

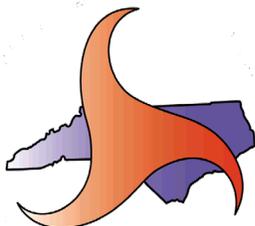


# **MECHANICALLY STABILIZED EARTH WALL AGGREGATE SAMPLING AND TESTING PROCEDURES**



**Materials and Tests Unit**

**August 6, 2020**



**North Carolina  
Department of Transportation**

## **1. SCOPE**

- 1.1 The purpose of this manual is to provide a uniform procedure for obtaining aggregate samples used as backfill material for mechanically stabilized earth (MSE) walls.
  - 1.2 The manual also describes the tests required on the backfill material.
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## **2. REFERENCED DOCUMENTS**

- 2.1 North Carolina Department of Transportation (NCDOT) *Standard Specifications for Roads and Structures*.
  - 2.2 NCDOT Policy for MSE Retaining Walls
  - 2.3 NCDOT Standard MSE Retaining Walls Provision
  - 2.4 AASHTO/NCDOT M&T Standards:
    - Sections 11.10.6.4.2a and 11.10.6.4.2b, AASHTO LRFD Bridge Design Specifications
    - T104, AASHTO Standard Method of Test for Soundness of Aggregate by Use of Sodium Sulfate
    - T112, AASHTO Standard Method of Test for Clay Lumps and Friable Particles in Aggregate
    - T267, AASHTO Standard Method of Test for Determination of Organic Content in Soils by Loss on Ignition
    - T288, AASHTO Standard Method of Test for Determining Minimum Laboratory Soil Resistivity
    - T289, AASHTO Standard Method of Test for Determining pH of Soil for Use in Corrosion Testing
    - NCDOT M&T Chemical Procedure C-Elec, *pH Analysis of Washed Stone Backfill (MSE Wall)*
    - T290, AASHTO Standard Method of Test for Determining Water-Soluble Sulfate Ion Content in Soil
    - T291, AASHTO Standard Method of Test for Determining Water-Soluble Chloride Ion Content in Soil
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## **3. SAMPLING EQUIPMENT**

- 3.1 Scoop, Shovel, or Tube
- 3.2 Distilled Water
- 3.3 5 Gallon Plastic Bag
  - 3.3.1 Use of porous sample bags (cloth or woven plastic) is not allowed.
- 3.4 Plastic Ties

## **4. SAMPLING**

- 4.1 **Sampling from Approved Sources Covered by QC/QA Program** – Samples shall be obtained by technicians certified as NCDOT Aggregate QC/QA Sampling Technicians or as NCDOT Aggregate QC/QA Sampling and Testing Technicians.
  - 4.2 Physical requirements will be determined based on routine QC/QA sampling performed by the Materials and Tests Unit.
  - 4.3 Chemistry/Electrochemical requirements will be based on samples taken on a wall specific basis. Take chemistry/electrochemical on all MSE walls except those utilizing Non-Coastal Plain Coarse Aggregate with steel reinforcement.
    - 4.3.1 The Materials and Tests Unit's Aggregate QC/QA Engineer shall be notified, by the project and Aggregate Producer, that a sample needs to be obtained from the aggregate source prior to use. The initial sampling for each wall will be performed by the Materials and Tests Unit at the quarry (Appendix A, Example 1).
    - 4.3.2 For walls exceeding 3000 yd<sup>3</sup>, additional project samples shall be taken by a certified technician (Appendix A, Example 2). Take one sample for every additional 3000 yd<sup>3</sup>
  - 4.4 Samples for testing of Chemistry/Electrochemical shall adhere to the following:
    - 4.4.1. The method and equipment used to obtain the sample will be the same used for obtaining QC Samples as described in the Aggregate QC/QA Program Manual.
    - 4.4.2 Sample size shall be 10 lbs.
    - 4.4.3 Make sure the large plastic bag is clean and the aggregate is not contaminated. Use of porous sample bags (cloth or woven plastic) is not allowed.
    - 4.4.4 Thoroughly clean and then rinse your scoop, shovel, or tube with distilled water prior to taking the sample.
    - 4.4.5 After taking the sample, ensure that you seal the plastic bag with a plastic tie rap (no metal ties)
    - 4.4.6 The type of reinforcement and connector material (steel or geosynthetic) for the MSE wall shall be stated on the sample card.
    - 4.4.7 Samples shall be submitted to the Central Materials and Tests Laboratory for analysis.
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## **5. GENERAL REQUIREMENTS**

- 5.1 Samples must meet both the chemical and physical requirements before being considered for acceptance.

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## 6. CHEMICAL REQUIREMENTS

- 6.1 The fine aggregate shall conform to the chemical requirements in Tables 1 and 3. Electrochemical testing shown in Table 1 is only required for fine aggregate used with steel reinforcement or connectors.
- 6.2 The coarse aggregate shall conform to the chemical requirements in Tables 2 and 3. Electrochemical testing shown in Table 2 is only required for Coastal Plain coarse aggregate used with steel reinforcement or connectors.
- 6.3 pH testing shown in Table 3 is only required for the following three conditions: (1) fine aggregate regardless of reinforcement or connector material; (2) coarse aggregate used with geosynthetic reinforcement or connectors; and (3) Coastal Plain coarse aggregate used with steel reinforcement or connectors.

**Table 1 – Chemical Composition of Fine Aggregate used with Steel Reinforcement or Connectors**

Property	Requirement*		Test Method
	Min.	Max.	
Resistivity, ( $\Omega\cdot\text{cm}$ )	3,000	--	AASHTO T288 (tested at 100% saturation)
Chlorides, (ppm)	--	100	AASHTO T291
Sulfates, (ppm)	--	200	AASHTO T290

\*Based on Section 11.10.6.4.2a of the AASHTO LRFD Bridge Design Specifications

**Table 2 – Chemical Composition of Coastal Plain\* Coarse Aggregate used with Steel Reinforcement or Connectors**

Property	Requirement		Test Method
	Min.	Max.	
Resistivity, ( $\Omega\cdot\text{cm}$ )	5,000	--	NCDOT Procedure, See Appendix B
Chlorides, (ppm)	--	100	NCDOT Procedure, See Appendix B
Sulfates, (ppm)	--	200	NCDOT Procedure, See Appendix B

\*Coastal Plain as defined by Subarticle 1018-2(B)(1) of the NCDOT Standard Specifications

**Table 3 – pH Composition of Aggregate**

Aggregate Material	Reinforcement or Connector Material	pH Requirement*		Test Method
		Min.	Max.	
Coarse or Fine	Steel	5	10	AASHTO T289 (fine), NCDOT Procedure (coarse), See Appendix B
Coarse or Fine	Geosynthetic	4.5	9	AASHTO T289 (fine), NCDOT Procedure (coarse), See Appendix B

\*Based on the following:

- Section 11.10.6.4.2a of the AASHTO LRFD Bridge Design Specifications for steel reinforcement or connector material
- Section 11.10.6.4.2b of the AASHTO LRFD Bridge Design Specifications for geosynthetic reinforcement or connector material

## 7. PHYSICAL REQUIREMENTS

7.1 The fine aggregate shall conform to the physical requirements in Table 1005-2 and Article 1014-1 of the NCDOT Standard Specifications, as shown in Tables 4 and 5 except fine aggregate is exempt from mortar strength in Subarticle 1014-1(E).

**Table 4 – Fine Aggregate Physical Properties**

Property	Requirement	
	Max.	Test Method
Sodium Sulfate Soundness, 5 cycle weighted average loss, (%)	15	AASHTO T104
Deleterious Substances, (% by weight)	2.0 (natural sand) 1.0 (manufactured sand)	AASHTO T112
Organic Impurities, (%)	1.0	AASHTO T267

**Table 5 – Fine Aggregate Gradation Requirements**

Std. Size # or Class/Type	Percentage of Total by Weight Passing							
	3/8"	#4	#8	#16	#30	#50	#100	#200
1S	100	90 - 100		40 - 85		0 - 20		0 - 3
2S	100	95 - 100	80 - 100	45 - 95	25 - 75	5 - 30	0 - 10	0 - 3
2MS		95 - 100	80 - 100	45 - 95	25 - 75	5 - 35	0 - 20	0 - 8
4S		100	95 - 100			15 - 45	0 - 10	0 - 5
Class III select material, Type 3	100	95 - 100	65 - 100	35 - 95	15 - 75	5 - 35	0 - 25	0 - 8

- 7.2 The coarse aggregate shall conform to the physical requirements in Table 1005-1 and Article 1014-2 of the NCDOT Standard Specifications for standard size No. 57, 57M, 67 or 78M except No. 57 or 57M stone may not be used in the reinforced zone of MSE walls with geosynthetic reinforcement.

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**8. REJECTION**

- 8.1 Samples not meeting specifications will be subject to possible removal and replacement or a pay reduction in accordance with Article 105-3 of the NCDOT Standard Specifications.

APPENDIX A

<p>★ <b>Required Field</b>          † <b>May Be Required Based on Material</b></p>		<p>HICAMS #: <span style="border: 1px solid black; padding: 2px;">1013293</span></p>
<p>★ <b>Material:</b> <u>Screenings - chemistry check</u></p>		<input type="checkbox"/> Metric <input type="checkbox"/> English
<p>† <b>Sample Owner:</b> <u>M&amp;T</u></p>	<p>† <b>Contract #:</b> <u>C204283</u></p>	
<p>★ <b>Testing Category:</b> <u>Acceptance</u></p>	<p>First ID: <u>CA-1A</u></p>	
<p>Check Sample?    <u>Y</u>   <u>N</u>   (circle one)</p>	<p>Proj/Po/Wo#: <u>I-5877</u></p>	
<p>† <b>Related Sample ID:</b> _____</p>	<p>Line Item #: <u>001-587</u></p>	
<p>† <b>Corr Sample ID:</b> _____</p>	<p>RE: <u>Randy Wise</u></p>	
<p># of Pieces: <u>1 Bag</u></p>	<p>★ <b>Rep Qty:</b> <u>3,000 Ton</u></p>	
<p>★ <b>To Be Used In:</b> <u>MSE Wall #1</u></p>		
<p>Comment: <u>Electrochemical for MSE wall</u>  <u>Strap = GeoSynthetic</u></p>		
<p>★ <b>Sampled Date:</b> <u>7-8-20</u></p>		<p>★ <b>Sampled By:</b> <u>Johnny Gilliam</u></p>
<p>★ <b>Sampled From:</b> <u>Stockpile</u></p>		<p>Truck/          Container #: <u>1</u></p>
<p>Structure Number: <u>57</u></p>	<p>Route Desc: <u>Long Branch Road</u></p>	
<p>Route Type:   US NC <input checked="" type="radio"/> SF (circle one)</p>	<p>Alignment: <u>Y13</u></p>	
<p>Route Number: <u>1002</u></p>	<p>★ <b>Location:</b> <u>CL</u>    Offset Dist: _____</p>	
<p>Map Number: _____</p>	<p>★ <b>Sta. From:</b> <u>30+30</u>    <b>Sta. To:</b> <u>30+30</u></p>	
<p>County: <u>Harnett</u></p>	<p>Coastal Plain: <input checked="" type="radio"/> N (circle one)</p>	
<p>† <b>Producer/Supplier:</b> <u>American Materials Co.</u></p>		<p>† <b>Plant ID#:</b> <u>FA483</u>    <input checked="" type="checkbox"/> Approved  <input type="checkbox"/> Other</p>
<p>† <b>Brand Name:</b> _____</p>		<p>Shelf Life Date: _____</p>
<p>† <b>Date Produced:</b> <u>7-8-20</u></p>		<p>† <b>Asphalt Mix/          JMF ID:</b> _____</p>
<p>† <b>Concrete Mix:</b> _____</p>		
<p>† <b>Alternate IDs type:</b></p>	<p>Prefix:</p>	<p>Range:</p>
<p>_____</p>	<p>_____</p>	<p>_____</p>
<p>_____</p>	<p>_____</p>	<p>_____</p>
<p>_____</p>	<p>_____</p>	<p>_____</p>
<p>_____ Description of Items:</p>		
<p>_____</p>		
<p>_____</p>		
<p>Please use reverse side for test data, comments, and additional information. Check here if more reverse <input type="checkbox"/></p>		

Example 1 - Initial chemistry/electrochemical sampled by M&T employee at quarry

* Required Field		HICAMS #: <u>1234567</u>	
† May Be Required Based on Material			
* Material:	<u>Screenings - chemistry check</u>	<input type="checkbox"/> Metric	<input type="checkbox"/> English
† Sample Owner:	<u>Project</u>	† Contract #:	<u>C204283</u>
* Testing Category:	<u>Verification</u>	Field ID:	<u>CA-1A</u>
Check Sample?	<u>Y</u> <u>N</u> (circle one)	Proj/PoWo#:	<u>I-5877</u>
† Related Sample ID:	_____	Line Item #:	<u>001-587</u>
† Corr. Sample ID:	_____	RE:	<u>Randy Wise</u>
# of Pieces:	<u>1 Bag</u>	* Rep. Qty:	<u>3,000 Tons</u>
* To Be Used In:	<u>MSE Wall #1</u>		
Comment: <u>Electrochemical for MSE wall</u> <u>Strap = geosynthetic</u>			
* Sampled Date:	<u>7-15-20</u>	* Sampled By:	<u>Mr. Inspector</u>
* Sample From:	<u>Project</u>	Truck/ Container#:	<u>3</u>
Structure Number:	<u>57</u>	Route Desc:	<u>Long Branch Rd</u>
Route Type:	<u>I</u> <u>US</u> <u>NC</u> <u>SR</u> (circle one)	Alignment:	<u>Y13</u>
Route Number:	<u>1202</u>	* Location:	<u>CL</u> Offset Dist.: _____
Map Number:	_____	* Sta. From:	<u>30+30</u> Sta. To: <u>30+30</u>
County:	<u>Harnett</u>	Coastal Plain:	<u>Y</u> <u>N</u> (circle one)
† Producer/Supplier:	<u>American Materials Co.</u>	† Plant ID#:	<u>FA483</u> <input checked="" type="checkbox"/> Approved <input type="checkbox"/> Other
† Brand Name:	_____	Shelf Life Date:	_____
† Date Produced:	<u>7-15-20</u>	† Asphalt Mix/ JMF ID:	_____
† Concrete Mix:	_____		
† Alternate IDs type:	Prefix	Range:	Description of Items:
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
Please use reverse side for test data, comments, and additional information. Check here if more reverse <input type="checkbox"/>			

Example 2 - Additional chemistry/electrochemical sampled by project employee at project

APPENDIX B

North Carolina Department of Transportation

Materials and Tests Unit

Chemical Laboratory

1801 Blue Ridge Road

Raleigh, NC 27607

Phone: 919-329-4090



## Electrochemical Testing of Stone

Document Number:
Author: Ronald Lichtenwalner, Chemistry Tech. II
Document Approver:
Version Release Date:
Next Review Date:

<b>Modification History</b>			
<b>Rev#:</b>	<b>Date:</b>	<b>Author/Editor /Approver:</b>	<b>Comments/Reasons:</b> (Minor Revisions become a 1.1, 1.2, etc. version; MAJOR revisions advance to 2.0 or 3.0, etc. See supervisors for definitions)
x	2015xxxx	Editor/Approver: NAME ? NAME	

*\* \* This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use, which includes disposal of hazardous materials in an appropriate manner.*

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## **1. SCOPE**

- 1.1 This test method describes the procedures for testing the electrochemistry of stone used as backfill for MSE walls.
- 1.2 Stone samples are tested for pH, conductivity/resistivity, chlorides, sulfates and sulfides.
- 1.3 Testing shall be performed at approximately 77°F (room temperature) unless otherwise noted.
- 1.4 Final test report is submitted to HiCAMS for supervisor approval.

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## **2. REFERENCED DOCUMENTS**

- 2.1 ASTM Standards
  - D1125 – Standard Test Method for Electrical Conductivity and Resistivity of Water
  - D1293 – Standard Test Method for pH of Water
  - D516 – Standard Test Method for Sulfate Ion in Water

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## **3. TERMINOLOGY**

- 3.1 PPE: Personal Protective Equipment.
- 3.2 pH: Measure of the acidity or basicity of an aqueous solution.
- 3.3 Conductivity: Measure of the ability of a solution to conduct electricity.
- 3.4 Resistance: Measure of the difficulty of passing an electrical current through a solution.
- 3.5 Supernatant: The liquid laying above the solid residue after settling.
- 3.6 ppm (parts per million): A measure of concentration.
- 3.7 Filtrate: The fluid that has passed through a filtration process.

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## **4. LIMITATIONS AND SAFETY**

- 4.1 Wear appropriate PPE: Safety Glasses, Lab Coat, and Latex/Nitrile Gloves.
- 4.2 Watch loose clothing and long hair when the rotary extractor is in operation.

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## 5. EQUIPMENT / APPARATUS

### 5.1 Equipment

*Balance*, Capable of reading to the nearest 0.1 g and capable of holding 2000 g, balance shall be verified by certified weight prior to use.

*Lars Lande Rotary Extractor*

*Conductivity Meter*

*pH Meter*

### 5.2 Materials

2000 mL graduated cylinder

1-Gallon wide-mouth plastic container with lid

MQuant sulfate test strips (200 – 1600 mg/L SO<sub>4</sub><sup>2-</sup>)

Hach chloride titrator strips (30 – 600 ppm Cl<sup>-</sup>)

Fisher lead acetate sulfide test strips

150 mL disposable plastic cups

Metal spatula

Pen and sharpie

Fisher Q8 filter paper

Funnel

Stopwatch/Timer

Stone Testing Book

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## 6. REAGENTS

6.1 Distilled Water

6.2 ThermoScientific Aquafast Sulfate Tablets

Avoid touching with hands, use gloves and pop the tablet directly from the foil into the solution.

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## 7. EXTRACTION PROCEDURE

7.1 Place an empty plastic container on to the balance and tare the balance.

7.2 Measure 2000 g of the material to be tested into the container.

7.3 Measure 2000 mL of distilled water in a graduated cylinder and add the water to the container with the 2000g of sample. Record the time and seal the container with the lid.

7.4 Place the container into the rotary extractor, but do not turn the extractor on until 30 minutes have passed since the water was added. Table 1 provides a quick reference for what steps to perform at the designated times after the water has been added.

Table 1: Steps to Perform at Designated Times after Water Added

Time	30 Minutes	2 Hours	4 Hours	24 Hours
Steps	7.5	7.5	7.5, 7.6	7.7

- 7.5 At the 30 minute, 2 hour, and 4 hour times after the water was added, start the extractor and allow the container to rotate for approximately 3 to 5 minutes. While the machine is operating, chain off access to the area to prevent potential injury.
- 7.6 After the final rotation performed at the 4 hour mark, remove the container from the rotary extractor and allow to sit on the benchtop for 20 hours (For a total of 24 hours since the water was added to the container).
- 7.7 After the 24 hour mark, use Fisher Q8 filter paper and a funnel to filter 50 to 100 mL of the supernatant water into a clean disposable plastic cup. Note: Using a separate 150 mL disposable cup to get the water for filtration is easier than trying to pour from the one-gallon jug.

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## 8. ANALYSIS OF FILTRATE

- 8.1 Measure and record the conductivity of the filtrate using a conductivity meter that has first been calibrated per SOP ###. After calibration, record the conductivity in  $\mu\text{s}/\text{cm}$ . Convert the conductivity to resistivity ( $\text{ohm}\cdot\text{cm}$ ) using Equation 1.

$$\rho = \frac{1}{\sigma} \times 1000 (\text{Equation 1})$$

$\rho$	=		The resistivity in $\text{ohm}\cdot\text{cm}$
$\sigma$	=		The conductivity in $\mu\text{s}/\text{cm}$

- 8.2 Measure and record the pH of the filtrate using a pH meter that has first been calibrated per SOP ###. The pH of the sample water should be between 4.5 and 9.5.
- 8.3 Measure the chloride ion concentration using a Hach chloride titrator strip. Insert the titrator strip into the water per the directions on the bottle and use the scale on the bottle to record the quantity in ppm.
- 8.4 Check for the presence of sulfides using a lead acetate test strip. After dipping the strip into the water, it will turn black in the presence of sulfides.
- 8.5 Measure the sulfate ion content using MQuant sulfate test strips. Insert the strip into the water per the instructions on the bottle of the strips. Record the quantity of sulfate in ppm based on how the color change matches the scale provided on the test strip bottle. Note: The bottle may have the scale in  $\text{mg}/\text{L}$ , but  $\text{mg}/\text{L}$  and ppm are approximately the same for the purposes of this test.

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## 9. COLORIMETRIC DETERMINATION OF SULFATE

- 9.1 Allow filtrate to sit for approximately 2 hours after other tests completed.
- 9.2 Turn on spectrophotometer and set wavelength to 420 nm.
- 9.3 Prior to testing actual samples, perform a check using the below procedure on 10, 30, and 50 ppm sulfate standard solutions.
- 9.4 Using a 10 mL disposable pipette, measure and dispense 10 mL of the sample into a 150 mL beaker. Take the water only from the top to minimize turbidity interfering with the results.
- 9.5 Using the same pipette, fill a cuvette with the sample being tested to account for interferences from the water sample itself. Place this blank into the spectrophotometer and zero the instrument.
- 9.6 Set a timer to 2 minutes, and without directly touching a tablet, pop an Aquafast Sulfate tablet into the beaker containing 10 mL of sample and begin the timer.
- 9.7 Crush the tablet using a clean stirring rod and stir at a constant speed until the timer stops.
- 9.8 Immediately after stirring has ceased, pour sample into the cuvette and record the absorbance.
- 9.9 To convert the absorbance to a concentration of sulfate in ppm, use the calibration curve constructed according to Appendix I.

## APPENDIX I: CALIBRATION CURVE

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In order to relate the measured absorbance to the concentration of sulfate in the sample, a calibration curve is constructed with sulfate solutions of known concentrations.

### A. Equipment/Apparatus

- 5 mL, 10 mL & 50 mL Volumetric Pipettes
- Pipette Bulb or Pump
- Nine 100 mL Volumetric Flasks
- Glass Stoppers
- Cuvettes
- Spectrophotometer: Capable of measuring absorbance at 420 nm

### B. Reagents

- ThermoScientific Aquafast Sulfate Tablets
- Distilled Water
- Ricca 100 ppm Sulfate Standard Solution

### C. Procedure

- i. Using an appropriate pipette add 0, 5, 10, 15, 20, 30, 50, 75, and 100 mL of the stock sulfate standard solution to separate labelled 100 mL volumetric flasks. These solutions will contain 0, 5, 10, 15, 20, 30, 50, 75 and 100 ppm of sulfate.
- ii. Dilute each flask to 100 mL with distilled water and mix by inversion.
- iii. Zero the spectrophotometer with a blank containing only distilled water.
- iv. Perform steps 9.4 to 9.8 for each standard solution and record the absorbance.
- v. Enter the data into Excel and perform a linear regression, which will yield the calibration curve that can be used to determine sulfate concentrations in samples.

### D. Calculations

To determine the concentration of sulfate in the original sample, the calibration curve is used. The calibration curve has the form:

$$y = mx + b \text{ (Eq. 1)}$$

Where  $y$  is the absorbance,  $x$  is the concentration in ppm,  $m$  is the slope determined by a linear regression of the data from the calibration curve procedure and  $b$  is the  $y$ -intercept, which is also provided by the linear regression.

This equation is used to solve for the concentration of sulfate in the solution measured with the spectrophotometer by solving for  $x$  and inputting the absorbance value measured for the sample (see Eq. 2)

$$x = \frac{y-b}{m} \text{ (Eq. 2)}$$