Standard Operating Procedure for:

# Total Suspended Solids

Missouri State University

and

Ozarks Environmental and Water Resources Institute (OEWRI)

Prepared by: _____ Date: _____
OEWRI Laboratory Manager

Approved by: _____ Date: _____
OEWRI Director

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**Table of Contents**

## 1 Identification of the test method

Determination of Total Suspended Solids.

## 2 Applicable matrix or matrices:

This method is suitable for the determination of suspended material in potable and surface waters and wastewaters with total suspended solids (TSS) of up to 20,000 mg/L.

## 3 Detection Limit

The desired detection limit for this method is 0.5 mg/L for a 1-L sample.

## 4 Scope of the test method

This standard operating procedure provides the Missouri State University (MSU) laboratory personnel with guidance on the procedure for determining TSS. This method is limited to the determination of TSS in water samples collected from natural bodies of water containing TSS of < 20,000 mg/L.

## 5 Summary of test method

6 The procedure described here follows published Standard Methods and U.S. Environmental Protection Agency (USEPA) protocols (USEPA 1971, Clesceri et al 1998). A well-mixed, measured volume of a water sample is filtered through a pre-weighed glass fiber filter (either 1.5 µm or 0.45 µm nominal pore size). The filter is heated to constant mass at 104 ± 1º C and then weighed. The mass increase divided by the water volume filtered is equal to the TSS in mg/L.

## 6 Definitions

6.1 Analytical batch: The set of samples processed at the same time

6.2 Laboratory reagent blank (LRB): An aliquot of deionized water treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

6.3 Laboratory duplicate (LD): Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation or storage procedures.

6.4 Method detection limit (MDL) -- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

## 7 Interferences

It is recognized that TSS measurements may include both positive errors (occluded water and waters of crystallization) and negative errors (decomposition and volatilization of minerals such as carbonates, as well as loss of solids that are small enough to pass through the filter). See Standard Method 2540 D.1.b for further discussion of interferences for this method.

## 8 Health and safety

8.1 The analysis involves handling of freshwater samples that may contain live microorganisms and therefore pose some threat of infection. Laboratory personnel who are routinely exposed to such water samples are encouraged to protect themselves from water borne illnesses by wearing clean disposable gloves and washing their hands frequently.

8.2 This analytical procedure uses a vacuum pump for the filtration steps. There is a risk of implosion under some circumstances. The analyst should ensure that the receiving flask is free from cracks or other imperfections.

## 9 Personnel qualifications

Laboratory and field personnel shall have a working knowledge of this analytical procedure and will have received training from an MSU employee knowledgeable of the proper sample analysis procedures. Prior to the first batch of sample analyses, the analyst will complete a demonstration of capability exercise as described below in the Quality Control section.

## 10 Equipment and supplies

10.1 Filters, Glass microfiber: 47 mm diameter, 1.5 µm nominal pore size, such as Whatman 934-AH (catalog number: 1827 047) or equivalent. And/or a 47 mm diameter, 0.45µm nominal pore size, such as Millipore Corp RW0304700 (Fisher catalog number: RW03 047 00). The 1.5 µm nominal pore size filter will be used for stormwater applications following Standard Method 2540 D (Clesceri et al. 1998). Alternatively, when used in conjunction with total dissolved solids protocol, a 0.45 µm nominal pore

size filter will be used following United States Geological Survey methods (Brown et al. 1970). Filter pore size used will be noted on the bench sheet for reporting purposes.

10.2 Filtration apparatus
- a. Filter pump
- b. A 1-L or 4-L receiving flask
- c. Filter funnel manifold, 3 positions
- d. Magnetic filter funnels 300 mL
- e. Vacuum tubing, ¼" inside diameter

10.3 Drying oven (Room 125 of Temple Hall, MSU), adjusted to 104 ± 1ºC.

10.4 Aluminum weighing dishes (or equivalent).

10.5 Analytical balance capable of reading to 0.1 mg.

## 11 Reagents and standards

11.1 Deionized water (DI): water that has been passed through a purification system (e.g., the Barnstead/Thermolyne system in 476 Temple).

11.2 There are no standards available for this method.

## 12 Sample collection, preservation, shipment and storage

12.1 See the SOP for water sample collection procedures (OEWRI 2007).

12.2 Bottles are sealed and placed on ice in a cooler for transport to the laboratory. The samples are placed in a refrigerator in the laboratory. The maximum holding time is seven days (SM, 2005) but analysis should begin as soon as possible upon delivery at the laboratory.

## 13 Quality control

13.1 Record the start and end temperatures for this analysis in the instrument log book.

13.2 Method detection limit: Initial demonstration of capability (for any new laboratory assistant) and quarterly thereafter;
- a. Carry ten filters through the procedure, filtering 1 liter of deionized water for each.

b. Calculate the standard deviation of the results. The method detection limit is three times the standard deviation. The detection limit should be less than or equal to 0.5 mg/L

13.3 Precision: Initial demonstration of capability (for any new laboratory assistant) and quarterly thereafter;

a. Collect 5 L of sample from the same site under conditions as close to identical as practical. Ideally, the site should be chosen to have TSS ≥ 10 mg/L.
b. Carry out ten analyses using 300 mL samples for each.
c. Calculate the average and standard deviation of the values.
d. The standard deviation should be less than or equal to 20% of the average value. If it is not, evaluate the procedures to identify sources of error.

13.4 Laboratory Duplicate (LD) reproducibility: Carry out with each analytical batch of filters.

a. Carry out two replicates on the same sample, using a sample volume sufficient to provide at least 5 mg solid (ideally, TSS > 10 mg/L).
b. The relative percent difference (RPD) between the two TSS values should be ≤ 20% of their average value.
c. Use equation 1 to calculate RPD:

$$\text{Equation 1:} \quad \text{RPD (\%)} = \frac{(A - B)}{(A + B)/2} \times 100\%$$

Where: A = mass of first aliquot (mg), and
B = mass of duplicate aliquot (mg)

d. Analyze one set of duplicates for every 10 samples analyzed.

13.5 Blank (LRB): At least one blank should be measured with each analytical batch of filters.

a. For the blank measurement, filter 1L deionized water.
b. The TSS value for the blank should be less than 0.5 mg/L. If it is not, evaluate the procedure and correct sources of error.
c. Analyze one blank for every 10 samples analyzed.

## 14 Calibration and standardization

There are no calibration or standardization procedures for this method.

## 15 Procedure

15.1 Balance operation:

a. The analyst should review the latest revision of the SOP for balance operation (OEWRI 2020) prior to using a balance for this method.

b. The balance should be level, clean and calibrated prior to use.

c. Perform a balance check prior to each batch of analyses:

1. Select a test weight (or combination of test weights) and place on the balance pan.

2. Record values on the bench sheet (see below) and in the balance instrument log book.

15.2 Preparation of the filters:

a. Place filter disks onto the filtration apparatus. Apply vacuum and rinse each with 300-mL volume of deionized water. Draw air through the filters until it appears that all water has been drawn off.

b. Place filters (in individual labeled aluminum evaporating dishes) in an oven set to 104 ± 1ºC.

c. Heat filters for two hours to ensure that filters are dry.

d. Remove filters from the oven and place them into a desiccator until they have cooled to balance temperature.

e. Record the standard weight before weighing the filters to ensure scale accuracy.

f. Weigh each filter, recording all measurements to 0.1 mg precision.

g. Record the mass of the filter on the bench sheet as the Filter Tare Mass in mg.

h. Store filters in a desiccator until use.

15.3 Selection of sample volume

a. The ideal mass increase for the TSS measurement is between 2 and 200 mg (minimum 1.0 mg). The volume of water sample needed to produce this mass change depends on the TSS value.

b. For water collected under base-flow conditions, the recommended starting volume is 300 mL. However, if the suspended solids collected in the filter are either too high or too low, or if the filtration becomes slow due to clogging (total filtration time > 10 minutes), the volume should be adjusted as indicated below.
    1. If the mass of the captured suspended solids is less than 1.0 mg, repeat the analysis using a larger sample volume, up to 2 L.
    2. If the filtration becomes slow because of clogging, estimate the filtration volume at which the filtration volume decreased and repeat the procedure with a fresh filter, using a volume less than that needed to significantly reduce filtration rate.
    3. Based on experience the analyst may adjust sample volume. If filtration of 500 mL produces little observable solids on the filter, the analyst may filter more of the water sample.
    4. If experience has shown that samples from a site normally have very high levels of suspended solids and/or frequently cause clogging, the analyst may reduce the sample volume, as long as the mass of solid collected falls in the range of 2 to 200 mg.

c. For highly turbid samples or samples with expected high amounts of TSS:
    1. Place a magnetic stir bar into the sample bottle.
    2. Place the bottle onto a magnetic stirring unit.
    3. Stir at sufficient speed to create a vortex.
    4. Use a 20 mL volumetric pipette to remove sample from the bottle; choose a point that is mid-depth and midway between the bottle wall and the vortex to obtain a homogenous sample.
    5. Place the measured sample onto the filter and continue suction.
    6. Continue steps 15.3.d.4 – 5 until the volume added starts to slow the filtration rate.
    7. Record the volume (in L) on the bench sheet.
    8. Rinse the filter pad with three 10 mL volumes of DI.

15.4 Procedures for TSS

a. Set up the filtration apparatus, insert a filter, and apply vacuum.

b. Wet the filter with a small volume of deionized water to seat it.
c. Shake the sample vigorously and then measure out the predetermined sample volume. Record the volume filtered in liters on the bench sheet.
d. Rinse the graduated cylinder and filter with three 100 mL volumes of DI, allowing complete drainage between washings.
e. Continue suction for three minutes after filtration is complete.
f. Carefully transfer the filter to an aluminum weighing dish, and place filter on a cookie sheet or similar device.
g. Place filters on sheet into an oven set to 104 ± 1ºC and dry for two hours.
h. Remove filters from oven and transfer them to a desiccator to cool to room temperature.
i. Record the standard weight before weighing the filters to ensure scale accuracy.
j. Weigh one sample filter to the nearest 0.1mg. On the bench sheet record the sample ID and the mass (Mass 1) in the "Weight check" section.
l. Record the Oven Dry Mass (in mg) on the bench sheet.
m. Calculate TSS as described below.
n. Dump remaining sample down the drain, remove label, and rinse with tap water to remove any solids from the bottle. Wash bottles according to the Bottle Prep non-Metals SOP 0150R01.

## 16 Data acquisition, calculations, and reporting

16.1 For each sample analyzed, including quality control samples, record the volume filtered and oven dry mass in the appropriate places on the bench sheet (see below). Calculate TSS using equation 2.

Equation 2. $$TSS\ (mg/L) = \frac{(A - B)}{V}$$

Where: A = mass of filter + dried residue (mg),
B = mass of filter (tare weight) (mg), and
V = volume of sample filtered (L)

16.2 Results should be reported to 0.1 mg/L precision.

16.3 If multiple bottles were used to collect a composite sample across a stream channel, analyze each bottle separately and calculate the average of the values for the final TSS concentration. If flow and discharge have been calculated for each subsection then calculate a flow-weighted average using the TSS concentrations for each aliquot.

## 17 Computer hardware and software

17.1 Word: This document and attached bench sheet are prepared using Microsoft Word. The Word document file name for this SOP is: 2010R01 TSS.doc

17.2 Excel: Quality control charts are created using Excel.

## 18 Method performance

18.1 The desired performance criteria for this measurement are:

a. Detection limit: 0.5 mg/L
b. Precision: ± 20% RPD
a. Minimum Quantification Interval: 0.1 mg/L

18.2 Below are values of reproducibility at different TSS values for the TSS process given in Standard Methods 2540 D (each for ten replicates by two different analysts; water volume not specified):

| TSS mass (mg/L) | Standard deviation | Coefficient of Variation (%) |
|---|---|---|
| 15 | 5.2 | 33 |
| 242 | 24 | 10 |
| 1707 | 13 | 0.8 |

Both Standard Methods 2540 D and EPA 160.2 indicate that replicates should agree within 5% of their average (e.g., a percent difference between the two values of 10%). The data noted here show that reproducibility is much poorer for samples with low TSS.

## 19 Pollution prevention

All wastes from these procedures shall be collected and disposed of according to existing waste policies within the MSU Geography, Geology, and Planning Department.

## 20 Data assessment and acceptable criteria for quality control measures

20.1 The analyst should review all data for correctness (e.g., calculations).

20.2 Precision values are calculated for pairs of duplicate analyses.

20.3 Record the precision values as a percent on the bench sheet.

20.4 The desired precision is ± 20% RPD.

20.5 The desired detection limit is 0.5 mg/L

20.6 The completed bench sheet is reviewed by the analyst's supervisor or the OEWRI QA coordinator.

## 21 Corrective actions for out-of-control or unacceptable data

21.1 Quality control charts will be created for charting precision and blank values.

21.2 The results for precision and blank data are compared to the acceptable values for this analysis; ± 20% and 0.5 mg/L, respectively.

21.3 If a precision value exceeds 20% RPD then the analyst should write in the comments section of the bench sheet: "These data are associated with an out-of-control duplicate analysis. The UCL = 20%." Note: "UCL" is the Upper Control Limit (i.e., 20%).

21.4 If a blank value exceeds 0.5 mg/L then the analyst should write in the comments section of the bench sheet: "These data are associated with a blank value that exceeds the detection limit of 0.5 mg/L."

21.5 The samples cannot be reanalyzed because the sample volume will be depleted after the initial analysis.

21.6 If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.

## 22 Waste management

The wastes generated in this method are not hazardous. They can be discarded in the following manner: the water, both filtrate and raw sample, can be discarded in the laboratory sink and filter papers can be discarded with the paper trash.

## 23 References


Brown, E., M. W. Skougstad, and M. J. Fishman (1970). Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases, Book 5, Chapter A1. Techniques of Water-Resources Investigations of the United States Geological Survey.

Clesceri, L. S., A. E. Greenberg, and A. D. Eaton (1998). Standard Methods for the Examination of Water and Wastewater, 20th Edition. United Book Press, Inc., Baltimore Maryland.

OEWRI (2006) Standard Operating Procedure for: Operation of Analytical Balances. Ozarks Environmental and Water Resources Institute, Missouri State University.

OEWRI (2007) Standard Operating Procedure for: Water Sample Collection. Ozarks Environmental and Water Resources Institute, Missouri State University.

USEPA (1971). Method #: 160.2, Residue, Non-Filterable (Gravimetric, Dried at 103-105°C). United States Environmental Protection Agency.


## 24 Tables, diagrams, flowcharts and validation data

24.1 There are no tables, diagrams, flowcharts or validation data for this method.

24.2 See page 11 for the bench sheet. The analyst should make a copy of this form for each analytical batch of samples to be analyzed.

Missouri State University
Ozarks Environmental and Water Resources Institute
Springfield, Missouri

**Total Suspended Solids**

Analyst: _____

Date analyzed: _____

| Data reviewed by: | |
|---|---|
| | |
| | |

Filter Size Used (Check One): ___ 1.5 µm ___ 0.45µm

Measured Mass (g) _____

| Sample Identification | Date Collected | Weigh Pan Label | Filter Tare Mass (g) (B) | Volume Filtered (L) (V) | Oven Dry Mass (g) (A) | Total Suspended Solids (mg/L) [(A - B) / V]*100 | RPD % |
|---|---|---|---|---|---|---|---|
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Date of Quality Control Check: _____

Comments: _____

# TSS - Bench Procedures

Preparing filters

1. Place filter disks onto the filtration apparatus. Apply vacuum and rinse each with three successive 100-mL volumes of deionized water. Draw air through the filters until it appears that all water has been drawn off.
2. Place filters (in individual labeled aluminum evaporating dishes) in an oven set to 104 ± 1ºC.
3. Heat filters for two hours to ensure that filters are dry.
4. Remove filters from the oven and place them into a desiccator until they have cooled to balance temperature.
5. Weigh each filter, recording all measurements to 0.1 mg precision.
6. Record the standard weight before every filter weight to ensure scale accuracy.
7. Record the mass of the filter on the bench sheet as the Filter Tare Mass in mg.
8. Store filters in a desiccator until use.

Sample Analysis

1. Set up the filtration apparatus, insert a filter, and apply vacuum.
2. Wet the filter with a small volume of deionized water to seat it.
3. Shake the sample vigorously and then measure out the predetermined sample volume using a graduated cylinder. Record the volume filtered in liters on the bench sheet.
4. Continue suction until it appears that all water has been drawn off to ensure filtration is complete.
5. Carefully transfer the filter to an aluminum weighing dish, and place filter on a cookie sheet or similar device.
6. Place filters on sheet into an oven set to 104 ± 1ºC and dry for two hours.
7. Remove filters from oven and transfer them to a desiccator to cool to room temperature.
8. Record the standard weight before every filter weight to ensure scale accuracy.
9. Record the Oven Dry Mass (in mg) on the bench sheet and calculate TSS.

Calculating TSS

- To calculate TSS, use the following equation:

$$\text{TSS (mg/L)} = \frac{(A - B)}{V}$$

Where: A = mass of filter + dried residue (mg),
B = mass of filter (tare weight) (mg), and
V = volume of sample filtered (L)

# TSS – Supply List

All supplies can be bought from fisher scientific.

## Filters

TSS Recommended-
GE Healthcare Whatman Binder-Free Glass Microfiber Filters, Grade 934-AH Circles.
Diameter: 47mm
Nominal pore size: 1.5 µm
Catalog Number: 09-873DD

SSC Recommended-
MilliporeSigma Durapore PVDM Membrane Filters
Diameter: 47mm
Nominal pore size: 0.45 µm
Catalog Number: HVWG04700

## Weighing Dishes

Recommended-
Fisherbrand Aluminum Weighing Dishes
Diameter: 68.2
Catalog Number: 08-732-103

## Vacuum Tubing

Recommended-
Fisherbrand Heavy-Pressure and Vacuum Rubber Tubing
Inside Diameter: 6.4 mm
Catalog Number: 14-173C

# TSS – Detection Limit

Analyst: _____

Date analyzed: _____

| Weigh Pan Label | Measured Mass (g) | Filter Tare Mass (g) (B) | Measured Mass (g) | Oven Dry Mass (g) (A) | Total Suspended Solids (mg/L) [(A - B) / V]*100 |
|---|---|---|---|---|---|
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Standard Deviation: _____
Detection Limit: _____