

SOP-C-107

**Determination of Nonfilterable  
Residue (Total Suspended Solids)**

Revision 18

Approval:

  
Laboratory Manager

8-30-22  
Date

  
Concurrence

8/30/2022  
Date

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*Texas Institute for Applied Environmental Research*

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- i. **Identification of the method**
  - a. Standard Methods 2540D, latest online edition (approved 2017).
  - b. Modification to published method: suction to filter is not left on for three minutes after rinsing, but filter is left on vacuum until dry (will hold when slightly bent).
- ii. **Applicable matrix or matrices**
  - a. Water
- iii. **Limits of detection and quantitation**
  - a. LOD= 4 mg/L
  - b. LOQ= 4 mg/L
  - c. PQL= 20 mg/L
- iv. **Scope and application, including parameters to be analyzed**
  - a. Suitable for the determination of solids in potable, surface, and saline waters, as well as domestic and industrial wastewaters in the range up to 20,000 mg/L.
- v. **Summary of the method**
  - a. Gravimetric analysis of suspended matter from filtered water samples that includes drying the residue retained on the filter to a constant weight at 103-105°C.
- vi. **Definitions**
  - a. See QAM-Q-100, "Quality Assurance Manual," and Q-101, "Laboratory Quality Control."
- vii. **Interferences**
  - a. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not representative. The Laboratory Manager can be contacted for guidance on this, if necessary.
  - b. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue.
  - c. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material.

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- d. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.
- e. Results for residues high in oil or grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.
- f. Some samples dry with the formation of a crust that prevents water evaporation. Special handling is required to deal with this.

**viii. Safety**

- a. See QAM-S-101 and MSDSs for hydrochloric acid and dust inhalation precautions (Celite™).
- b. Use the oven mitts to remove hot sample trays from the oven.

**ix. Equipment and supplies**

- a. Desiccator, provided with a desiccant containing a color indicator of moisture concentration or an instrumental indicator.
- b. Drying oven, for operation at 103 to 105°C.
- c. Analytical balance, capable of weighing to 0.1 mg.
- d. Magnetic stirrer with TFE stirring bar.
- e. Wide-bore pipets.
- f. Graduated cylinder, class A or calibrated by TIAER, "To Contain" glassware is used.
- g. Low-form beaker
- h. Glass-fiber filter disks without organic binder (pre-washed or prepared in lab), Millipore AP40, Reeves Angel 934-AH, Gelman type A/E or equivalent
- i. Aluminum weighing pans
- j. Filtration apparatus with reservoir and a meshed plate as filter support
- k. Suction flask, 1000 mL or more capacity
- l. Forceps

**x. Reagents and standards**

- a. Hydrochloric acid, 0.1 N
  - i. Carefully mix 8.3 mL of conc. HCL into about 800 mL of DI water in a 1 L volumetric flask. Dilute to volume.

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- b. Celite™ standard, 100 mg/L, **LCS/LCSD**
  - i. Rinse about 500 g of Celite™ 545, or equivalent, with three 250mL portions of 0.1 N HCL by mixing in a large beaker, allowing settling and decanting each time.
  - ii. Rinse the Celite™ with three portions of DI water in the same manner and decant.
  - iii. Dry the rinsed Celite™ overnight at 180C
  - iv. Stir the solid with a glass rod to ensure homogeneity of drying and place in a desiccator
  - v. Weigh 0.1000 g of prepared Celite™ and mix with about 800 mL DI water in a 1 L volumetric flask. Dilute to volume and stir for at least 15 minutes to mix well. Standard may be transferred to a sample bottle. Standard is ready for analysis. Mix well each time prior to use.
- x. **Sample collection, preservation, shipment and storage**
  - a. Holding Time: 7 days, analyze as soon as possible
  - b. Preservation: Refrigerate sample to  $>0-6^{\circ}$  C in type "A" plastic bottle.
- xii. **Quality control**
  - a. See QAM-Q-100 and Q-101.
  - b. Record all drying times and max/min temperatures used.
- xiii. **Calibration and standardization**
  - a. The analytical balance is calibration checked daily with the 0.1 g class S weight before the initial and final weights of the filters are taken. Calibrate per QAM-I-101, "Operation and Calibration of the Analytical Balance".
  - b. Other than the LCS/LCSD check, no standardization or calibration is applicable to this SOP.
- xiv. **Procedure**
  - a. Preparation of glass-fiber filter disk: *If pre-prepared glass fiber filter disks are used, eliminate this step.* Insert disk with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three successive 20-mL portions of DI water. Continue suction to remove all traces of water, turn vacuum off, and discard washings. Remove filter from

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filtration apparatus and transfer to an inert aluminum weighing dish. Dry in an oven at 103 to 105°C for at least 1 hr. If volatile solids are to be measured, ignite at 550°C for at least 15 min in a muffle furnace. Cool in desiccator to balance temperature and weigh the filter only, not the support pan or dish. Repeat cycle of drying or igniting, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 0.5 mg. Store in desiccator until needed.

- b. Selection of filter and sample sizes: Choose sample volume to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L. If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume. Clean samples and blanks will require 1 liter aliquots; LCS/LCSD and most samples will require 250 mL or so; very turbid samples may be as low as to 1 mL. If insufficient sample is submitted, initiate a Corrective Action Report, but continue with analysis.
- c. Samples may be brought to room temperature before analysis. Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of reagent-grade water to seat it.
- d. Stir or mix sample and use a pipet or graduated cylinder to transfer a measured volume onto a glass fiber filter with applied volume.
- e. For volumes less than 100 mL use a pipet to transfer a measured volume onto the filter.
- f. For volumes of 100 mL or greater, use a Class A graduated cylinder to transfer a measured volume onto the filter.
- g. Pour or apply the sample to the filter as evenly as possible. Rinse the pipet or graduated cylinder at least 3 times.
- h. If larger pieces are present, stir sample with a magnetic stirrer at a speed to shear the larger particles, if practical, to obtain a more uniform (preferably homogeneous) particle size. Centrifugal force may separate particles by size and density, resulting in poor precision when point of

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sample withdrawal is varied. To avoid this, don't stir too quickly. While stirring, pipette a measured volume onto the seated glass-fiber filter. For homogeneous samples, pipette from the approximate midpoint of container but not in vortex. Choose a point both mid-depth and midway between wall and vortex and apply to filter as above.

- i. After sample is filtered, wash walls and filter with at least three successive volumes of  $\geq 10$ -mL DI water, allowing complete drainage between washings, and continue suction after filtration is complete until filter appears dry and no water continues to drip through. Samples with high dissolved solids may require additional washings.
- j. Rinse the bottom of the filter cup on to the filter to remove any particles that are adhering to the underside.
- k. Carefully remove filter from filtration apparatus with forceps and transfer to an aluminum dish as a support. If the filter is sufficiently dry it will hold a bent shape. If not return it to the filtration apparatus and run the filter pump to further dry the filter.
- l. Dry for at least 1 hr at 103 to 105°C in an oven, cool in a desiccator to balance temperature, and weigh. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or weight change is less than 0.5 mg.
- m. If volatile solids are to be determined, treat the residue according to SOP-C-108, "Determination of Nonfilterable Volatile and Fixed Solids." If the filtrate is used for TDS, refer to SOP-C-109, "Determination of Total Dissolved Solids," to continue.

**xv. Data analysis and calculations**

$$\text{Non-filterable residue mg/L} = \{(A-B)*1000\}/C$$

A = weight of filter + residue in mg

B = weight of filter in mg

C = mL of sample

- a. Data is entered into the Solids worksheet Elog in ESDMS. A paper copy is printed and affixed to the personal logbook. Calculations and data flags should automatically appear in the worksheet. If not, notify the LM.

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**xvi. Method performance**

- a. QC Samples: For every 10 samples (QCB), analyze at least one sample duplicate. Duplicate determinations agree within 5% of their average weight or the QCB is reanalyzed. Method blanks and LCS/LCSD pair are analyzed for every set of 20 samples or less (PB).
- b. Only analysts with approved DOCs on file are allowed to perform this test without the direct supervision of another trained analyst. Technicians may help in filter and equipment preparation without a DOC, but only with documented training.
- c. No CCV, CCB, AWRL/RL or LOQ standard is required for this procedure.

**xvii. Pollution prevention**

- a. See section on Waste Management

**xviii. Data assessment and acceptance criteria for quality control measures**

- a. If part of a sample adheres to the sample container, consider this in evaluating and reporting results.
- b. The LM or LQAO reviews the QC data in the control charting system for tracking and trending.
- c. Duplicate acceptance limits are  $\pm 10\%$  RPD for sample duplicates,  $\pm 20\%$  RPD for LCS/LCSD and  $\pm 30\%$  RPD for Field Splits. FSs are evaluated by project personnel.

**xix. Corrective actions for out-of-control data**

- a. Reanalyze the QC batch if the sample duplicate precision is unacceptable and holding time remains.
- b. Reanalyze the Preparation batch if the method blank or LCS/LCSD accuracy and precision do not pass QC requirements and holding time remains.

**xx. Contingencies for handling out-of-control or unacceptable data**

- a. If any method blank, sample duplicate or LCS/LCSD does not pass the acceptance criteria as described in QAM-Q-101, "Laboratory Quality Control," complete a Corrective



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Action Report in accordance with QAM-Q-105,  
"Corrective Actions," and inform the Laboratory Manager.

**xxi. Waste management**

- a. Waste management and pollution prevention: refer to SOP-W-101, "Disposal of Laboratory Waste". Other than weak acid rinsate, no hazardous waste is generated by this SOP.

**xxii. References**

- a. Standard Methods for the Examination of Water and Wastewater, Online Edition (approved 2020), ed. by Arnold E. Greenberg, et al., APHA, AWWA, Washington, D.C., methods 2020, 2540D.
- b. National Environmental Laboratory Accreditation Conference (NELAC) TNI Standard, 2016, The NELAC Institute.

**xxiii. Any tables, diagrams, flowcharts and validation data**

- a. Example TSS Map (showing formulas)



## Attachment 1: Example TSS Map

[illegible]