>The KPA is calibrated daily when in use. The order for performing instrument calibration is:</p> <ul style="list-style-type: none"> <li>• background,</li> <li>• calibration curve, and</li> <li>• calibration check standard.</li> </ul> <p><i>QSAS, Appendix D.1.7.c.1; D.1 DOE-11; ASTM Method D5174 - 02, Section 9.2</i></p>		

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29.7	<p>The following requirements are implemented for calibration curves:</p> <ul style="list-style-type: none"> <li>• the background is sufficiently low to permit attaining RDLs;</li> <li>• at least three standards are used for each calibration range;</li> <li>• the calibration range includes the range of the sampled to be measured; and</li> <li>• the linear regression coefficient (<math>R^2</math>) for the calibration curve is <math>&gt; 0.99</math></li> </ul> <p><i>QSAS, Appendix D; Section 1.7.c.2 and 1.7.c.3; D.1 DOE-11</i></p>		
29.8	<p>The following requirements are implemented for calibration check standards:</p> <ul style="list-style-type: none"> <li>• the performance check is performed using a separately prepared standard at a different concentration from the calibration standards;</li> <li>• the performance check is performed upon completion of calibration and subsequently after every 10 samples are analyzed; and</li> <li>• the relative bias of the calibration check standards is in the range -0.10 to +0.10.</li> </ul> <p><i>QSAS, Appendix D, Sections 1.7.d.1 and 1.7.d.2; D.1 DOE-11</i></p>		
29.9	<p>The LCS is measured in the same calibration range as the samples in a batch. If measurements are performed in more than one calibration range, then a separate LCS is prepared for each range.</p> <p><i>QSAS, Appendix D.1.7.c.4; D.1 DOE-11</i></p>		

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29.10	The method detection limit (MDL) is determined for each matrix analyzed and represents the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.  <i>QSAS, Appendix D, Section D. 1.7.b.1; D.1 DOE-11</i>		

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<b>30.0</b>	<b>Sample Dilutions</b>		
30.1	<p>Samples with concentrations that exceed the calibration range are diluted to fall within the range.</p> <p><i>QSAS, Section 5.5 DOE-6</i></p>		
30.2	<p>Samples are initially diluted so that all of the project-required quantitation limits can be met. Otherwise, all samples are reanalyzed at a lower dilution. All exceptions are discussed with the technical point of contact.</p> <p><i>QSAS, Section 5.5 DOE-6</i></p>		

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<b>31.0</b>	<b>Data Review</b>		
31.1	Data review is documented and records are maintained.  <i>QSAS, Section 4.12.2.5.2 h</i>		
31.2	Measures are in place (e.g., SOPS) to ensure that reported data are free from transcription and calculation errors.  <i>QSAS, Section 5.4.7.1</i>		
31.3	Records are maintained of data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.  <i>QSAS, Section 4.12.2.5.3 k</i>		

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### Record of Revision for Checklist 3 Data Quality for Inorganic Analyses

Revision Number	Effective Date	Reason for Revision	Line of Inquiry
4.3	11/2009	Changed reference for laboratory notebooks from Section 4.12 DOE-5 to DOE-6	1.3
4,3	11/2009	Changed reference for closure of laboratory notebooks from Section 4.12 DOE-5 to DOE-6	1.4
4.3	11/2009	Add requirement purity checks for reagents (QSAS Section 5.6 DOE-1)	7.6
5.3	11/2010	No Changers or Revisions	
5.3	1/23/2012	<b>Added to the Note section of the checklist: Fully document any deviation from the LOI or requirements of QSAS 2.7</b>	Page 1