NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION

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| LABORATORY NAME: | | CERT #: | |
| PRIMARY ANALYST: | | DATE: | |
| NAME OF PERSON COMPLETING CHECKLIST (PRINT): | | | |
| SIGNATURE OF PERSON COMPLETING CHECKLIST: | | | |

**Parameter: HEXAVALENT CHROMIUM (Aqueous)**
**Method: SW-846 Method 7196A**

Equipment:

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| | Spectrophotometer, 540 nm, 1 cm light path or longer | | Filter photometer- greenish-yellow, 540 nm, 1 cm light path or longer | | |

**PLEASE COMPLETE CHECKLIST IN INDELIBLE INK**
**Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice and in the column labeled SOP to indicate whether it is addressed in the SOP.**

| | GENERAL | LAB | SOP | EXPLANATION |
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| 1 | Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 2H .0805 (a) (7)]<br><br>**Date:** | | | Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made.<br>Verify proper method reference. During review notate deviations from the approved method and SOP. |
| 2 | Are all revision dates and actions tracked and documented? [15A NCAC 2H .0805 (a) (7)] | | | Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents. |
| 3 | Is there North Carolina data available for review? | | | If not, review PT data |
| | **PRESERVATION and STORAGE** | **LAB** | **SOP** | **EXPLANATION** |
| 4 | Are samples iced to above freezing but ≤6°C during shipment and storage? [SW-846, Chapter Three, Inorganic Analytes, Table 3-2] | | | **SW-846 7196A states**: To retard the chemical activity of hexavalent chromium, the samples and extracts should be stored at 4°C until analyzed.<br>**SW-846 Chapter 3, Table 3-2** however, lists ≤6°C.<br>**We will allow ≤6°C.** |
| 5 | Are samples analyzed within 24 hours of collection? [SW-846, Chapter Three, Inorganic Analytes, Table 3-2] | | | **SW-846 7196A states**: The maximum holding time prior to analysis of the samples or extracts is 24 hr.<br>**SW-846 Chapter 3, Table 3-2** also lists 24 hours as the hold time for aqueous samples. |
| | **PROCEDURE – Calibration** | **LAB** | **SOP** | **EXPLANATION** |
| 6 | What is your laboratory's reporting limit? [15A NCAC 2H .0805 (a) (7) (H)]<br><br>**ANSWER:** | | | One of the standards shall have a concentration equal to or less than the laboratory's lowest reporting concentration for the parameter involved. |
| 7 | List the values of standards used for the daily calibration: [15A NCAC 2H .0805 (a) (7) (H)(v)]<br><br>**ANSWER:** | | | **SW-846 7196A states**: Accordingly, pipet a chromium standard solution in measured volumes into 250 ml beakers or conical flasks to generate standard concentrations ranging from 0.5 to 5 mg/L Cr(VI) when diluted to the appropriate volume.<br>**15A NCAC .0805 (a) (7) (H) (v)**: For analytical procedures requiring analysis of a series of standards, the concentrations of these standards shall bracket the range of the |

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| | | | | sample concentrations measured. One of the standards shall have a concentration equal to or less than the laboratory's lower reporting concentration for the parameter involved. For colorimetric analyses, a series of five or more non-zero standards for a curve prepared every 12 months or three or more non-zero standards for curves established each day, or standards as set forth in the analytical procedure, shall be analyzed to establish a calibration curve. |
| 8 | Are calibration standards treated with the same procedure as the samples? [SW-846 7196A, Section 7.2.1 and 7.2.2] | | | **Section 7.2.1**: To compensate for possible slight losses of chromium during digestion or other operations of the analysis, treat the chromium standards by the same procedure as the sample.<br>**Section 7.2.2**: Develop the color of the standards as for the samples. Transfer a suitable portion of each colored solution to a 1-cm absorption cell and measure the absorbance at 540 nm. |
| 9 | Is a reagent blank used to correct the absorbance readings of the standards by subtracting the reagent blank absorbance? [SW-846 7196A, Section 7.2.2] | | | As reference, use reagent water. Correct the absorbance readings of the standards by subtracting the absorbance of a reagent blank carried through the method. |
| 10 | Is a calibration curve constructed by plotting corrected absorbance values against mg/L of Cr(VI)? [SW-846 7196A, Section 7.2.2] | | | |
| 11 | Do calibration curves meet a minimum correlation coefficient of 0.995? [NC WW/GW LC Policy] | | | When linear regression is used, use the minimum correlation coefficient specified in the method. If the minimum correlation coefficient is not specified, then a minimum value of 0.995 (or a coefficient of determination, $r^2$, of 0.99) is required. |
| | **PROCEDURE – Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
| 12 | What volume of sample is analyzed? [SW-846 7196A, Section 7.1]<br>**ANSWER:** | | | Transfer 95 ml of the extract to be tested to a 100-mL volumetric flask. |
| 13 | Is 2.0 mL diphenylcarbazide solution added to the sample and mixed? [SW-846 7196A, Section 7.1] | | | Add 2.0 ml diphenylcarbazide solution and mix. |
| 14 | Is $H_2SO_4$ solution added to give a pH of 2 ± 0.5 s.u., then diluted to 100mL? [SW-846 7196A, Section 7.1] | | | Add $H_2SO_4$ solution to give a pH of 2 ± 0.5, dilute to 100 ml with reagent water, |
| 15 | Is this pH adjustment documented? [15A NCAC 2H .0805 (a) (7) (F) (x)]] | | | All laboratories shall use printable laboratory benchsheets. Certified Data shall be traceable to the associated sample analyses and consist of: sample preparation, where applicable. |
| 16 | After pH adjustment, are samples allowed to let stand 5 to 10 minutes for full color development? [SW-846 7196A, Section 7.1] | | | let stand 5 to 10 min for full color development. |
| 17 | Is absorbance measured at 540 nm? [SW-846 7196A, Section 7.1] | | | Transfer an appropriate portion of the solution to a 1-cm absorption cell and measure its absorbance at 540 nm. |
| 18 | Is a reagent blank used to correct the absorbance readings of the samples by subtracting the reagent blank absorbance? [SW-846 7196A, Section 7.1] | | | Use reagent water as a reference. Correct the absorbance of a blank carried through the method. |
| 19 | If the sample is turbid after dilution to 100mL, is an absorbance reading taken before adding the carbazide reagent and subtracted from the final absorbance measurement? [SW-846 7196A, Section 7.1] | | | Note: if the solution is turbid after dilution to 100mL id Step 7.1, above, take an absorbance reading before adding the carbazide reagent and correct the absorbance reading of the final colored solution by subtracting the absorbance measured previously. |
| | **QUALITY ASSURANCE** | **LAB** | **SOP** | **EXPLANATION** |
| 20 | Are samples diluted when they are more concentrated than the highest calibration standard? [SW-846 7196A, Section 8.2] | | | Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve. |

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| 21 | Is a method/reagent blank analyzed with each sample batch? [SW-846 7196A, Section 8.3] | | | Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring. |
| 22 | Is the reagent/method blank concentration less than or equal to ½ the concentration of the lowest calibration standard or as otherwise specified by the method? [15A NCAC 2H .0805 (a) (7) (H) (i)] | | | The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method. |
| 23 | What corrective action is taken if the reagent/method blank is not acceptable? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. |
| 24 | Is the initial calibration verified by analyzing a **second source standard**? [15A NCAC 2H .0805 (a) (7) (H) (ii)]<br><br>**List acceptance criterion and value of standard used:** | | | Laboratories shall analyze one known second source standard to verify the accuracy of standard preparation if an initial calibration is performed and in accordance with the referenced method requirements thereafter. |
| 25 | What corrective action does the laboratory take if the calibration verification standard is outside established control limits? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. |
| 26 | Is the calibration verified by analyzing a calibration blank initially, after every 10th sample and at the end of the run? [15A NCAC 2H .0805 (a) (7) (H)] | | | A calibration blank and calibration verification standard shall be analyzed prior to sample analysis, after every tenth sample, and at the end of each sample group, unless otherwise specified by the method, to check for carryover and calibration drift. |
| 27 | Is the calibration blank concentration less than or equal to ½ the concentration of the lowest calibration standard or as otherwise specified by the method? [15A NCAC 2H .0805 (a) (7) (H) (i)] | | | The concentration of reagent, method, and calibration blanks shall not exceed 50 percent of the lowest reporting concentration or as otherwise specified by the reference method. |
| 28 | What corrective action is taken if the calibration blank results are greater than one-half the reporting level? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. |
| 29 | Does the laboratory analyze an independently prepared check standard after every 15 samples to verify calibration? [SW-846 7196A, Section 8.4] **List value of standard used for second source.**<br><br>**ANSWER:** | | | Verify calibration with an independently prepared check standard every 15 samples. |
| 30 | What is the acceptance criterion of the check standard? [15A NCAC 2H .0805 (a) (7) (A)]<br><br>**ANSWER:** | | | **SW-846 7196A does not prescribe an acceptance criterion.**<br>NC Rule: Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses. Each laboratory shall calculate and document the precision and accuracy of all quality control analyses with each sample set. When the method of choice specifies performance acceptance criteria for precision and accuracy, and the laboratory chooses to develop laboratory-specific limits, the laboratory-specific limits shall not be less stringent than the criteria stated in the approved method. |

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| 31 | What corrective action is taken if the check standard recovery is outside of established control limits? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. |
| 32 | Is a lower reporting limit standard analyzed or back-calculated with each analysis? [15A NCAC 2H .0805 (a) (7) (H)] | | | Laboratories shall analyze or back-calculate a standard at the same concentration as the lowest reporting concentration each day samples are analyzed. |
| 33 | What is the acceptance criterion of the lower reporting limit standard? [15A NCAC 2H .0805 (a) (7) (A)]<br><br>**ANSWER:** | | | Establish laboratory control limits<br><br>Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses. |
| 34 | Is a Laboratory Fortified Matrix (LFM) analyzed for each sample matrix analyzed and at least every 20 samples? [SW-846 7196A Section 7.3.1][NC WW/GW LC Policy] | | | For every sample matrix analyzed, verification is required to ensure that neither a reducing condition nor chemical interference is affecting color development. This must be accomplished by analyzing a second 10-mL aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI).<br>Note: wastewater, groundwater and surface water are considered different matrix types. |
| 35 | How is the LFM (spike) prepared? [NC WW/GW LC Policy] [SW-846 7196A Section 7.3.1]<br><br>**ANSWER:** | | | See Matrix Spike Technical Assistance document. Use the same solution as the LFB for the LFM to evaluate bias attributed to matrix and accuracy of the LFM.<br>**SW-846 7196A Section 7.3.1** states: This must be accomplished by analyzing a second 10-mL aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstances should the increase be less than 30 µg Cr(VI)/liter. |
| 36 | What is the accuracy acceptance criterion for the LFM? [SW-846 7196A Section 7.3.1]<br><br>**ANSWER:** | | | To verify the absence of an interference, the spike recovery must be between 85% and 115%. |
| 37 | What corrective action does the laboratory take if the LFM results are outside of established control limits for accuracy? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | **15A NCAC 2H .0805 (a) (7) (B)**: If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.**SW-846 7196A Section 7.3.3 states**: If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed.<br>**SW-846 7196A Section 7.3.4 states**: If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used. |
| 38 | Are acidic extracts that yield spike recoveries of less than 85% retested to determine if the low spike recovery is due to the presence of residual reducing agent? [SW-846 7196A, Section 7.4] | | | Acidic extracts that yield recoveries of less than 85% should be retested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the extract alkaline (pH 8.0-8.5) using 1N sodium hydroxide and then respiking and analyzing. If a spike recovery of 85-115% is obtained in the alkaline aliquot of an acidic |

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| | | | | extract that initially was found to contain less than 5 mg/L Cr(VI), one can conclude that the analytical method has been verified. |
| 39 | Is a laboratory-fortified matrix duplicate or sample duplicate analyzed for every 10 samples? [SW-846 7196A, Section 8.4] | | | Run one matrix spike replicate or one replicate sample for every ten samples. |
| 40 | What is the precision acceptance criterion for LFMDs or sample duplicates? [15A NCAC 2H .0805 (a) (7) (A)]<br><br>**ANSWER:** | | | Unless specified by the method or this Rule, each laboratory shall establish performance acceptance criteria for all quality control analyses. |
| 41 | What corrective action does the laboratory take if the LFMD or sample duplicate results are outside of established control limits for precision? [15A NCAC 2H .0805 (a) (7) (B)]<br><br>**ANSWER:** | | | If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. |
| 42 | Is the data qualified on the Discharge Monitoring Report (DMR) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)] | | | If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such. If data qualifiers are used to qualify samples not meeting QC requirements, the data may not be useable for the intended purposes. It is the responsibility of the laboratory to provide the client or end-user of the data with sufficient information to determine the usability of the qualified data. |

Additional Comments:

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Inspector: _____ Date: _____