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# Evaluation of a Roller Method for Geochemical Leach Testing

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EVALUATION OF A ROLLER METHOD FOR  
GEOCHEMICAL LEACH TESTING

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# EVALUATION OF A ROLLER METHOD FOR GEOCHEMICAL LEACH TESTING

By

Troy Allen Svede

TROY ALLEN SVEDE, B.S. Civil Engineering

THESIS

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## **Abstract**

Currently, multiple tests are being used when evaluating an MSE wall backfill material for pH, conductivity, chloride concentration, and sulfate concentration. There is no one test to measure all four characteristics when testing a soil. Texas Department of Transportation (TxDOT) uses four different tests to measure these characteristics. The goal of this research was to refine a simplified test procedure that was created by David Borrok et al., the Tex-620-M. The method that was developed to replace the current TxDOT test procedures Tex-620-J, Tex-128-E, and Tex-145-E test procedures is the Tex-620-M2. Leaching kinetics were analyzed for gravel and fines by performing 24-hour tests, and a duration of one hour was selected for the Tex-620-M2 method. The results from tests carried out as per Tex-620-M2 were compared to current TxDOT test procedures. Each TxDOT test was compared based on the respective analyte (*i.e.*, pH, electrical conductivity, chloride concentration, and sulfate concentration). Finally, the results from Tex-620-M2 were compared to the results from the current standards used by other agencies.

Comparison of results of the Tex-620-M2 test procedure to the current TxDOT standards for soils passing a No. 40 sieve showed that the Tex-620-M2 method generally leached more chloride and sulfate and resulted in greater conductivity than the current TxDOT standards. Comparison of results from Tex-620-M2 analysis of TxDOT AS, BS, and DS size distributions against results from current standards showed that the current standards are biased to show greater conductivity and chloride and sulfate concentrations because the current methods specify using a finer material. Overall, the proposed test procedure Tex-620-M2 seems to be a simple, comprehensive, and reliable test for highway agencies to consider.

## Table of Contents

Acknowledgements.....	iii
Abstract.....	iv
Table of Contents.....	v
List of Tables .....	vii
List of Figures.....	viii
1. Introduction.....	1
1.1 Background.....	1
1.2 Goals and Objectives .....	2
2. Methodology.....	3
2.1 Soil/Geomaterial Samples.....	3
2.2 Comparison of Soil Leach Test Methods.....	4
2.3 AASHTO Test Procedures.....	5
2.3.1 AASHTO T-289 Determine pH of soil for use in corrosion testing.....	5
2.3.2 AASHTO T-290 Determine sulfate ion content in soil (Method B) .....	5
2.3.3 AASHTO T-291 Determine chloride ion content in soil (Method A).....	6
2.4 ASTM Test Procedures.....	7
2.4.1 ASTM D-4972 Standard test method for pH of soil (Method A).....	7
2.4.2 ASTM D-18 Determine pH of lightweight aggregate .....	8
2.5 NCHRP Test Procedures.....	8
2.5.1 NCHRP-21-06 Measuring pH of soil samples.....	8
2.6 SCDOT Test Procedures.....	8
2.6.1 SC T-143 Coarse aggregate sample for pH and resistivity.....	8
2.7 TxDOT Test Procedures .....	9
2.7.1 Tex-128-E Determining soil pH .....	9
2.7.2 Tex-145-E Determine sulfate content in soils .....	9
2.7.3 Tex-146-E Conductivity test for field detection of sulfate in soil .....	10
2.7.4 Tex-620-J Determine chloride and sulfate contents in soil .....	10
2.8 Development of Tex-620-M2, a modified version of Tex 620-J.....	11
2.8.1 Tex-620-M2 Determine chloride, sulfate, conductivity, and pH in a soil .....	11

2.8.2 Analysis of dissolution kinetics .....	13
2.8.3 Reproducibility of Tex-620-M2.....	15
3. Results and Discussion .....	19
3.1 Comparison of Tex-620-M2 on fines: Comparison to current TxDOT test procedures .....	19
3.2 Comparison of Tex-620-M2 to current standards.....	23
4. Conclusion .....	26
References.....	27
Appendix.....	28
Vita .....	30

## **List of Tables**

Table 2.1 Source location and principal geologic type of soil materials .....	4
Table 2.2 Soil leach tests organized by agency .....	5
Table 2.3 Reproducibility Analysis for pH.....	15
Table 2.4 Reproducibility Analysis for Normalized Conductivity ( $\mu\text{S}/\text{cm}\cdot\text{mL}/\text{g}$ ) .....	16
Table 2.5 Reproducibility Analysis for Chloride Concentration (mg of $\text{Cl}^-/\text{kg}$ of soil) .....	17
Table 2.6 Reproducibility Analysis for Sulfate Concentration (mg of $\text{SO}_4^{2-}/\text{kg}$ of soil).....	18



## List of Figures

Figure 2.1 TxDOT AS, BS, DS gradation size distributions .....	3
Figure 2.2 AASHTO T-290 sulfate titration calibration curve at 400nm .....	6
Figure 2.3 Sample of 200 grams passing sieve 1 ¾” .....	12
Figure 2.4 Bottle Roller (Thermo model 88881003) .....	12
Figure 2.5 Normalized dissolution kinetics: (a) conductivity, (b) chloride, and (c) sulfate .....	14
Figure 3.1 Comparison of Tex-620-M2: pH vs. Tex-128-E .....	19
Figure 3.2 Comparison of Tex-620-M2: normalized conductivity vs. Tex-146-E .....	20
Figure 3.3 Comparison of Tex-620-M2: chloride mass fraction vs. (a) Tex-145-E and (b) Tex-620-J .....	21
Figure 3.4 Comparison of Tex-620-M2: sulfate mass fraction vs. (a) Tex-145-E and (b) Tex-620-J .....	22
Figure 3.5 Comparison of Tex-620-M2: pH vs. current standards .....	23
Figure 3.6 Comparison of Tex-620-M2: normalized conductivity vs. current standards .....	24
Figure 3.7 Comparison of Tex-620-M2: chloride mass fraction vs. current standards .....	25
Figure 3.8 Comparison of Tex-620-M2: sulfate mass fraction vs. current standards .....	25

# **1. Introduction**

## **1.1 BACKGROUND**

Backfill material used for mechanically stabilized earth (MSE) walls must be tested and approved to minimize corrosion of embedded metal reinforcement. In general, the most corrosive soils contain large concentrations of soluble salts, especially in the form of sulfates, chlorides, and bicarbonates and may be characterized as very acidic (low pH) or highly alkaline (high pH) [1]. The characteristic evaluation parameters of the leach water specified by the Texas Department of Transportation (TxDOT) are pH, sulfate concentration, chloride concentration, and electrical conductivity. Other states may refer to the American Society for Testing and Materials (ASTM) or the American Association of State Highway and Transportation Officials (AASHTO) test procedures to analyze these parameters. Regardless of which body of standards is used, multiple tests must be performed to fully analyze a given material since there is not a single test procedure to analyze all four major parameters. Many of the current test procedures specify testing a finer material (*e.g.*, passing No. 10 or No. 40 sieve), which would generally have a higher concentration of dissolved ions in the leachate. However, there is a growing trend of using coarser aggregate as backfill material, and coarse backfills with good drainage and ample aeration may corrode steel at a much lower rate than clay and silt [2]. The current TxDOT standard test procedure used to measure chloride and sulfate ion content is Tex-620-J, which is a 12-hour test at elevated temperature ( $66\pm 11^{\circ}\text{C}$ ). Thus, there is a desire to develop a simplified and comprehensive test procedure that can accommodate coarse aggregate.

In this research, we not only used gradations specified by the methods but also different gradations such as TxDOT AS, BS, and DS gradations. These three gradations had a variation of

gravel, medium, and fine materials. The development of a single leach test to reduce the amount of time to analyze the four main leach parameters and provide consistency in evaluating materials was first proposed by Borrok et al. [2], which resulted in the Tex-620-M (a simplification of the current Tex-620-J method).

## **1.2 GOALS AND OBJECTIVES**

The goal of this research was to standardize the Tex-620-M geomaterial leach test proposed by Borrok et al. [2] to improve the practicability, reliability, consistency, and efficiency in evaluating backfill materials used in MSE walls with metallic reinforcements. The objectives of this research were to: (1) standardize some of the steps proposed in test procedure Tex-620-M that can accommodate larger aggregate (*i.e.*, passing 1  $\frac{3}{4}$ " sieve), as well as a smaller aggregate (*e.g.*, sand), and (2) compare the results from the proposed test procedure to those from existing state and federal standards. The maximum aggregate size of passing 1  $\frac{3}{4}$ " sieve was selected because this size is commonly used throughout Texas.

## 2. Methodology

### 2.1 SOIL/GEOMATERIAL SAMPLES

Twenty backfill materials were collected for use in this research: 13 from Texas, three from Oklahoma, two from Arkansas, one from Florida, and one from South Carolina. The materials are listed in Table 2.1 with their principal geologic type. Figure 2.1 shows the particle size distribution of three TxDOT gradations (AS, BS, and DS) used in the analyses of Tex-620-M. The AS and DS gradations have a higher percent of gravel in the mixture, whereas BS has a higher percentage of fines.

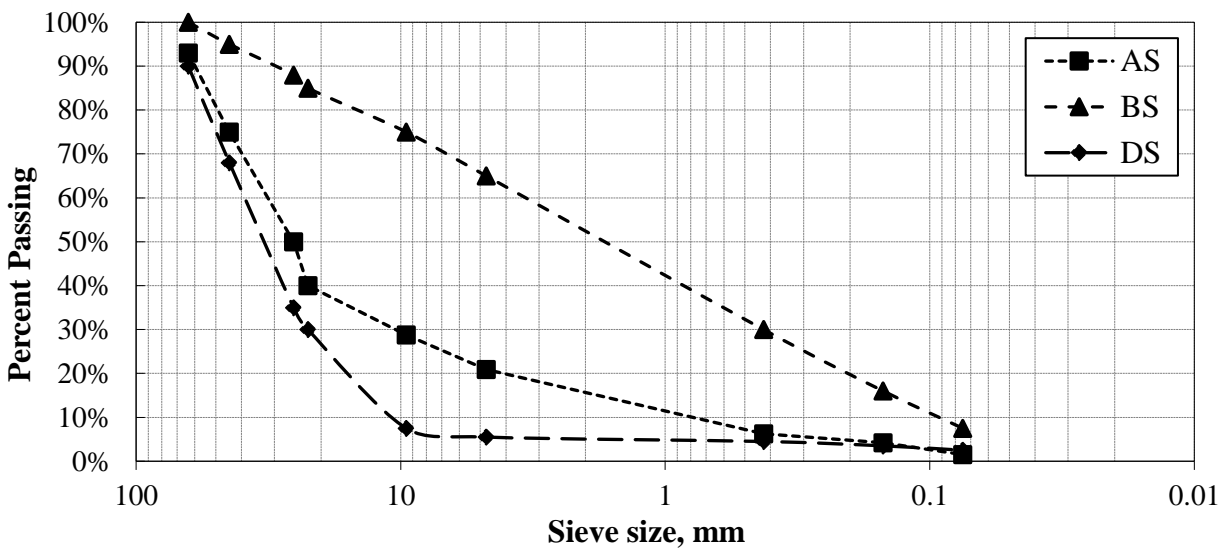


Figure 2.1 TxDOT AS, BS, DS gradation size distributions

Table 2.1 Source location and principal geologic type of soil materials

<b>Location</b>	<b>Geologic Type</b>
Amarillo, TX	Limestone
Apple, OK	Sandstone
Ashdown, AR	Sandstone/Quartz
Austin, TX	Limestone
Bridgeport, TX	Limestone
Brownwood, TX	Limestone
Corpus Christi, TX	Limestone
Dallas, TX	Shale
El Paso, TX	Limestone
FL	
Garden City, TX	Limestone
Graham, TX	Limestone
Jacksboro, TX	Limestone
Lawton, OK	Limestone
Malvern, AR	Shale
Pharr, TX	Sandstone/Quartz
, SC	
San Antonio, TX	Limestone
Spencerville, OK	Sandstone
Waco, TX	Limestone

## 2.2 COMPARISON OF SOIL LEACH TEST METHODS

A list of soil leach test procedures is provided in Table 2.2, which compares sieve gradation, the water/soil ratio, temperature, mixing method, and duration. All test procedures listed in Table 2.2 were performed in the manner specified by the respective agency. The results from different test procedures were compared based on the analyte for which each test procedure was intended.

Table 2.2 Soil leach tests organized by agency

Method	Analyte	Gradation	Water/Soil Ratio (mL/g)	Temp. (°C)	Mixing Method	Duration (hr.)
<b>AASHTO T-289</b> [3]	pH	Passing No. 10	1	Amb.	Shake by hand	1
<b>AASHTO T-290</b> [4]	Sulfate	Passing No. 10	3	Amb.	Shake by hand	~5 min
<b>AASHTO T-291</b> [5]	Chloride	Passing No. 10	3	Amb.	Shake by hand	1
<b>ASTM D-18</b> [6]	pH	Retained on No. 4	2	Amb.	Shake by hand	1
<b>ASTM D-4972</b> [7]	pH	Passing No. 10	1	Amb.	Shake by hand	1
<b>NCHRP 21-06</b> [8]	pH	Passing No. 10	1	Amb.	Shake by hand	0.5
<b>SCDOT T-143</b> [9]	pH, Cond.	Retained on No. 4	1	Amb.	Shake by hand	24
<b>Tex-128-E</b> [10]	pH	Passing No. 4	5	50±5	Stirring plate	1
<b>Tex-145-E</b> [11]	Sulfate	Passing No. 40	20	Amb.	Shake by hand	12
<b>Tex-146-E</b> [12]	Conductivity	Passing No. 40	20	Amb.	Shake by hand	12
<b>Tex-620-J</b> [13]	Chloride/Sulfate	Passing No. 40	10	66±11	Stirring plate	12
<b>Tex-620-M2</b>	pH, SO <sub>4</sub> <sup>2-</sup> , Cl <sup>-</sup> , Cond.	Passing 1 ¾"	10	Amb.	Roller	1

## 2.3 AASHTO TEST PROCEDURES

### 2.3.1 AASHTO T-289 Determine pH of soil for use in corrosion testing

A 125-mL high-density polyethylene (HDPE) bottle was filled with 30±0.1 mL of DI water (< 1 µS/cm) and 30±0.1 g of soil passing sieve No. 10 (water/soil ratio of 1 mL/g). A magnetic stirring bar was added to the slurry, and the bottle was capped. The bottle was placed on a magnetic stirring hot plate (VWR model 97024-634). The slurry was mixed for approximately 20 seconds every 15 minutes for a total of one hour. After the one hour, a calibrated symphony H30PCO probe was used to measure the pH of the slurry immediately after the final mixing. The specimen passes the T-289 test if the pH is within the range of 5 to 10.

### 2.3.2 AASHTO T-290 Determine sulfate ion content in soil (Method B)

A 500-mL HDPE bottle was filled with 300±0.1 mL of DI water (< 1 µS/cm) and 100±0.1 g of soil passing sieve No. 10 (water/soil ratio of 3 mL/g). The bottle was shaken by hand for one minute (approximately 120 shakes) and was left to stand/settle for 5.0±0.1 minutes. After settling, the sample was filtered through a 0.45-µm membrane. Ten milliliters of the filtered sample were

dispensed into a 15-mL plastic vial, along with 2 mL of a NaCl solution and 1 mL of a glycerin solution (1:1 ratio of glycerin and DI water). The vial was then capped and mixed by hand for one minute (approximately 120 shakes). After mixing, 3 mL was dispensed into a quartz cuvette to analyze as a blank on the UV-VIS spectrophotometer (Agilent model 8453). The 3 mL was poured back into the original vial and 60 mg of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  was mixed by hand (approximately 120 shakes) in with the solution. After mixing, 3 mL was dispensed into the quartz cuvette and measured on the UV-VIS to measure the amount of light absorbed. Prior to the blanks being analyzed, eight different known sulfate concentrations were measured on UV-VIS to make a calibration curve at a wavelength of 400 nm (Figure 2.2). The concentrations of 60 mg of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  to precipitate sulfate were measured in mg/L based on the absorbance of the standard curve. The evaluation limit for sulfate content is 200 ppm.

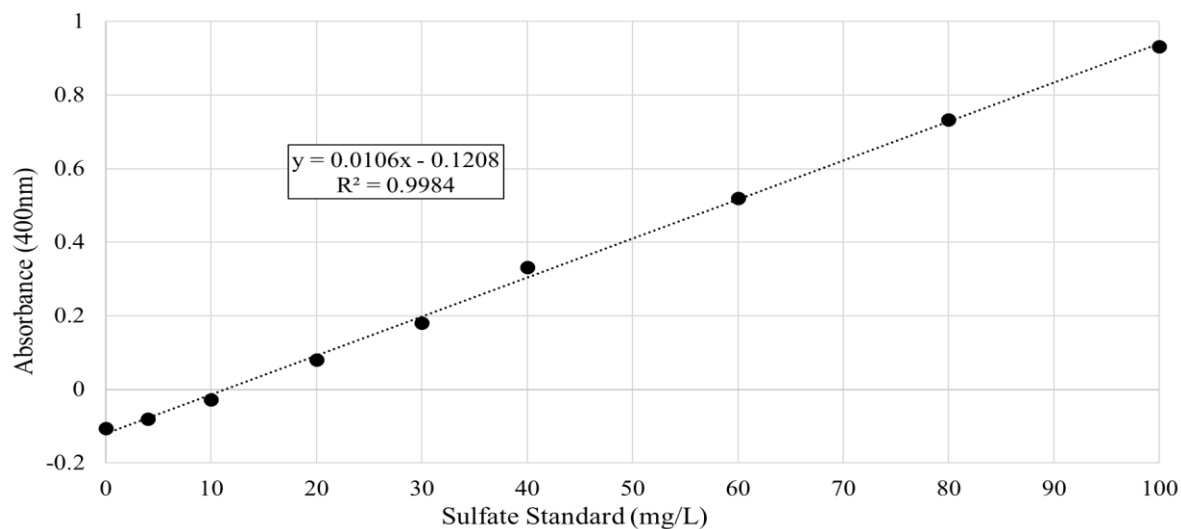


Figure 2.2 AASHTO T-290 sulfate titration calibration curve at 400nm

### 2.3.3 AASHTO T-291 Determine chloride ion content in soil (Method A)

A 500-mL HDPE bottle was filled with  $300 \pm 0.1$  mL of DI water ( $< 1 \mu\text{S}/\text{cm}$ ) and  $100 \pm 0.1$  g of soil passing sieve No. 10 (water/soil ratio of 3 mL/g). The sample was shaken by hand for

20 seconds (approximately 40 shakes) and left to settle for one hour. After one hour, the sample was shaken again for 20 seconds (approximately 40 shakes) and filtered through a 0.45- $\mu$ m membrane. The pH of the filtered water was checked. If the pH was out of the range of pH 6 through pH 8, the pH was adjusted using sodium bicarbonate or nitric acid. A 50-mL burette was filled with a silver nitrate solution (1 mg of Cl/mL equivalent). Using Micro-Lab FS-522, the number of drops from the counter were counted when 5 mL was dispensed to calculate the volume per drop used in a titration. In a 100-mL glass beaker, 30 mL of the filtered water and two drops of a 5% potassium dichromate solution was dispensed into the beaker. The beaker was placed onto a magnetic stirring plate with a magnetic stir bar placed inside. The mixture was thoroughly mixed for approximately two minutes, and the titration was started. Once the solution turned from yellow to red, the volume of silver nitrate was recorded. The volume from the titration was used to calculate the concentration in milligrams of chloride per kilogram of soil (*i.e.*, parts per million, ppm). The limit for the chloride content is 100 ppm.

## **2.4 ASTM TEST PROCEDURES**

### **2.4.1 ASTM D-4972 Standard test method for pH of soil (Method A)**

A 125-mL HDPE bottle was filled with  $10 \pm 0.1$  mL of DI water ( $< 1$   $\mu$ S/cm) and  $10 \pm 0.1$  g of soil passing sieve No. 10 (water/soil ratio of 1 mL/g). The sample was shaken by hand for approximately 15 seconds and left to settle for one hour. The bottle was tilted to allow most of the soil to settle out. A calibrated sympHony H30PCO was used to measure the pH of the slurry. (This test method is similar to the AASHTO T-289, but this method does not involve periodic mixing.)



## **2.4.2 ASTM D-18 Determine pH of lightweight aggregate**

A 1-L HDPE bottle was filled with  $400 \pm 0.1$  mL of DI water ( $< 1 \mu\text{S/cm}$ ) and  $200 \pm 0.1$  g of soil (water/soil ratio of 2 mL/g) retained on a No. 4 sieve. The bottle was capped and shaken by hand for approximately one minute. The sample was shaken by hand for one minute every 15 minutes for a total of one hour. After the one hour, a calibrated symphony H30PCO was used to measure the pH of the slurry.

## **2.5 NCHRP TEST PROCEDURES**

### **2.5.1 NCHRP-21-06 Measuring pH of soil samples**

Approximately 25 g of soil passing sieve No. 10 was weighed and oven dried at  $110^\circ\text{C}$  for 6-12 hours until a consistent weight was obtained. After drying the material, the soil was weighed again to calculate the percent of moisture in the soil. With the moisture content measured,  $10 \pm 0.1$  g was weighed out plus 10 g times the moisture content. To bring the total amount of the soil and water mixture to 20 g, the mass of the soil was subtracted from 20 g, giving the mass of the amount of DI water ( $< 1 \mu\text{S/cm}$ ) needed (water/soil ratio of  $\sim 1$  mL/g). Once the soil and DI water were mixed in a 125-ml plastic bottle and capped, the soil was shaken by hand for approximately 20 seconds and left to settle for 30 minutes. The bottle was tilted to allow most of the soil to settle out. A calibrated symphony H30PCO was used to measure the pH of the slurry. The pH was continuously measured until the pH did not change more than 0.05 pH units.

## **2.6 SCDOT TEST PROCEDURES**

### **2.6.1 SC T-143 Coarse aggregate sample for pH and resistivity**

A 5-gallon food grade bucket was filled with  $2000 \pm 1$  mL of DI water ( $< 1 \mu\text{S/cm}$ ) and  $2000 \pm 0.1$  g of soil retained on sieve No. 4 (water/soil ratio of 1 mL/g). After the soil and DI water

are mixed, the sample is set aside to settle for 30 minutes. After 30 minutes, the soil was mixed with a glass stirring rod for three minutes. The stirring process was repeated at two hours and then four hours. The sample was set aside to settle for 20 hours to allow the soil to settle. The slurry was then filtered through a coarse filter membrane and a calibrated sympHony H30PCO was used to measure the pH and conductivity of the filtered water.

## **2.7 TxDOT TEST PROCEDURES**

### **2.7.1 Tex-128-E Determining soil pH**

A 300-mL glass beaker was filled with  $150 \pm 1$  mL of DI water ( $< 1 \mu\text{S}/\text{cm}$ ) and was heated to  $45\text{-}60^\circ\text{C}$  ( $50 \pm 3^\circ\text{C}$  for consistency in this research). A beaker containing 150 mL of DI water was mixed with  $30 \pm 0.1$  g of air dried soil, passing sieve No. 4 (water/soil ratio of 5 mL/g). After the soil was mixed with the DI water, the heat was turned off and a stirring bar was added. The beaker was placed on a mixing plate and was stirred vigorously for 20 seconds. The slurry was mixed to completely disperse the soil. The slurry was stirred for approximately 20 seconds every 15 minutes for one hour. After one hour, a calibrated sympHony H30PCO probe was used to record the pH of the slurry by placing the probe approximately  $\frac{3}{4}$  inches into the aqueous slurry and avoiding the soil in the bottle. The probe remained in the slurry at  $30 \pm 3^\circ\text{C}$  until the pH was stable. The acceptable range for the pH is between 5.5 to 10.

### **2.7.2 Tex-145-E Determine sulfate content in soils**

A 500-mL (HDPE) bottle was filled with  $400 \pm 1$  mL of DI water ( $< 1 \mu\text{S}/\text{cm}$ ) and  $20 \pm 0.1$  g of soil passing sieve No. 40 (water/soil ratio of 20 mL/g). The cap was placed on the bottle and was shaken vigorously by hand for one minute (approximately 120 shakes). After shaking, the bottle was set aside to settle for 12 hours. After the 12 hours, the bottle was then shaken again for

one minute and filtered through a 0.45- $\mu$ m membrane. A 5-mL IC vial was filled using the filtered water from the slurry. Sulfate ion concentration was measured on a DIONEX ICS-2100 IC instrument using an IonPac AS16 4x250 mm analytical column with 18 mM KOH eluent at 1 mL/min. This test method is used for soils that have high concentrations of sulfate. The limit for the sulfate content is 200 ppm.

### **2.7.3 Tex-146-E Conductivity test for field detection of sulfate in soil**

A 250-mL HDPE bottle was filled with  $100 \pm 1$  mL of DI water ( $< 1$   $\mu$ S/cm) and  $5 \pm 0.1$  g of soil passing sieve No. 40. (water/soil ratio of 20 mL/g). After mixing the DI water and soil, the bottle was capped and shaken vigorously by hand for one minute (approximately 120 shakes). The bottle was set aside for 12 hours to allow the particles to settle. After 12 hours, the sample was shaken vigorously by hand for one minute (approximately 120 shakes). A calibrated symphony H30PCO probe was used to measure the conductivity immediately after shaking.

### **2.7.4 Tex-620-J Determine chloride and sulfate contents in soil**

A 600-mL glass beaker was filled with  $350 \pm 1$  mL of DI water ( $< 1$   $\mu$ S/cm) and heated to  $60 \pm 3^\circ\text{C}$  for consistency in this research. Soil passing sieve No. 40 was dried in an oven at  $60 \pm 2^\circ\text{C}$ . After both the soil and DI water were heated,  $30 \pm 0.1$  g of the sieved soil was added to the glass beaker (water/soil ratio of 10 mL/g). A stir bar was added to the glass beaker and was stirred for approximately 20 seconds. The temperature was held constant for the full 12 hours of the test. The slurry was mixed every hour for approximately 20 seconds. After the 12 hours, the slurry was filtered then set aside to cool to room temperature. Once the sample was at room temperature, it was filtered through a 0.45- $\mu$ m membrane and prepared to be analyzed by IC. A 5-mL vial was filled using the filtered water. Chloride and sulfate ion concentrations were measured on a

DIONEX ICS-2100 IC instrument using an IonPac AS16 4x250 mm analytical column with 18 mM KOH eluent at 1 mL/min. The limits of chloride and sulfate are 100 and 200 ppm, respectively.

## **2.8 DEVELOPMENT OF TEX-620-M2, A MODIFIED VERSION OF TEX 620-J**

The development of the Tex-620-M2 was an iterative process. The first iteration was an interim version of the current TxDOT test procedure Tex-620-J, which was called the Tex-620-M which was a simplified version of the current Tex-620-J. After the development of the Tex-620-M, the Tex-620-M1 was developed, which used a Gilson Rice shaker for maximum specific gravity (Gmm) filled with 1 liter of DI water and  $100 \pm 0.1$  g of soil. (A brief comparison of the interim Tex-620-M1 test procedure results for pH, conductivity, chloride, and sulfate to the current Texas test procedures is provided in Appendix A.) Then, the Tex-620-M2 was developed to standardize the test method even further. Ultimately, the Tex-620-M2 test procedure was developed using a bottle roller for mixing, which provided a very consistent mixing intensity.

### **2.8.1 Tex-620-M2 Determine chloride, sulfate, conductivity, and pH in a soil**

A 2-liter HDPE bottle (24.4 cm tall, 12.0 cm diameter) was filled with  $2 \text{ L} \pm 1 \text{ mL}$  of DI water ( $< 1 \text{ } \mu\text{S/cm}$ ) and  $200 \pm 0.1$  g of soil passing a  $1 \frac{3}{4}$ -inch sieve (water/soil ratio of 10 mL/g). An example photo of a 200-g soil sample is shown in Figure 2.3. The cap was screwed on, and the bottle was placed on a bottle/tube roller (Thermo model 88881003) shown in Figure 2.4. The bottle was set to roll for one hour at a roller speed of 80 rpm, which causes a bottle rotation speed of approximately  $18 \pm 1$  revolutions per minute (rpm). After one hour, the slurry was filtered through a 0.45- $\mu\text{m}$  membrane. A calibrated sympHony H30PCO was used to measure the pH and the conductivity of the slurry. A 5-mL vial was filled using the filtered slurry to be measured for

chloride and sulfate ion content. Chloride and sulfate ion concentrations were measured on a DIONEX ICS-2100 IC instrument using an IonPac AS16 4x250 mm analytical column with 18 mM KOH eluent at 1 mL/min.



Figure 2.3 Sample of 200 grams passing sieve 1  $\frac{3}{4}$ "



Figure 2.4 Bottle Roller (Thermo model 88881003)

### **2.8.2 Analysis of dissolution kinetics**

A 24-hour dissolution kinetics test was performed on the Tex-620-M2 test procedure before selecting a duration for the test. Three materials were tested (El Paso, Brownwood, and Ashdown), and two gradations were tested (gravel and fines). Samples were collected at five different times of 0.1, 1, 2, 8, and 24 hours, and normalized results are shown in Figure 2.5. Analysis of the 24-hour kinetics testing of the fines (samples labeled “F”) revealed that the conductivity of the sample at 1 hour was over 90% of the conductivity of the sample at 24 hours (Figure 2.5a). Analysis of the 24-hour kinetics testing of the gravel (samples labeled “GR”) revealed that the conductivity of the Ashdown sample at 1 hour was almost 70% of the conductivity of the Ashdown sample at 24 hours (Figure 2.5a). While the fraction of chloride and sulfate at one hour were not as high as the fraction of conductivity, the final concentrations of chloride and sulfate were very low ( $< 6$  mg/L) for gravel, and thus, not significantly impacting corrosion rates. Thus, one hour was selected for the duration of the Tex-620-M2 test procedure because most of the conductivity can be detected within one hour, even for gravel gradations of a sorted river-stone material.

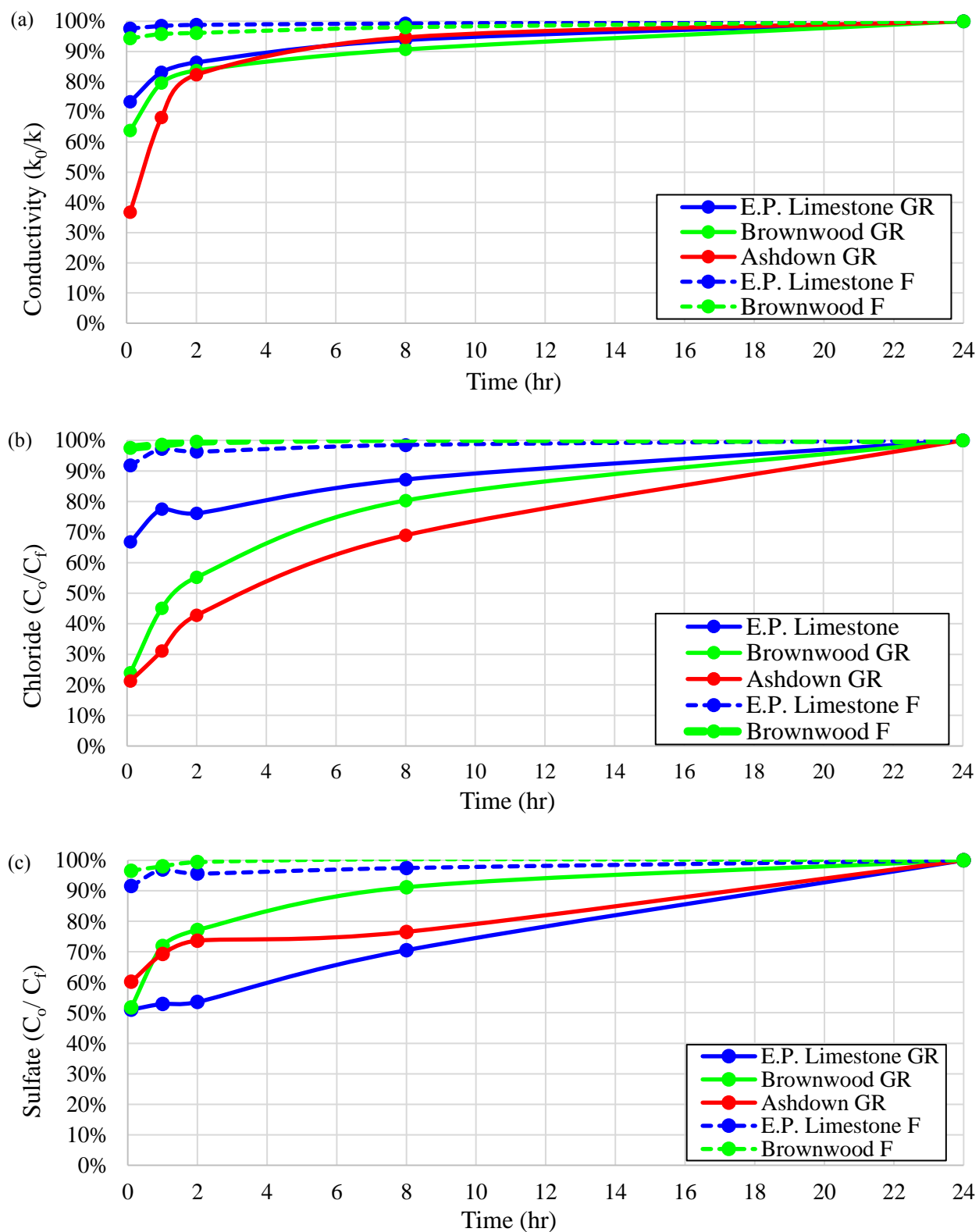


Figure 2.5 Normalized dissolution kinetics: (a) conductivity, (b) chloride, and (c) sulfate

### 2.8.3 Reproducibility of Tex-620-M2

The reproducibility of the Tex-620-M2 test was analyzed by performing five replicates on three different materials. The mean, standard deviation (SD), and coefficient of variation (CV) of the pH of the five replicates of each test are provided in Table 2.3. The maximum CV observed for five replicates of one material was for the SCDOT T-143 test on the El Paso material with a CV of 3.5%. The average of the CV values for the Tex-620-M2 analysis of the three materials was approximately 0.5%, which shows that the pH of the Tex-620-M2 was very reproducible.

Table 2.3 Reproducibility Analysis for pH

	Replicate/Stat.	AASHTO T-289	ASTM D-18	ASTM D- 4972 (A)	NCHRP 21-06	SCDOT T-143	TX 128-E	TX 620-M2
EPLS	1	7.69	9.55	8.18	8.29	8.07	8.95	9.20
	2	8.23	9.61	8.28	8.35	8.26	8.84	9.26
	3	8.29	9.60	8.28	8.35	8.73	8.78	9.31
	4	8.26	9.49	8.32	8.36	8.76	8.92	9.39
	5	8.31	9.60	8.26	8.30	8.54	8.90	9.33
	Mean	8.16	9.57	8.26	8.33	8.47	8.88	9.30
	S.D.	0.26	0.05	0.05	0.03	0.30	0.07	0.07
	CV	<b>3.22%</b>	<b>0.53%</b>	<b>0.63%</b>	<b>0.39%</b>	<b>3.54%</b>	<b>0.77%</b>	<b>0.77%</b>
SC	1	8.04	8.09	8.35	8.08	7.33	8.74	9.15
	2	8.09	8.16	8.09	8.09	7.14	8.92	9.15
	3	8.09	8.25	8.06	8.09	7.33	8.87	9.19
	4	8.11	8.22	8.23	8.06	7.24	8.81	9.16
	5	8.12	8.25	8.19	8.17	7.44	8.59	9.16
	Mean	8.09	8.19	8.18	8.10	7.30	8.79	9.16
	S.D.	0.03	0.07	0.12	0.04	0.11	0.13	0.02
	CV	<b>0.4%</b>	<b>0.8%</b>	<b>1.4%</b>	<b>0.5%</b>	<b>1.5%</b>	<b>1.5%</b>	<b>0.2%</b>
FL	1	8.03		8.07	7.95		7.99	8.53
	2	7.94		8.12	8.08		8.07	8.62
	3	8.02		8.14	8.12		8.15	8.62
	4	8.08		8.18	8.1		8.25	8.58
	5	8.08		8.19	8.16		8.25	8.67
	Mean	8.03		8.14	8.082		8.142	8.604
	S.D.	0.06		0.05	0.08		0.11	0.05
	CV	<b>0.72%</b>		<b>0.60%</b>	<b>0.98%</b>		<b>1.40%</b>	<b>0.61%</b>



The conductivity results (Table 2.4) showed more variation than the pH results. The range of the CV was from 2% to 26%. The average of the three CV values for the Tex-620-M2 was approximately 4%.

Table 2.4 Reproducibility Analysis for Normalized Conductivity ( $\mu\text{S}/\text{cm}\cdot\text{mL}/\text{g}$ )

Matl.	Replicate/ Stat.	AASHTO T-289	AASHTO T-290 (B)	AASHTO T-291 (A)	ASTM D-18	ASTM D- 4972 (A)	NCHRP 21-06	SCDOT T-143	Tex 128-E	Tex 620-J	Tex 620-M2
EP	1	1763	1668	1890	338	1253	1669	216	1625	798	1426
	2	1563	1752	1905	383.2	1438	1702	198.5	1620	729	1546
	3	1749	1320	2034	314.8	1518	1662	171.4	1765	720	1648
	4	1616	1746	1950	493.2	1508	1614	183.2	1600	789	1468
	5	1649	1761	1827	357.6	1590	1790	187.4	1725	849	1755
	Mean	1668	1649.4	1921.2	377.36	1461.4	1687.4	191.3	1667	777	1568.6
	SD	86.13	187.86	76.91	69.47	128.35	65.40	16.87	73.19	53.20	134.18
	CV	<b>5.2%</b>	<b>11.4%</b>	<b>4.0%</b>	<b>18.4%</b>	<b>8.8%</b>	<b>3.9%</b>	<b>8.8%</b>	<b>4.4%</b>	<b>6.8%</b>	<b>8.6%</b>
SC	1	864	813	930	2600	414	718	1113	478.5	439.2	641
	2	800	876	816	2664	734	712	1085	496.5	476.4	631
	3	823	858	996	2516	788	691	1080	442	472.8	638
	4	799	912	834	2428	511	785	1085	397.5	394.8	606
	5	847	882	1080	2336	737	671	1070	479	472.5	629
	Mean	826.60	868.20	931.20	2508.80	636.80	715.40	1086.60	458.70	451.14	629.00
	SD	28.71	36.47	110.75	131.19	164.18	43.10	15.98	39.55	34.93	13.77
	CV	<b>3.5%</b>	<b>4.2%</b>	<b>11.9%</b>	<b>5.2%</b>	<b>25.8%</b>	<b>6.0%</b>	<b>1.5%</b>	<b>8.6%</b>	<b>7.7%</b>	<b>2.2%</b>
FL	1	115.2	204.3	218.7		144.8	101.8		361	261.6	537
	2	104.3	219.0	195.9		115.9	99.5		353.0	283.5	530
	3	114.5	208.5	213.6		106.3	100.1		349.5	294.3	523
	4	117.6	206.7	211.2		106.3	97.4		343.5	274.2	515
	5	113	203.4	209.7		113.0	97.1		345.5	292.5	520
	Mean	112.92	208.38	209.82		117.26	99.18		350.50	281.22	525.00
	SD	5.10	6.27	8.50		15.96	1.96		6.92	13.58	8.63
	CV	<b>4.5%</b>	<b>3.0%</b>	<b>4.1%</b>		<b>13.6%</b>	<b>2.0%</b>		<b>2.0%</b>	<b>4.8%</b>	<b>1.6%</b>

Results for chloride concentrations (Table 2.5) and sulfate concentrations (Table 2.6) showed more variability than pH and conductivity. The CV values for chloride concentrations ranged from 3% to 49% with the greatest CV values associated with the lowest chloride

concentrations. Similarly, the CV values for sulfate concentrations ranged from 3% to 54%, with the greatest CV values associated with the lowest sulfate concentrations.

Table 2.5 Reproducibility Analysis for Chloride Concentration (mg of Cl<sup>-</sup>/kg of soil)

Matl.	Replicates/ Stat.	AASHTO T-291 (A)	Tex 620-J	Tex 620-M2
EP	1	247.71	115.40	100.75
	2	252.11	101.88	108.26
	3	243.35	101.63	149.03
	4	253.54	103.31	103.44
	5	236.08	113.19	144.56
	Mean	246.56	107.08	121.21
	SD	7.09	6.66	23.56
	<b>CV</b>	<b>3%</b>	<b>6%</b>	<b>19%</b>
SC	1	345.38	240.43	30.90
	2	389.06	242.24	29.49
	3	424.05	228.68	32.06
	4	375.97	234.14	29.05
	5	384.66	252.38	26.89
	Mean	383.82	239.57	29.68
	SD	28.22	8.94	1.96
	<b>CV</b>	<b>7.4%</b>	<b>3.7%</b>	<b>6.6%</b>
FL	1	-	6.82	3.19
	2	-	5.06	7.74
	3	-	6.97	2.43
	4	-	3.85	5.12
	5	-	5.67	3.28
	Mean	-	5.67	4.35
	SD	-	1.29	2.14
	<b>CV</b>	-	<b>23%</b>	<b>49%</b>

Table 2.6 Reproducibility Analysis for Sulfate Concentration (mg of SO<sub>4</sub><sup>2-</sup>/kg of soil)

Matl.	Replicate/ Stat.	AASHTO T-290 (B)	Tex 620-J	Tex 620-M2
EP	1	340.1	409.1	184.1
	2	424.2	328.2	211.2
	3	288.8	320.7	202.2
	4	346.8	326.9	201.9
	5	368.7	402.3	271.4
	Mean	353.7	357.5	214.2
	SD	49.1	44.2	33.5
	<b>CV</b>	<b>13.9%</b>	<b>12.4%</b>	<b>15.6%</b>
SC	1	230.7	112.3	24.1
	2	278.1	107.5	27.5
	3	232.1	133.8	22.2
	4	248.3	85.5	26.0
	5	285.6	101.6	20.9
	Mean	255.0	108.1	24.1
	SD	25.7	17.5	2.7
	<b>CV</b>	<b>10.1%</b>	<b>16.2%</b>	<b>11.1%</b>
FL	1	4.0	8.3	2.9
	2	4.2	7.1	7.4
	3	4.1	9.2	1.9
	4	4.3	6.4	4.9
	5	4.2	8.2	3.0
	Mean	4.2	7.9	4.0
	SD	0.1	1.1	2.2
	<b>CV</b>	<b>2.7%</b>	<b>14.2%</b>	<b>54.1%</b>

### 3. Results and Discussion

#### 3.1 COMPARISON OF TEX-620-M2 ON FINES: COMPARISON TO CURRENT TxDOT TEST

##### PROCEDURES

The Tex-620-M2 test procedure was compared to the current TxDOT test procedures (Table 2.1) for each of the key analytes (pH, conductivity, chloride, and sulfate) by testing nine geomaterials. While the Tex-620-M2 specifies testing material passing a 1 <sup>3</sup>/<sub>4</sub>-inch sieve, the nine materials were sieved to passing No. 40 for this comparison because three of the four current Texas test procedures specify passing No. 40 sieve. With respect to pH, the Tex-620-M2 test procedure yields a higher pH compared to the Tex-128-E test procedure for all nine materials tested, as shown in Figure 3.1. Both are one-hour tests, but Tex-128-E is heated to 50±5°C. When comparing a material passing No. 40 sieve compared to a soil passing No. 4 sieve for the pH comparison was would not vary much.

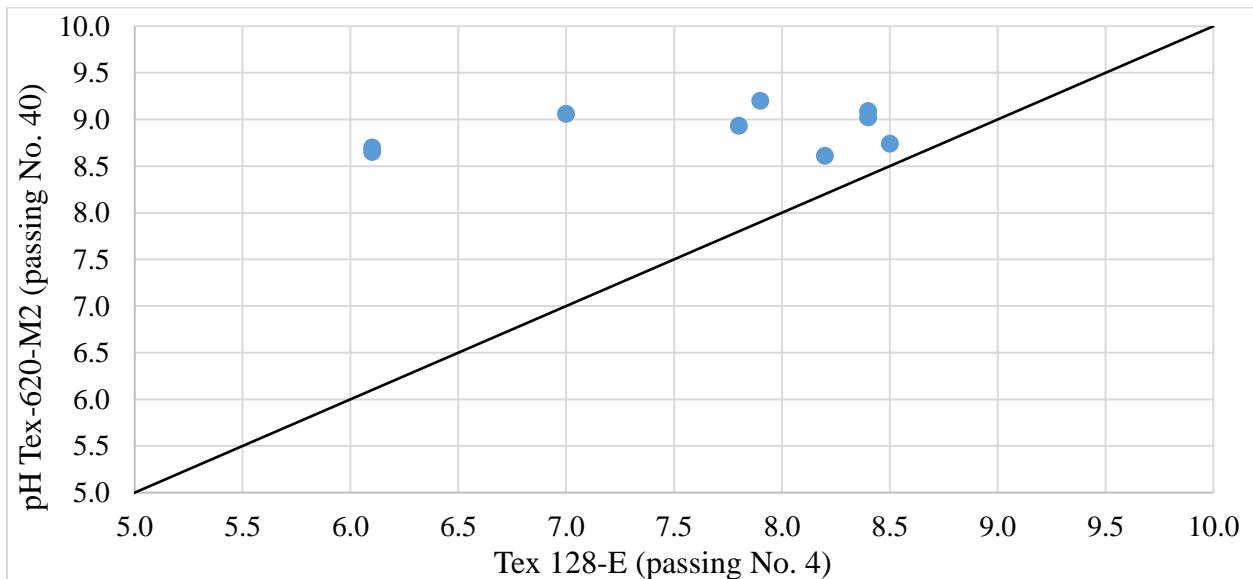


Figure 3.1 Comparison of Tex-620-M2: pH vs. Tex-128-E

With respect to conductivity, the Tex-620-M2 test procedure yielded greater normalized conductivities than the 12-hour Tex-146-E test procedure for six of the nine geomaterials, as shown in Figure 3.2. This indicates that the Tex-620-M2 test procedure is generally more effective at leaching the ionic content from the geomaterial than the Tex-146-E test procedure, most likely due to the continuous mixing. The conductivity was normalized by multiplying the measured conductivity by the water/soil ratio (mL/g).

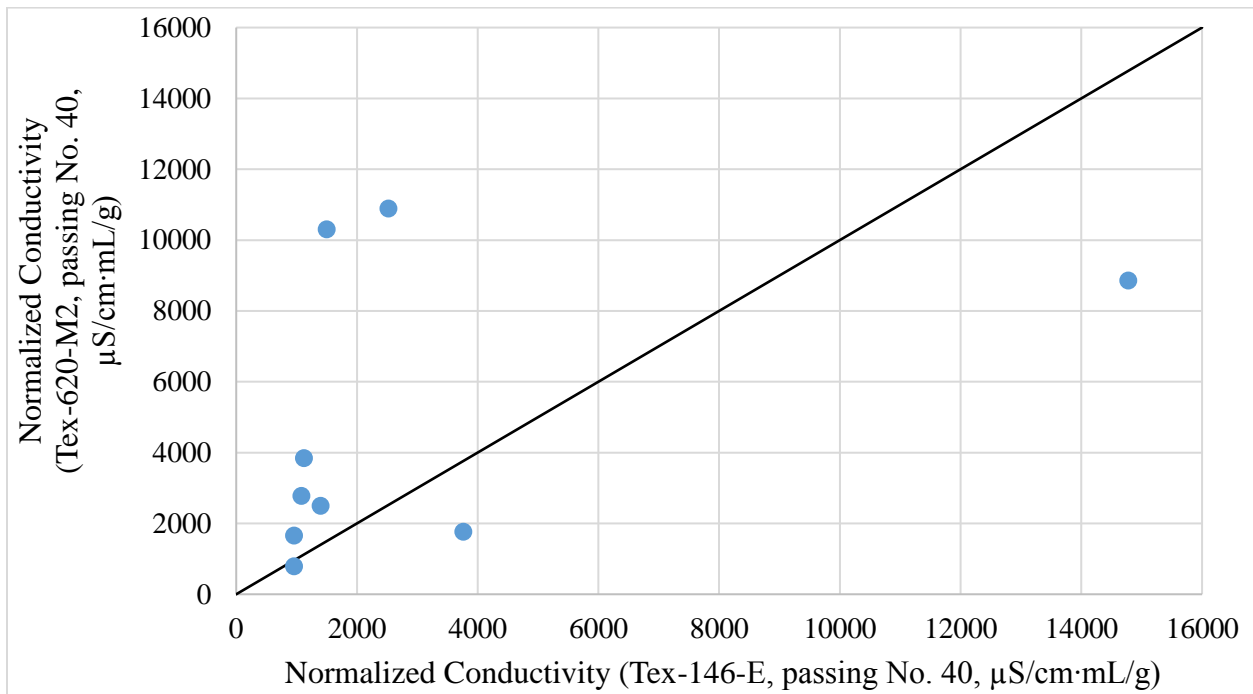


Figure 3.2 Comparison of Tex-620-M2: normalized conductivity vs. Tex-146-E

Similarly, as shown in Figure 3.3, the Tex-620-M2 test procedure yielded greater chloride concentrations for six of the seven materials tested with the 12-hour Tex-146-E test procedure (Figure 3.3a) and for seven of the eight materials tested with the heated 12-hour Tex-620-J test procedure (Figure 3.3b). This indicates that the Tex-620-M2 test procedure is generally more effective than the Tex-146-E and Tex-620-J test procedures at leaching the ionic content from the geomaterial, most likely due to the continuous mixing.

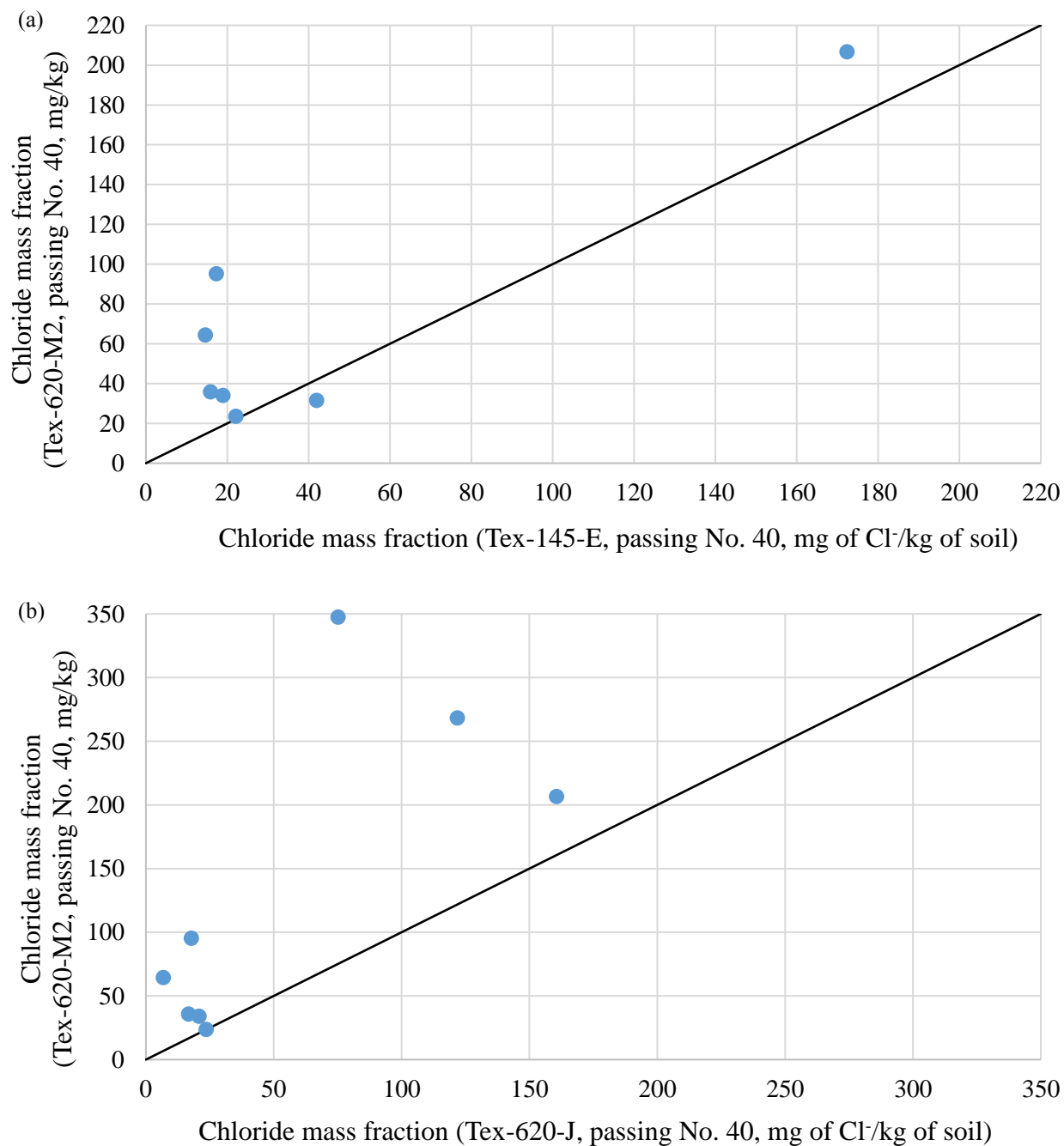


Figure 3.3 Comparison of Tex-620-M2: chloride mass fraction vs. (a) Tex-145-E and (b) Tex-620-J

Similarly, as shown in Figure 3.4, the Tex-620-M2 test procedure yielded greater sulfate concentrations for seven of the nine materials tested with the 12-hour Tex-145-E test procedure (Figure 3.4a) and for six of the eight materials tested with the heated 12-hour Tex-620-J test procedure (Figure 3.4b). This indicates that the Tex-620-M2 test procedure is generally more

effective than the Tex-145-E and Tex-620-J test procedures at leaching the ionic content from the geomaterial, most likely due to the continuous mixing.

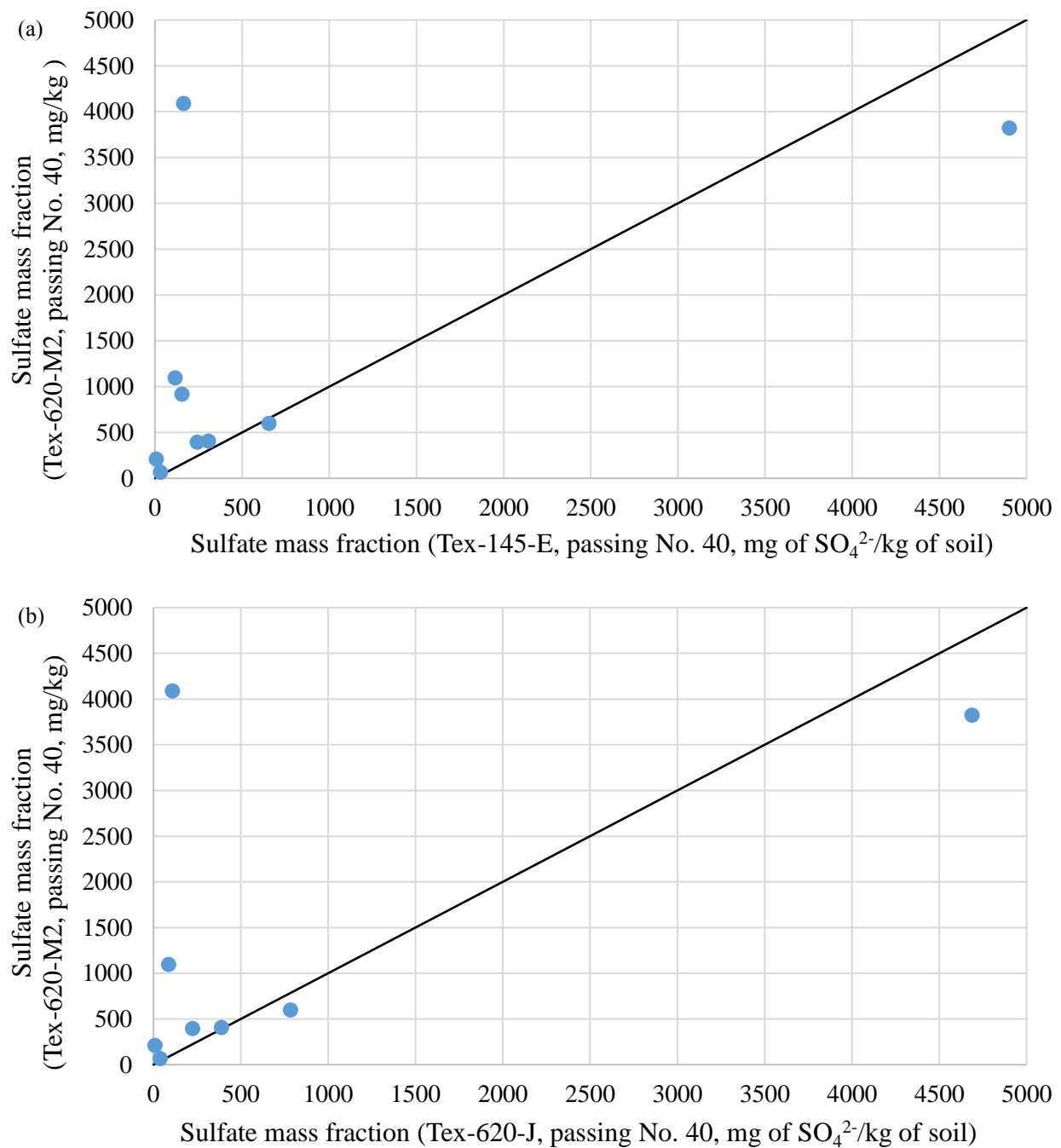


Figure 3.4 Comparison of Tex-620-M2: sulfate mass fraction vs. (a) Tex-145-E and  
(b) Tex-620-J

## 3.2 COMPARISON OF TEX-620-M2 TO CURRENT STANDARDS

When comparing the Tex-620-M2 test procedure to the current standards listed in Table 2.2, all the test procedures were performed as specified in each standard. It is important to note that the Tex-620-M2 test procedure was performed with coarse gradations (as intended) while the existing standards specify testing a relatively fine size (*e.g.*, passing a No. 40 sieve). As shown in Figure 3.5, in all but one of the tests, the pH of the Tex-620-M2 test procedure was greater than the pH of the current standard. (Standards are identified by the symbol color, while Tex-620-M2 gradation is identified by symbol shape.) Since many of the materials are limestone, the greater leaching effectiveness of the Tex-620-M2 test procedure would result in higher pH values.

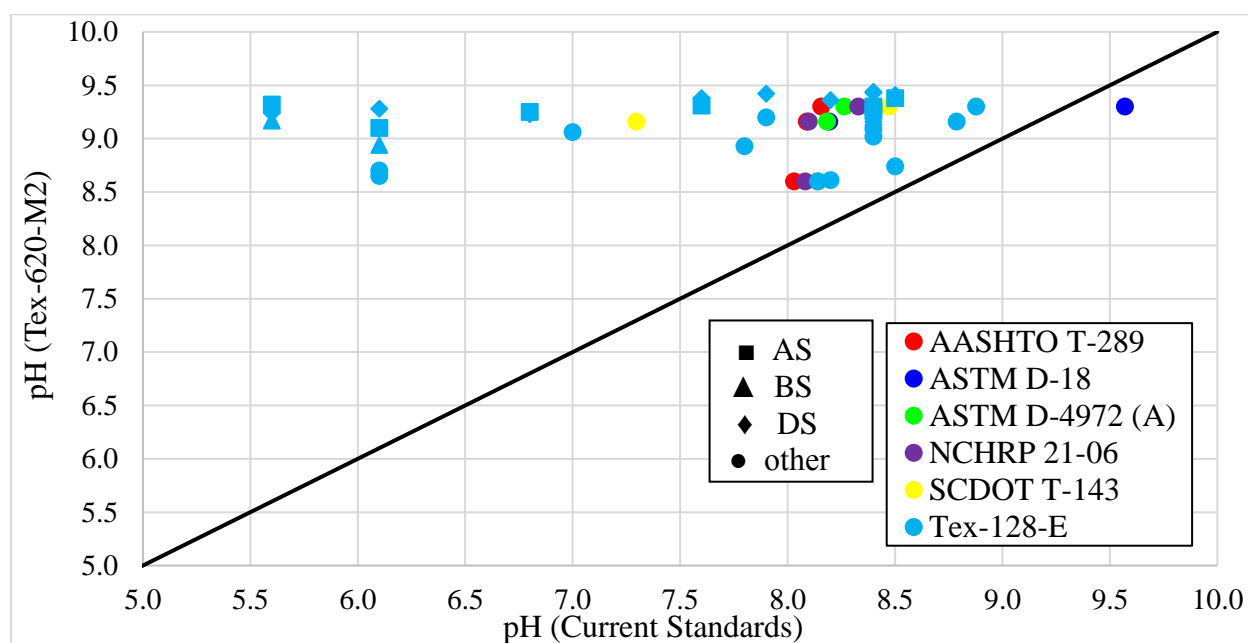


Figure 3.5 Comparison of Tex-620-M2: pH vs. current standards

To compare conductivity results from leach test procedures of differing water/soil dilution ratios, the measured conductivity measured for each test procedure was normalized by multiplying by the respective water/soil dilution ratio (mL/g). As shown in Figure 3.6, most of the Tex-620-M2 normalized conductivity results were less than the normalized conductivity of current standards.



This trend is likely due to the relatively fine size (*e.g.*, passing a No. 40 sieve) of the current standards because the finer size fractions (especially fine sand and fines) contain greater ionic content. Similarly, most of the chloride and sulfate contents measured by Tex-620-M2 were less than results from the current standards, as shown in Figure 3.7 and 3.8, respectively. Again, this is due to the current standards specifying a relatively fine size distribution (*e.g.*, passing a No. 40 sieve), which biases the current standards to show greater ionic content. Implementing Tex-620-M2 as a leach test procedure for aggregates passing a 1 3/4-inch would allow a more representative measurement of the actual gradation of backfill material. Unfortunately, the current test procedures may be unnecessarily restricting some materials from qualification and use as backfill material due to the implicit bias of testing finer material (*e.g.*, passing No. 40). Some of the circular data points in Figure 3.6 above the 1:1 line are the same data shown in Figure 3.2.

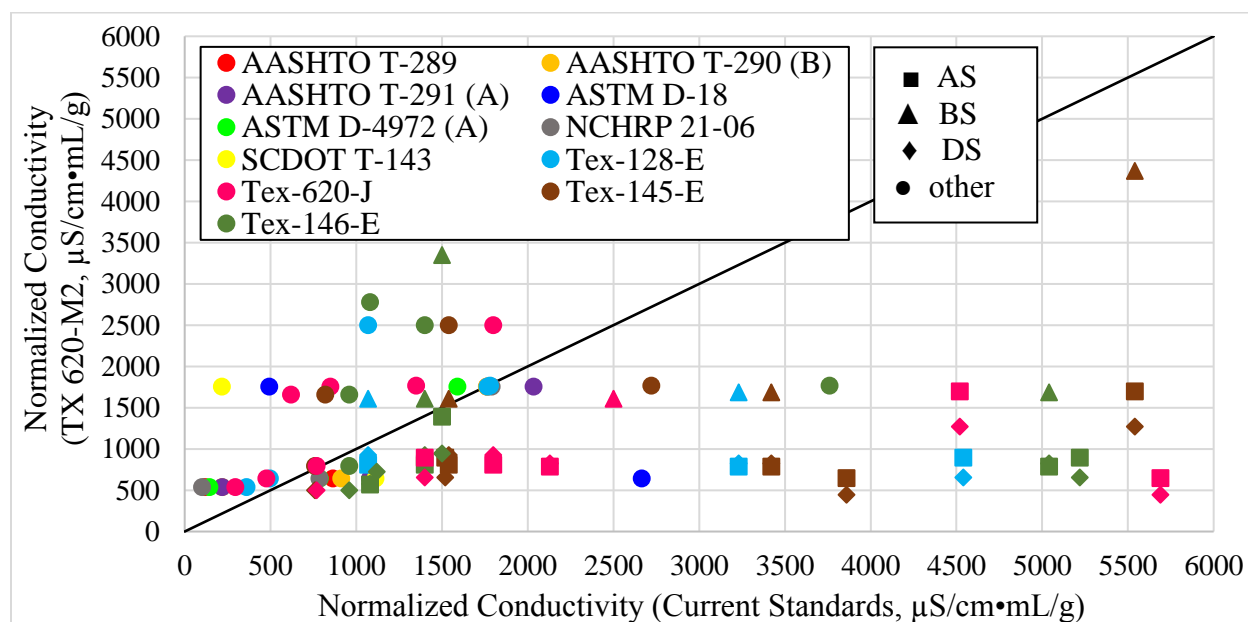


Figure 3.6 Comparison of Tex-620-M2: normalized conductivity vs. current standards

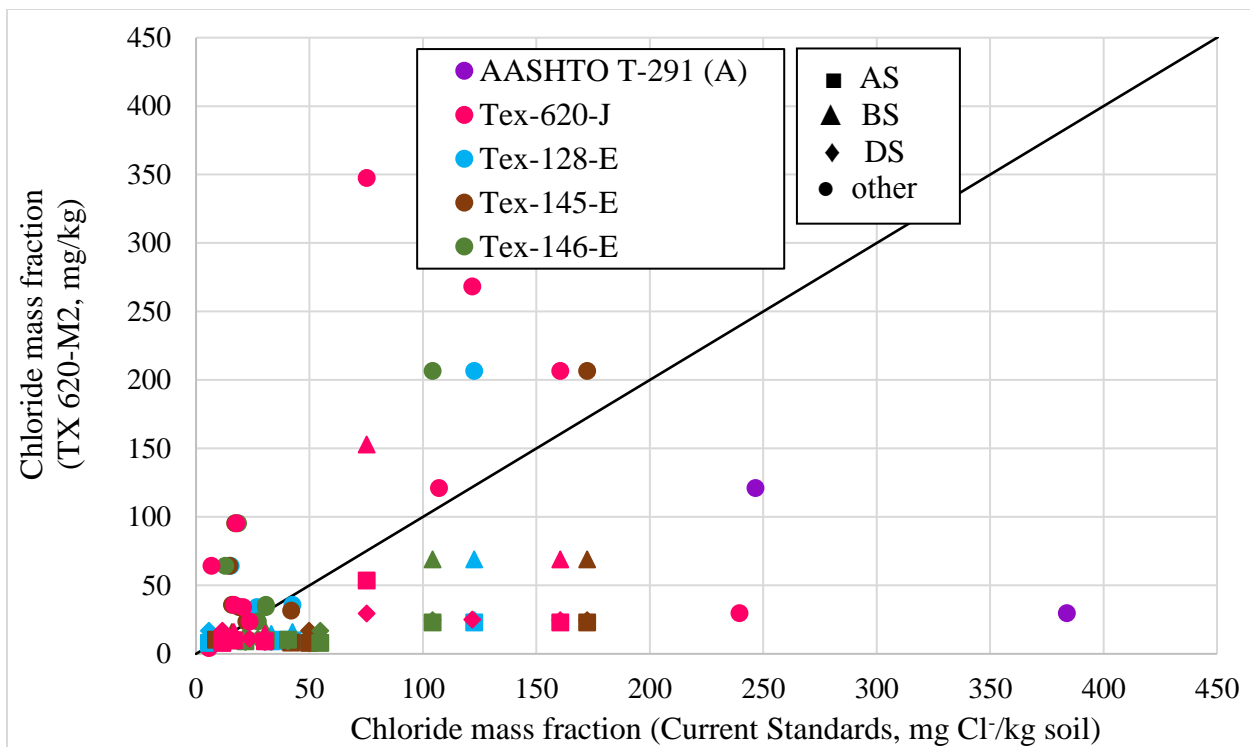


Figure 3.7 Comparison of Tex-620-M2: chloride mass fraction vs. current standards

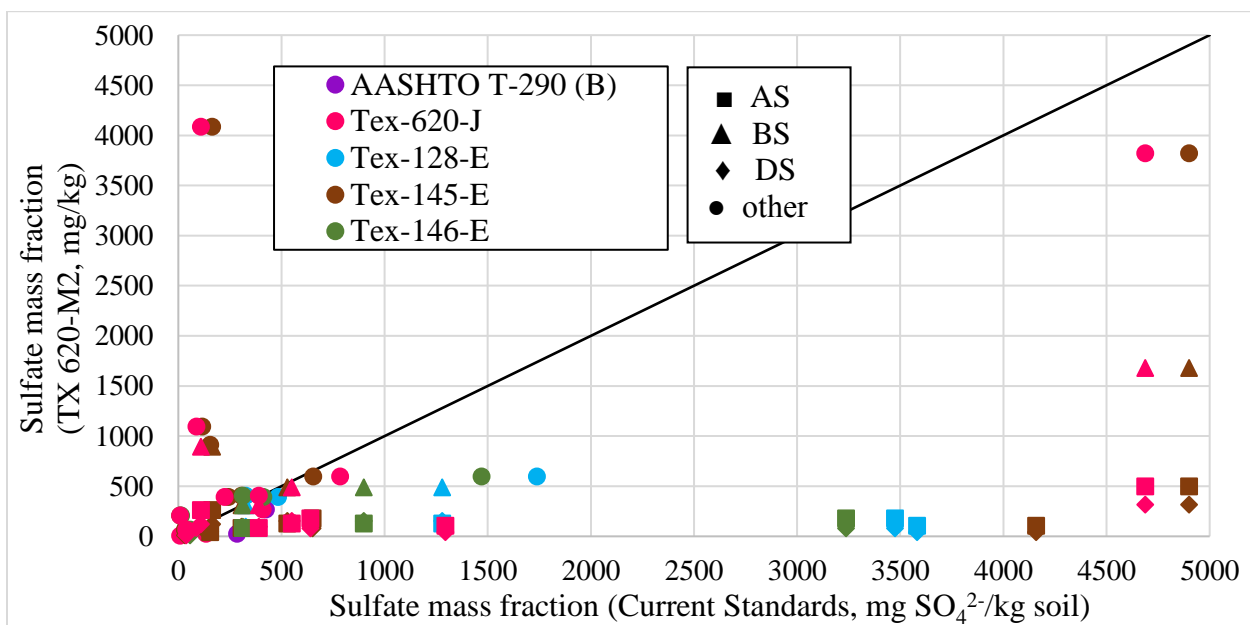


Figure 3.8 Comparison of Tex-620-M2: sulfate mass fraction vs. current standards

## 4. Conclusion

The development of the Tex-620-M2 test procedure based on Tex-620-M was successful with respect to accommodating the testing of material passing a 1 3/4-inch sieve and using a commercially available laboratory bottle roller with 2 L bottles for standardizing the test procedure. This test procedure is also shown to be sufficiently reproducible by running five replicates tests on three different materials. Using the Tex-620-M2 test procedure will reduce the amount of time required to obtain the current parameters used in TxDOT specifications. Conducting all currently specified TxDOT tests for measuring the pH, conductivity, chloride concentration, and sulfate concentration takes approximately two days. However, the Tex-620-M2 test procedure can be used to characterize all four parameters in approximately three hours. This would significantly expedite the evaluation of MSE backfill material.

The Tex-620-M2 test procedure was compared by testing material passing a No. 40 sieve against current TxDOT standards. Based on pH, conductivity, chloride, and sulfate analyses, the Tex-620-M2 test was shown to leach more ionic content than current TxDOT standards.

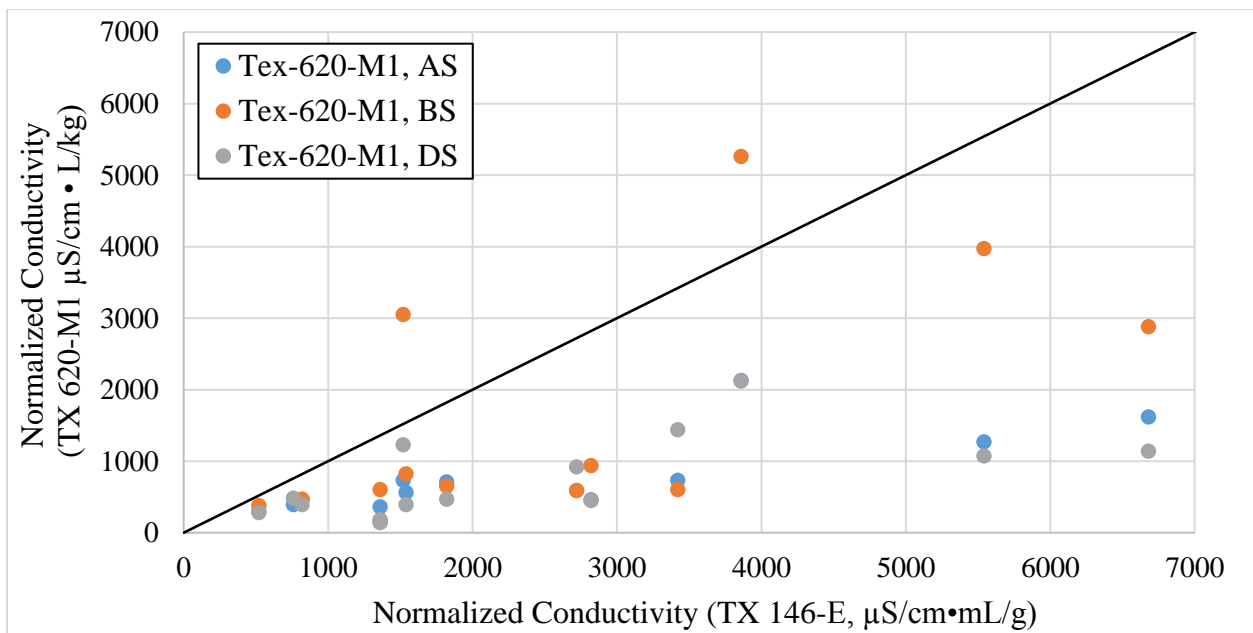
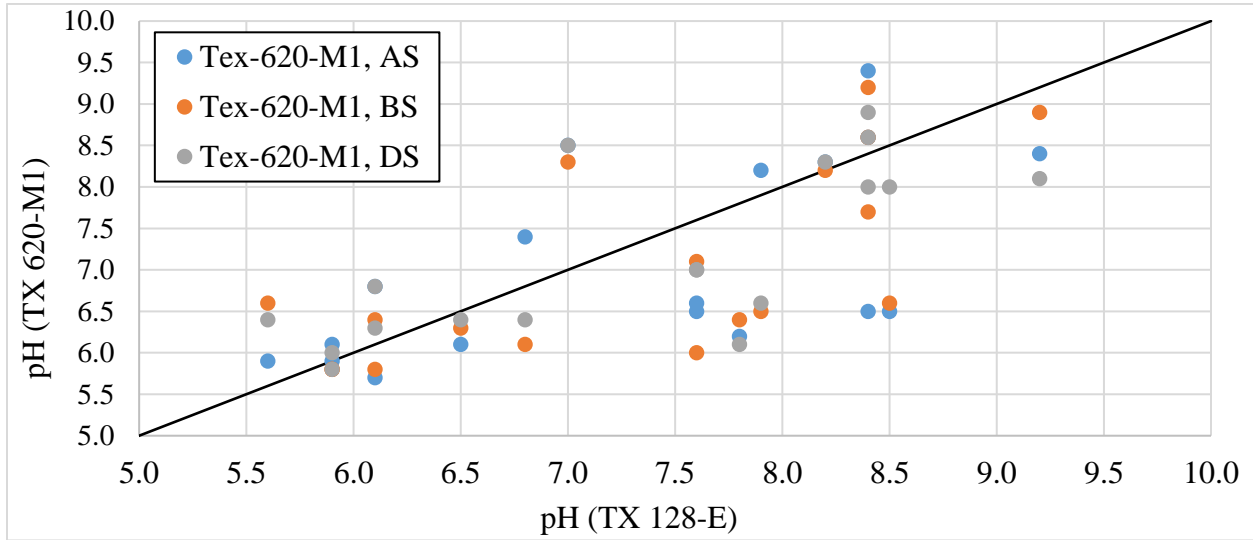
When the results from Tex-620-M2 test procedure were compared to the current standards, the current test procedures were performed with the specified size aggregate (typically a relatively fine size distribution such as passing a No. 40 sieve). The pH values measured with the Tex-620-M2 test procedure were greater than results from the current standards, whereas, results for conductivity, chloride, and sulfate by the Tex-620-M2 test procedure were lesser than the current standards. The finer size categories (*i.e.*, fine sand and fines) contain greater ionic content, and thus, the Tex-620-M2 test procedure provides a more representative analysis of coarse aggregate gradations. (Future work should validate the pH sensitivity of the Tex-620-M2 test.)

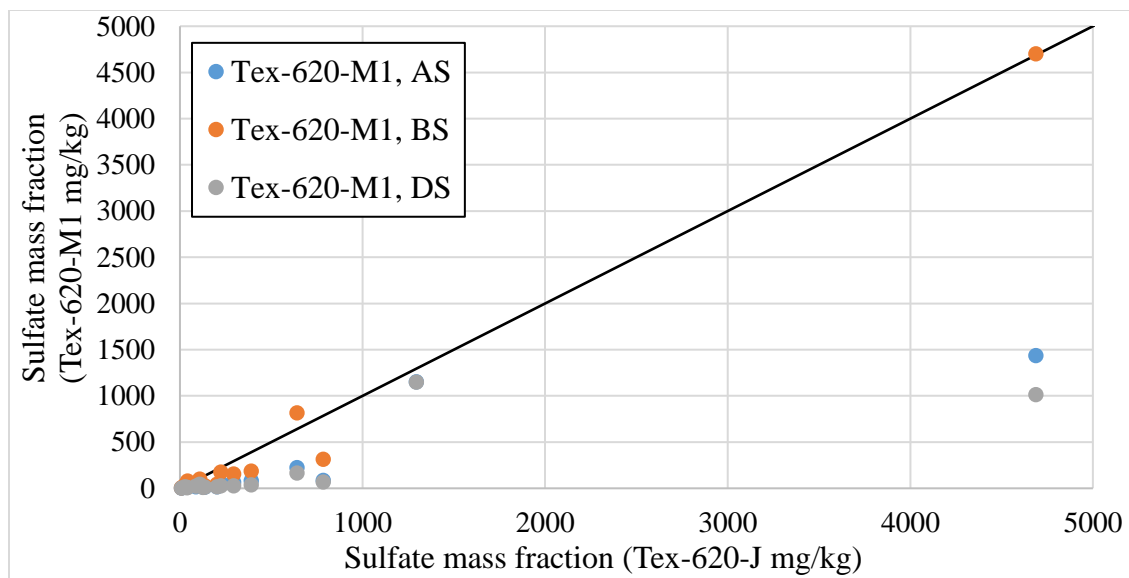
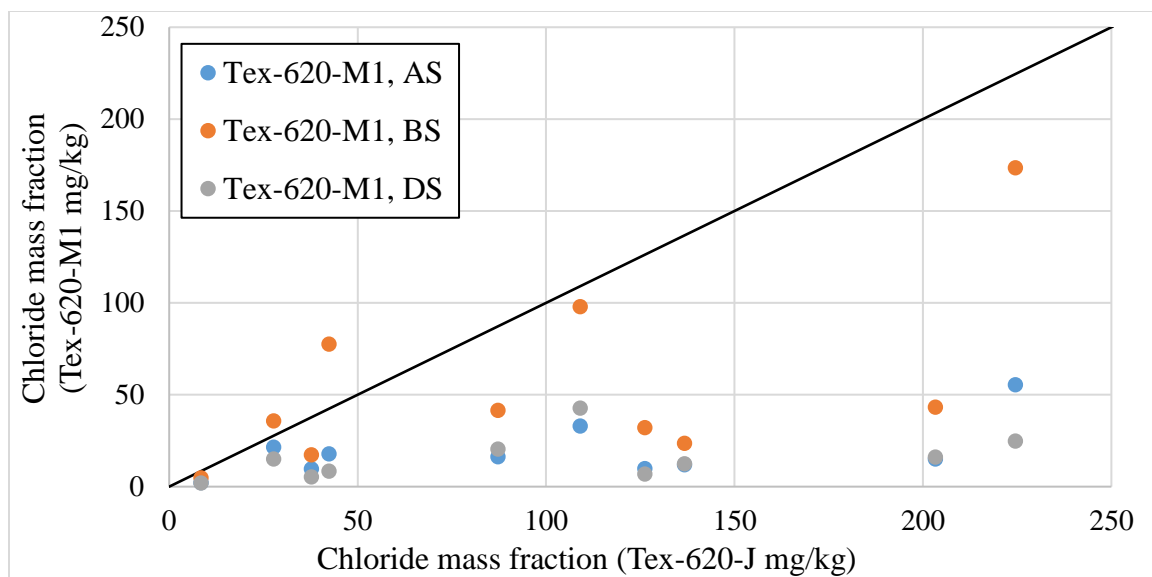
The Tex-620-M2 test procedure could be used as a reliable test procedure for evaluating backfill material to (1) allow the qualification and use of coarse aggregate materials that cannot be fairly tested with current test procedures, and (2) restrict the qualification of unacceptable coarse or fine material.

## References

- [1] V. Elias, K. Fishman, B. Christopher, and R. Berg, "CORROSION / DEGRADATION OF SOIL REINFORCEMENTS FOR MECHANICALLY STABILIZED EARTH WALLS AND REINFORCED SOIL SLOPES," *U.S Dep. Transp. Fed. Highw. Adm.*, no. 132042, 2009.
- [2] D. Borrok, A. Bronson, and S. Nazarian, "Characterization of Coarse Backfill Materials for Prevention of Corrosion of MSE Metallic Wall Reinforcement," vol. 79968, April, 2013.
- [3] American Association of State Highway and Transportation Official (AASHTO), "Standard Method of Test for Determining pH of Soil for Use in Corrosion Testing," *AASHTO T-289*, 1991.
- [4] American Association of State Highway and Transportation Official (AASHTO), "Determining Water Soluble Sulfate Ion Content in Soil," *AASHTO T-290*, pp. 1–5, 1996.
- [5] American Association of State Highway and Transportation Official (AASHTO), "Determining Water Soluble Chloride Ion Content in Soil," *AASHTO T-291*, pp. 2104–2113, 2010.
- [6] American Society for Testing and Materials (ASTM), "Standard Test Method for Determining pH of Lightweight Aggregate," *ASTM D-18*, pp. 1–7, 2014.
- [7] American Society for Testing and Materials (ASTM), "D4972 - Standard Test Method for pH of Soils," *ASTM D-4972*, pp. 1–3, 2001.
- [8] S. V. I. William, I. Corpro Companies, and O. C. R. Group, "CORROSION IN THE SOIL ENVIRONMENT : SOIL RESISTIVITY AND PH MEASUREMENTS," January 2009, 2009.
- [9] South Carolina Department of Transportation, "Method of preparing Coarse Aggregate Sample for pH and Resistivity Testing in the Laboratory," *SC T 143*, pp. 1–2.
- [10] Texas Department of Transportation (TXDOT), "Test Procedure for DETERMINING SOIL pH TxDOT Designation : Tex-128-E," no. 40, pp. 1–3, 1999.
- [11] Texas Department of Transportation (TXDOT), "Test Procedure for DETERMINING SULFATE CONTENT IN SOILS — COLORIMETRIC METHOD TxDOT Designation : Tex-145-E PART I — FIELD DETERMINATION OF SULFATES," vol. 4, no. 4, pp. 1–7, 2005.
- [12] Texas Department of Transportation (TXDOT), "Test Procedure for CONDUCTIVITY TEST FOR FIELD DETECTION OF SULFATES IN SOIL TxDOT Designation : Tex-146-E," pp. 6–8, 2005.
- [13] Texas Department of Transportation (TXDOT), "Test Procedure for DETERMINING SODIUM CHLORIDE IN DEICING TxDOT Designation : Tex-620-J," *TxDOT Des. TEX-620-J*, vol. 60, no. 60, pp. 1–15, 2014.

## Appendix





## **Vita**

Troy Allen Svede, an El Paso Texas native, obtained his B.S. degree in Civil Engineering at the University of Texas at El Paso, El Paso, Texas in December 2015. During his bachelors, Troy was given the opportunity to intern with a local company HNTB. His academic research work included building a demonstration Reverse Osmosis Unit for the KBH Water Treatment Facility in El Paso Texas. His passion for water treatment led him to further his education and obtain his masters in Environmental Engineering, and his master's thesis research work focused on the Evaluation of a Roller Method for Geochemical Leach Testing. With his education and work experience, he hopes to work for an engineering firm in El Paso to continue the process of providing clean drinking water to the community.

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