*Name of Facility*

Standard Operating Procedure

for the Analysis of

Settleable Residue

Method: SM 2540 F-2015

Effective Date:

Supervisor Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date:\_\_\_\_\_\_\_\_\_\_

Supervisor Name (print):\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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*Blue text is replaceable instructional language to be customized for your facility.*

1. Summary of Method
   1. 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 Section (b) (3).
   2. One liter of water is allowed to settle for 1 hour in an Imhoff Cone and the amount of residue which settles to the bottom of the cone is visually determined.

* 1. *State what type of samples are analyzed, e.g., wastewater effluent, ground water monitoring well, etc.*
  2. *State what the smallest gradation of the cone is, which will be the reporting limit*

1. Definitions
   1. mL/L: Units for the measurement of settleable residue.
   2. NC WW/GW LCB: North Carolina Wastewater Groundwater Laboratory Certification Branch
   3. *Add any acronyms or terms used by your facility that may need to be defined*
2. Safety and Waste Handling
   1. *Items that would be included in this section are things such as:*

* *Precautionary measures (list here and at the critical steps in the procedure)*
* *Personal protective equipment (e.g., gloves, eye protection, lab coat, work in a hood, etc.)*

1. Apparatus and Equipment
   1. Imhoff Cone
   2. Graduated cylinder
   3. *Glass rod (remove if not using to stir)*
   4. *Timer (when used)*
2. Interferences
   1. N/A
3. Sample Collection, Preservation and Holding Time
   1. A minimum of 1 liter is collected.
   2. *State what type of container samples are collected in. Samples must be collected in glass, fluoropolymer or polyethylene containers.*
   3. When sample analysis is not begun within 15 minutes, the sample must be transported and stored above freezing and ≤ 6°C.

* 1. The holding time is 48 hours.

1. Procedure
   1. Ensure the Imhoff Cone is dry prior to analysis. Mix the sample well and pour exactly 1 liter into the Imhoff Cone.
   2. Set timer and allow the sample to settle for 45 minutes.
   3. At 45 minutes, *gently stir the sides of the cone with a glass rod (or) gently spin the cone (remove the procedure that is not done).*
   4. Set timer and allow the sample to settle for an additional 15 minutes. Do not allow sample to settle for more than 1 hour total.
   5. Document the observed volume of residue in the bottom of the cone in mL. If the settled matter contains pockets of liquid between large settled particles, estimate volume of pockets of liquid and subtract from the volume of settled solids. NOTE: Where a separation of settleable and floating material occurs, do not estimate the floating material as settleable matter.

1. Documentation
   1. All documentation errors shall be corrected by drawing a single line through the error so that the original entry remains legible. Entries shall not be obliterated by erasures or markings. Wite-Out®, correction tape, or similar products designed to obliterate documentation are not to be used; instead the correction shall be written adjacent to the error. The correction shall be initialed by the responsible individual and the date of change documented. All manual data and log entries shall be written in indelible ink.

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. Units of measure (mL/L)
  8. Parameter analyzed
  9. Final value to be reported
  10. Method reference (refer to Certified Parameters Listing (CPL) for correct method description)
  11. Data qualifier(s), where applicable.

1. Proficiency Testing Procedures
   1. Analysis of a blind PT Sample is required at least once during every 9-month PT calendar year (January 1- September 30).
      1. A list of approved PT Sample Providers may be found on the NELAC website at

<http://nelac-institute.org/content/NEPTP/ptproviders.php>. Check this list yearly to assure the chosen vendor is approved.

* + 1. A PT Sample can be analyzed as early as January 1 and the graded result must be reported to NC WW/GW LC office from the PT Sample Provider no later than September 30.
  1. PT Samples must be analyzed in accordance with the routine testing, calibration and reporting procedures, unless otherwise specified in the instructions supplied by the PT Sample Provider.
     1. PT Samples are logged in and analyzed using the same staff, sample tracking systems, standard operating procedures including the same equipment, reagents, calibration techniques, analytical methods, and the same quality control acceptance criteria.
     2. PT Samples shall not be analyzed with additional quality control. They are not to be replicated beyond what is routine for Compliance Sample analysis.
     3. PT Sample analysis must be documented on the laboratory’s daily benchsheet.

* 1. The PT Sample Provider’s instructions for preparing the PT Sample must be followed and the practice documented by the analyst. The instruction sheet will be initialed and dated when the PT sample is prepared and retained for 5 years.
  2. The following information must be included when reporting the PT Sample result.
     1. EPA Lab Code: *(enter here so it is easy to retrieve)*
     2. State Lab Certification number: *(enter here so it is easy to retrieve)*
     3. Method description (refer to CPL for current method description): *(enter here so it is easy to retrieve)*
     4. Mailing address for NC WW/GW LC: 1623 Mail Service Center, Raleigh, NC 27699-1623

1. Reporting
   1. Data must be reported in mL/L.
   2. Results that fall below the smallest gradation on the Imhoff Cone (i.e., *state lowest gradation)* must be reported as less than (*state that value again)*.
   3. 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 the result qualified on the report. Calculate results as follows:

(mL of residue) X 1000 = Result in mL/L

(mL of sample)

* 1. *State who is transcribing the data to the DMR and whether anyone peer reviews (checks) it. Peer review is recommended, but if that is not possible, it is recommended that the employee rechecks their own transcription for errors after a certain amount of time has passed*

1. Employee Training

Employee training must be documented and kept on file.

* 1. *Include required education, training, experience and/or demonstrated skills*
  2. Employee must have read and acknowledged understanding of this SOP *– may also include reading the Approved Procedure for the Analysis of Settleable Residue*
  3. *Employee must obtain acceptable results on Proficiency Testing samples or other demonstrations of proficiency (e.g., Initial Demonstration of Capability (IDOC), side-by-side comparison with established analyst, etc.) before analyzing compliance samples for reporting. Specify how proficiency is demonstrated and how the results are evaluated.*

1. References
   1. Standard Methods, 2540 F-*2015*.

* 1. North Carolina Wastewater/Groundwater Laboratory Certification Approved Procedure for the Analysis of Settleable Residue, Revision *03/2021* (consult WW/GW LCB website for latest revision).
  2. 15A NCAC 02H .0800

1. Revision History

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| Type: Review or Revision | Date | Summary of Changes Made if Revision |
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