Title: Total Dissolved Solids; Filterable Residue Methods SM 2540C & EPA 160.1

Approvals (Signature/Date):

Tung Nguyen — 9/30/13 — Date Department Manager	William Nash — 09/30/2013 — Date Environmental Health & Safety Coordinator
Maria Friedman — 9-27-2013 — Date Quality Assurance Manager	Kirk Miltimore — 09/30/13 — Date Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the filterable residue (total dissolved solids) in drinking water, surface water, saline water, domestic waste and industrial waste.

1.2 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated to near dryness in an oven below 100°C, and then dried to constant weight at 180°C.

1.3 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.

1.4 This method has been modified for the analysis of soils in one of two ways:

- Based on soil ("soluble" TDS), by preparing a 10-fold leachate prior to filtration. Results are reported as mg/kg.
- Based on a leachate, by preparing a leachate using a 1:1 preparation factor prior to filtration. Results are reported as mg/L.

1.5 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

1.6 The reporting limit is for waters and leachates is 10 mg/L when using 100 mL sample aliquot. The reporting limit for soils is 100 mg/kg. Note: Although lower reporting limits may be achieved using larger sample aliquots, the logistics of handling significantly large volumes may make the analysis impractical.

1.7 EPA Method 160.1 is approved only for the reporting of drinking water samples.

2.0 SUMMARY OF METHOD

A measured aliquot of aqueous sample is filtered through a glass fiber filter into a tared glass beaker. The beaker is dried to constant weight at 180°C. The weight of residue divided by the sample aliquot volume yields Total Dissolved Solids in the sample.

3.0 DEFINITIONS

There are no additional specific definitions associated with this test. See the laboratory QA manual Standard Methods 2540C for general definitions.

4.0 INTERFERENCES

Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.

Wear gloves to pick up a weighing dish since oil from your skin can slightly increase the weight of the dish.

Too many residues in the evaporating dish will crust over and entrap water that will not be driven off during drying. **Total residue should be limited to about 200 mg.**

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. 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

5.1.1 Personal Protective Equipment Required: Safety Glasses, Labcoat, Nitrile gloves

5.1.2 When working with the drying oven use temperature resistant gloves to handle hot material

5.2 Primary Materials Used

There are no materials used in this method that have a significant or serious hazard rating.

6.0 EQUIPMENT AND SUPPLIES

6.1 Equipment

6.1.1 Glass fiber filter disc, Whatman 934AH, Gelman A/E, Millipore AP40, Environmental Express ProWeigh, or equivalent

6.1.2 Filter holder, membrane filter funnel

6.1.3 500 mL suction flask

6.1.4 Drying oven, 180± 2°C

6.1.5 Desiccator

6.1.6 Analytical balance

6.2 Supplies

6.2.1 Graduated cylinders, class A

6.2.2 150 mL glass beakers

7.0 REAGENTS AND STANDARDS

7.1 Standards

7.1.1 Sodium chloride, 1000 mg/L

All purchased standards must be accompanied by a Certificate of Analysis (C of A) which is kept available at the laboratory in order to demonstrate traceability of the standard to certified (NIST-traceable) source material, if available.

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 Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	1L Poly	150 mL	Cool >0 to 6°C	7 Days	40 CFR Part 136.3
Soils	4 oz. Jar	100 g	Cool >0 to 6°C	28 Days*	N/A

* 28 days from collection to preparation (leaching); 7 days from preparation to analysis.

9.0 QUALITY CONTROL

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Method Blank (MB)

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 samples are ND (qualify the result accordingly).
- The sample result is > 10x the blank level (qualify the result accordingly).

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 of (see attachment 1). If the LCS is outside of these limits, re-prepare the entire batch unless:

- The LCS recovery is above the upper limit and samples are ND. Qualify sample results accordingly.

9.1.3 Sample Duplicate.

Prepare and analyze a sample duplicate for each matrix and with 10 samples, or less. The relative percent difference (%RPD) must be ≤ 5% of the average result of the pair if the sample residue is ≥ than 10 mg or a third analysis must be performed. If the third analysis still does not ≤ 5% of the average, report the results with an NCM.

If the residue is < 10 mg then the RPD must be ≤ 20%.

10.0 PROCEDURE

10.1 Standard Preparation

10.1.1 Prepare the laboratory control sample solution by dissolving 1.000 g of NaCl in 1000 mL of Laboratory Reagent Grade water to obtain a final concentration of 1000 mg/L
10.1.2 Assign a six month expiration date to the standard.
10.1.3 Enter the standards information into LIMS and have it reviewed by a department manager or peer before using.
10.1.4 Store the solution in the refrigerator at >0 to 6°C

10.2 Sample Analysis

10.2.1 Heat the evaporating glass beakers 180± 2°C for 1 hour.

10.2.2 Cool the evaporating glass beakers in a desiccator and store until needed. Weigh and record the weight of each weighing dish immediately before use. .

10.2.3 Assemble the filtering apparatus with a glass fiber filter. Prepare the glass fiber filter disc by placing the disc on the filter apparatus and wetting it to help it adhere. Rinse the disc with three successive 20 mL aliquots of Laboratory Reagent Grade water while applying a vacuum after the water has passed through the filter. Discard the resulting rinse water.

10.2.4 For all samples, Method Blank and LCS, the analysis volume must be measured in a graduated cylinder and recorded prior to filtration. In addition, after filtration of the initial aliquot, the graduated cylinder must be rinsed with three successive 10 mL aliquots of Laboratory Reagent Grade water. Each 10 mL aliquot is to be poured through filtration apparatus, allowing complete drainage between rinses. After the final rinse, suction must be maintained for approximately 1 minute.

10.2.5 Measure 100 mL of Laboratory Reagent Grade water in a clean graduated cylinder. Filter and rinse. This will be used as the method blank.

10.2.6 Measure 50 mL of the LCS solution into graduated cylinder. Filter and rinse

10.2.7 Check and record the pH and conductivity of all samples.

- If sample pH is <2 or >9, verify correct (unpreserved) container is being used. Notify the project manager and write an NCM indicating that the sample may be improperly preserved.
- Use sample conductivity to determine the volume to filter.

10.2.8 Shake the sample vigorously and then transfer 100 mL (or less) of sample into a graduated cylinder or use a pipette for smaller volume (20 mL or less). Record the volume. Transfer this volume to the filtration apparatus. Vacuum filter the sample directly into a 150 mL glass beaker. Rinse the graduated cylinder with three successive 10 mL aliquots of water. If the total filterable residue is expected to be high, based on conductivity results (>2000 umhos/cm), use smaller volume as follows:

Sample conductivity (uS)	Sample volume to use (mL)
<2,000	100
2,000 to 4,000	50
4,000 to 5,000	20
5,000 to 20,000	10
20,000 to 50,000	5
>50,000	1

10.2.9 For soluble TDS in soil, first prepare a 10X deionized water leachate of the soil and then analyze an appropriate volume of the leachate in the same manner as water samples. Report in mg/Kg.

10.2.10 For leachable TDS in soil, prepare a 1:1 Laboratory Reagent Grade water leachate of the soil and then analyze an appropriate volume of leachate in the same manner as water sample. Report in mg/L.

10.2.11 Evaporate the filtrate to near dryness in an oven below 100°C to prevent splattering. After the filtrate has been evaporated to near dryness, dry the filtrate for 2 hours at **180 ± 2°C** to obtain a constant weight.

10.2.12 Cool the sample in a desiccator. Weigh the sample after it is cool. Repeat the drying cycle until a constant weight is obtained or until the weight loss is less than 0.5 mg. Use the lowest of the final two constant weights weighing in the calculation.

10.2.13 The final residue weight should be no greater than 200mg. If it the residue exceeds this level, there is a potential for high bias in the result. Samples must be either re-analyzed or qualified as estimates.

10.3 Preventative Maintenance

If any equipment is unusable or has limitation to its use, it must be tagged "out of service" until such a time the problem has been corrected. Record the problem, solution and verification of proper operation into the instrument maintenance logbook.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2 Concentration

$$\text{TDS (mg/L)} = \frac{(A - B) \times 1{,}000{,}000}{C} \qquad \text{TDS (mg/Kg)} = \frac{(A - B) \times 1{,}000{,}000}{D}$$

Where:
A = weight of dried residue + dish (g)
B = weight of dish (g)
C = volume of sample or leachate (mL)
D = weight of sample in leachate volume filtered (g)

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 Demonstration of Capabilities

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 at 1 to 4 times the RL with an average recovery and RSD within laboratory acceptance limits. An on-going DOC must be performed annually. An ODOC can be 4 consecutive LCSs at mid-level 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

- **Non-Hazardous Waste**

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.

15.0 REFERENCES / CROSS-REFERENCES

15.1 Standard Method 2540C, Standard Methods for the Examination of Water and Wastewater, 20th Edition 1998.

15.2 EPA Method 160.1, EPA Methods for Chemical Analysis of Water and Waste, Revised March 1983.

16.0 METHOD MODIFICATIONS

Item	Method	Modification
1	*SM 2540C*	*The method has been modified to report TDS in solid samples based on the weight of sample in the leachate volume filtered.*
2	*SM2540C 3.d*	*Sample/Sample Duplicate RPD for samples with residues less than 10 mg is allowed to be up to 20%.*

17.0 ATTACHMENTS

17.1 **Attachment 1**: Analysis information
17.2 **Attachment 2:** Data Review Checklist
17.3 **Attachment 3:** Datatypes

18.0 REVISION HISTORY

18.1 Revision 0, dated 31 December 2007

- Integration for TestAmerica and STL operations
- This revision supersedes 160_1.SOP, revision 8 (06/07/07)

18.2 Revision 1, dated 09 March 2009

- Inserted table of screening conductivity levels
- Prepared by DD.

18.3 Revision 2, dated 23 April 2010

- Addition of Pollution Control wording.
- Addition of Waste Management wording.
- Addition of table for method modifications.
- Revision of Demonstration of Capabilities Section.
- Revised by LH

18.4 Revision 3, dated 29 April 2011

- This revision supersedes IR-WET-TDS, revision 2 (04/23/10)
- Added requirement to flag samples with pH from 2 to 5.
- Clarified requirement that final residue weight must not exceed 200mg
- Added Data Review Checklist
- Revised by MC and LH

18.5 Revision 4, dated 28 September 2012

- This revision supersedes IR-WET-TDS, revision 3 (04/29/11)
- Revised Sample Analysis section
- Removed Logbook Page
- Added datatypes attachment
- Revised by XL and LH

18.6 Revision 5, dated 30 September 2013

- Updated signatories to the SOP
- Added reference to EPA 160.1
- Changed SA/DU requirement from every 20 to every 10 samples
- Revised by DD

Attachment 1
Analysis Information

TestAmerica Irvine

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
TDS - in Water (SM2540C & EPA 160.1)								
Preservation: 4 C, Cool								
Container: 1 Liter Poly		**Amount Required:** 100 ml				**Hold Time:** 7 days		
Total Dissolved Solids	5	10 mg/l		5			90 - 110	10
TDS – in Solid (SM2540C Mod.)								
Preservation: 4 C, Cool								
Container: 4 oz Jar		**Amount Required:** 100 g				**Hold Time:** 28 days		
Total Dissolved Solids	50	100 mg/kg		5			90 - 110	10

Attachment 2
Data Review Checklist

DAILY DATA CHECKLIST
Total Dissolved Solids –SM2540C & EPA 160.1

Analyst: _____ 2nd Level Review: _____
Analysis Date: _____ Date: _____
Batch ID: _____

Analyst Rev	2nd Level Rev	
		Calibration
_____	_____	Daily balance calibration verification is recorded
_____	_____	Beginning and ending oven temperatures are recorded in LIMS
_____	_____	Time IN and time OUT are recorded in LIMS
_____	_____	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 (SA/DU RPD<5)
_____	_____	Duplicate pair (SA/DU) analyzed every 10 samples
		Analysis
_____	_____	Constant weight is achieved for all samples and QC (diff <0.5mg or 5%)
_____	_____	Final residue no greater than 200 mg

Comments: _____

TDS_check_r3.DOC
8/30/2012

Attachment 3
Datatypes

Method Code	Level	Datatype Description	Value to Enter	Units
2540C_Calcd	BATCH	Nominal Amount Used	[100]	mL
2540C_Calcd	BATCH	Oven ID	[Specify]	None
2540C_Calcd	BATCH	Date samples were placed in the oven	Date/time	None
2540C_Calcd	BATCH	Oven Temperature Verification	N/A	Celsius
2540C_Calcd	BATCH	Oven Temp when samples are put in	[Specify]	Celsius
2540C_Calcd	BATCH	Uncorrected In Temperature	N/A	Celsius
2540C_Calcd	BATCH	Date samples were removed from oven	Date/time	None
2540C_Calcd	BATCH	Oven Temp when samples removed from oven	[Specify]	Celsius
2540C_Calcd	BATCH	Uncorrected Out Temperature	N/A	Celsius
2540C_Calcd	BATCH	Balance ID	[Specify]	None
2540C_Calcd	BATCH	Pipette ID	[Specify]	None
2540C_Calcd	BATCH	ID number of the thermometer	[Specify ID and CF]	None
2540C_Calcd	BATCH	Filter Paper Lot Number	[Specify]	None
2540C_Calcd	BATCH	Constant Weight (WT2) Date/Time in	Date/time	None
2540C_Calcd	BATCH	Constant Weight (WT2) Temp In	[Specify]	Celsius
2540C_Calcd	BATCH	Constant Weight (WT2) Date/Time Out	Date/time	None
2540C_Calcd	BATCH	Constant Weight (WT2) Temp Out	[Specify]	Celsius
2540C_Calcd	BATCH	Constant Weight (WT3) Date/time In	Date/time	None
2540C_Calcd	BATCH	Constant Weight (WT3) Temp In	[Specify]	Celsius
2540C_Calcd	BATCH	Constant Weight (WT3) Date/Time Out	Date/time	None
2540C_Calcd	BATCH	Constant Weight (WT3) Temp Out	[Specify]	Celsius
2540C_Calcd	BATCH	Batch Comment	Enter as needed	None