# NORTH CAROLINA WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION

## APPROVED PROCEDURE FOR THE ANALYSIS OF VECTOR ATTRACTION REDUCTION (VAR): OPTION 12 – Raising the pH of Domestic Septage

This document provides an approved procedure for the analysis of VAR: Option 12 for compliance monitoring per 15A NCAC 2H .0805 (a) (7) and (g) (4).

**Holding Time:**

- Samples must be analyzed within 15 minutes of collection.

**General Information:**

- This option applies only to domestic septage applied to agricultural land, forest, a reclamation site, or surface disposal site. The method is geared to the practicalities of the use or disposal of domestic septage, which is typically treated by lime addition in the domestic septage hauling truck. Every container (truckload) must be monitored to demonstrate that it meets the requirement. Alkali may be added to the septic tank or to the septic tank truck while domestic septage is being pumped from a septic tank into the tank truck. The agitation from the high velocity incoming stream of septage distributes the lime and mixes it with the domestic septage. The pH is measured when the truck loading is complete. The truck then moves to the use or disposal site. Agitation generated by the motion of the truck may help in mixing and distributing the lime however, supplemental mixing in the tank may be needed.
- When pH is measured with alkaline pH paper, it must be micro-range type pH paper that gives different colors for each 0.3 S.U. change in pH. Since pH paper can age and become contaminated, samples must be measured with pH paper from two different containers. If the two measurements do not agree, compare with pH paper from a third container and reject the one that disagrees with the others. If the pH is below 12 S.U., either initially or after 30 minutes, more lime must be added and mixed in. After an additional waiting period of at least 30 minutes, the pH must again be measured to ensure that it is greater than 12 S.U.
- When the pH measurement is made with a pH meter, it must be equipped with an automatic temperature compensator and a low-sodium glass electrode.
- Vector attraction is reduced if the pH is raised to at least 12 S.U. through alkali addition and maintained at 12 S.U. or higher for 30 minutes without adding more alkali.

**Meter Calibration:**

- Use a pH meter accurate and reproducible to 0.1 S.U. (as demonstrated daily by acceptable performance of a check standard buffer) with a range of 0 to 14 S.U. and equipped with an automatic temperature compensator and low-sodium electrode.

- The pH meter must be calibrated each day of analysis. Calibration must include at least two standard buffers. The meter calibration must be verified with a third standard buffer (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.). A portion of the standard buffer is not to be used for more than one calibration. Discard any used buffer portions. Do not pour unused portions back into the original bottle.

  Note: High pH buffers absorb $CO_2$ and acidic vapors from the atmosphere which alters the pH. Storing with minimal headspace will reduce these effects.

- All check standard buffers (pre- and post-analysis) must read within ±0.1 S.U. to be acceptable. If the meter verification does not read within ±0.1 S.U., corrective actions must be taken before any samples are analyzed. Possible corrective actions may be found at the end of this document.

**Procedure:**

- Alkali is mixed with septage to raise the pH to at least 12 S.U. A representative sample is analyzed to verify the pH.
- The pH is measured again after at least 30 minutes at the use or disposal site to assure it is still ≥ 12 S.U. without the addition of more alkali.
- If domestic septage is not applied within 15 minutes of the final acceptable pH measurement, the pH must be retested, and additional alkali be added to the domestic septage to raise the pH to 12 S.U. for an additional 30 minutes, if necessary.
- If samples are not analyzed at 25°C, the results must be adjusted based on the septage temperature at the same location and depth at which the pH is measured and the following calculation [a quick reference for correction factors can be found in Attachment A]:

$$\text{Correction Factor} = \frac{0.03 \text{ pH units} \times (T_{meas} - 25°C)}{1.0°C}$$

$$\text{Actual pH} = \text{Measured pH} \pm \text{the Correction Factor}$$

Where $T_{meas}$ is the septage temperature at the same location and depth at which the pH is measured.

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.

**Documentation:**

The following must be documented in indelible ink whenever sample analysis is performed:

1. Date and time of sample collection. If pH measurement is made *in situ*, it must be notated.
2. Date and time of sample analysis
3. Facility name or permit number, and sample site (ID or location)
4. Collector's/analyst's name or initials
5. True value of the buffers used for calibration, if applicable
6. True value of the buffer check standard, if applicable
7. Value obtained for the buffer check standard (within ±0.1 S.U.), if applicable
8. True value and value obtained for the post-analysis calibration verification, if applicable
9. Indication of when the post-analysis calibration verification was performed (e.g., time of analysis, end-of-day analysis, etc.), if applicable
10. Temperature of sample
11. Measured pH of sample
12. Actual pH of Sample
13. Units of measure
14. Traceability for chemicals, reagents, standards and consumables
15. Instrument identification (serial number preferred)
16. Parameter analyzed
17. Method reference or Standard Operating Procedure
18. Data qualifiers, when necessary
19. Equipment maintenance (recommended)

**TROUBLESHOOTING:**

If the check buffer does not read within ±0.1 S.U., the lab should first try pouring a new aliquot of the check buffer and reading it again. If it still does not read within ±0.1 S.U., the meter must be recalibrated. Possible corrective actions include: check the meter calibration procedure, refer to the trouble shooting section in the instrument manual, and check the buffers. If, after recalibration, the check buffer does not read within ±0.1 S.U., the meter and/or probe operation may be suspect and may require servicing. If the laboratory does not have a back-up meter/electrode, or another meter/electrode cannot be procured, it is recommended that the

lab report the measured pH results with a qualifier that indicates the value is estimated.

The true values of buffers are temperature dependent. Check the manufacturer's label on the bottle for the true value.

This document was prepared using Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003), Code of Federal Regulations, Title 40, Part 136; Federal Register Vol. 82, No. 165, August 28, 2017; Table II and EPA SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; 3rd Edition, Methods 9040 C and 9045 D as references.

Attachment A: pH Temperature Correction Factors at Varying Temperatures

| Temp °C | Correction Factor |
|---|---|
| 40 | Plus 0.45 |
| 39 | Plus 0.42 |
| 38 | Plus 0.39 |
| 37 | Plus 0.36 |
| 36 | Plus 0.33 |
| 35 | Plus 0.30 |
| 34 | Plus 0.27 |
| 33 | Plus 0.24 |
| 32 | Plus 0.21 |
| 31 | Plus 0.18 |
| 30 | Plus 0.15 |
| 29 | Plus 0.12 |
| 28 | Plus 0.09 |
| 27 | Plus 0.06 |
| 26 | Plus 0.03 |
| 25 | 0.00 |
| 24 | Minus 0.03 |
| 23 | Minus 0.06 |
| 22 | Minus 0.09 |
| 21 | Minus 0.12 |
| 20 | Minus 0.15 |
| 19 | Minus 0.18 |
| 18 | Minus 0.21 |
| 17 | Minus 0.24 |
| 16 | Minus 0.27 |
| 15 | Minus 0.30 |
| 14 | Minus 0.33 |
| 13 | Minus 0.36 |
| 12 | Minus 0.39 |
| 11 | Minus 0.42 |
| 10 | Minus 0.45 |

Actual pH = Measured pH +/- the Correction Factor at the Measured Temperature