

SOP CF No.: IR-WET-TSS\_r4-CF2 Effective Date: 12/06/2013 Page No.: 1 of 4

# FACILITY SOP ATTACHMENT

# SOP NUMBER: IR-WET-TSS, Rev 4 (8/28/2013)

CHANGE FORM ID: CF2

# **SOP TITLE:** SM 2540D/ASTM D 3977 TOTAL SUSPENDED SOLIDS (NON-FILTERABLE RESIDUE) AND SEDIMENT CONCENTRATION

# **REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary):**

1) Addition of preparation of MRL check standard.

2) Clarification on when an NCM is required for samples with a residue of less than 2.5mg.

3) Requirement to filter a full liter for the method blank.

4) Addition of checks to ensure that method blanks and ND samples are NOT reported with an elevated RL.

# CHANGE OR ADDITION (Use additional sheets if necessary):

Add section 9.1.4

9.1.4 <u>Reporting Limit Check Standard (MRL Check)</u>

Prepare and analyzed an MRL check with each batch of 20 samples or less only **if required by a specific program or client project**. MRL check recovery must be within the specified program or client recovery limits.

# Add section 9.1.5

9.1.5 Minimum Residue Requirement

SM2540D requires that a full 1L (1000 mL) of sample must be filtered if the final residue is less than 2.5 mg. If the final residue is less than 2.5 mg **AND** less than 1 L of sample is filtered, an NCM must be written to state why this method requirement was not followed. The 2.5 mg requirement must be met REGARDLESS of the reporting limit requested for the sample and REGARDLESS of whether or not the sample has a result above the reporting limit.

# Add section 10.1.4

10.1.4 Reporting Limit Standard (MRL Check)

MRL check levels are prepared, if required, at the program or client-specified RL.

Prepare an X mg/L MRL Check Standard (where X= the reporting limit required) by pipetting X mL of 1000 mg/L LCS Stock Standard into a 1 L volumetric flask and bringing to volume with laboratory reagent grade water. Store at >0 to 6°C for up to one month.

# **Revise section 10.1.2**

Currently:

A method blank is processed by filtering 100 mL of Reagent Grade water through a filter in the same manner as the samples.

Change to read:

A method blank is processed by filtering **1000** mL of Reagent Grade water through a filter in the same manner as the samples. 1000 mL is <u>always</u> filtered for the method blank regardless of the program or client-specified RL.

# **Revise section 10.3.4**

Currently:

Rinse the filtering apparatus prior to use. Apply a vacuum while rinsing the disc with 3 successive 20 mL volumes of ultrapure water. Remove all traces of water by continuing to apply vacuum after the water has passed through the filtration device. Discard the resulting rinse water.



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# CHANGE FORM ID: CF2

Change to read:

Rinse the filtering apparatus prior to use. Apply a vacuum while rinsing the disc with a minimum of 60 mL of laboratory reagent water applied using the directed spray from a wash bottle. Remove all traces of water by continuing to apply vacuum after the water has passed through the filtration device. Discard the resulting rinse water.

#### **Revise section 10.3.7**

#### Currently:

Filter the sample through a glass fiber filter. Rinse the graduated cylinder, the filter, the non-filterable residue and the funnel walls with 3 portions of ultrapure water while applying a vacuum. Continue to apply a vacuum until the filtration is complete to remove as much water as possible. Allow time for complete drainage between each rinsing.

#### Revise to read:

Filter the sample through a glass fiber filter. Rinse the graduated cylinder, the filter, the non-filterable residue and the funnel walls with a minimum of 60 mL of laboratory reagent water applied using the directed spray from a wash bottle while applying a vacuum. Continue to apply a vacuum until the filtration is complete to remove as much water as possible. Allow time for complete drainage between each rinsing.

# Add section 11.4 11.4 Reporting limit Calculation

**Final RL** =  $RL_{base} \times \frac{V_{no \min al}}{V_{sample}}$ 

Where  $RL_{base}$  = the requested RL (mg/L)  $V_{nominal}$  = the nominal or "final" volume in the TALS batch (mL)  $V_{sample}$  = the volume of sample filtered (mL)

#### **Revised data review checklist**

Added a check that ND results are not reported at an elevated RL. Added a check that MB RL does not exceed base RL requested.

| Prepared By: D. Dawes     |                          |
|---------------------------|--------------------------|
| APPROVED BY:              |                          |
| Department Manager        | Date 5/13                |
| Quality Assurance Manager | <u> 2-3-20 3</u><br>Date |



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# FACILITY SOP ATTACHMENT

| SOP NUMBER: IR-WET-TSS, Rev 4 (8/28/2013)        | CHANGE FORM ID: CF2 |
|--|---------------------|
| Environmental Health and Safety Coordinator Date | 03/2013             |
| Laboratory Director Date 12/0                    | 03/13               |
| Control Copy Number                              |                     |
| unontholiph                                      |                     |



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| Data | Review | Checklist |
|------|--------|-----------|
|      |        |           |

|                               | DAILY DATA CHECKLIST   |
|-------------------------------|--|
|                               | Total Suspended Solids –SM2540D  |
|                               |  |
|                               |  |
| Analyst:<br>Analysis Date:    | 2 <sup>nd</sup> Level Review:<br>Date:                                     |
| Batch ID:                     |  |
|                               |  |
| Analyst 2 <sup>nd</sup> Level |  |
| Rev Rev                       |  |
|                               | Calibration  |
| <u> </u>                      | Daily balance calibration verification has been performed                  |
|                               | Beginning and ending oven temperatures are recorded                        |
|                               | Date/Time IN and time OUT are recorded                                     |
|                               | Temperatures within the required temperature range of the method           |
|                               | Sample Preparation Batch   |
|                               | Batch contains no greater than 20 samples                                  |
|                               | Batch contains a passing Method Blank (< 10 mg/L)                          |
|                               | Batch contains a passing LCS (%R= 85-115)                                  |
|                               | Batch contains a Duplicate for every 10 (or fewer) samples (SA/DU RPD<5)   |
|                               |  |
|                               | Analysis   |
|                               | Constant weight is achieved for all samples and QC (diff <0.5mg)           |
|                               | Total residue for each sample does not exceed 200mg                        |
|                               | Final residues less than 2.5mg must be based on 1000 mL of filtered sample |
|                               | Elevated RLs are NOT reported for ND samples                               |
|                               | Method Blank RL does not exceed base RL requested                          |
|                               |  |
|                               | Documentation  |
|                               | All required TALS datatypes are entered                                    |
|                               | All standards used are uniquely identified and are not expired             |
|                               | All data flags correctly applied and NCMs written, as required             |
| Comments:                     |  |
|                               |  |
|                               |  |



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# FACILITY SOP ATTACHMENT

| SOP NUMBER: IR-WET-TSS, Rev 4 (8/28/2013)  | CHANGE FORM ID: CF1   |
|--|---|
| <b>SOP TITLE:</b> SM 2540D/ASTM D 3977 TOTAL SUSPENDED<br>RESIDUE) AND SEDIMENT CONCENTRATION  | SOLIDS ( NON-FILTERABLE   |
| REASON FOR ADDITION OR CHANGE (Use additional sh   | eets if necessary):   |
| SM2540D specifies a sample duplicate to be analyzed at a 10% f specifies a frequency of 5%.  | requency. The SOP currently   |
| CHANGE OR ADDITION (Use additional sheets if necessary   |   |
| Section 9.1.3 Sample Duplicate   |   |
| <u>Currently:</u><br>Prepare and analyze a sample duplicate for every batch of 20 sams sample and duplicate readings should be 10% if the sample residue be performed.       | ples or less. The RPD between the e is $> 10$ mg or a third analysis must |
| Revise to read:<br>Prepare and analyze a sample duplicate for 10 samples or less. The duplicate readings should be 10% if the sample residue is $> 10 \text{ mg}$ performed. |   |
| Prepared By: D. Dawes  | 0   |
| *APPROVED BY:<br>Department Manager Date   | 6/25/2013   |
| Quality Assurance Manager Date   | -25-2013  |
| Environmental Health and Safety Coordinator Date   | 5/2013  |
| Laboratory Director 10/2   | 25/13   |

\*Should be the same signature authorities of SOP being revised. Control Copy Number \_\_\_\_



SOP No. IR-WET-TSS, Rev. 4 Effective Date: 08/28/2013 Page No.: 1 of 12

# Title: Total Suspended Solids (Non-filterable residue) and Sediment Concentration SM2540D

|                           | Approvals (Si     | gnature/Date):              |
|---------------------------|-------------------|-----------------------------|
| Tung Nguyen               | <u>8/27/1</u> 3   | William Nash                |
| Department Manager        | Date              | Health & Safety Coordinator |
| Maria Friedman            | <u>8-27-2</u> 013 | Kirk Miltimore              |
| Quality Assurance Manager | Date              | Laboratory Director         |

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# 1.0 SCOPE AND APPLICATION

**1.1** This procedure is used to determine the non-filterable residue (Total Suspended Solids) in drinking water, surface water, saline water, domestic waste and industrial waste.

**1.2** The standard reporting limit for water is 10 mg/L. Lower reporting limits can be achieved using a larger sample volume. Note that RLs are subject to change based on annual method detection limit (MDL) studies.

**1.3** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in "Validation of Methods" in the Quality Assurance Manual.

# 2.0 SUMMARY OF METHOD

**2.1** For TSS, a well-mixed sample is filtered through a glass fiber filter and the residue retained on the filter is dried to constant weight at 103°C to 105°C.

**2.2** For sediment concentration, a thoroughly mixed aliquot of sample is either weighed or measured volumetrically and then filtered through a tared glass-fiber filter. Dry the filter and filtrate to constant weight at 105°C.

# 3.0 DEFINITIONS

**3.1** <u>Total Suspended Solids</u> is also referred to as non-filterable residue. It is those solids that are retained by a glass fiber filter and dried to a constant weight at 103°C to 105°C.

**3.2** <u>Sediment concentration</u> is defined as either the ratio of the mass of dry sediment in a water-sediment mixture to the mass of the mixture or, as the ratio of the mass of the dry sediment to the volume of the mixture. At concentrations below 8000 mg/L these two definitions are the same.

# 4.0 INTERFERENCES

**4.1** Filtration apparatus, filter material, pre-washing, post-washing and drying temperature are specified because these variables have been shown to affect the results.

**4.2** Sample high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

# 5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

## 5.1 Specific Safety Concerns or Requirements

Personal Protective Equipment Required: Safety Glasses/Face Shield, Lab coat, Nitrile/Cutresistant Gloves

# 5.2 Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

| Material   | Hazards  | Exposure<br>Limit (1) | Signs and symptoms of exposure   |
|--|----------|-----------------------|--|
| Sigmacell<br>Cellulose   | Irritant | 15 mg/m3<br>TWA       | May be harmful if inhaled. May cause respiratory tract<br>irritation. May be harmful if absorbed through skin. May<br>cause skin irritation. May cause eye irritation. May be<br>harmful if swallowed. |
| 1 – Exposure limit refers to the OSHA regulatory exposure limit. |          |                       |  |

#### 6.0 EQUIPMENT AND SUPPLIES

#### 6.1 <u>Instrumentation</u>

- 6.1.1 Filtering apparatus: filter holder, membrane filter funnel
- 6.1.2 Suction Flask
- 6.1.3 Drying oven
- 6.1.4 Desiccator
- 6.1.5 Analytical balance, capable of weighing to 0.1mg

## 6.2 <u>Supplies</u>

- **6.2.1** Glass fiber filter disc, Environmental Express PreWeigh F93447MM pre-weighed 47mm, pore size: 1.5μm, or equivalent
- 6.2.2 Graduated cylinder
- 6.2.3 50mL aluminum weighing dishes
- 6.2.4 Tongs and gloves

# 7.0 REAGENTS AND STANDARDS

- 7.1.1 Laboratory reagent water
- 7.1.2 SigmaCell® Cellulose -20µ

## 8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

| Matrix | Sample<br>Container | Min. Sample<br>Size | Preservation   | Holding Time | Reference         |
|--------|---------------------|---------------------|----------------|--------------|-------------------|
| Waters | Poly                | 1L                  | Cool >0 to 6°C | 7 Days       | 40 CFR Part 136.3 |
| Soils  | 4 oz. Jar           | 100 g               | Cool >0 to 6°C | 7 Days       | N/A               |

#### 9.0 QUALITY CONTROL

9.1 <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

#### 9.1.1 <u>Method Blank (MB)</u>

Prepare and analyze a method blank (MB) for each matrix and with every batch of 20 samples, or less. Check that there are no analytes detected at or above the reporting limit. If the method blank shows contamination, re-prepare all samples in the batch unless:

- The sample is ND (flag and write an NCM).
- The sample result is > 10x the blank level (flag and write an NCM).

#### 9.1.2 Laboratory Control Sample (LCS).

Prepare and analyze a primary source laboratory control sample (LCS) for every batch of 20 samples or less. The LCS recovery must be within laboratory acceptance limits (see attachment 1). If the LCS is outside of these limits, re-prepare the whole batch and/or re-calibrate the system unless:

• The LCS recovery is above the upper limit and samples are ND. Flag and write an NCM.

#### 9.1.3 <u>Sample Duplicate.</u>

Prepare and analyze a sample duplicate for every batch of 20 samples or less. The RPD between the sample and duplicate readings should be 10% if the sample residue is  $\geq$  10 mg or a third analysis must be performed.

• If the third analysis still does not fall within10%, report the result with an NCM.

If the residue is < 10 mg, flag the data to indicate "due to the low levels of analyte in the sample, the duplicate RPD calculation does not provide useful information".

For result with a final residue of more than 10 mg, duplicate analysis results must agree within 5% of the <u>average</u> result of the pair. This is equivalent to an RPD of 10%.

#### 9.2 Instrument QC

#### 9.2.1 Balance Calibration Verification Tolerance Levels

The tolerance levels for the balance are indicated in the following table.

|      | Tolerance Level  | S                   |
|------|--|---------------------|
|      | Top Loaders<br>(Balances with 2 and 3<br>decimal places) | Analytical balances |
| 0.5g | 0.49 g – 0.51 g  | 0.4999 g – 0.5001 g |

| 1 g   | 0.99 g – 1.01 g    | 0.9999 g – 1.0001 g    |
|-------|--------------------|------------------------|
| 50 g  | 49.95 g – 50.05 g  | 49.9995 g – 50.0005 g  |
| 100 g | 99.90 g – 100.10 g | 99.9990 g – 100.0010 g |

#### 10.0 PROCEDURE

#### 10.1 <u>Standard Preparation</u>

#### 10.1.1 LCS Stock Standard

Prepare a 1000 mg/L LCS standard by adding 1.00 g of cellulose to 1 L of DI Water. Shake thoroughly before use. Discard after one month. Store the solution in the refrigerator at >0 to  $6^{\circ}$ C.

#### 10.1.2 Method Blank-MB

A method blank is processed by filtering 100 mL of Reagent Grade water through a filter in the same manner as the samples.

#### 10.1.3 Laboratory Control Sample-LCS

An LCS is processed by filtering 100 mL of TSS LCS solution through a filter in the same manner as the samples.

#### 10.2 Instrument Initialization and Calibration

Verify the analytical balance has been leveled and its calibration checked prior to use.

## 10.3 Sample Preparation and Analysis

**10.3.1** Purchase pre-rinsed and pre-weighed filters from the supplier. Each filter comes in its own aluminum weighing dish.

**10.3.2** Wear gloves when handling a weighing dish since oils from your skin can slightly increase the weight of the dish.

**10.3.3** Label each dish with the sample number prior to filtering the sample.

**10.3.4** Rinse the filtering apparatus prior to use. Apply a vacuum while rinsing the disc with 3 successive 20 mL volumes of ultrapure water. Remove all traces of water by continuing to apply vacuum after the water has passed through the filtration device. Discard the resulting rinse water.

**10.3.5** Assemble the filtering apparatus by placing the pre-rinsed and pre-weighed glass fiber filter disc on the membrane filter apparatus.

**10.3.6** Shake the sample vigorously and transfer an appropriate volume of sample to the filter using a graduated cylinder.

- Final residues must be at least 2.5 mg (up to a filtered volume of 1L) but no more than 200 mg
- Rely on analyst judgment and experience to adjust the filtered volume in order to meet these residue criteria.

• If the final residue exceeds 200mg, re-analyze the sample with a smaller aliquot. To estimate the re-analysis volume, divide 200 by the actual residue; multiply this by the volume originally used and round down to an easily measured multiple of 10.

$$new\_vol = \frac{200}{residue} \times original\_vol$$

• If the final residue is less than 2.5mg, re-analyze the sample with a larger aliquot (not to exceed 1L). To estimate the re-analysis volume, divide 2.5 by the actual residue; multiply this by the volume originally used and round UP to an easily measured multiple of 10. If the re-analysis volume is greater than 1L, use 1L for re-analysis.

$$new\_vol = \frac{2.5}{residue} \times original\_vol$$

**10.3.7** Filter the sample through a glass fiber filter. Rinse the graduated cylinder, the filter, the non-filterable residue and the funnel walls with 3 portions of ultrapure water while applying a vacuum. Continue to apply a vacuum until the filtration is complete to remove as much water as possible. Allow time for complete drainage between each rinsing.

**10.3.8** Remove the filter from the filter support and transfer it to its aluminum weighing dish using tongs only.

**10.3.9** Dry the filter for a minimum of 2 hours, or overnight at 103°C to 105°C. Record the time in, time out, and the temperature of the oven into the TALS Batch Notes when the sample is in the oven.

**10.3.10** Cool the sample in a desiccator for about 30 minutes. Weigh the sample after it is cool.

**10.3.11** Repeat the drying cycle until constant weight is obtained (weight loss is less than 0.5 mg). Record all the weights directly into TALS worksheet. Use the lowest dried residue weight in the final calculation.

# 10.3.12 Procedure for soil samples

Weigh 5  $\pm$  0.05 grams of the well mixed sample into a disposable 50 mL centrifuge tube. Add 40mL Laboratory Reagent Grade water using a Class A graduated cylinder. All initial and final amounts must be documented. Shake samples by hand to ensure water and soil are mixed and then place on an orbital shaker for minimum of 10 minutes. If necessary, centrifuge for 3 – 5 min or until separation of the phases occurs. Filter the resultant supernatant water through a 0.2um filter. This filtrate can now be analyzed in the same manner as regular water samples.

# 10.4 Preventative Maintenance

- **10.4.1** Clean the balance after every use. This is a courtesy as well as a safety matter. This also adds to the operational life of the balance.
- **10.4.2** If the balance is unusable or has limitation to its use, it must be tagged immediately and reported to QA staff.

# 11.0 CALCULATIONS / DATA REDUCTION

## 11.1 Accuracy

LCS % Recovery = <u>observed concentration</u> x 100 known concentration

# 11.2 Precision (RPD)

Sample Duplicate = <u>|orig. sample value - dup. sample value|</u> x 100 [(orig. sample value + dup. sample value)/2]

## 11.3 <u>TSS Concentration</u>

TSS (mg/L) =  $(A - B) \times 1,000,000$ 

Where:

A = weight of filter + residue in grams (the lowest of the replicate weightings)

B = weight of filter (gram)

C = volume of sample filtered (ml)

# 12.0 METHOD PERFORMANCE

# 12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure as described in laboratory's SOP, IR-QA-MDL.

# 12.2 <u>Demonstration of Capabilities</u>

Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples. An IDOC consists of 4 consecutive LCS samples with an average recovery and RSD within the in-house statistical limits. An ODOC can be 4 consecutive LCSs or a passing PT.

## 12.3 Training Requirements

The analyst must have documented training, including reading of the SOP and source methods, conducted by the department manager, senior chemist, or other analyst with training documentation and a passing DOC.

# 13.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in the "Waste Management and Pollution Prevention" section of the Corporate Environmental Health and Safety Manual (CW-E-M-001).

## 14.0 WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures

are incorporated by reference to the laboratory's Waste Disposal SOP (IR-EHS-WASTE). The following waste streams are produced when this method is carried out:

#### Non-Hazardous Waste: Total Suspend Solids.

Non-Hazardous waste is disposed of by pouring the samples water that have been extracted into the sink, measuring the pH and neutralizing the water using soda ash, and then draining the neutralized contents into the sewer system. The soil generated in these tests is collected in the 55-gallon closed head metal drum in the wetchem area. Sample archive technicians label the drum with a preprinted label of Non-RCRA Hazardous waste solid.

Wetchem analysts/technicians are responsible for neutralizing this waste in the glassware washing room.

<u>Unused standards</u> All these departments generate unused and expired standards. If the standard is hazardous and can not be collected with one of the waste streams generated in the method, it must be labeled as hazardous waste with a date that the material became waste. The analyst or technicians take this standard and placed it on the shelves labeled "hazardous waste" in the main waste storage area. The standard will be lab packed (example: mercury standard). If the standard can be collected in the satellite waste container for one of the waste streams of the method, then pour the standard in the right satellite container, rinse the original container, and collect the rinsate in the satellite container. The original container can be placed in the regular trash. (Example, buffer solutions pH 4)

## 15.0 REFERENCES / CROSS-REFERENCES

- **15.1** Method 2540D, Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition 1998.
- 16.0 METHOD MODIFICATIONS None
- 17.0 ATTACHMENTS
- 17.1 <u>Attachment 1:</u> Analysis Information
- 17.2 Attachment 2: Datatypes
- 17.3 Attachment 3: Data Review Checklist
- 18.0 **REVISION HISTORY**
- 18.1 Revision 0, dated 31 January 2008
  - Integration for TestAmerica and STL operations
  - This revision supersedes 160\_2.SOP, revision 8 (06/07/07)

#### 18.2 <u>Revision 1, dated 17 February 2010</u>

- This revision supersedes IR-WET-TSS, revision 0 (01/31/08)
- Changes made in accordance with "Template\_Method SOP Format\_R2-1"
  - Addition of Safety sections 5.1, 5.2, and table in 5.2
  - Addition of Pollution Control wording
  - Addition of Waste Management wording

- Addition of determination of Sediment Concentration by ASTM D 3977, Method B
- Revision of Demonstration of Capabilities section.
- Prepared by LH

#### 18.3 Revision 2, dated 01 March 2011

- This revision supersedes IR-WET-TSS, revision 1 (02/17/10)
- Removal of reference to EPA 160.2
- Revised Sample Duplicate RPD requirement
- Corrected MDL specification
- Added data review checklist
- Revised by DK and LH

#### 18.4 <u>Revision 3, dated 01 August 2012</u>

- This revision supersedes IR-WET-TSS, revision 2 (03/01/11)
- Added Balance Calibration Verification Tolerance Levels
- Removed Analysis Logbook page; added datatypes
- Revised by DK and LH

#### 18.5 Revision 4, dated 28 August 2013

- This revision supersedes IR-WET-TSS, revision 3 (08/01/12) and IR-WET-TSS\_r3-CF1 (07/02/13)
- Specify that a minimum residue of 2.5mg is required and to increase filtered volume up to 1000mL to meet this minimum.
- Removed requirement that MDL is based on lowest discernable unit of measure on the balance.
- Removed the nominal sample volume used
- Removed ASTM D 3977-97
- RLs are subject to change based on annual method detection limit (MDL) studies.
- Revised by DK, LH and DD.

| Analysis Information<br>TestAmerica Irvine                                    |     |                    |                 |                  |                        |                             |
|---|-----|--------------------|-----------------|------------------|------------------------|-----------------------------|
|   |     | Analytical I       | Method Info     | rmation          |                        |                             |
| Analyte   | MDL | Reporting<br>Limit | Surrogate<br>%R | Duplicate<br>RPD | Matrix Spike<br>%R RPD | Blank Spike / LCS<br>%R RPD |
| TSS - in Water (SM 2540D)<br>Preservation:4 C, Cool<br>Container:1 Liter Poly |     | Am                 | ount Required:  | 100 ml           | Hold Time              | e:7 days                    |
| Total Suspended Solids  | 5   | 10 mg/l            | l               | 10               | -NO                    | 85 - 115                    |
|   |     | ·                  | $\langle$       | S                |                        |                             |
|   |     |                    | 5               | ¥                |                        |                             |
|   | 5   |                    | v               |                  |                        |                             |
|   |     |                    |                 |                  |                        |                             |

# Attachment 2 Datatypes

| Method Code | Datatype Description                     | Value to Enter                  | Units   |
|-------------|--|---------------------------------|---------|
| 2540D       | Perform Calculation (0=No, 1=Yes)        | Enter [1]                       | NONE    |
| 2540D       | Nominal Amount Used                      | Enter volume                    | mL      |
| 2540D       | Oven ID                                  | [Specify]                       | NONE    |
| 2540D       | ID number of the thermometer             | Enter ID# and Correction factor | NONE    |
| 2540D       | Date samples were placed in the oven     | Enter date & time IN            | NONE    |
| 2540D       | Uncorrected In Temperature               | N/A                             | Celsius |
| 2540D       | Oven Temp when samples are put in oven   | Enter corrected temp            | Celsius |
| 2540D       | Date samples were removed from oven      | Enter date & time OUT           | NONE    |
| 2540D       | Uncorrected Out Temperature              | N/A                             | Celsius |
| 2540D       | Oven Temp when samples removed from oven | Enter corrected temp            | Celsius |
| 2540D       | Filter Paper Lot Number                  | [Specify]                       | NONE    |
| 2540D       | Constant Weight (WT2) Temp In            | Enter temperature               | Celsius |
| 2540D       | Uncorrected CW (Wt2) Temp In             | NA                              | Celsius |
| 2540D       | Constant Weight (WT2) Temp Out           | Enter temperature               | Celsius |
| 2540D       | Uncorrected CW (Wt2) Temp Out            | NA                              | Celsius |
| 2540D       | Constant Weight (WT3) Date/time In       | Enter date & time               | NONE    |
| 2540D       | Constant Weight (WT3) Date/Time Out      | Enter date & time               | NONE    |
| 2540D       | Constant Weight (WT3) Temp In            | Enter temperature               | Celsius |
| 2540D       | Uncorrected CW (Wt3) Temp In             | NA                              | Celsius |
| 2540D       | Constant Weight (WT3) Temp Out           | Enter temperature               | Celsius |

# <u>Attachment 3</u> Data Review Checklist

|                               | DAILY DATA CHECKLIST<br>Total Suspended Solids – SM2540D  |
|-------------------------------|---|
|                               | Total Suspended Solids – Sm2340D  |
|                               |   |
| Analyst                       | 2 <sup>rd</sup> Level Review:   |
| Analysis Date:<br>Batch ID:   | Date:   |
|                               |   |
|                               |   |
| Analyst 2 <sup>nd</sup> Level |   |
| Rev Rev                       | Calibration   |
|                               | Daily balance calibration verification has been performed   |
|                               | Beginning and ending oven temperatures are recorded   |
|                               | Date/Time IN and time OUT are recorded  |
|                               | Temperatures within the required temperature range of the method  |
|                               | Samuela Barrantia Batta   |
|                               | Sample Preparation Batch<br>Batch contains no greater than 20 samples   |
|                               | Batch contains a passing Method Blank (< 10 mg/L)   |
|                               | Batch contains a passing LCS (%R= 85-115)   |
|                               | Batch contains a Duplicate (SA/DU RPD<5)  |
| 2.5 <u> </u>                  |   |
|                               | Analysis  |
|                               | Constant weight is achieved for all samples and QC (diff <0.5mg)  |
|                               | Total mg residue for each sample does not exceed 200  |
|                               | Final residues less than 2.5mg must be based on 1000 mL of filtered sample  |
|                               |   |
|                               | Documentation   |
|                               | All required TALS datatypes are entered   |
|                               | All standards used are uniquely identified and are not expired  |
|                               | All data flags correctly applied and NCMs written, as required  |
| Comments:                     | (a) Solution (Constraint) (C |
|                               |   |
|                               |   |
| <u> </u>                      |   |
|                               |   |
|                               |   |
|                               |   |
|                               | TSS_check_r2.DOC  |
|                               | Rev 08/07/13  |
|                               |   |
|                               |   |