

# NORTH CAROLINA WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

## APPROVED PROCEDURE FOR THE ANALYSIS OF SETTLEABLE RESIDUE

This document provides an approved procedure for the volumetric analysis of Settleable Residue for compliance monitoring per 15A NCAC 02H .0805 (a) (7) and (g) (4).

Settleable Residue is considered a method-defined parameter per the definition in the Code of Federal Regulations, Part 136.6, Section (a) (5). This means that the method may not be modified to reduce the sample volume per Part 136.6, Section (b) (3).

### Holding Time:

- Samples must be analyzed within 48 hours of collection (40 CFR Part 136 Table II).
- Samples must be stored above freezing and  $\leq 6^{\circ}\text{C}$ , if not analyzed within 15 minutes of collection.

### General Information:

- Collect a minimum of 1 liter of sample.
- Ensure the Imhoff Cone is clean and dry prior to analysis.
- Sample duplicates are not a required quality control element for Field parameters.

### Analysis:

- Pour exactly 1 liter of well mixed sample into the Imhoff Cone and allow the sample to settle for 45 minutes.
- After 45 minutes, gently stir on the sides of the cone or gently spin the cone.
- Allow to settle for another 15 minutes.
- Do not allow the sample to settle for more than 1 hour.
- Measure the volume of residue in the bottom of the cone in mL. Subtract pockets of liquid from the final volume of residue reported and document calculation on the benchsheet. Results which fall below the lowest graduation on the Imhoff Cone (this is generally in the 0.1 to 1.0 mL/L range), must be reported as less than that value. For example, if the lowest graduation is 0.1 mL, then results which fall below 0.1 mL must be reported as "<0.1 mL/L" when 1 liter of sample is analyzed.
- If the laboratory does not receive 1 liter of sample and another sample cannot be collected, the results and reporting limit must be calculated based upon the volume analyzed and qualified. Calculate results as follows:

$$\frac{(\text{mL of residue}) \times 1000}{(\text{mL of sample})} = \text{Result in mL/L}$$

### Documentation:

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

1. Date and time of sample collection
2. Date and time of sample analysis (must document start and end times) to verify the 48-hour holding and the 1-hour settling times are met
3. 45-minute stir time (use of a check box is acceptable)
4. Permitted facility name or permit number, and sample site (ID or location)
5. Collector's/analyst's name or initials
6. Volume of sample analyzed
7. Final value to be reported
8. Units of measure
9. All data must be reported in mL/L
10. Parameter analyzed
11. Method reference
12. Data qualifier(s), when applicable

Ref: Standard Methods 2540 F - 2020

Refer to the NC DEQ WW/GW LCB website for additional resources.