Illinois State Water Survey
Health and Environmental Applications Laboratory

Standard Operating Procedure
For
Total Suspended Solids Dried at 103 to 105 °C

(based on SM 2540 D 2012, 22nd ed.)

SOP Number: AN.HEAL.MN.TSS.2.2

Revision 2.3, effective 8 October 2024

Illinois State Water Survey
2204 Griffith Drive
Champaign, IL 61820-7495

Prepared by: Margarita Bargon Date: 12/19/2024

Rita Bargon, Quality Assurance Officer

Approved by: Evan Rea Date: 12/19/2024

Evan Rea, HEAL Director

The following individuals (in addition to those listed on the cover) have reviewed this SOP.

Name (printed)	Signature	Date
Anthony Kilber	Anthony Kilber	12/19/2024
Tatyana Grandt	Tatyana Grandt	01/02/2025

Beginning Revision	Ending Revision	Revision Date	Changes
2.1	2.2	7/27/24	Added QC calculations and updated QC scheme
2.2	2.3	10/8/24	Edited section 11 to be more clear about sample volume.

Table of Contents

Page

1.0 Scope and Applicability

1.1 Solids refer to matter suspended or dissolved in water or wastewater. Water high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitation.

1.2 Total suspended solids (TSS) refer to the portion of solids that is retained on a 2.0 µm (or smaller) nominal pore size filter. The HEAL uses a 1.5 µm filter. The MDL is 1.8 mg/L.

2.0 Summary of Method

2.1 A well-mixed, 250 mL sample is filtered through a weighed standard glass-fiber filter, and the residue retained on the filter is dried at 103 to 105°C. The increase in the weight of the filter represents the total suspended solids.

2.2 If the suspended material clogs the filter and prolongs filtration, it may be necessary to decrease the sample volume.

3.0 Definitions

LFB Laboratory Fortified Blank
LRB Laboratory Reagent Blank
MDL Method Detection Limit
RPD Relative Percent Difference
TSS Total Suspended Solids

4.0 Safety Warnings and Waste Management

4.1 Always wear eye protection in the laboratory.

4.2 Food, drinks, and smoking are not allowed in the laboratory.

4.3 Safety Data Sheets (SDS) applicable to this SOP can be found online by using the University of Illinois Division of Research Safety (DRS) website: https://www.drs.illinois.edu/Programs/SafetyDataSheets.

4.4 The Illinois State Water Survey Chemical Hygiene Plan covers the ISWS laboratory safety program, including, but not limited to, personal protective equipment used, control equipment inventory and operations (such as vented hoods), employee training programs, medical programs, and safety. The ISWS Chemical Hygiene Plan is available at https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan.

4.5 The University of Illinois DRS has a laboratory safety plan available at https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf. The ISWS has their own laboratory safety manual, available at https://go.illinois.edu/ISWS-Laboratory-Safety-Manual.

4.6 The HEAL practices pollution prevention, which encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The quantity of chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material.

4.7 Laboratory waste management practices must be consistent with all applicable rules and regulations. Excess reagents and samples and method process wastes should be characterized and disposed of by DRS. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations.

4.8 The HEAL has listed known health and safety warnings for this SOP, but this list should not be assumed to comprise all health and safety issues.

5.0 Cautions

5.1 Use forceps to handle filters and weighing dishes. Do not use bare hands to touch filters or dishes.

6.0 Interferences

6.1 Large floating particles or other not homogenous material may not properly represent the suspended solids of the sample. Exclude these particles.

6.2 Limit sample size to that yielding no more than 200 mg residue. Excessive residue on the filter may form a water-entrapping crust leading to an inaccurate result.

6.3 Dissolved material may remain trapped on filter if the filter is not properly washed following filtration.

6.4 Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal material captured on the clogged filter.

7.0 Personnel Qualifications

Analysts in training must complete at least five days of training or a manufacturer's training course, and a satisfactory demonstration of capability before analyzing routine samples.

8.0 Equipment and Supplies

8.1 Equipment

8.1.1 Electronic analytical balance: Mettler-Toledo AX304 or equivalent, capable of weighing up to 0.1 mg (Max 310 g), connected to a computer with LabX software installed.

8.1.2 Desiccator with a desiccant containing a color indicator of moisture concentration.

8.1.3 Drying oven for operation at 104°C

8.1.4 Glass Microfiber Filter, Whatman grade 934AH (1.5 µm particle retention), 47 mm, or other products that give demonstrably equivalent results.

8.1.5 Magnetic stir plate

8.1.6 Magnetic stir bars

8.1.7 250 mL glass filter funnel

8.1.8 Vacuum filtering set up

8.1.9 25 mL plastic serological pipettes

8.1.10 Forceps with flat tips, so they don't poke holes in filters

8.1.11 Aluminum weighing dishes

8.1.12 250 mL graduated cylinder

8.2 Reagents and solutions

8.2.1 TSS spiking solution, 1000 mg/L celite (diatomaceous earth) in water for the laboratory fortified blank (Aqua Solutions, P/N SPE509).

8.2.2 LRB: used fresh deionized water with resistivity of at least 18.2 Mohms

8.2.3 LFB: add 225 mL of deionized water and pipette 25 mL of TSS spiking solution stock to a 250 mL Nalgene bottle, and shake.

8.2.4 MDL verification solution: pipette 30 mL TSS spiking solution, 1000 mg/L to a 1000 mL volumetric flask filled halfway with DI water. Add DI water to the 1000 mL mark.

9.0 Instrument Setup and Calibration

9.1 Balance calibration should be tested daily by the analyst, using at least 3 weights traceable to NIST calibration/certification. If the scale is going to be calibrated, perform it before the analysis.

10.0 Sample Collection, Handling and Preservation

10.1 Samples should be collected in 1L HDPE bottles supplied by the laboratory to ensure sufficient volume and cleanliness. All bottles must be thoroughly cleaned and rinsed with reagent water. A volume of 500 mL is usually sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

10.2 Ship and store samples at 4°C. Begin analysis as soon as possible after sample receipt. The holding time is 7 days after receipt in the laboratory.

10.3 Filters should be handled carefully with filter forceps (flat-tipped), and only on the edge of the filter. Filter pans should also be handled with forceps to ensure no oils or debris from hands are transferred into the dish.

11.0 Sample Preparation and Analysis

11.1 Use the Lab-X software to check the calibration of the scale.

11.1.1 Open the Lab-X software. If the quick start wizard opens, click next twice to proceed to step 3 of 4. Do not change any default settings on step 2.

11.1.2 Use the dropdown menu titled “Layout Name” to select the proper format for the task at hand. Usually this is either “TDS or TSS”, or “Balance Calibration Checks”. Don’t change any other settings and then click next to proceed to step 4.

11.1.2 Use the dropdown menu to select the file or application you are transferring data into (usually this is Excel or a specific spreadsheet). There is also the option to use the cursor location to enter the data, which works the same way.

11.1.3 Place 50 g weight on the balance. Right-click on ‘50’ in the weight column, then left click to select balance. When the circle disappears from balance read out, click out of the ‘50’ box to save the reading.

11.1.4 If the scale needs to be re-calibrated, it should be done before the analysis.

11.2 Preparation of glass fiber filter

11.2.1 Write a unique ID on the tab of each aluminum dish with a marker. Do not touch the dishes or filters with bare hands, either wear gloves or ideally use forceps.

11.2.2 Open the “SolidsPreWeigh” excel spreadsheet the file in “\\pri-fs1\HEAL\Data\Lab Templates”. Enter pan IDs in the spreadsheet, making sure to include enough pans for all samples and duplicates/QCs. The pan ID numbers will be matched up with sample IDs later.

11.2.3 Insert a filter disk into vacuum filtration apparatus with forceps. Wetting the filter with a few drops of DI water will help with suction. Apply vacuum and rinse disk with three, 20 mL portions of deionized water. Continue suction to remove all traces of water. Discard washings.

11.2.4 Place filter in aluminum weighing dish and dry in 104°C drying oven overnight. Keep drying times consistent.

11.2.5 Place weighing dish with filter into a desiccator for at least 2 h. It is best to leave the desiccators in the same room as the balance to minimize temperature changes when weighing. Keeping the desiccation time consistent across all weighing is important to achieve consistent results.

11.2.6 Weigh each dish/filter to four decimal places and record weights in the SolidsPreWeigh file. Keep a running list of filters prepared for analysis in this file (no need to make a new file for each analysis).

11.2.7 Repeat steps 11.2.4 through 11.2.6 to re-dry and re-weigh the filters, until the weights are consistent (difference is less than 0.0005 g or 4%, whichever is less). The excel sheet includes logic to check the weights and will display a flag in the “passed” column as either ‘pass’ or ‘fail’. When two weights are consistent, then the filter is ready for the analysis.

11.2.8 Take desiccators containing the prepared filters to the same room as the analytical balance.

11.3 Sample Analysis

11.3.1 Create an excel spreadsheet by opening the TSS template file in “\\pri-fs1\HEAL\Data\Lab Templates” and saving a new copy with a filename that includes the analysis date.

11.3.2 Use the SolidsPreWeigh spreadsheet to record a final passing pre-analysis weight for each filter immediately before sample analysis. Then, copy passing weights and pan numbers into the analytical template named “TSS_Template”. These weights are the “filter date weight” (cells colored in dark green). Be sure to use filters for all samples and QCs.

11.3.3 Create Prep Batch and Analytical batch in LIMs. Copy an Excel file from LIMs to the TSS working spreadsheet. Each sample should have a corresponding ‘passing’ filter/pan number assigned to it at this point.

11.3.4 Begin each run with the following Quality Control samples: LRB, LFB. Then, run samples, being sure to include a duplicate every 10 samples (at least one per batch).

11.3.5 Stir or shake each sample vigorously before pouring, and rinse graduated cylinder/ filter funnel after filtering sample with three ~10 mL portions of DI water. Allow up to 3 minutes for the vacuum to completely drain all moisture from the filter. Shut off the vacuum and remove the filter using forceps. Be careful not to rip the filter, and to grip it only by the outer edges to avoid disturbing the sample.

Note: Rinse stir bars with DI. When using stir bars, be sure to adjust the mixer speed to get a good vortex in the sample and avoid a 'jumpy' stir bar.

11.3.6 Samples: Place a clean stir bar in each sample bottle and mix before filtering. Place the filter on the filtration assembly. Wetting the filter with a few drops of DI helps with suction.

11.3.7 Pour ~half of target volume of sample into a graduated cylinder, then shake the sample again and pour the remaining amount onto the filter. Typical sample size is 250 mL, but this depends on available sample volume. Adjust volume to be filtered based on turbidity of the sample: use less volume if sample is very turbid or more volume if the sample is very clear. If the filter becomes clogged and filtration stops, then repeat the analysis with less volume. Be careful not to use the entire sample volume, to ensure there is enough sample left over for additional analyses or reanalysis. Record volume used on the excel spreadsheet.

11.3.8 Wash with three successive 10 mL portions of DI water. Apply vacuum for ~3 minutes. Place the filter back into the weighing dish. Repeat for each sample and duplicate.

11.3.8 Transfer weighing dishes with filters back into the 104°C oven. Let samples dry overnight.

11.3.9 Remove samples and transfer to a desiccator for two hours. Preferably, leave the desiccators in the same room as the balance to reduce temperature variations when weighing.

11.3.10 Verify balance calibration with certified weights (as in section 11.1).

11.3.11 Retrieve the TSS analytical spreadsheet, then use it to record the weights of all filters. Repeat the cycle of drying, cooling, and weighing (steps 11.3.5 to 11.3.8) until a constant weight is achieved (difference is less than 0.0005 g). Be sure to check all QCs before sending the data for review.

11.3.11 Save a copy of the TSS prep sheet as a PDF, and a copy of the completed TSS analytical spreadsheet to "\\pri-fs1\HEAL\Data\Lab Data In\TSS".

11.3.12 Place all the weighing dishes and filters in a box with sample ID numbers and store on a shelf in room 303 until complete batch is approved.

11.3.13 Empty the waste jug, rinse funnels and stir bars with DI, and put everything back on the cart with the rest of the TSS supplies.

12.0 Troubleshooting

12.1 If sample weights are out of limits, they can be reweighed. If stable weights cannot be obtained within 10 weighing, consult with the QA Officer.

12.2 If there is insufficient residue on the filter, repeat analysis with a higher volume. If there is too much residue on the filter, then repeat the analysis with a lower volume.

12.3 Use good weighing practices to ensure that interferences are reduced: do not lean on the counter while weighing, do not leave windows open, ensure there are no excessive air currents in the room, weigh at a time when temperature is consistent, and check the level bubble on the balance to make sure it is level.

13.0 Data and Record Management

13.1 Precision and bias data (5/2/2014- 1/16/2015) for the ISWS laboratory are as follows:

Table 1. ISWS Precision and bias of LFB measurements

Number of Samples	True value (mg/L)	Mean (mg/L)	Mean % Recovery	Standard Deviation (mg/L)
20	100	95.7	95.7	3.3

13.2 The MDL is 1.8 mg/L.

13.3 Calculate the concentration of TSS as follows:

$$\text{Total suspended solids, mg/L} = \frac{(A - B) \times 1000}{\text{sample volume (mL)}}$$

where: A = weight of dried sample + filter (mg)
B = weight of filter (mg)

13.2 Upload analytical file in LIMs and attach a copy of it to the analytical batch for the QA Officer for review. Complete Prep Batch in LIMs.

14.0 Quality Control and Quality Assurance

14.1 Laboratory Reagent Blank (LRB) – A LRB is analyzed with each preparation batch. LRB results should be less than the MDL (1.8 mg/L).

14.2 Laboratory Fortified Blank (LFB) - A LFB shall be analyzed with each preparation batch of samples with the same matrix. If the recovery of any analyte falls outside the required control limits of 85 to 115%, that analyte is judged out of control.

14.3 Duplicate sample analyses will be performed for every 10 samples in a preparation batch, with a minimum of one per matrix type or preparation procedure. Over time, samples representing all routine sample sources and types should be analyzed in duplicate. Because of the wide variety of projects, choice of a specific sample for duplication is at the discretion of the analyst.

14.3.1 Duplicate values should agree within 10% relative percentage difference (RPD) when the concentration is > 10*MDL.

$$Duplicate\ acceptance\ range = \left(\frac{Dup\ 1 + Dup\ 2}{2}\right) \pm \left[\left(\frac{Dup\ 1 + Dup\ 2}{2}\right) * 0.05\right]$$

14.5 Corrective Action

14.5.1 The procedure for nonconforming work is given in the Corrective Action Policy (AD.HEAL.0.CorrectiveAction).

Table 14–1 Summary of quality control samples and acceptance criteria

QC Type	Frequency	Acceptance Criteria
LRB	1 per batch	<7mg/L
LFB	1 per batch	± 85-115%
Duplicate	Every 10 samples	±10% when >10*MDL

15.0 References

15.1 American Public Health Association, American Water Works Association, Water Environment Federation. (2012), Total Suspended Solids Dried at 103-105°C, 2540 D, In E.W. Rice, R.B. Baird, A.D. Eaton, L.S. Clesceri (Eds.), Standard Methods for the Examination of Water and Wastewater, 22nd Edition. Washington, DC: American Public Health Association.

15.2 Rea, E. 2023. Health and Environmental Applications Laboratory Quality Assurance Plan. Illinois State Water Survey, Champaign, IL.

AN.HEAL.MN.TSS.2.3

Final Audit Report 2025-01-02

Created:	2024-12-19 (Central Standard Time)
By:	Evan Rea (erea@illinois.edu)
Status:	Signed
Transaction ID:	CBJCHBCAABAA5uk4r_TSLXSTCfcK88pNHPErfmEwURro
Number of Documents:	1
Document page count:	11
Number of supporting files:	0
Supporting files page count:	0

"AN.HEAL.MN.TSS.2.3" History

Document created by Evan Rea (erea@illinois.edu)
2024-12-19 - 2:40:44 PM CST- IP address: 130.126.105.85

Document emailed to Margarita Bargon (rbargon@illinois.edu) for signature
2024-12-19 - 2:42:26 PM CST

Email viewed by Margarita Bargon (rbargon@illinois.edu)
2024-12-19 - 2:43:03 PM CST- IP address: 130.126.105.134

Agreement viewed by Margarita Bargon (rbargon@illinois.edu)
2024-12-19 - 2:43:03 PM CST- IP address: 130.126.105.134

Margarita Bargon (rbargon@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-19 - 2:43:23 PM CST

Document e-signed by Margarita Bargon (rbargon@illinois.edu)
Signature Date: 2024-12-19 - 2:43:23 PM CST - Time Source: server- IP address: 130.126.105.134

Document emailed to Evan Rea (erea@illinois.edu) for signature
2024-12-19 - 2:43:25 PM CST

Email viewed by Evan Rea (erea@illinois.edu)
2024-12-19 - 3:16:15 PM CST- IP address: 130.126.105.85

Agreement viewed by Evan Rea (erea@illinois.edu)
2024-12-19 - 3:16:29 PM CST- IP address: 130.126.105.85

Evan Rea (erea@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-19 - 3:16:34 PM CST

Document e-signed by Evan Rea (erea@illinois.edu)
Signature Date: 2024-12-19 - 3:16:34 PM CST - Time Source: server- IP address: 130.126.105.85

Document emailed to Anthony Kilber (awkilber@illinois.edu) for signature
2024-12-19 - 3:16:35 PM CST

Email viewed by Anthony Kilber (awkilber@illinois.edu)
2024-12-19 - 4:01:42 PM CST- IP address: 130.126.105.96

Agreement viewed by Anthony Kilber (awkilber@illinois.edu)
2024-12-19 - 4:01:57 PM CST- IP address: 130.126.105.96

Anthony Kilber (awkilber@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-19 - 4:03:48 PM CST

Document e-signed by Anthony Kilber (awkilber@illinois.edu)
Signature Date: 2024-12-19 - 4:03:48 PM CST - Time Source: server- IP address: 130.126.105.96

Document emailed to Tatyana Grandt (tgrandt@illinois.edu) for signature
2024-12-19 - 4:03:49 PM CST

Reminder sent to Tatyana Grandt (tgrandt@illinois.edu)
2024-12-27 - 1:02:38 PM CST

Email viewed by Tatyana Grandt (tgrandt@illinois.edu)
2025-01-02 - 9:13:33 AM CST- IP address: 130.126.105.117

Agreement viewed by Tatyana Grandt (tgrandt@illinois.edu)
2025-01-02 - 9:13:37 AM CST- IP address: 130.126.105.117

Tatyana Grandt (tgrandt@illinois.edu) authenticated with Adobe Acrobat Sign.
2025-01-02 - 9:13:45 AM CST

Document e-signed by Tatyana Grandt (tgrandt@illinois.edu)
Signature Date: 2025-01-02 - 9:13:45 AM CST - Time Source: server- IP address: 130.126.105.117

Agreement completed.
2025-01-02 - 9:13:45 AM CST