NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

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

Parameter: Vector Attraction Reduction

Method: Option 6: Addition of Alkali [40 CFR 503.33(b)(6)]

Pathogens and Vector Attraction in Sewage Sludge EPA/600/R-22/194

Equipment:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | pH meter with:  temperature-compensation adjustment  low-sodium glass electrode (recommended) |  | pH Buffers |  | pH Buffers |
|  |
|  |
|  | Temperature measuring device (if separate from pH meter/probe) |  | Value:\_\_\_\_\_\_\_ Exp: \_\_\_\_\_\_\_ |  | Value:\_\_\_\_\_\_\_ Exp: \_\_\_\_\_\_\_ |
|  | 0.01 M CaCl2 (if necessary) |  | Value:\_\_\_\_\_\_\_ Exp: \_\_\_\_\_\_\_ |  | Value:\_\_\_\_\_\_\_ Exp: \_\_\_\_\_\_\_ |

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| **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** |
|  | Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 02H .0805 (g) (4)]  **Date:** |  |  | Standard Operating Procedure documentation shall state the effective date of the document and shall be reviewed every two years and updated if changes in procedures are made.  Verify proper method reference. During review notate deviations from the approved method and SOP. |
|  | Are all review/revision dates and procedural edits tracked and documented? [15A NCAC 02H .0805 (g) (4)] |  |  | Each laboratory shall have a formal process to track and document review dates and any revisions made in all Standard Operating Procedure documents. |
|  | Is there North Carolina data available for review? |  |  |  |
|  | Are the following items documented with each analysis? [15A NCAC 02H .0805 (g) (2)] |  |  |  |
|  | The method or SOP reference |  |  |  |
|  | Laboratory identification |  |  |  |
|  | Instrument identification |  |  |  |
|  | Sample collector |  |  |  |
|  | Signature or initials of the analyst |  |  |  |
|  | Date of sample collection |  |  |  |
|  | Time of sample collection |  |  |  |
|  | Date of sample analysis |  |  |  |
|  | Time of sample analysis |  |  | One time may be documented for sample collection and analysis if there is documentation showing that the analysis is performed *in situ*, or immediately on the sample site. |
|  | Sample identification |  |  |  |
|  | Proper units of measure |  |  |  |
|  | Temperature of sample [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | pH of sample [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Final value to be reported (temperature adjusted pH value) |  |  |  |
|  | Facility name or permit number [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Parameter analyzed [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Meter calibration time [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | True value of the buffers used for calibration [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | True value of the buffer check standard [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Value obtained for the buffer check standard [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Acceptance criterion for check buffers (i.e., ± 0.1 S.U.) [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Evaluation of the check buffers (check box acknowledging that it passed is acceptable) [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | True value and value obtained for the post-analysis calibration verification [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  | If applicable. A post-analysis calibration verification using the check standard buffer must be analyzed at the end of the run any time the meter is transported by vehicle to another location after calibration. |
|  | Indication of when the post-analysis calibration verification was performed (e.g., time of analysis, end-of-day analysis, etc. [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  | If applicable |
|  | **PRESERVATION and STORAGE** | **LAB** | **SOP** | **EXPLANATION** |
|  | Are samples analyzed as soon as possible? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | **PROCEDURE – Meter Calibration** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is the pH meter equipped with temperature-compensation adjustment? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  | A low-sodium glass electrode is recommended to prevent low-biased results. |
|  | How is the pH meter calibrated? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali]  **List buffers used:** |  |  | Calibrate per manufacturer’s instructions. Calibration must include at least two buffers. The meter calibration must be verified with a third standard buffer solution (i.e., check buffer) prior to sample analysis. The calibration and check standard buffers must bracket the range of the samples being analyzed (i.e., 12 ± 0.5 S.U.). |
|  | Is the meter calibration documented each day analyses are performed? [15A NCAC 02H .0805 (g) (3)] |  |  | A record of instrument calibration or calibration verification shall be documented and available for inspection upon request. |
|  | Is the sample temperature measured with an NIST traceable or NIST-verified temperature-measuring device? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | **PROCEDURE – Sample Preparation** | **LAB** | **SOP** | **EXPLANATION** |
|  | What is the sludge type (e.g., liquid, dewatered cake, etc.)?  **Answer:** |  |  |  |
|  | If the sludge is a dewatered cake, is a slurry prepared by adding 20 mL of distilled water (containing 0.01 M CaCl2) to 10 g of sludge cake and mixing occasionally for half an hour while waiting for the sample to clarify if necessary? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | **PROCEDURE – Sample Analysis** | **LAB** | **SOP** | **EXPLANATION** |
|  | Is the temperature-corrected pH value after addition of alkali documented to be ≥ 12 S.U.? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Is the temperature-corrected pH value documented to be ≥ 12 S.U. after 2 hours without the addition of more alkali? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | If the temperature-corrected pH of the sample after 2 hours is < 12 S.U., is alkali added to the sludge to bring the pH back up to at least 12 S.U. and the process repeated? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Is the temperature-corrected pH value documented to be ≥ 11.5 S.U. after an additional 22 hours without the addition of more alkali? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | If the temperature-corrected pH of the sample after additional 22 hours is < 11.5 S.U., is alkali added to the sludge to bring the pH back up to at least 12 S.U. and the 24-hr process repeated? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Is the proper correction factor applied to the measured pH values if the sample is not 25 °C? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  | Either sewage sludge samples may be taken and heated or cooled to 25°C or results can be adjusted based on the ambient temperature where pH is measured and the following calculation:  Note: Temperature compensation devices on pH meters correct only for variations in the conductance of pH probes, and not for the variability in solution concentration. Therefore, the temperature correction noted above must be applied to pH measurements, even though a pH meter with temperature compensation is used. |
|  | **QUALITY ASSURANCE** | **LAB** | **SOP** | **EXPLANATION** |
|  | Does the Compliance Temperature-Measuring Device have a valid (i.e., not expired) NIST certificate? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | If the Compliance Temperature-Measuring Device does not have a valid NIST certificate, is the device checked against a Reference Temperature-Measuring Device initially before use? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  | This may be performed by a contract laboratory. Maintain comparison data and documentation of NIST traceable temperature-measuring device information listed below. |
|  | Are all Compliance Temperature-Measuring Devices checked against a Reference Temperature-Measuring Device every 12 months after first use or after certificate expiration, whichever comes first? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | Is the Reference Temperature-Measuring Device only used to verify the calibration of other devices? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | Is the Reference Temperature-Measuring Device within its expiration date? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | Is the serial number, stated accuracy (must be ± 0.5 °C) and expiration date of the Reference Temperature-Measuring Device used in the comparison documented? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  | Reference Temperature-Measuring Device  Serial Number: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_  Stated accuracy: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_  Expiration date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ |
|  | Is the Compliance Temperature-Measuring Device checked at two temperatures that bracket the range of compliance samples? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | Is the date of the verification, the serial number of the Compliance Temperature-Measuring Device and all four temperatures documented? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  | Compliance Temperature-Measuring Device  Verification Date:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_  Serial Number:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_  Temperatures:  Compliance device Reference device  \_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_  \_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_ |
|  | Do the readings from both devices agree within 0.5°C? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | If the Compliance Temperature-Measuring Device does not agree within ±0.5°C, is the device taken out of use for compliance temperature monitoring? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  | If the device reading differs by more than 0.5°C from the Reference Temperature-Measuring Device, it may not be used. No temperature correction factors are allowed for this parameter. |
|  | Is the verification documentation kept on file for 5 years? [NC WW/GW LCB Approved Procedure for the Analysis of Temperature] |  |  |  |
|  | Is a check buffer analyzed after calibration and before sample analysis? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Is a post-analysis check buffer analyzed at the end of the run any time the meter is transported by vehicle to another location after calibration? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Are the check buffers (pre- and post-analysis) within ±0.1 S.U. of the true value? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Are corrective actions taken if the standard buffer checks do not read within ±0.1 S.U. of the true value? [NC WW/GW LCB Approved Procedure for the Analysis of VAR Option 6: Addition of Alkali] |  |  |  |
|  | Is the data qualified on the EPA Biosolids Annual Report (NPDES Form 6100-035) or client report if Quality Control (QC) requirements are not met? [15A NCAC 02H .0805 (e) (5)] |  |  | Reported data associated with quality control failures, improper sample collection, holding time exceedances, or improper preservation shall be qualified as such. |

Additional Comments:

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Inspector: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_Date:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_