

1. Report No. FHWA/TX-11/0-6362-1	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle RAPID FIELD DETECTION OF SULFATE AND ORGANIC CONTENT IN SOILS: TECHNICAL REPORT		5. Report Date October 2010 Published: June 2011	
		6. Performing Organization Code	
7. Author(s) Pat Harris, Omar Harvey, and Stephen Sebesta		8. Performing Organization Report No. Report 0-6362-1	
9. Performing Organization Name and Address Texas Transportation Institute The Texas A&M University System College Station, Texas 77843-3135		10. Work Unit No. (TRAIS)	
		11. Contract or Grant No. Project 0-6362	
12. Sponsoring Agency Name and Address Texas Department of Transportation Research and Technology Implementation Office P. O. Box 5080 Austin, Texas 78763-5080		13. Type of Report and Period Covered Technical Report: September 2009–August 2010	
		14. Sponsoring Agency Code	
15. Supplementary Notes Project performed in cooperation with the Texas Department of Transportation and the Federal Highway Administration. Project Title: Rapid Field Detection of Sulfate and Organic Content in Soils URL: http://tti.tamu.edu/documents/0-6362-1.pdf			
16. Abstract In recent years, the Texas Department of Transportation (TxDOT) has experienced problems chemically stabilizing moderate to high plasticity clay soils with calcium-based additives. Many of the problems are the result of soluble sulfate minerals in the soil reacting with the lime or cement added for stabilization. The occurrence of these deposits is unpredictable and often restricted to small areas. To address this problem, the researchers set about identifying a technique that provides a map showing the sulfate content of the soil over a large area to a depth of 3 to 4 ft. Two technologies were identified that provide an indirect measurement of sulfate salts (an electromagnetic device – EM-38, and a soil conductivity device – VERIS 3150). We tested these devices on three different TxDOT projects in different parts of the state that have been known for high sulfate contents. We collected soil samples at 1 ft intervals to a depth of 4 ft where the data varied. We measured the PI, moisture content, sulfate content, and organic content in each sample. We then ran multivariate statistical analyses to correlate the conductivity data collected with the VERIS 3150 to laboratory-measured soil properties. We observed that soil conductivity is related to the soil texture/clay content, moisture content, and dissolved salts (i.e., sulfate and other salt minerals). We noted that for all of the projects tested, a soil conductivity over 100 mS/m may contain problematic sulfate levels, but it may also be due to high plasticity clay soils and/or high moisture contents with other dissolved salts. What is noteworthy about this research is it provides a tool to intelligently decide where to collect soil samples to analyze for problematic sulfate levels versus the current method of collecting soil samples in a grid pattern of a specified interval that may be too large and not detect problematic sulfate levels until the road explodes.			
17. Key Words XRF, DC, EM, PACES		18. Distribution Statement No restrictions. This document is available to the public through NTIS: National Technical Information Service Springfield, Virginia 22161 http://www.ntis.gov	
19. Security Classif.(of this report) Unclassified	20. Security Classif.(of this page) Unclassified	21. No. of Pages 96	22. Price

RAPID FIELD DETECTION OF SULFATE AND ORGANIC CONTENT IN SOILS: TECHNICAL REPORT

by

Pat Harris
Associate Research Scientist
Texas Transportation Institute

Omar Harvey
Post Doctoral Research Associate
Texas Transportation Institute

and

Stephen Sebesta
Assistant Research Scientist
Texas Transportation Institute

Report 0-6362-1

Project 0-6362

Project Title: Rapid Field Detection of Sulfate and Organic Content in Soils

Performed in cooperation with the
Texas Department of Transportation
and the
Federal Highway Administration

October 2010

Published: June 17, 2011

TEXAS TRANSPORTATION INSTITUTE
The Texas A&M University System
College Station, Texas 77843-3135

DISCLAIMER

The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official view or policies of the Federal Highway Administration (FHWA) or the Texas Department of Transportation (TxDOT). This report does not constitute a standard, specification, or regulation. The researcher in charge was Pat Harris, P.G. (Texas# 1756).

The United States Government and the state of Texas do not endorse products or manufacturers. Trade or manufacturers' names appear herein solely because they are considered essential to the object of this report.

ACKNOWLEDGMENTS

Dr. German Claros, P.E., and Dr. Zhiming Si, P.E., from the Texas Department of Transportation are program coordinator and project director, respectively, of this important project and have been active in providing direction to the research team. Mr. Al Aramoon, P.E.; Mr. Cliff Coward; Mr. Billy Pigg, P.E.; Ms. Caroline Herrerra, P.E.; Mr. Mike Arellano, P.E.; Mr. Tracy Cain, P.E.; Mr. Ramon Rodriguez; and Ms. Adriana Geiger, from TxDOT, have also been active in assisting the researchers. Both TxDOT and the FHWA provided funds for this project.

TABLE OF CONTENTS

	Page
List of Figures	viii
List of Tables	x
Chapter 1. Literature Review of Potential Techniques to Rapidly Identify Soil Organic Carbon and Soluble Sulfate Minerals in Texas Soils.....	1
Introduction.....	1
Sulfate Detection.....	2
Chapter 2. Evaluation of Existing Technologies for Measuring Organic Matter and Sulfates.....	15
Summary of Raman Spectroscopy.....	16
Summary of Infrared (IR) Spectroscopy	16
Summary of XRF Results	17
Summary of Multispectral Analysis (MSA).....	28
Summary of Electromagnetic Induction Using EM-38	28
Summary of Electrical Conductivity Using Veris 3150	28
Chapter 3. Field Trial of Existing Technologies for Measuring Organic Matter and Sulfates.....	29
Introduction.....	29
Testing	31
Results	32
Discussion.....	38
Conclusions.....	40
Chapter 5A. Evaluating the Veris 3150 and EM-38 Conductivity Devices	41
Introduction.....	41
Eagle Pass Loop.....	42
Grapevine, Texas	47
Sherman, Texas.....	55
Sherman, Texas, Round Two.....	70
Chapter 5B. Evaluating the NIR Attachment to Estimate Soil Organic Matter	77
Introduction.....	77
Results	78
Chapter 6. Conclusions and Recommendations.....	81
References.....	83

LIST OF FIGURES

	Page
Figure 1.1. Heaves Observed at Joe Pool Lake due to Sulfate in the Soil (Left). Localized Sulfate Deposits Exposed in a Drainage Ditch (Right).....	1
Figure 1.2. Map of High Organic Matter and High Smectite Contents.....	2
Figure 1.3. Efflorescence of Gypsum in Drainage Ditch along Highway.....	4
Figure 1.4. Gypsum Crystals Present in Embankment Adjacent to Highway.....	4
Figure 1.5. Two Devices [EM-38 (Left), PACES (Right)] Tested at Riverside Campus for Sulfate Detection.....	6
Figure 1.6. Veris 3150 Field Conductivity Measuring Machine.....	7
Figure 1.7. False Color Image of White Sands, New Mexico, Shows Concentration of Gypsum.....	8
Figure 1.8. Use of Rockhound to Identify Minerals in a Rock Sample.....	9
Figure 1.9. Emission of Characteristic X-Rays Explain the Theory behind XRF.....	10
Figure 1.10. Portable XRF Used to Detect Sulfur in Experimental Site at Riverside Campus, Texas A&M.....	10
Figure 1.11. Colorimetric Technique (Tex-408-A) Can Yield Artificially High Organic Contents.....	12
Figure 1.12. Veris VIS-NIR Spectrophotometer Used to Measure Soil Carbon.....	13
Figure 1.13. Absorbance of Humic Acid Present in Soil at a Wavelength of 300 nm.....	14
Figure 2.1. Niton XL3t Handheld XRF Detector with He Purge System.....	17
Figure 2.2. XRF Results from Kaolinite Treated with Gypsum.....	18
Figure 2.3. Calibrating Sulfur Content to Measured Ca Content.....	19
Figure 2.4. Calibrating XRF to Sulfur Concentration.....	20
Figure 2.5. XRF Results from Kaolinite Treated with Pyrite.....	21
Figure 2.6. Calibrating Iron Content to Sulfur Content from Kaolinite Treated with Pyrite.....	21
Figure 2.7. Known Sulfur Content versus XRF Measurements with Natural Soil.....	23
Figure 2.8. Calibrating XRF Measurements to Sulfur Concentration.....	26
Figure 3.1. Test Site at Riverside Campus.....	29
Figure 3.2. Diagram of Gypsum and SOM Contents in Test Pad at Riverside Campus.....	30
Figure 3.3. EM-38DDRT Used to Measure Electromagnetic Properties of Soil.....	31
Figure 3.4. Portable XRF with Extendapole Used for Measurement in the Field.....	31
Figure 3.5. Veris 3150 Conductivity Device Used to Measure Conductivity of the Soil.....	32
Figure 3.6. EM-38DDRT Data Collection at Heights of 0, 2, and 4 Ft above Ground.....	32
Figure 3.7. Electrical Conductivity Maps for the EM-38DDRT at Ground Level.....	34
Figure 3.8. Electrical Conductivity Maps for the EM-38DDRT at 2 Ft above Ground Level.....	35
Figure 3.9. Electrical Conductivity Maps for the EM-38DDRT at 4 Ft above the Ground.....	36
Figure 3.10. Aftermath of Storm where Sulfates and Organics Were Rearranged.....	37
Figure 3.11. (A) Known Sulfate Content versus (B) Sulfate Measured with the Handheld XRF.....	38
Figure 3.12. Causes of Conductivity Variation in Soils Show Salinity Is Largest Contributor.....	39
Figure 5a.1. US 67 in the Dallas District Showing Distress due to Sulfate Heave.....	41

Figure 5a.2. Devices Used in This Study to Measure Conductivity of Subgrade: A) Veris 3150; and B) EM-38DDRT.	42
Figure 5a.3. New Construction Site in Eagle Pass, Texas, Shows Dry Conditions.	43
Figure 5a.4. Electrical Conductivity Maps of the Shallow and Deep Sections in Eagle Pass, Texas.	44
Figure 5a.5. Electromagnetic Induction Data Collected with the EM-38DDRT Device in Horizontal (Shallow) and Vertical (Deep) Modes.	45
Figure 5a.6. Soils Map Showing Gypsum Content from the NRCS Website of the Area Analyzed with the Veris 3150.	47
Figure 5a.7. Image Taken from Google Earth Showing the Location (Area inside Red Square) of Our First Test Site in Grapevine.	48
Figure 5a.8. Auger Used to Collect Soil Samples Every 1 Ft to a Depth of 4 Ft in Areas of Contrasting Conductivity.	49
Figure 5a.9. Conductivity Data for Site 1 Collected with the Veris 3150.	50
Figure 5a.10. Section Adjacent to Access Ramp for Eastbound SH 121 is the Second Location in Grapevine.	52
Figure 5a.11. Grapevine Site 2 Showing Embankment with Steep Slope that Prevented Collecting Soil Samples Using the Drilling Rig from Some Areas.	53
Figure 5a.12. Conductivity Data For Site 2 Collected with the Veris 3150.	54
Figure 5a.13. Image Taken from Google Earth Showing the Location (Area inside Red Rectangle) of Our First Test Site in Sherman.	56
Figure 5a.14. US 82 Site 1, East of Sherman Shows Fill Material in Choctaw Creek Drainage Basin.	57
Figure 5a.15. Bobcat with 9-Inch Auger Used to Collect Soil Samples to a Depth of 4 Ft.	57
Figure 5a.16. Veris Conductivity Data for US 82 Site 1 East of Sherman.	58
Figure 5a.17. EM-38DDRT Electromagnetic Data for US 82 Site 1 East of Sherman.	59
Figure 5a.18. Sulfate Content versus Veris 3150 EC.	62
Figure 5a.19. Natural Log of Sulfate Content versus Veris 3150 EC.	62
Figure 5a.20. Correlation among Soil Parameters upon Zero-Order Conditioning.	65
Figure 5a.21. Correlation among Soil Parameters after Zero and First-Order Conditioning.	66
Figure 5a.22. Predicting Sulfates with Veris EC.	66
Figure 5a.23. Grouped and Ungrouped Transformed Sulfate and EC Data.	68
Figure 5a.24. Predicting Sulfates with Grouped Data.	69
Figure 5a.25. Predicted Sulfates with and without Intercept Value.	70
Figure 5a.26. Aerial View of US 82 Project Shows High Sulfate Zone (Red Arrow) and Extent of Data Collection (Blue Arrow).	71
Figure 5a.27. View Looking from High Sulfate Area (Foreground) Back to Low Sulfate Area (Background) to the West.	72
Figure 5a.28. Electrical Conductivity Measurements Taken with the Veris 3150 for US 82.	73
Figure 5a.29. Veris Deep Conductivity Data Plotted before and after a Rain Event from Stations 1528+00 to 1533+00 in Sherman.	75
Figure 5b.1. Black Implement NIR Device that Is Attached to Veris 3150.	77
Figure 5b.2. NIR Detector (Red Arrow) Attached between the Rubber Wheels.	78

LIST OF TABLES

	Page
Table 2.1. Techniques to Measure Sulfate Evaluated in This Project.	15
Table 2.2. XRF Results for Gypsum.....	18
Table 2.3. Calibrating XRF Data to Sulfur Concentration.	19
Table 2.4. XRF Measurements for Natural Soil Treated with Gypsum.	22
Table 2.5. Calibrating XRF Data to Sulfur Concentration with Natural Soil.....	23
Table 2.6. XRF Data.	25
Table 2.7. Statistical Summary of XRF Calcium Measurements.	25
Table 2.8. Maximum Error from Varying Number of XRF Tests.....	27
Table 5a.1. Soil Properties of Samples Collected From Eagle Pass.	46
Table 5a.2. Engineering Properties of Soil Samples from Grapevine Site 1 (Teardrop Site).....	51
Table 5a.3. Engineering Properties of Soil Samples from Grapevine Site 2.	55
Table 5a.4. Veris EC, Sulfate, Organic Content, Water Content, and Plasticity Index for Shallow Measurements.	60
Table 5a.5. Multivariate Regression Output for Predicting Veris EC.	61
Table 5a.6. Data for Predicting EC Using Natural Log of Sulfate Content, Organic Content, Water Content, and Plasticity Index.....	63
Table 5a.7. Multivariate Regression Output for Predicting Veris EC where Sulfates Are Transformed Using the Natural Log.	64
Table 5a.8. Correlation Matrix and Test Statistics.	64
Table 5a.9. Regression Output Predicting ln(Sulfate Concentration) from Veris EC.	66
Table 5a.10. Data Grouped According to Geospatial Regions for Input into Model Development.	67
Table 5a.11. Regression Output from Shallow Grouped Data.	68
Table 5a.12. Regression Output from Deep Grouped Data.	69
Table 5a.13. Regression Output from All Grouped Data.	69
Table 5a.14. Engineering Properties of Soil Samples from Sherman Site 2.	74
Table 5b.1. Organic Carbon Contents of Samples Collected from US 183 and SH 29.....	79

CHAPTER 1

LITERATURE REVIEW OF POTENTIAL TECHNIQUES TO RAPIDLY IDENTIFY SOIL ORGANIC CARBON AND SOLUBLE SULFATE MINERALS IN TEXAS SOILS

INTRODUCTION

In recent years, the Texas Department of Transportation (TxDOT) has experienced problems chemically stabilizing moderate to high plasticity clay soils with calcium-based additives. Many of the problems are the result of soluble sulfate minerals in the soil (Figure 1.1, left) reacting with the lime or cement added for stabilization (Petry and Little, 1992; Burkart et al., 1999; Harris et al., 2004). The occurrence of these deposits is unpredictable and often restricted to small areas (Figure 1.1, right).



Figure 1.1. Heaves Observed at Joe Pool Lake due to Sulfate in the Soil (Left). Localized Sulfate Deposits Exposed in a Drainage Ditch (Right).

Additionally, soils in some regions of the state with moderate to high concentrations of organic matter have caused rapid deterioration of the roadway. Evidence suggests that this is due to a combination of the loss of subgrade stabilizer effectiveness through organic acids neutralizing the stabilizer, calcium from the stabilizer forming complexes with organic molecules, and organics coating clay particles, which physically prevent clay/stabilizer reactions (Clare and Sherwood, 1954; Clare and Sherwood, 1956). Figure 1.2 depicts as red and brown areas the regions of the state that contain moderate to high concentrations of smectite (expansive clay) and moderate to high concentrations of organic matter. These areas are where most efforts at stabilizing organic rich soils will be concentrated. As one can see, most organic rich clay soils

are concentrated along river drainages, the coast, and associated with the Balcones, Mexia-Talco, and Luling fault zones. TxDOT personnel in western and northwestern districts report minor problems with organics (primarily along stream drainages), which lends credence to the data presented in Figure 1.2.

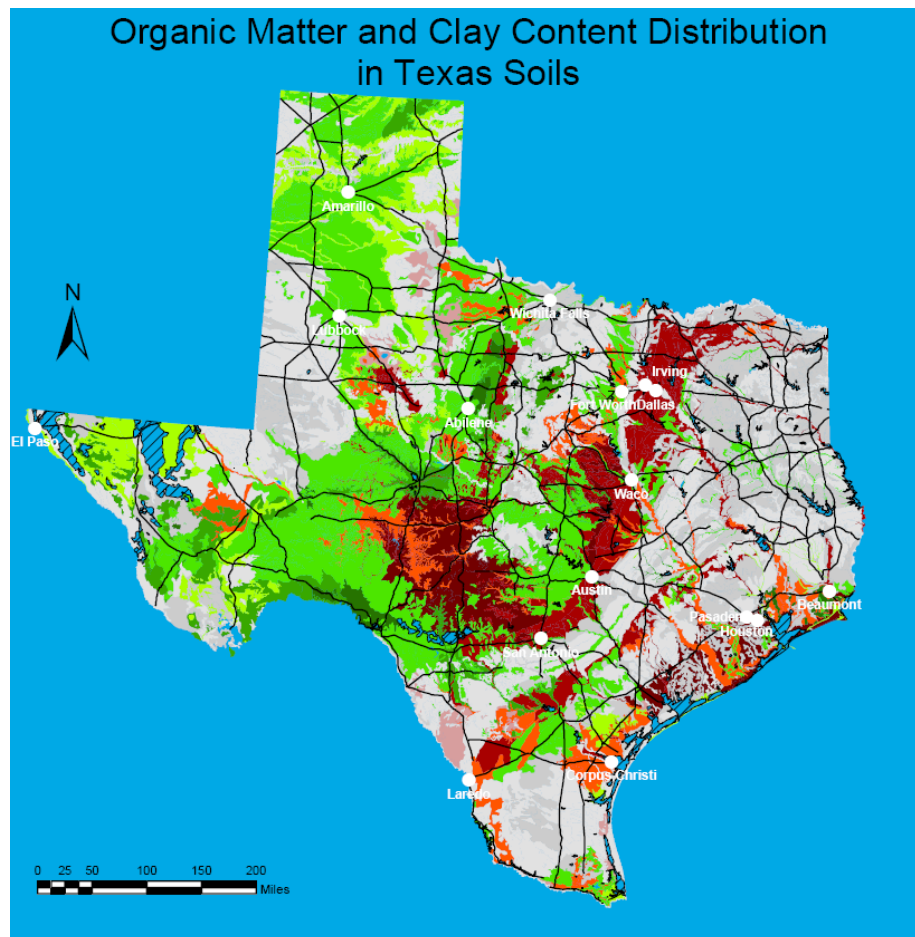


Figure 1.2. Map of High Organic Matter and High Smectite Contents.

SULFATE DETECTION

In the paragraphs that follow, we explore some of the existing technology for detecting sulfate minerals and organic matter. A good way to get a general idea of potential problems due to sulfate minerals in the soil is to look at the Geologic Atlas of Texas published by the Bureau of Economic Geology in Austin. It has 38 sheets printed at a scale of 1:250,000 that cover the entire state. A digital version of the Atlas is available in geographic information systems (GIS) format. It has a detailed description of all of the formations, listing the minerals expected in each rock formation. Sulfate and sulfide minerals are commonly listed in the descriptions of rock formations. One of the most problematic formations in Texas with respect to sulfate heave is the Eagle Ford. The Dallas, Fort Worth, and Paris Districts are familiar with this formation and automatically test for sulfates any time construction is planned in areas where this formation is present. These maps were discussed further in research project 0-4240 (Report 4240-1).

The United States Department of Agriculture (USDA) has an excellent web-based mapping system that can be accessed by visiting the web soil survey (<http://websoilsurvey.nrcs.usda.gov/app/HomePage.htm>). Sulfate and organic matter are two parameters that can be mapped using this database by selecting an area of interest, the features to be mapped, and to what depth in the soil to be mapped. We have done some field checking of these maps that are sometimes fairly accurate and other times are not accurate. Inaccuracies can be attributed to the scale at which the maps are made and the sulfate content fluctuating with the wet and dry seasons. However, these resources are a good place to start when planning a new or rehabilitating an old construction project.

The best way to detect sulfates is by visual observation of the subgrade to be stabilized. Sulfate minerals can often be seen if an inspector is trained to look for them. For example, gypsum is highly soluble in water and will dissolve in the rainy season and be transported to depth in the soil; as the summer dry season arrives the dissolved salt is transported to the surface of the soil where it will precipitate out as a white crust (Figure 1.3). Actual gypsum crystals may also be observed in ditches or embankments adjacent to the highway (Figure 1.4). Gypsum is a very soft mineral that can be easily scratched with a fingernail. If you see translucent to transparent crystals lying on the ground, then pick one up and try scratching it with your fingernail.

An absence or low diversity of vegetation may also be an indication of dissolved salts like gypsum. Researchers in northern Queensland, Australia, used vegetation to detect salinity, but there was a large scatter in vegetation type and salinity so the correlation was not that good (Bui and Henderson, 2003). In Texas, Dr. Fares Howari (2009) tried using vegetation to map salinity in soils. However, he determined that there were too many types of vegetation with too broad a range in salinity tolerance to be useful for mapping salinity.



Figure 1.3. Efflorescence of Gypsum in Drainage Ditch along Highway.



Figure 1.4. Gypsum Crystals Present in Embankment Adjacent to Highway.

Dozens of tests have been promoted for measuring sulfate in soils. Many of the tests are a variation of measuring conductivity generated by the salt dissolved in water. Bower and Huss (1948) used conductivity to measure sulfate content in soils by mixing air-dried soil with distilled water. The water dissolved the gypsum; acetone was then added to reprecipitate the gypsum. The reprecipitated gypsum was washed to remove salts (NaCl, etc.) and then redissolved in distilled water. The conductivity was measured and compared to a calibration curve to determine gypsum concentration in the soil. Bredenkamp and Lytton (1995) used a similar conductivity technique to measure sulfate in soil. TxDOT currently employs a conductivity test (Tex-146-E) as a screening tool for sulfates. However, these techniques all rely on collecting spot samples and measuring the conductivity of a selected sample. These techniques do not provide a continuous measurement of the electrical properties of a soil, so localized concentrations of sulfate may go unnoticed until the damage is done.

Conductivity is a measure of how well a solution will carry a current (i.e., pass electrons usually via ions). Two factors influence conductivity: first, the number of displaceable electrons each ion carries (e.g., an anion with a -2 charge will carry twice as many electrons as an anion with a -1 charge); second, the speed at which each ion travels through the solution (Robinson, 1970).

Conductivity is the sum of the conductivities of the ions present; it cannot distinguish between different types of ions. For example, if two salts (halite and gypsum) are present in a soil, gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) will dissolve releasing Ca^{2+} and $(\text{SO}_4)^{2-}$ ions while halite (NaCl) will add Na^+ and Cl^- to the solution. Both salts will contribute to the conductivity. Also at higher

concentrations the ions may form some un-ionized molecules which will reduce the conductivity (Robinson, 1970).

The conductivity of soil is further complicated because the current is traveling through a complex mixture of minerals, organic matter, liquids, and gases. Soil conductivity will be influenced by a number of parameters which include: porosity (shape and volume of pores), permeability (interconnectedness of pores), moisture content, temperature and phase state (solid/liquid/gas) of water, and the amount and composition of clays (Spies and Woodgate, 2005).

Indirect Measurement of Sulfate

The following discussion concerns techniques for indirectly measuring sulfates in soils by determining the conductivity/resistivity of the soil.

Direct current (DC) electrode devices that require electrical contact with the ground can be used to measure resistivity (reciprocal of conductivity). These techniques offer better resolution than the inductive conductivity devices, but they require much more time and manpower to collect data because the electrodes have to be placed in the ground.

There have been several technological developments in portable analytical devices. One such device is the Geonics EM-38 (Figure 1.5, left), which is a portable electromagnetic induction device that directly measures conductivity at shallow depths. In the horizontal dipole mode, these devices give fairly accurate measurements of ground conductivity to approximately 3 ft depth. Research done at Texas Transportation Institute (TTI) and the Department of Soil and Crop Sciences at the Texas A&M University System showed that this device worked well at distinguishing sandy versus clay rich soils, but it was not sensitive enough to detect differences in sulfate content in the soil. The sulfate signal tends to be masked by clay and is dependent on soil moisture conditions. However, Bennett et al. (2000) found that salinity was the dominant contributor to electromagnetic (EM) response in all soils they studied. They further state that for practical purposes, ground-based EM can be confidently used in salinity mapping. Other researchers in Australia recommend the EM-38 device for mapping saline soils (gypsum is a dominant phase in saline soils), but they warn that soils must be sufficiently moist (~20 percent volumetric moisture) for the measurable conduction of electricity (Spies and Woodgate, 2005).

The device in Figure 1.5 (right) uses the pulled array continuous electrical sounding (PACES) method to measure the electrical properties of the soil. This technique is used in lieu of putting electrodes into the ground. It has a current electrode that sends an electric current through the ground and a tail of potential electrodes that measure the electrical resistivity at fixed distances from the current electrode. This instrument produces a depth profile of resistivity along a path. Data covered 10 to 15 km can be collected in a day with this technique.



Figure 1.5. Two Devices [EM-38 (Left), PACES (Right)] Tested at Riverside Campus for Sulfate Detection.

The PACES device actually could distinguish different sulfate contents in our test site by means of differences in electrical properties of the soil. However, neither of these techniques directly measure sulfate content; they are indirect measurements of electrical conductivity or resistivity (reciprocal of conductivity) of a soil.

Another device used extensively in agriculture is a disk harrow that has been equipped with heavy duty coulter electrodes to measure the conductivity of a soil as the disk cuts through the soil in a field. We rented the Veris 3150, one of these devices (Figure 1.6) and tried it on the site at Riverside campus of the Texas A&M University System. This device does not measure sulfates, but it does measure the soil conductivity at two different depths, a shallow reading down to a depth of 18 inches and a deep reading down to a depth of 3 ft. However, the instrument we rented had been modified for the wider rows farmers typically use in Texas fields. The discs/electrodes were spread farther apart resulting in shallow and deep readings down to about 2 ft and 4 ft deep. Soil moisture, salts, and clay content will affect the readings. The soil moisture content needs to be ~10 percent by volume to get good results; higher moisture contents do not negatively impact results, although the overall conductivity values may increase with increased soil moisture, the relative values remain consistent. According to the manufacturer, soil compaction will not affect the readings enough to make a difference in the overall readings. Dissolved salts will increase the conductivity of the soil more than clay. The air temperature will not affect readings from this device because the electrodes are coupled to the ground negating air temperature corrections that are necessary with non contact electromagnetic induction devices.



Figure 1.6. Veris 3150 Field Conductivity Measuring Machine.

Techniques to Measure Sulfate Directly

The following techniques measure sulfur directly or identify sulfur-bearing minerals. Another technique for detecting sulfates that has a lot of potential is multispectral analysis (MSA), which is used in remote sensing. Multispectral analysis is the study of data in different spectral bands. The term spectral bands, refers to the electromagnetic spectrum, more specifically the ultraviolet, visible, and infrared regions. This technology is used in laboratory, field, airborne, and spacecraft applications. The spectra obtained with laboratory instruments (infra-red [IR], ultraviolet-visible [UV-Vis]) use the same spectrometers that are used in airplanes and satellites. There will be more interferences with airborne and satellite-borne systems due to interactions with the atmosphere. Therefore, more data manipulation is required to filter out all of the noise and obtain useful data. Figure 1.7 is a false color image generated from Airborne Visual and Infra-Red Imaging Spectrometer (AVIRIS) satellite data. The false color image is generated by assigning a color to a particular infra-red wavelength (e.g. cyan is assigned to a wavelength typical of gypsum so the gypsum shows up as cyan in the image in Figure 1.7). Using infra-red spectra collected by the satellite, Dr. Fares Howari was able to map the gypsum distribution at White Sands, New Mexico. Dr. Howari successfully delineated gypsum, which is a hydrous calcium sulfate mineral and is the predominant sulfate mineral responsible for sulfate-induced soil heave in Texas.

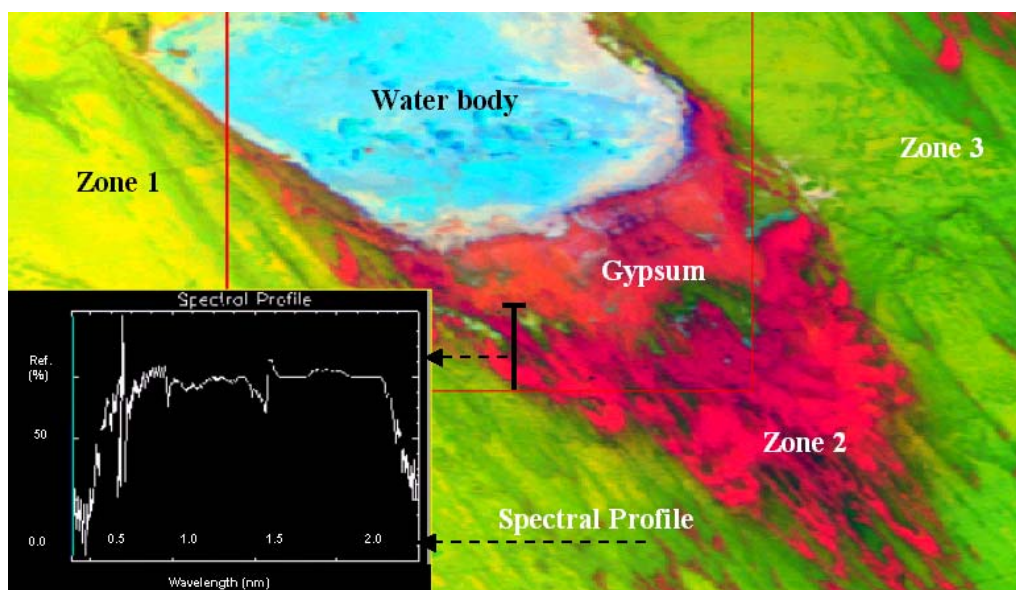


Figure 1.7. False Color Image of White Sands, New Mexico, Shows Concentration of Gypsum.

Image provided by Dr. Fares Howari, University of Texas Permian Basin.

MSA has also been used to map slight variations in vegetation. Spies and Woodgate (2005) successfully used visual observations of vegetation to delineate saline soils in Australia because only certain types of vegetation will grow in saline soils. Dr. Howari has been trying to use vegetation in Texas to delineate areas of hypersalinity. He states that there are many types of vegetation in Texas that are tolerant to high salinity conditions and if any vegetation appears to be stressed, it may be due to high salinity, or it may be due to a lack of water. Dr. Howari's point is that one needs to integrate a large amount of data from diverse disciplines to determine the extent of gypsum deposits in the soil and that one technique will likely not be enough to unequivocally define the extent of sulfate deposits.

MSA advantages are that it can directly identify gypsum, and it can cover very large areas. Drawbacks to MSA are the initial expense of obtaining data/equipment, requirement of having someone specifically trained to do the data manipulation, and it only detects minerals visible at the surface (cannot penetrate vegetation to detect sulfate minerals).

Raman spectroscopy is another technique that has been touted as a good tool to identify minerals in a rapid and non-destructive manner. The Raman Effect occurs when light impinges on a molecule and interacts with the electron cloud and the bonds of the molecule. The light energy excites the vibrational state of molecules in a specimen. The vibrational energy is shifted to a higher or lower level, which generates Raman lines of energy that are going to be different for various molecules and can be used like a fingerprint to identify minerals (Robinson, 1970). One device specifically designed to analyze rock samples in the field, the Rockhound (Figure 1.8) measures these spectra in a sample and compares them to a library of over 500 reference minerals; the software then identifies what minerals are in a sample. Raman spectra are not negatively impacted by water, which makes the technique more appropriate for soils than

Fourier transform infrared analysis (FTIR). Drawbacks to this technique are reduced sensitivity due to fluorescence of certain minerals in soil, the high initial cost of the equipment, and it is a spot test.



Figure 1.8. Use of Rockhound to Identify Minerals in a Rock Sample.

Image taken from <http://DeltaNu.com/rockhound>.

Another technique recently evaluated for measuring sulfate in soil is FTIR. This technique is very similar to Raman spectroscopy, but it requires a changeable electric dipole to absorb the IR radiation. A molecule has an electric dipole when there is a slight positive and a slight negative electrical charge on its component atoms (Robinson, 1970). The data obtained from this technique are generally complimentary to Raman data. Samples bearing water cannot be analyzed with midIR because water is a polar molecule that masks the effects of other polar molecules. Drawbacks to FTIR are the expense of equipment, potential interference from water, and it is a spot test.

X-ray fluorescence (XRF) is another technique that can measure sulfur in soils. A drawback to this technique is that it cannot differentiate between sulfur present in the oxidized form (sulfate) or the reduced form (sulfide). XRF instruments detect the emission of characteristic X-rays from individual atoms. When an element is placed in a beam of X-rays, the X-rays are absorbed. The absorbing atoms become excited and then emit X-rays with wavelengths characteristic of the emitting atoms (Robinson, 1970). Electrons from other shells (L, M, N, etc.) drop in to replace the ejected electron in the K shell as illustrated in Figure 1.9. If an L shell electron replaces the ejected K shell electron, then the energy is called K_{α} . If the M shell electron replaces the ejected K shell electron, then the energy is called K_{β} . If an L shell electron were ejected and replaced with electrons from outer shells, then it would be called L_{α} , L_{β} , etc. Heavier elements are more easily detected than lighter elements because there is a larger difference in the energy required to eject an electron and the energy it takes for an outer shell electron to fill that space. Therefore, detection limits are commonly lower for heavier elements. However, there can be interferences between the emission energies of different elements, which may decrease the detection of certain element combinations.

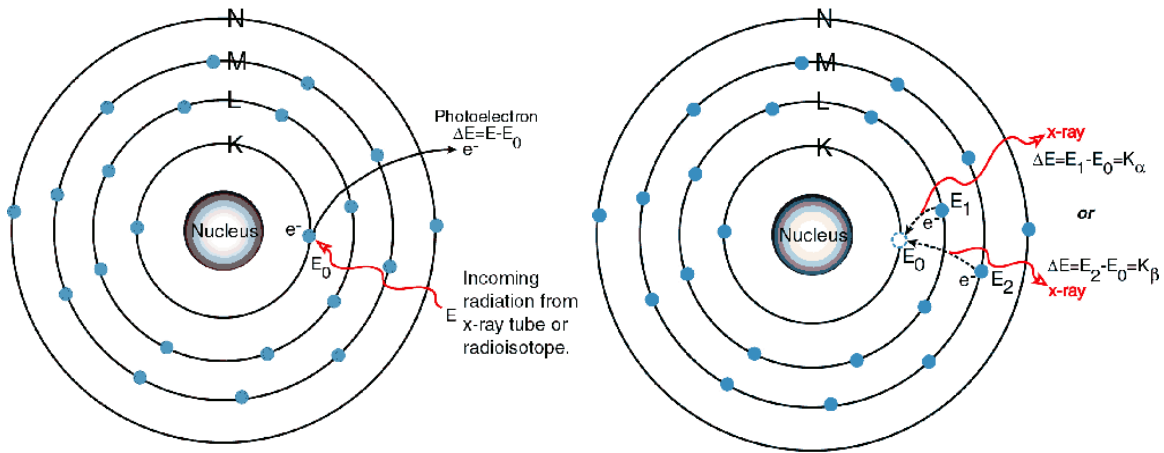


Figure 1.9. Emission of Characteristic X-Rays Explain the Theory behind XRF.

Figure taken from <http://www.amptek.com/xrf.html>.

XRF is again a spot technique that measures a very small sample at the surface (Figure 1.10). The unit we tested (Niton XL3t) also had difficulty measuring sulfur in small concentrations due primarily to the configuration of the detector on these portable units. Sulfur with an atomic mass of 32.064 is the lightest element the Niton XL3t will detect unless a helium (He) purge is added to the unit. With a helium purge, magnesium, aluminum, silicon, and phosphorus can be detected. The portable units do not have the sensitivity of a laboratory instrument. However, the vendor showed the researchers a new portable XRF detector and claim it will detect sulfur, magnesium, aluminum, silicon, and phosphorus without the need for a He purge.



Figure 1.10. Portable XRF Used to Detect Sulfur in Experimental Site at Riverside Campus, Texas A&M.

Measurement of Organic Matter

There are a vast number of techniques to measure organic matter in soils. Soil scientists have developed many methods to analyze different fractions of organic matter; all of the methods have their drawbacks. Most of these techniques are restricted spot samples analyzed in the laboratory. We selected a few methods that are relevant to soil stabilization, which will be described in the paragraphs that follow.

A number of TxDOT districts use an ignition oven to estimate the organic content of soils. The technique is called Loss On Ignition (LOI). They use ASTM 2974-00, which calls for drying the soil in an oven at 105°C (to remove moisture) prior to placing in the ignition oven to burn off the organic matter. The dried soil is placed in the ignition oven at 450°C and left until constant weight is achieved. The weight lost is attributed to organics. The LOI technique may yield false high percentages of organic matter due to hydrated (water-bearing) minerals (expansive clays) losing water that contribute to the loss in weight, which can create some large errors in organic carbon measurement. For soils with zero to low contents of expansive clay minerals, the ignition oven is remarkably accurate at measuring total organic matter in the soil. The errors believed to occur with carbonate rocks are negligible based on findings from research project 0-5540.

Another technique used by TxDOT for fine aggregate used in concrete and mortar is Tex-408-A, a colorimetric technique. It is simply a 3 percent sodium hydroxide (NaOH) solution that is added to the soil. The color of the supernatant is compared to color standards (right side of Figure 1.11) to determine the approximate organic content. This procedure is not reliable; concentrations of organic matter ranging from 0.5 percent to 5 percent all yield similarly high results. Different types of organic matter at the same concentration will yield varying results as well. The bottles from left to right in Figure 1.11 are duplicates of 3 percent, 5 percent, and 0.5 percent organic matter in a sandy soil. As can be seen, these bottles all look to have similar concentrations of organic matter.



Figure 1.11. Colorimetric Technique (Tex-408-A) Can Yield Artificially High Organic Contents.

Another technique commonly employed for measuring organic matter in soils is a Carbon, Hydrogen, Nitrogen (CHN) analyzer. The Department of Soil and Crop Sciences at Texas A&M University uses an Elementar VarioMax CHN analyzer. The procedure used in the Department of Soil and Crop Sciences calls for a wet chemical treatment to remove carbonates, which can give false high organic C (carbon) results. The samples are heated to 650°C (for organic C) in purified O₂ stream and heated to 900°C (for total C). The CO₂ evolved is measured, and percents of organic carbon and total carbon are calculated. Organic matter measurements obtained by researchers at TTI using the CHN analyzer have provided inconclusive results. It is sensitive to mineralogy as well as different types of organic matter, so assumptions about calculating percent organic matter using the organic C evolved will need to be revised.

Sherwood (1962a, b) developed a practical test for cement-soil mixtures. He correlated the pH of soil-cement mixtures during early stages of hydration to unconfined compressive strength (UCS). He concluded that the measurement of the pH of the soil-cement mixture 1 hour after being mixed could be used to identify the presence, but not the quantity, of deleterious organic matter.

Spurred by environmental climate change legislation pertaining to carbon emissions, there has been more research into ways to quickly estimate the carbon content in soils. Recent work has been done with near infrared diffuse reflectance spectrometry for measuring moisture, organic carbon, and total nitrogen (Dalal and Henry, 1986). They concluded that organic C can be measured in soils containing moderate amounts of organic matter (0.3–2.5 percent). He and Song (2006) used diffusely reflected near infrared (NIR) to measure soil organic matter (SOM). They determined that it works well at predicting SOM and can be easily done in the field. A white paper

on the Veris Technologies website, www.veristech.com, contains case studies illustrating their NIR sensor to measure soil carbon. Results from their VIS-NIR spectrophotometer (Figure 1.12) show an R^2 value of 0.93 when compared to laboratory-measured carbon contents.



Figure 1.12. Veris VIS-NIR Spectrophotometer Used to Measure Soil Carbon.

From www.veristech.com.

Current research at TTI shows that UV-Vis spectroscopy can be used to measure the humic acid fraction of organic matter (Figure 1.13). The peaks at a wavelength of 300 nm in Figure 1.13 represent the humic acid fraction of soil organic matter. As stated in the background, humic acids are the bad actors when it comes to soil stabilization issues. Based upon analyses of 146 soils from around Texas, the predicted concentration of soil organic matter using the UV-Vis, humic acid peak at 300 nm, correlates extremely well with the actual measured concentration. The R^2 value was 0.885 for the analyses.

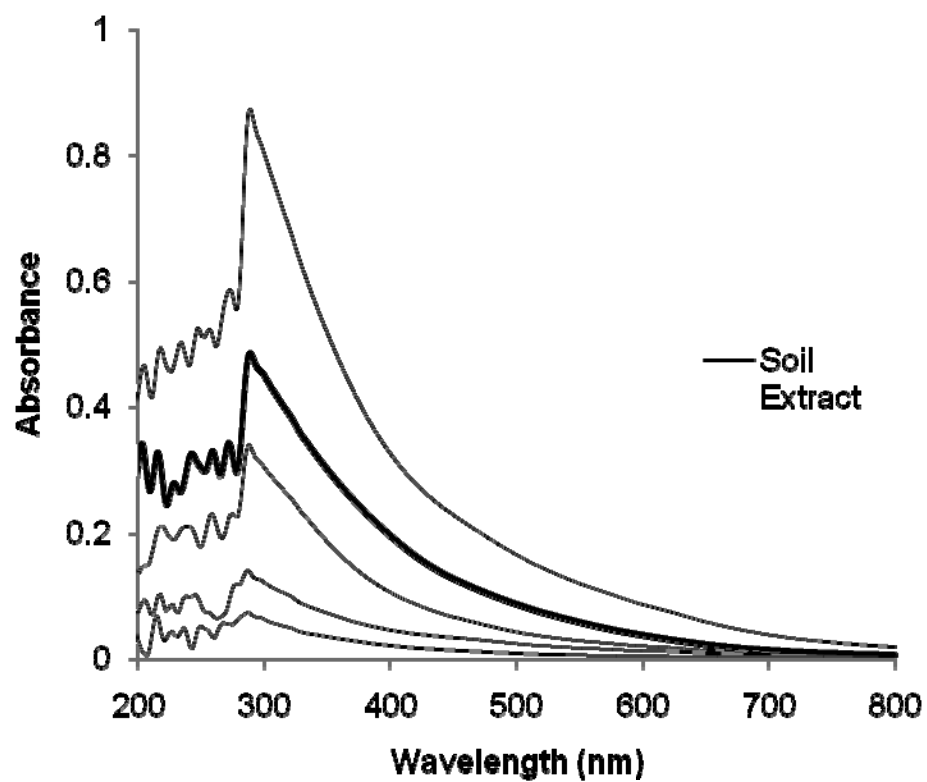


Figure 1.13. Absorbance of Humic Acid Present in Soil at a Wavelength of 300 nm.

CHAPTER 2

EVALUATION OF EXISTING TECHNOLOGIES FOR MEASURING ORGANIC MATTER AND SULFATES

After an extensive literature review, the researchers picked several tools that looked promising with respect to improving on existing techniques to measure sulfates and organic matter in the soil. More specifically, TxDOT wanted a technique that could be run continuously over a project and collect data without any gaps. Currently, TxDOT collects spot samples at specified intervals (usually between 500 and 2500 ft) and returns them to the laboratory for testing (TEX-145-E, and TEX-146-E). If there is a zone smaller than the specified interval that has high sulfates or organic matter, then the current testing plan may miss the problematic soil altogether.

Table 2.1 lists the methods researchers evaluated in this phase of the research project. The table lists the type of data gathered from each technique and some advantages and disadvantages of each technique.

Table 2.1. Techniques to Measure Sulfate Evaluated in This Project.

Method	Type of Data	Pros	Cons
BEG Maps	S minerals	good data for mineralogy	not high enough resolution
Web Soil Survey	S minerals	good for SOM and Sulfates	not high enough resolution soil properties change with season
Visual Observation	gypsum pyrite, etc.	definitive	trained/observant personnel cannot tell actual concentrations
Spectrophotometer	gypsum	can measure low sulfate contents inexpensive, simple	spot test
Satellite Imaging (MultiSpectral Analysis)	gypsum pyrite, etc.	cover large areas, high resolution (2m) can identify gypsum, pyrite, etc.	expense, need expert user to process only surface features, vegetation covers
X-ray Fluorescence	S minerals	can measure S content not too affected by moisture	spot test, detection limits at edge time consuming to do detailed analysis
IR Spectroscopy	gypsum	can measure low sulfate contents	expense, spot test, not field amenable
Raman Spectroscopy	gypsum	fast, can detect many S-bearing mins	cannot work in soils at low concentrations
Conductivity Test	salinity	can detect small salinity differences inexpensive, simple	spot test, cannot directly measure sulfate
Ground Penetrating Radar	stratigraphy	high resolution to 10s of feet	cannot penetrate high salinity
EM-38	salinity	fast, easy, 3 ft depth penetration	MC >20%, poor resolution <10 inch, errors in magnetic soils, frequent recalibration
Veris 3150	salinity	cover large areas, high resolution shallow and deep data (18 and 36 in.)	need soil moisture for electrical conductivity (EC) cannot directly measure sulfate

The researchers started by contacting vendors of specific instruments to obtain access to their equipment to determine if their techniques were better than the current methods employed by TxDOT. Specific techniques we looked at were: (1) a portable Raman Spectrometer developed by DeltaNu for analysis of geological samples; (2) an IR instrument developed by A2 Technologies; (3) a portable XRF unit developed by ThermoFisher; (4) MultiSpectral Analysis; (5) electromagnetic induction using the EM-38 developed by Geonics Limited; and (6) conductivity using an instrument developed by Veris Technologies for precision agriculture.

SUMMARY OF RAMAN SPECTROSCOPY

Researchers contacted DeltaNu, a vendor of a handheld Raman spectrometer that is used to identify minerals in geological rock samples. The spectrometer has a database of over 500 minerals that are used to match with spectra generated in an unknown sample. Unlike infrared spectroscopy, Raman spectroscopy does not have interference from water, which is one less interference to deal with in soils. However, some minerals fluoresce, which makes obtaining a clean spectrum difficult. Another drawback to this device is the small sample area ($\sim 35 \mu\text{m}$).

Researchers submitted soil samples to the manufacturer. The soils were mixed with predetermined amounts of reagent grade gypsum; the samples contained 0, 1000, 3000, 5000, and 8000 ppm of sulfate. Travis Thompson from DeltaNu tested the samples and determined that there was too much interference from fluorescent phases in the soil samples to detect the sulfates at levels as high as 8000 ppm. They never sent any data because they could not detect sulfates in any of the samples we sent to them. We can conclude that Raman will not detect sulfates at concentrations up to 8000 ppm sulfate due to interferences from fluorescence.

SUMMARY OF INFRARED (IR) SPECTROSCOPY

A company, A2 Technologies, contacted researchers about using Fourier Transform Infrared/Attenuated Total Reflectance (FT-IR/ATR) to measure sulfate in soils. We submitted soil samples spiked with sulfate contents of 0, 500, 2500, 19,000, and 40,000 ppm to Jim Fitzpatrick from A2 Technologies.

The company analyzed pure $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to identify infrared peaks that could be integrated to calculate the percentage of gypsum in a soil sample. From the analysis of the pure gypsum sample, they identified two peaks that would be useful for quantitative analysis: a peak located at 1104 cm^{-1} or a peak at 670 cm^{-1} . The area under these peaks is directly proportional to the gypsum content in the soil.

They analyzed samples containing ~ 500 ppm, 2500 ppm, 19,000 ppm, and 40,000 ppm of sulfate and determined that the peak at 670 cm^{-1} tracks linearly with concentration and does not have any interferences with other common soil minerals. It provides a quantitative measure of the sulfate content of the soil.

The technique they recommend is to take 1 gram of homogenized soil and place it in 1 ml of water and agitate it. After the sample has settled, $25 \mu\text{l}$ of water is deposited on the diamond attenuated total reflection (ATR) and allowed to dry. An analysis is then made of the dried residue.

The technique worked well with the samples we supplied to A2 Technologies; however, this technique does not provide any benefit over the spectrophotometric technique TxDOT currently uses (TEX-145-E). Additionally, the cost of one of these

instruments is several thousand dollars compared to approximately \$400 for the spectrophotometer currently used by TxDOT.

SUMMARY OF XRF RESULTS

A representative from ThermoFisher loaned the researchers a handheld XRF unit to try to measure sulfur in subgrade soils. The instrument used by researchers initially was a Niton XL3t handheld XRF analyzer (Figure 2.1) equipped with a Si-pin detector. The compressed gas container is filled with helium, which has to be used to detect light elements like Mg, Si, Al, and P. Following less than stellar results at detecting sulfate in concentrations typically found in Texas soils, the manufacturer loaned researchers the next generation of XRF, the Niton XL3t Geometrically Optimized Large Drift Detector (GOLDD), which improves detection of light elements (i.e., sulfur) without the need for a helium purge.



Figure 2.1. Niton XL3t Handheld XRF Detector with He Purge System.

Experiment #1: Measuring Gypsum in Soil

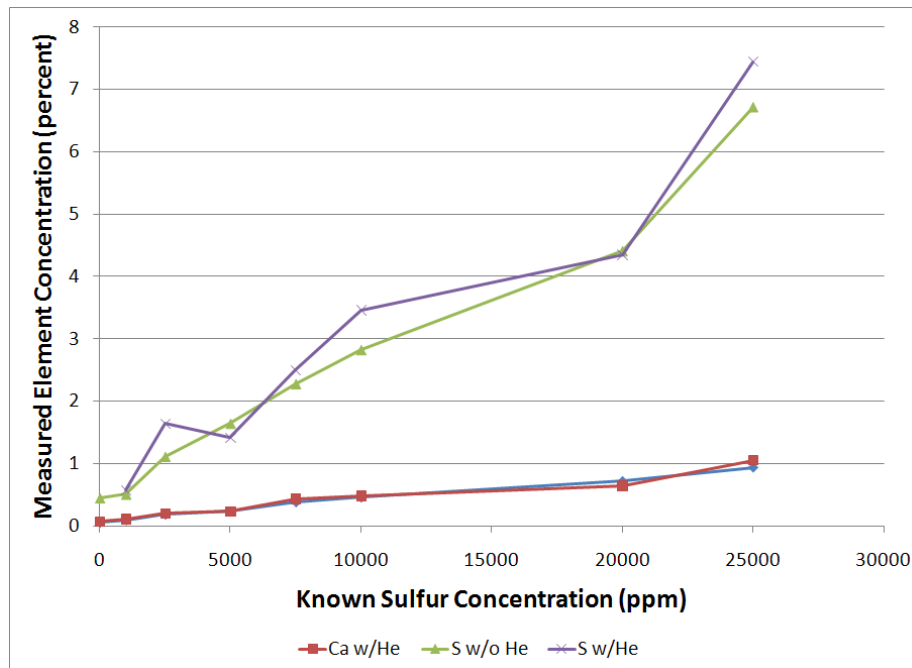
In this experiment, researchers mixed Kaolinite clay with gypsum to generate mixtures with sulfur content ranging from 0 to 25000 ppm and collected three measurements on each treatment level, both with and without the Helium purge. A sulfur concentration of 1000 ppm translates to approximately 3000 ppm soluble sulfate content, which is the maximum recommended threshold for conventional calcium-based soil stabilization. Elements the XRF unit tested for included calcium and sulfur, since the chemical composition of gypsum is $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. Researchers analyzed the results for both calcium and sulfur percentages present in the sample. Table 2.2 presents the results.

Table 2.2. XRF Results for Gypsum.

Without He Purge								
	Ca #1	Ca #2	Ca #3	Ca Avg.	S #1	S #2	S #3	S Avg.
Kaol. 0% gyp.	0.052	0.055	0.05	0.052333	LOD	LOD	0.441	0.441
Kaol. 1000 ppm S.	0.089	0.089	0.094	0.090667	0.518	LOD	0.485	0.5015
Kaol. 2500 ppm S.	0.175	0.185	0.181	0.180333	0.722	1.35	1.25	1.107333
Kaol. 5000 ppm S.	0.223	0.243	0.222	0.229333	1.83	2.03	1.05	1.636667
Kaol. 7500 ppm S.	0.385	0.359	0.387	0.377	1.98	2.16	2.68	2.273333
Kaol. 10000 ppm S.	0.448	0.466	0.445	0.453	3.38	3.19	1.89	2.82
Kaol. 20000 ppm S.	0.602	0.917	0.615	0.711333	4.09	4.85	4.28	4.406667
Kaol. 25000 ppm S.	0.988	0.892	0.913	0.931	6.76	5.97	7.42	6.716667
With He Purge								
	Ca #1	Ca #2	Ca #3	Ca Avg.	S #1	S #2	S #3	S Avg.
Kaol. 0 ppm S.	0.08	0.069	0.065	0.071333	LOD	LOD	LOD	
Kaol. 1000 ppm S.	0.108	0.108	0.114	0.11	LOD	0.579	LOD	0.579
Kaol. 2500 ppm S.	0.185	0.208	0.207	0.2	1.22	2.24	1.46	1.64
Kaol. 5000 ppm S.	0.217	0.216	0.247	0.226667	1.51	1.42	1.31	1.413333
Kaol. 7500 ppm S.	0.449	0.441	0.393	0.427667	2.58	2.77	2.16	2.503333
Kaol. 10000 ppm S.	0.443	0.472	0.516	0.477	2.72	3.84	3.82	3.46
Kaol. 20000 ppm S.	0.637	0.625	0.664	0.642	4.35	4.03	4.66	4.346667
Kaol. 25000 ppm S.	1.02	1.04	1.08	1.046667	7.53	7.28	7.51	7.44

LOD—Limit of Detection

Figure 2.2 illustrates the observed relationship between the known sulfur content and measured calcium (Ca) and sulfur (S) content. The data clearly show as sulfur (and therefore gypsum) content increased, the measured calcium and sulfur contents increased. Oddly, the data seem to show a better fit when the helium purge was not used. Regardless, the data look promising for generating a calibration curve to predict sulfur (and therefore gypsum) content based upon either XRF-measured Ca or S concentrations.

**Figure 2.2. XRF Results from Kaolinite Treated with Gypsum.**

To develop calibration curves and determine which measurements achieved the best calibration, researchers regressed the known sulfur concentration on the result from the XRF unit. This regression was performed for all four measurement scenarios shown in Figure 2.2. Table 2.3 presents the results, which show that Ca without the He purge provided the best predictor, as indicated by this calibration exhibiting the lowest standard error and highest R^2 . Figure 2.3 shows this dataset with the calibration curve.

Table 2.3. Calibrating XRF Data to Sulfur Concentration.

Predictor Variable	Slope	Intercept	Standard Error	R^2
Ca w/o He	29,301	-2204	1010	0.989
Ca w/He	27,247	-2028	2230	0.949
S w/o He	4189	-1549	1477	0.978
S w/He	3784	-1454	3049	0.906

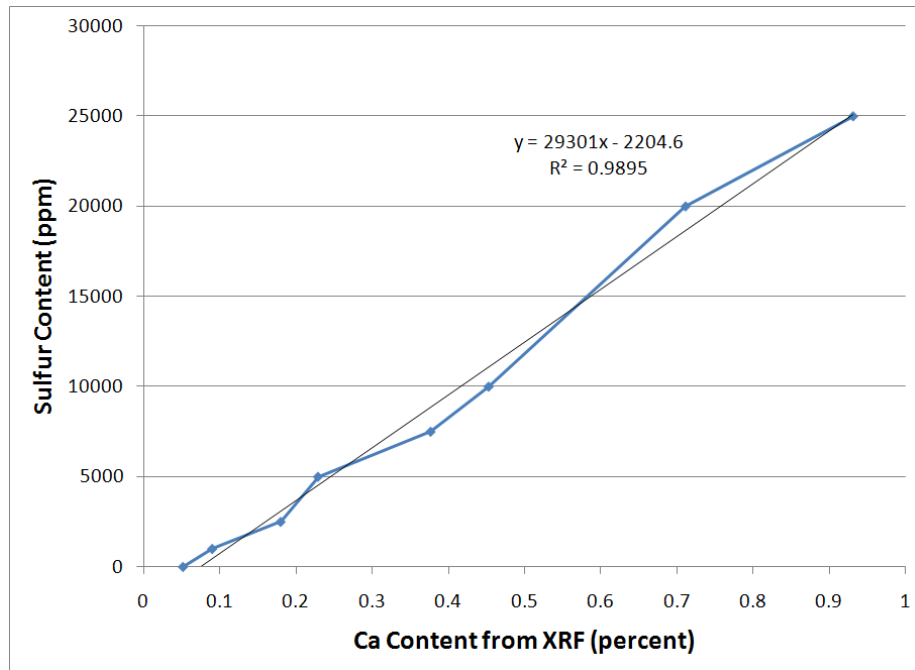


Figure 2.3. Calibrating Sulfur Content to Measured Ca Content.

Unfortunately, even the best calibration relationship had a standard error of approximately 1000 ppm sulfur, which equates to approximately 3000 ppm soluble sulfate. Since the recommended threshold sulfate concentration is 3000 ppm, this relationship may not be adequate to distinguish sulfate-free soils from those requiring more investigation. However, this relationship was developed at sulfate concentrations from 0 to approximately 75,000 ppm. The goal of the XRF unit is to rapidly achieve in the field a go or no-go result (i.e., does the soil have sufficiently high sulfate content to require sampling and further testing). Therefore, researchers also analyzed the data at sulfur concentrations from 0 to only 2500 ppm, which equates to about 7500 ppm sulfate. In this range of concentrations, the data generate a predictive relationship with an R^2

value of 0.987 and a standard error of only 200 ppm sulfur. Figure 2.4 shows this calibration. The standard error of this relationship is low enough that the prediction could reasonably be used to distinguish soils requiring further testing from those that do not require additional testing.

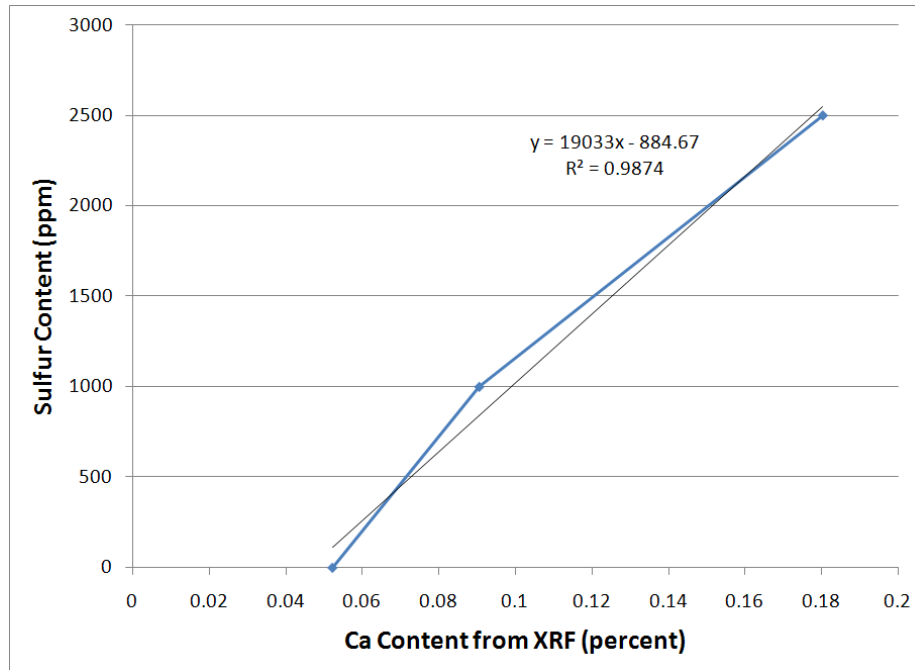


Figure 2.4. Calibrating XRF to Sulfur Concentration.

Experiment #2: Measuring Pyrite Content in Soil without Helium Purge

Since gypsum is the most common, but not the only, sulfur-bearing mineral that may be encountered in the field, researchers also performed experiments to detect pyrite in Kaolinite clay by using the XRF to analyze for iron (Fe) and sulfur. The chemical composition of pyrite is FeS_2 . The results in Figure 2.5 clearly show that the sulfur measurements did not work well. However, the relationship between measured iron content and sulfur concentration proved promising. Figure 2.6 shows how sulfur content could be predicted from measured iron concentration.

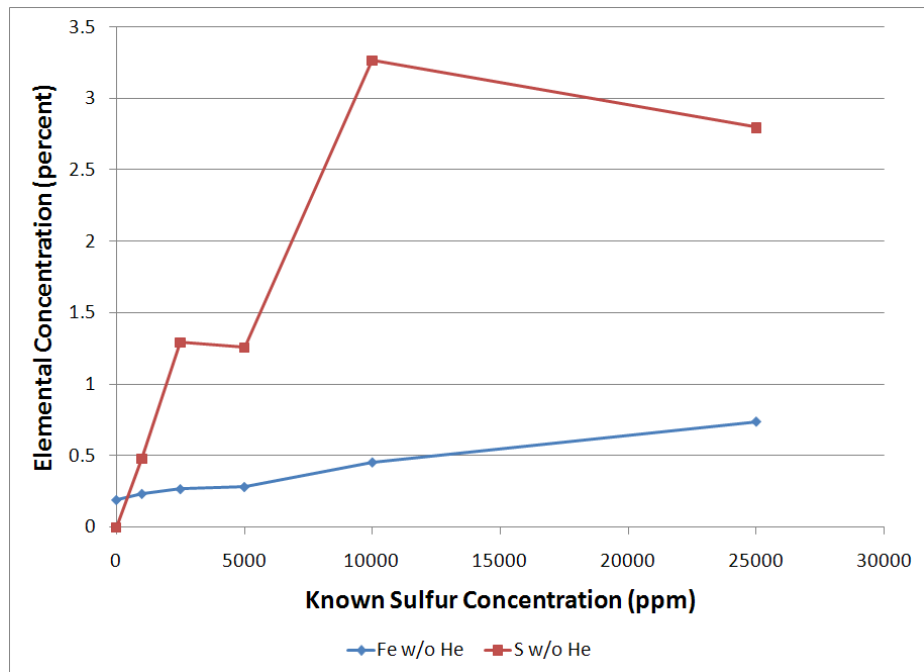


Figure 2.5. XRF Results from Kaolinite Treated with Pyrite.

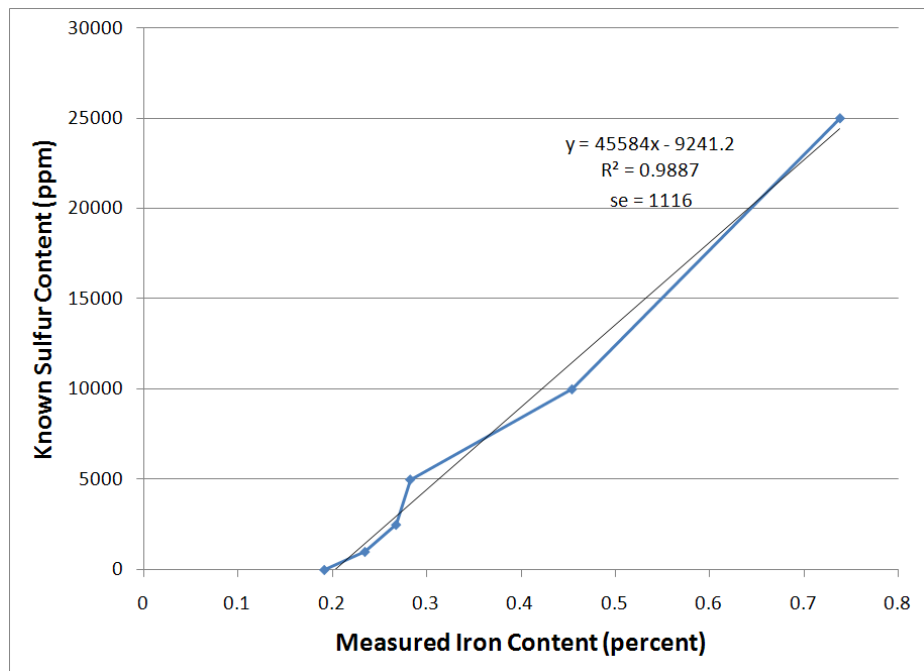


Figure 2.6. Calibrating Iron Content to Sulfur Content from Kaolinite Treated with Pyrite.

As with the soil treated with gypsum, the calibration shown in Figure 2.6 was generated over a large sulfur concentration range and exhibits a standard error sufficiently large to hinder reliable distinction of soils needing further testing from soils that do not. Focusing the analysis window to a narrower range of sulfur contents (from 0 to 2500 ppm) enables generating a relationship with a standard error of 338 ppm sulfur according to the following relationship:

$$S \text{ (ppm)} = -6340 + 32405 \cdot (\text{Fe } \%) \quad R^2 = 0.964; \text{ s.e.} = 338$$

Experiment #3: Measuring Sulfur Content in Natural Soil without Helium Purge

In this experiment, researchers mixed gypsum into a natural soil with a plastic index of 35 to generate mixtures with sulfur content ranging from 0 to 25,000 ppm and collected two sets of three measurements on each treatment level. Table 2.4 presents the results, and Figure 2.7 shows the relationship between known sulfur content and average measured Ca and S content.

Table 2.4. XRF Measurements for Natural Soil Treated with Gypsum.

First Set of Measurements						
	Ca #1	Ca #2	Ca #3	S #1	S #2	S #3
0% S	0.264	0.287	0.264	LOD	LOD	LOD
0.1% S	0.483	0.405	0.406	4.31	4.1	2.63
0.25% S	0.715	0.812	0.697	9.55	10.51	8.28
0.5% S	1.42	1.57	1.3	19.26	21.43	19.8
1.0% S	2.51	2.29	2.09	37.94	36.44	33.72
2.0% S	5.3	4.65	5.29	86.57	76.83	90.11
2.5% S	5.69	5.69	4.3	90.87	93.76	70.68
Second Set of Measurements						
	Ca #1	Ca #2	Ca #3	S #1	S #2	S #3
0% S	0.289	0.286	0.268	LOD	LOD	LOD
0.1% S	0.592	0.440	0.476	6.560	3.940	4.350
0.25% S	0.689	0.849	0.786	8.730	9.540	9.530
0.5% S	1.460	1.410	1.200	22.540	20.490	16.840
1.0% S	2.720	2.310	2.790	40.360	38.030	44.260
2.0% S	5.170	5.070	5.250	84.160	83.250	85.610
2.5% S	4.470	4.470	5.480	72.510	72.440	91.880

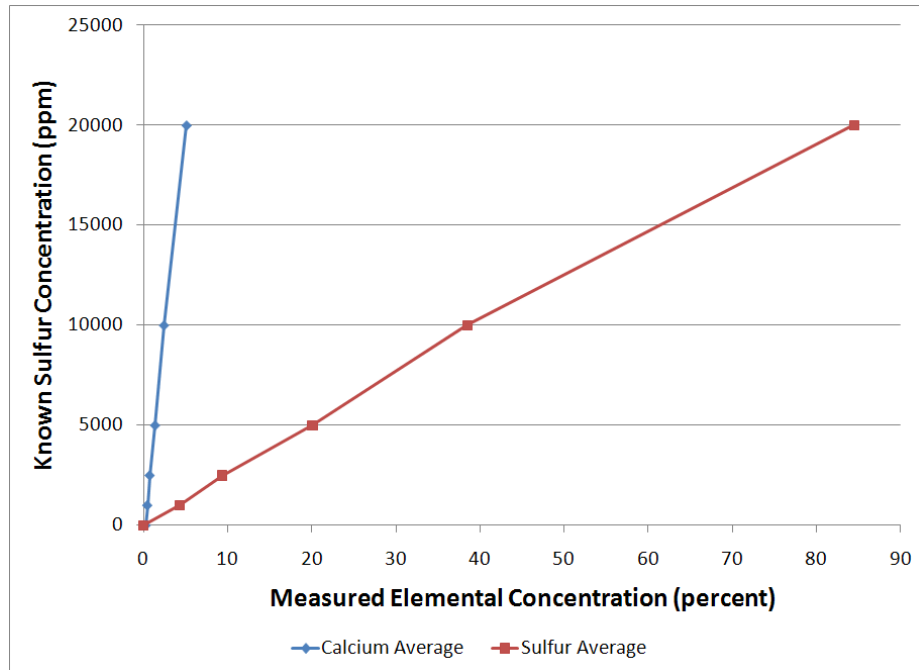


Figure 2.7. Known Sulfur Content versus XRF Measurements with Natural Soil.

With the data presented in Figure 2.7, the calibrations shown in Table 2.5 calibrate the XRF data to predict sulfur content. Again, when calibrated over a large range of sulfur contents, the standard error of the estimate is probably too high to reliably distinguish between soils needing further testing. However, the predictive relationship does not need to be developed through 25,000 ppm sulfur since the threshold soluble sulfate content of interest is equivalent to approximately 1000 ppm sulfur.

Table 2.5. Calibrating XRF Data to Sulfur Concentration with Natural Soil.

Predictor Variable	Slope	Intercept	Standard Error	R ²
Ca	4116	-765	430	0.997
S	238	206	388	0.998

Since calibrating the XRF to such high sulfur concentrations is not necessary for the intended use, researchers focused on developing a predictive relationship for the lower range of sulfur contents. A multivariate relationship predicting sulfur content up to 2500 ppm was obtained as follows:

$$S \text{ (ppm)} = -1343 + 4860 \cdot (\text{Ca}\%) + 17 \cdot (\text{S}\%) \quad R^2 = 1; \text{ s.e.} = 0$$

The best relationship when calibrated up to 5000 ppm S was:

$$S \text{ (ppm)} = 5.54 + 251.3 \cdot (\text{S}\%) \quad R^2 = 0.998; \text{ s.e.} = 124$$

The researchers do not believe calibration above 5000 ppm S (which would be equivalent to approximately 15,000 ppm soluble sulfates) is necessary since the intended purpose is to screen out soils that may have soluble sulfates exceeding only 3000 ppm.

Conclusions and Recommendations

The results presented support the following conclusions:

- The handheld XRF can be used to develop predictive relationships between measured elemental concentration and sulfur (and therefore sulfate or pyrite) content.
- When calibrated over a large sulfur concentration range (up to 25,000 ppm), the standard error of the estimate is too high to reliably distinguish soils with soluble sulfate concentrations above 3000 ppm from those with soluble sulfate concentrations below 3000 ppm.
- By focusing efforts at a lower sulfur concentration range, calibrations were achieved that could serve to check for soils needing sampling and further testing.
- Additional work should be performed focusing on lower sulfur concentration ranges, up to the equivalent of 5000 ppm soluble sulfates.

Based on the results from the initial experiments, and questions generated by the project monitoring committee, the researchers designed an experiment and prepared samples with lower sulfate contents to evaluate the XRF's ability to detect lower sulfate levels.

Experiment #4: XRF Tests of Low Levels of Sulfate in a Manufactured Soil

In this experiment, researchers treated a pure Kaolinite soil with gypsum to obtain sulfur concentrations of 0, 500, 1000, 1500, 2000, and 2500 ppm. This translates to soluble sulfate levels from approximately 0 to 7500 ppm. Researchers then performed single spot measurements to evaluate measurement repeatability, as measured by the standard deviation.

Table 2.6 shows the data collected. Because the concentrations of sulfur were generally below the limits of detection of the XRF device at the sulfate concentrations of interest, the statistical summary of the data presented in Table 2.7 focuses only on the calcium measurements.

Table 2.6. XRF Data.

XRF Kaol + Gypsum (low Concentrations) Repeatability Center of Sample						
	Ca #1	Ca #2	Ca #3	S #1	S #2	S #3
0% S	0.045	0.049	0.049	LOD	LOD	LOD
500 ppm S	0.099	0.093	0.103	1.26	LOD	LOD
1000 ppm S	0.121	0.129	0.123	LOD	LOD	LOD
1500 ppm S	0.111	0.109	0.102	0.933	LOD	LOD
2000 ppm S	0.171	0.172	0.178	1.15	1.54	1.37
2500 ppm S	0.196	0.208	0.202	1.21	LOD	1.35

XRF Kaol + Gypsum (low Concentrations) Repeatability Right of Center of Sample						
	Ca #1	Ca #2	Ca #3	S #1	S #2	S #3
0% S	0.064	0.067	0.063	LOD	LOD	LOD
500 ppm S	0.078	0.086	0.082	LOD	LOD	LOD
1000 ppm S	0.115	0.121	0.125	LOD	LOD	LOD
1500 ppm S	0.098	0.09	0.102	0.948	1.3	0.936
2000 ppm S	0.16	0.159	0.15	LOD	1.3	1.14
2500 ppm S	0.229	0.23	0.239	1.44	1.65	1.12

Table 2.7. Statistical Summary of XRF Calcium Measurements.

Known Sulfur PPM	Center of Sample			Right of Center		
	Avg	Standard Deviation	Coefficient of Variation	Avg	Standard Deviation	Coefficient of Variation
0	0.04767	0.00231	0.04845	0.06467	0.00208	0.03219
500	0.09833	0.00503	0.05119	0.08200	0.00400	0.04878
1000	0.12433	0.00416	0.03349	0.12033	0.00503	0.04183
1500	0.10733	0.00473	0.04403	0.09667	0.00611	0.06321
2000	0.17367	0.00379	0.02180	0.15633	0.00551	0.03523
2500	0.20200	0.00600	0.02970	0.23267	0.00551	0.02367

Results from Data

Data analysis showed the following observations and results:

- Paired t-tests show equivalency between the average, standard deviation, and coefficient variation values among the center of sample and right of center data sets.
- Once beyond 0 ppm S, the observed standard deviations were stationary as the sulfur level increased. The observed pooled standard deviation, which indicates repeatability, was 0.00505 from 500 through 2500 ppm.
- The data produced a predictive relationship with a standard error of 366 ppm sulfur, as Figure 2.8 shows. This translates to a standard error of approximately 1100 ppm soluble sulfate.
- The data point at 1500 ppm known sulfur concentration may be suspect. Without this data point, the standard error of the estimate is 195 ppm sulfur, or approximately 585 ppm sulfate.

- Since the standard error of the estimate in the calibration relationship is already 1100 ppm sulfate, any error from uncertainty in the XRF measurement should be kept as low as possible. This error from the XRF measurement is a function of the desired confidence level (here assumed to be 95 percent), the standard deviation of the measurement (already determined to be 0.00505), and the number of tests performed. Table 2.8 shows performing replicate XRF measurements would limit error from uncertainty in the XRF measurement to at most 320 ppm sulfate.
- Most uncertainty in predicting sulfate content from XRF measurements comes from scatter in the calibration relationship. The standard error of the estimate in the calibration relationship far outweighs the potential error from repeatability uncertainty in the XRF unit.

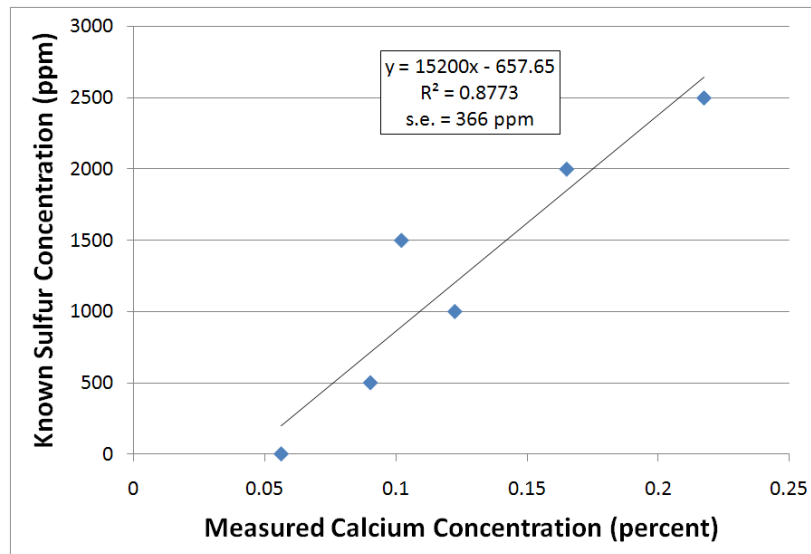


Figure 2.8. Calibrating XRF Measurements to Sulfur Concentration.

Table 2.8. Maximum Error from Varying Number of XRF Tests.

Number of Tests	Maximum Error (% Ca)	Maximum Error (ppm S)*	Maximum Error (ppm SO4)**
1	0.00990	150	451
2	0.00700	106	319
3	0.00571	87	261
5	0.00443	67	202
10	0.00313	48	143
20	0.00221	34	101
30	0.00181	27	82
40	0.00157	24	71
50	0.00140	21	64
100	0.00099	15	45
200	0.00070	11	32

*based on potential error in the percent Ca measurement and the slope factor shown in Figure 2.8.

**3 times the maximum error in ppm S.

Conclusions

Researchers performed regression analysis to investigate the suitability of using the XRF data as a predictor for sulfur, and thus sulfate content. Results showed:

- The limits of detection of the device are not suitable for predicting low concentrations (< ~4500 ppm) of sulfate when using sulfur as the predictor variable.
- The limits of detection of the device for calcium are suitable for predicting low levels of sulfate.

With the data sets collected, the best calibration between the XRF and known sulfur concentration had an R^2 of 0.9 and a standard error of the estimate of 334 ppm sulfur. This standard error would be approximately 1000 ppm sulfate.

Recommendations

The XRF can be used to predict sulfate content in soils with reasonable precision. The relationship should be calibrated to each soil. Although this technology will work for measuring sulfates, the time saved as compared with existing TxDOT conductivity and colorimetric methods, along with the high cost of the XRF device and the fact that the measurement is still spot specific, suggests the XRF device should not be a serious candidate for everyday implementation as a sulfate-screening tool.

SUMMARY OF MULTISPECTRAL ANALYSIS (MSA)

Researchers met with Fares Howari on June 4, 2009, at the Bureau of Economic Geology in Austin to discuss the potential of MSA for detecting sulfate-bearing soils. He informed us that there was great potential for using MSA to detect sulfate as well as other sulfur-bearing minerals (Figure 1.7). As shown in the literature review, Dr. Howari has been using this technology to detect high sulfate soils in desert regions of the world. The lack of data in forested soils is due to vegetation masking the soil; MSA is a surface technique that only detects what is present on the surface. There would still be a need to ground truth the data after collection and processing like any other indirect technique.

After discussions with TxDOT, researchers decided to not pursue this technology any further due to the expense of initial data collection and the need to have a highly trained professional to manipulate and interpret the data.

SUMMARY OF ELECTROMAGNETIC INDUCTION USING EM-38

Researchers did an extensive literature review of electromagnetic induction and determined that the EM-38 device had been used successfully to delineate saline soils in Australia and Africa (Spies and Woodgate, 2005). However, this instrument is not conducive to laboratory testing, so it will be discussed in Task 3 under field testing of existing technologies.

SUMMARY OF ELECTRICAL CONDUCTIVITY USING VERIS 3150

Soil and crop scientists have been using this technology for precision agriculture for about the last 10 years to map the texture and salinity of fields before planting crops. They determine where the variations in soil texture and salinity are high and amend the soil to make it more hospitable for crops (Veris Technologies website, 2009). Like the EM-38, this instrument is not conducive to laboratory testing, so it will also be discussed in Task 3 under field testing.

CHAPTER 3

FIELD TRIAL OF EXISTING TECHNOLOGIES FOR MEASURING ORGANIC MATTER AND SULFATES

INTRODUCTION

To test the usefulness of the different technologies in detecting organic matter and sulfate (gypsum) in the field, researchers constructed a test site at Riverside campus (Figure 3.1). The test pad has the dimensions of 12 ft wide \times 40 ft long \times 1 ft deep. The plasticity index of the soil used is 25. We added gypsum to 4 ft \times 4 ft \times 1 ft pads in concentrations of 3000 and 10,000 ppm sulfate. We also added organic matter to pads in concentrations of 0.5, 1, and 3 percent by weight (Figure 3.2). Some pads contain both gypsum and organic matter, while others contain either organic matter or gypsum.



Figure 3.1. Test Site at Riverside Campus.

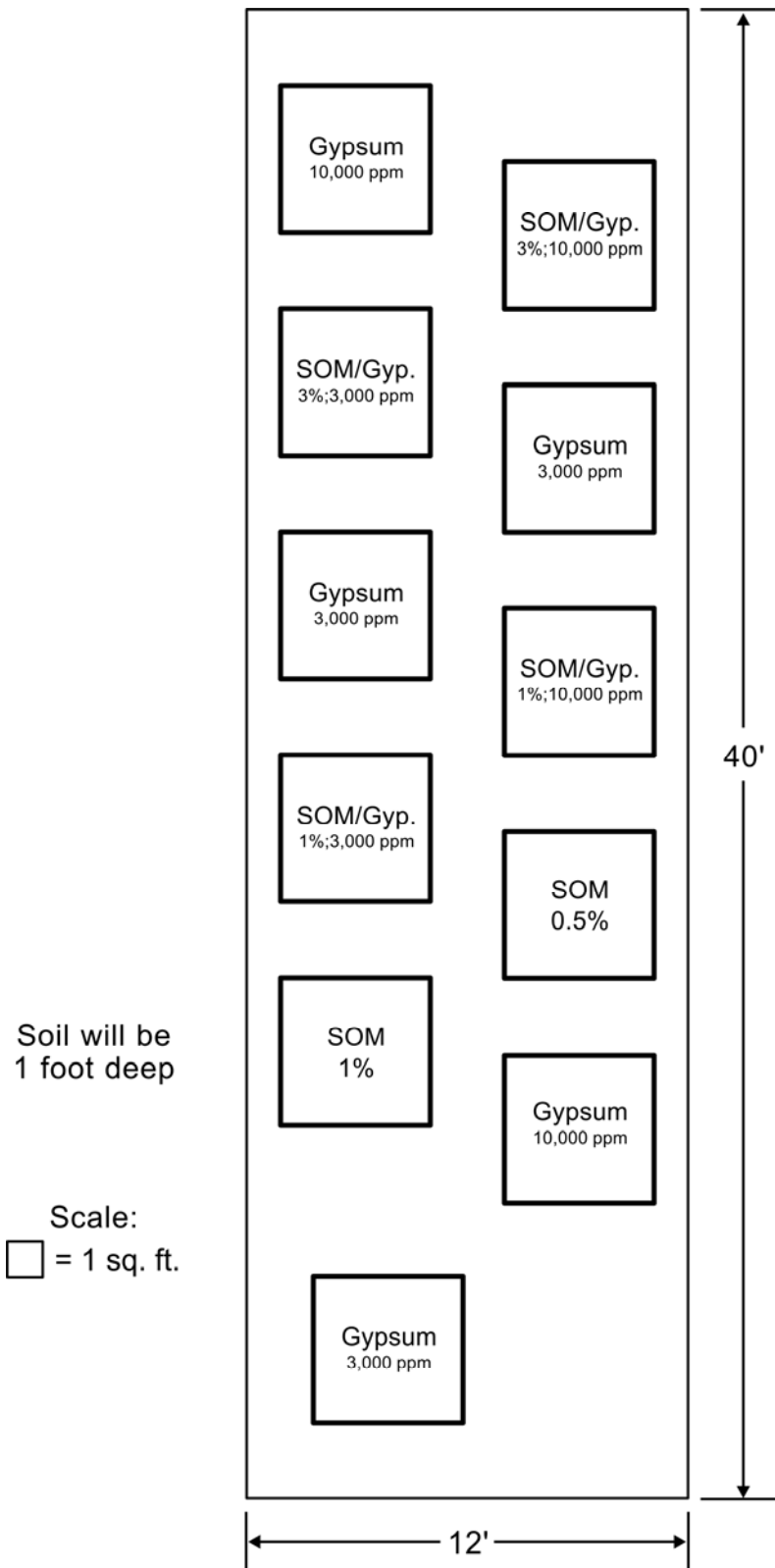


Figure 3.2. Diagram of Gypsum and SOM Contents in Test Pad at Riverside Campus.

TESTING

After consulting with TxDOT, we tested three instruments at the test pad constructed on the Riverside campus. We used the EM-38 DDRT electromagnetic device (Figure 3.3) developed by Geonics Limited, the Niton XL3t portable XRF unit (Figure 3.4) made by Thermofisher, and the Veris 3150 conductivity device (Figure 3.5) produced by Veris Technologies. All tests were run during the summer of 2009, which was an especially hot and dry summer in Texas.



Figure 3.3. EM-38DDRT Used to Measure Electromagnetic Properties of Soil.



Figure 3.4. Portable XRF with Extendapole Used for Measurement in the Field.



Figure 3.5. Veris 3150 Conductivity Device Used to Measure Conductivity of the Soil.

RESULTS

Researchers measured the electrical conductivity of the Riverside test pad using the EM-38DDRT device, which measures in both the vertical and horizontal modes simultaneously. This procedure cuts data collection time in half. Data were collected at heights of 0, 2, and 4 ft above the ground (Figure 3.6) and plotted in Surfer 9 software (Figures 3.7-3.9).



Figure 3.6. EM-38DDRT Data Collection at Heights of 0, 2, and 4 Ft above Ground.

Figure 3.7A shows the known sulfate content of the site as determined when the site was constructed. The middle plot (Figure 3.7B) shows the shallow conductivity (horizontal dipole) measurement using the EM-38DDRT device at a measurement distance of 0 ft off the ground. Figure 3.7C shows data collected in the vertical mode,

which represents an average of conductivities to a depth of approximately 3 ft. As one can see, the data from the shallow and deep readings of the EM-38DDRT are similar, but they do not resemble the known sulfate contents.

Figures 3.8B and 3.8C are data collected with the EM-38DDRT at a measurement distance of 2 ft off the ground. These data are similar to the data collected at 0 ft off the ground: again they correlate poorly with the known sulfate content of the soil (Figure 3.8A). Figures 3.9B and 3.9C are conductivity maps of data collected at a distance of 4 ft off the ground using the EM-38DDRT electromagnetic device. Figure 3.9B data show a trend that more closely correlates with the known sulfate content shown in Figure 3.9A than the other measurements with the EM-38DDRT device. These data are from the horizontal dipole mode collected at 4 ft off the ground, which represents the shallowest penetration into the soil at the test site. The vertical dipole data (Figure 3.9C) are very similar to data collected at heights of 0 and 2 ft off the ground.

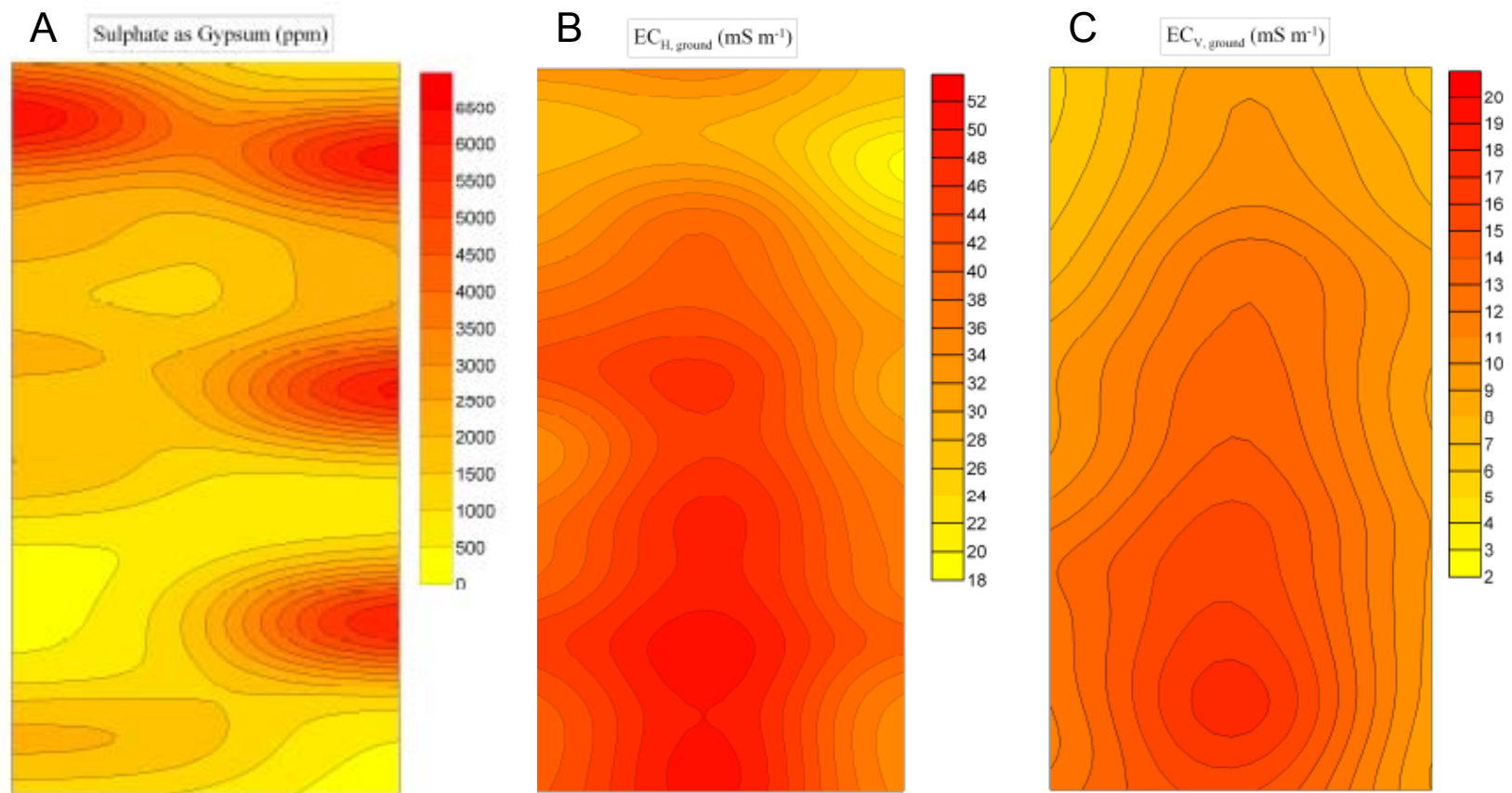


Figure 3.7. Electrical Conductivity Maps for the EM-38DDRT at Ground Level.

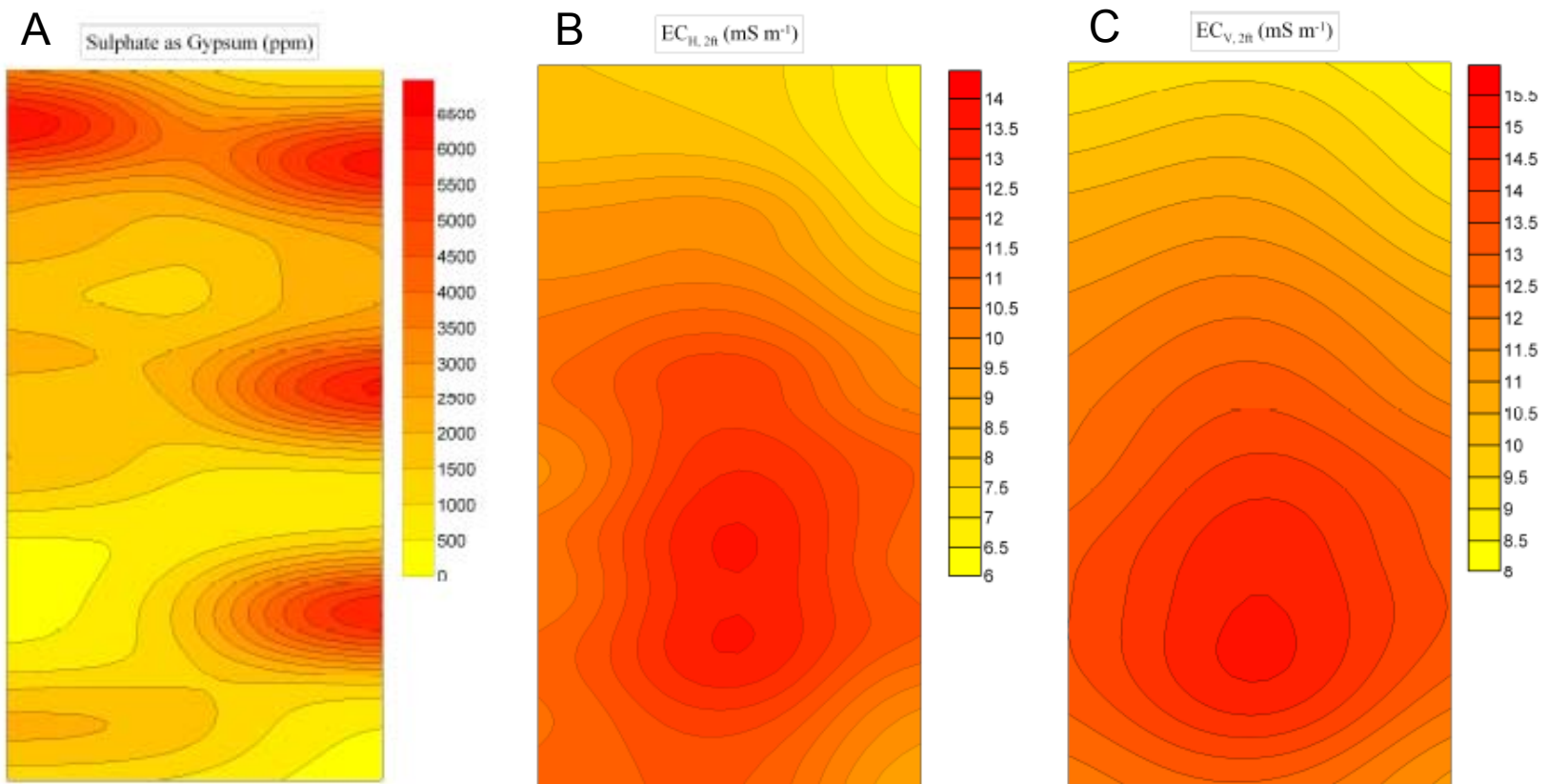


Figure 3.8. Electrical Conductivity Maps for the EM-38DDRT at 2 Ft above Ground Level.

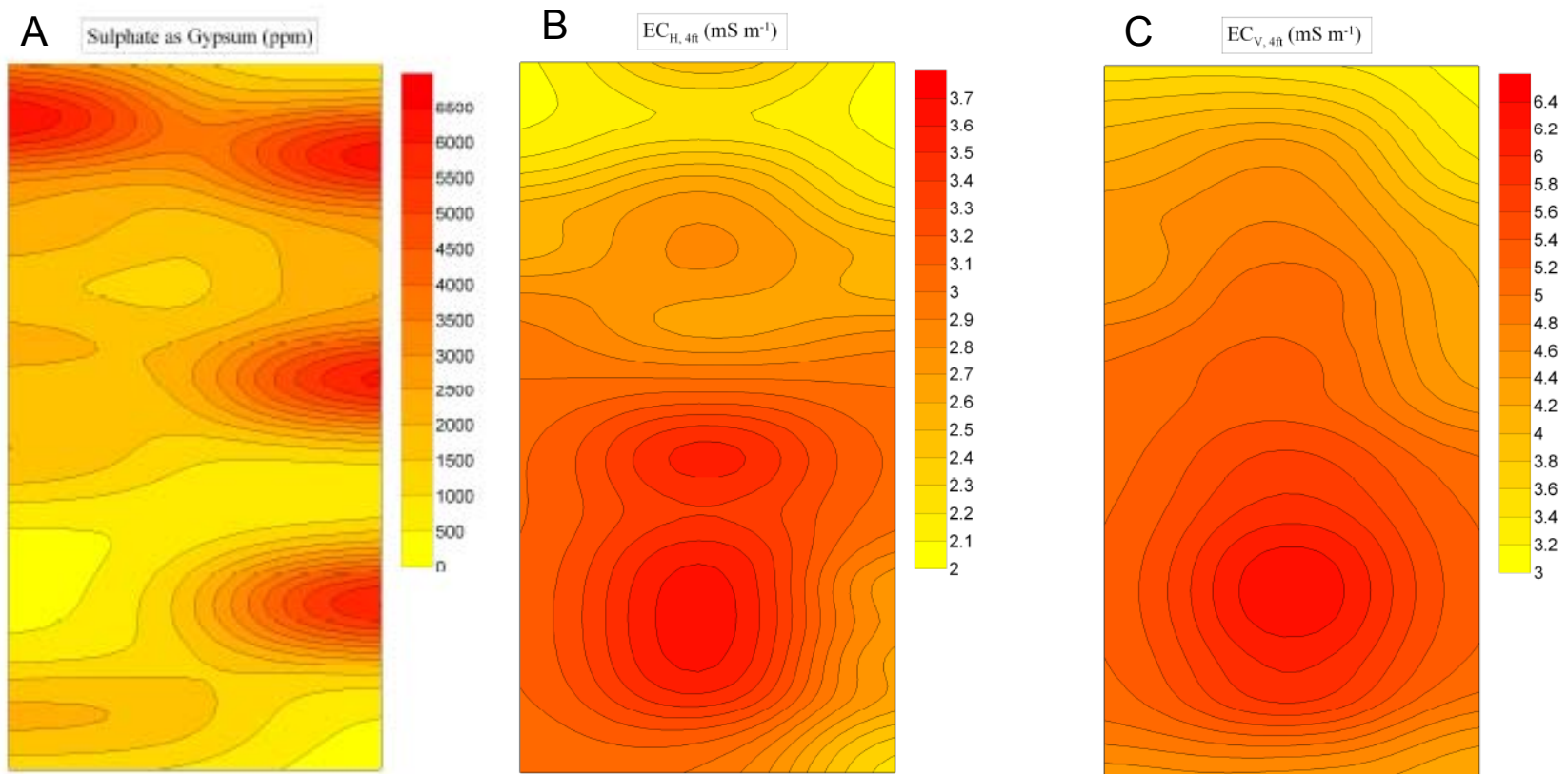


Figure 3.9. Electrical Conductivity Maps for the EM-38DDRT at 4 Ft above the Ground.

We collected XRF data on a 2 ft by 2 ft grid with a total of 95 points where researchers made analyses. Each analysis took 1 minute, and soil samples were collected at each location to measure the sulfate content by TEX-145-E. During the data collection phase, a storm came through and blew the cover off part of the test pad (Figure 3.10), which rearranged the location of the soluble sulfates on the west end of the site.



Figure 3.10. Aftermath of Storm where Sulfates and Organics Were Rearranged.

As shown in Figure 3.11, the sulfate contents measured by the handheld XRF (Figure 3.11B) unit do show some correlation with the known content (Figure 3.11A). There is a discrepancy in the concentration of sulfate determined with the XRF unit and the known; the XRF unit detected up to 36 percent sulfate, but sulfate was added up to 1 percent or 10,000 ppm, as illustrated in Figure 3.11A. There are a number of possible explanations for the discrepancy in results.

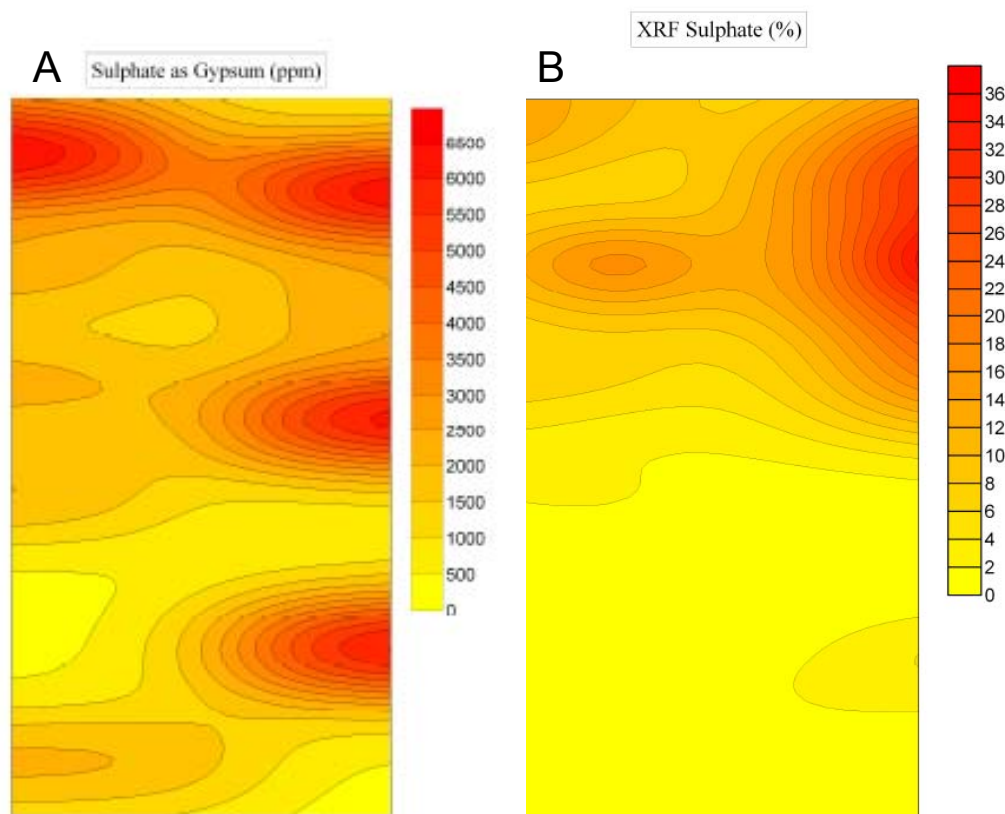


Figure 3.11. (A) Known Sulfate Content versus (B) Sulfate Measured with the Handheld XRF.

Figure 3.5 shows the Veris 3150 conductivity device that we used to measure soil conductivity of the test site at the annex. However, this instrument was so large that it could not readily detect differences in conductivity at such a small scale. We did not plot the data from this device because the GPS system on the unit could not be resolved at such a fine resolution, and there was no correlation with the known sulfate content at the test site.

DISCUSSION

One technique, XRF, is a direct measurement of sulfur content in the medium being analyzed. As stated in the literature review, XRF instruments detect the emission of characteristic X-rays from individual atoms. Using this device, one can identify different elements in the soil. For the instrument we were using, sulfur is at the edge of the detection limit due to a smaller difference in energies required to eject electrons from their orbitals. To overcome this limitation, the manufacturer installed numerous correction factors in the software to boost the signal of sulfur. However, if a medium of a different density is used to calibrate the instrument, one will not be able to obtain absolute values of the sulfur content in the soil. That is one possible explanation for the high sulfur concentrations determined with the handheld XRF device. Second, the sulfur could be concentrated at the surface by efflorescence following the

summer rain storm when the tarp covering the site was blown off and the ground absorbed a lot of water (Figure 3.10). However, there was no visual evidence of efflorescence when the measurements were made with the XRF, and concurrent sampling followed by sulfate measurement in the laboratory did not reveal any high concentrations of sulfate near the surface.

The other devices used to detect variations in sulfate content rely on variations in the conductivity of the soil. As stated in the background, these instruments use an indirect method to estimate sulfate content. Figure 3.12 shows how conductivity varies in different textures or grain sizes of the soil. Clay soils have much higher conductivities than sandy soils. Salinity is the biggest contributor to soil conductivity, which is what researchers rely on to detect the sulfates in the soil. Salinity is caused by ions released into the soil by mineral weathering or saline groundwater intrusion (Sposito, 1989). According to Sposito (1989), the ligands that contribute most to salinity are carbonate, sulfate, and chloride. Solubility of the chloride minerals is very high, so chloride minerals are only expected in the most arid environments. Solubility of the carbonates is much lower than the chlorides and sulfates, so one would expect the salinity in east and central Texas to be dominated by sulfate minerals.

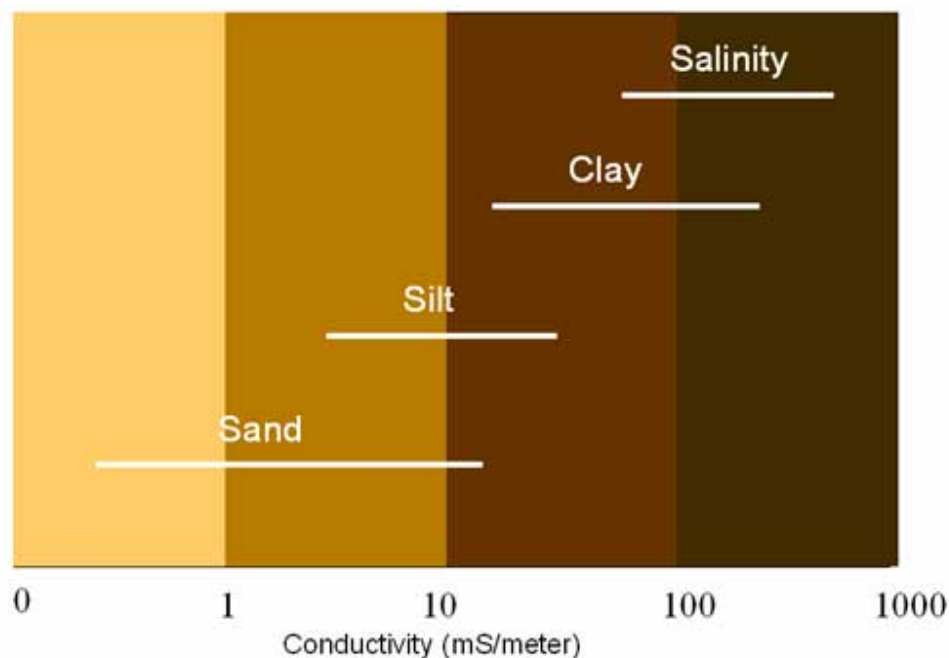


Figure 3.12. Causes of Conductivity Variation in Soils Show Salinity Is Largest Contributor.

<http://www.veristech.com/images/3100/3100cbig.jpg>

As stated before, there are several factors that affect soil conductivity (McNeill, 1980):

- Clay content/clay type (smectite high/kaolinite low).
- Moisture profile with depth.
- Moisture salinity (i.e., dissolved electrolytes in pore water).
- Moisture temperature.

One or more of these factors will control the conductivity of a soil at a particular location. McNeill (1980) states that the most complex of these factors is usually the moisture profile. The moisture profile varies with depth and is a product of the other three factors, along with the pore structure. That is why it is important to collect samples, because conductivity cannot be used to estimate sulfate content in the soil. Where conductivity is high in a soil with a moderate to high moisture content, samples have to be collected and analyzed specifically for sulfate due to the many other causes of high conductivity in the soil.

CONCLUSIONS

Due to the spurious results obtained at the test site constructed at the annex, researchers along with the project monitoring committee decided to test the Veris 3150 and the EM-38DDRT at larger scale sections on real construction projects. We also determined that the handheld XRF instrument was not any better than the current test methods available to TxDOT (test methods Tex-145-E and Tex-146-E). It is still a spot test using a very small sample and is less accurate than current test methods at low sulfate contents as shown in Chapter 2. Researchers proceeded with testing the Veris 3100 and EM-38DDRT on several TxDOT projects throughout the state, which will be discussed in Chapter 5a.

CHAPTER 5A

EVALUATING THE VERIS 3150 AND EM-38 CONDUCTIVITY DEVICES

INTRODUCTION

The research team identified sites in three different geographic regions of Texas that had documented cases of problems caused by the stabilization of the subgrade using calcium-based additives. These sites are located in the Laredo, Ft Worth, and Paris Districts. The problems stem from the presence of sulfate minerals in the soil. The sulfate minerals react with the stabilizer to form an expansive phase called ettringite, which may cause the subgrade to expand after stabilization and cause severe distress in a construction project (Figure 5a.1).



Figure 5a.1. US 67 in the Dallas District Showing Distress due to Sulfate Heave.

The Texas Department of Transportation has a test to measure sulfate in the field (Tex-146-E); however, it is a spot test. The sulfate minerals commonly are present in a limited geographic region (commonly less than 100 ft wide). The current testing protocol calls for testing every 500 ft on a project. If a high sulfate zone lies between these 500 ft intervals, testing

will miss the zone of sulfates; when this occurs, addition of the calcium-based stabilizer will cause a distress like the one observed in Figure 5a.1.

As part of this research project TxDOT wanted to determine if there were any techniques that could provide a continuous measurement of sulfate content. The Veris 3150 (Figure 5a.2A) is used in precision agriculture to measure the salt content of soils to aid in distribution of fertilizers. Conductivity as presented earlier is an indirect measure of sulfate content. Samples still need to be collected to verify observations made with the equipment.

The EM-38 device (Figure 5a.2B) uses electromagnetic induction to measure the conductivity of soil. This device gives similar data to the Veris 3150 except it is portable and takes longer to collect over the same geographic area.

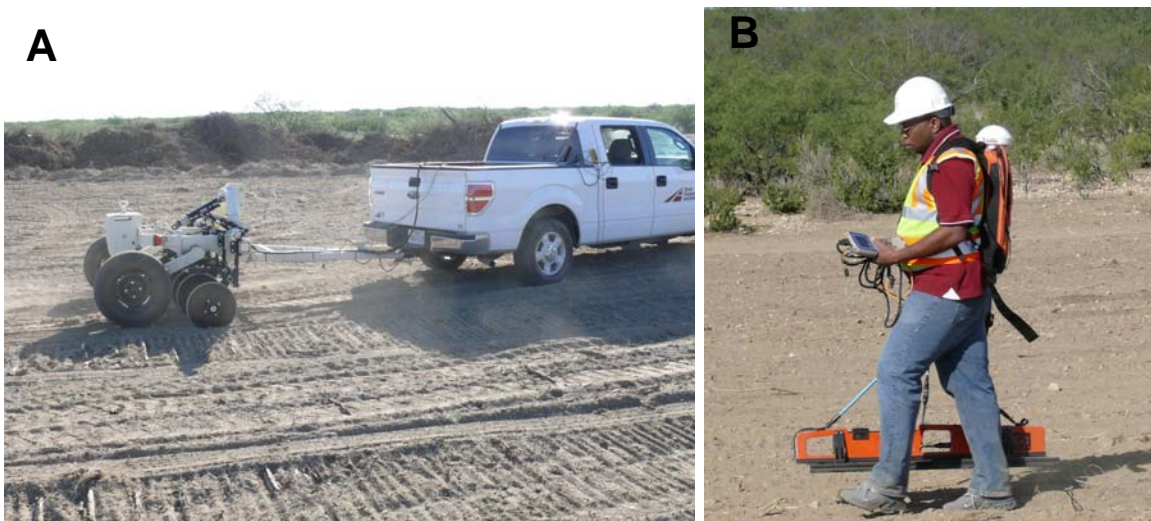


Figure 5a.2. Devices Used in This Study to Measure Conductivity of Subgrade: A) Veris 3150; and B) EM-38DDRT.

Both of these devices can provide continuous coverage of the subgrade over the length of a project and to a depth of approximately 3.5 ft. The data that follow were obtained from several projects that the researchers used to correlate with sulfate content in the soil.

EAGLE PASS LOOP

Researchers went to Eagle Pass, Texas, to collect data using the EM-38DDRT and the Veris 3150 devices. The manufacturer states that the Veris 3150 does not work well if the soil moisture content is less than 10 percent by volume. The soil in Eagle Pass was extremely dry when we collected data (Figure 5a.3). We were testing a new construction site where a loop was being constructed, east of Loop 431, around Eagle Pass to accommodate traffic coming and going from Mexico. The contractor identified this site as having high sulfate contents.



Figure 5a.3. New Construction Site in Eagle Pass, Texas, Shows Dry Conditions.

Eagle Pass Results

We collected data with the Veris 3150 at both shallow (~2 ft) and deep (~4 ft) readings (Figure 5a.4). The yellow dots on the deep data represent GPS locations that show we made four passes along the transect. We also collected data using the EM-38 device over much smaller areas to compare the results with data collected with the Veris instrument (Figure 5a.5). The yellow dots again represent GPS locations that show a much tighter grid pattern because the EM-38DDRT has much less coverage in one swath: 3 ft for the EM-38DDRT versus 12 ft for the Veris 3150.

A comparison of data between the two instruments reveals similar trends. The area near the top of figures 5a.4 and 5a.5 are particularly similar. The other two areas are not as well correlated. Researchers decided to collect more data with the EM-38DDRT at the next location to obtain more of a comparison with the Veris data.

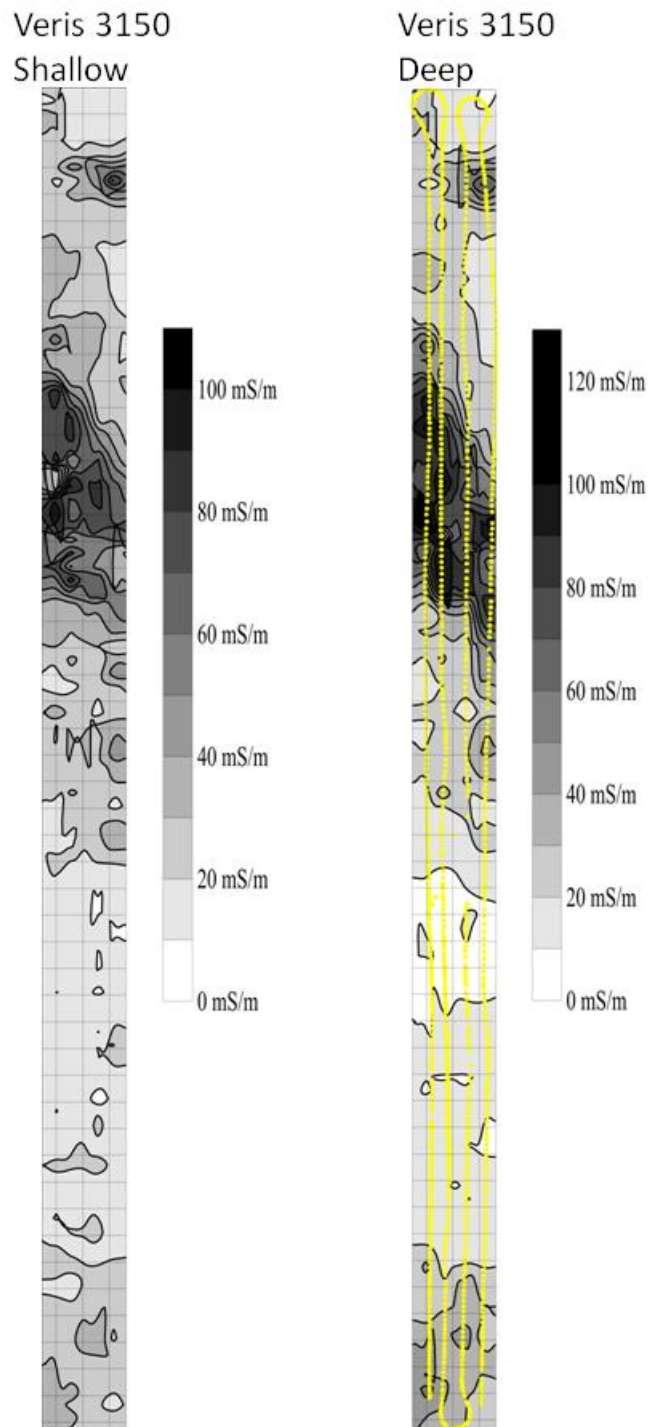
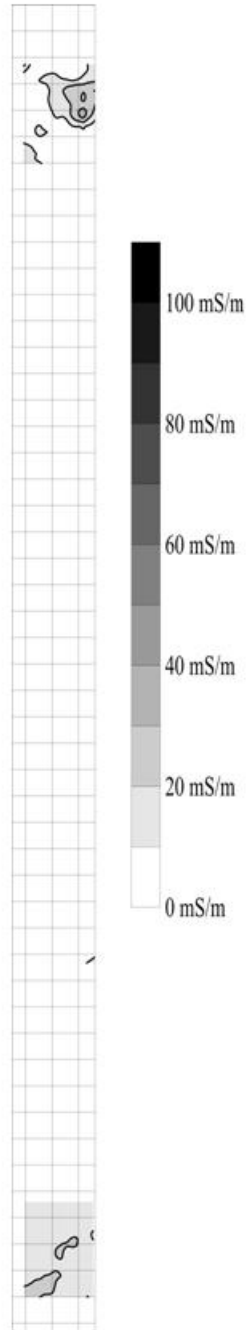


Figure 5a.4. Electrical Conductivity Maps of the Shallow and Deep Sections in Eagle Pass, Texas.

These data were collected using the Veris 3150.

EM-38
Horizontal (QH)



EM-38
Vertical (QV)

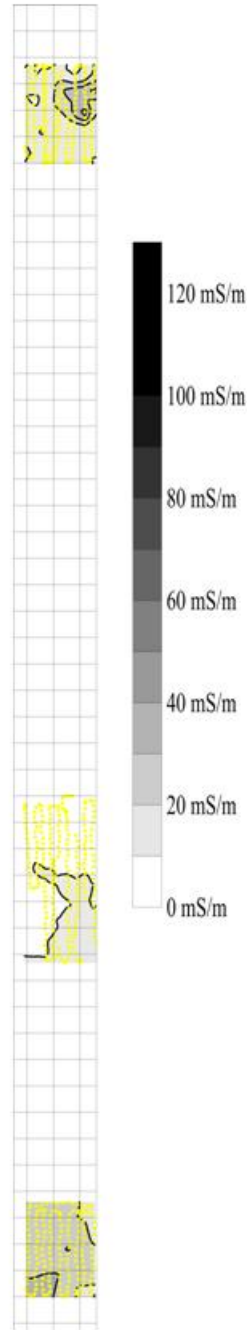


Figure 5a.5. Electromagnetic Induction Data Collected with the EM-38DDRT Device in Horizontal (Shallow) and Vertical (Deep) Modes.

To verify that the measurements using the two devices were detecting sulfates, we collected 10 surface samples and 10 samples 1 ft deep at the site (Table 5a.1) and measured moisture contents, sulfate content, percent organic matter, and plasticity index (PI).

Table 5a.1. Soil Properties of Samples Collected From Eagle Pass.

Sample ID	Sulfate (ppm)	Organics (%)	PI
#1 Surface	140	1.146	15
#1 1 ft Deep	140	1.27	16
#2 Surface	180	1.124	15
#2 1 ft Deep	120	1.299	16
#3 Surface	3660	0.945	17
#3 1 ft Deep	2660	0.873	18
#4 Surface	100	1.341	14
#4 1 ft Deep	100	1.207	12
#5 Surface	ND	2.657	11
#5 1 ft Deep	ND	3.388	15
#6 Surface	ND	3.779	18
#6 1 ft Deep	ND	3.754	24
#7 Surface	ND	2.491	16
#7 1 ft Deep	ND	2.898	21
#8 Surface	ND	2.038	16
#8 1 ft Deep	ND	2.218	14
#9 Surface	ND	1.419	15
#9 1 ft Deep	ND	1.594	16
#10 Surface	ND	1.359	18
#10 1 ft Deep	100	1.638	18

Eagle Pass Discussion

There was not a correlation among any of the parameters measured and the nondestructive testing (i.e., conductivity) data obtained with the Veris 3150 and the EM-38. There are a couple of explanations for the lack of correlation. First, the soil was extremely dry, less than 10 percent moisture. Second, we only collected samples at the surface and at a depth of 1 ft. Both instruments average the data over at least 2 ft for the shallow or horizontal mode and about 4 ft for the deep or vertical mode. The samples we collected were not representative of the area being measured. These data helped us change our strategy for the next site we investigated.

A map showing the gypsum content, over the section where we collected data was plotted using the Web Soil Survey at the natural resources conservation service (NRCS) website (Figure 5a.6). The red areas represent less than 1 percent (10,000 ppm) gypsum; green represents between 1 percent and 5 percent gypsum; and blue represents between 5 and 7 percent gypsum. The shallow and deep conductivity data from the Veris correlate very well with the soils map in spite of the soil being very dry. Where high conductivities are measured (Figure 5a.4), the sulfate content is high (Figure 5a.6), and low conductivities generally correlate with low sulfates.

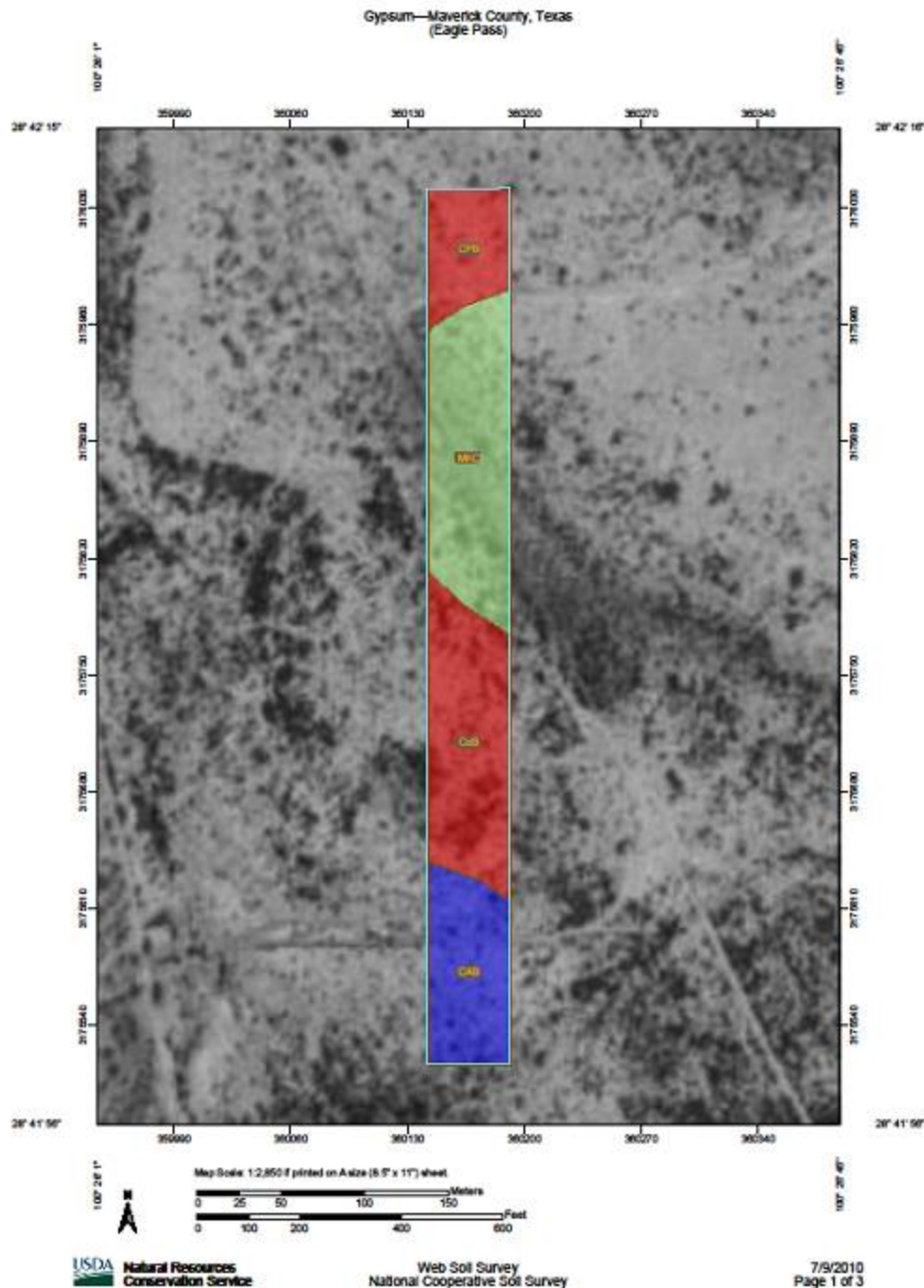


Figure 5a.6. Soils Map Showing Gypsum Content from the NRCS Website of the Area Analyzed with the Veris 3150.

GRAPEVINE, TEXAS

After collecting data in far south Texas where the soil is very dry, researchers had an opportunity to evaluate two projects in Grapevine, Texas. The soil was very wet and very plastic

at the Grapevine sites. The first project was dubbed the teardrop and is at the intersection of SH 114 and SH 121 (Figure 5a.7). Researchers used both the Veris 3150 and EM-39DDRT to collect conductivity data. Based on conductivity changes recorded with the Veris 3150 and EM-38DDRT instruments, we picked sites to collect soil samples. We collected soil samples in 1 ft increments to a depth of 4 ft in 10 areas that represented large variations in conductivity. Samples were taken using an auger on a TxDOT drilling rig (Figure 5a.8).

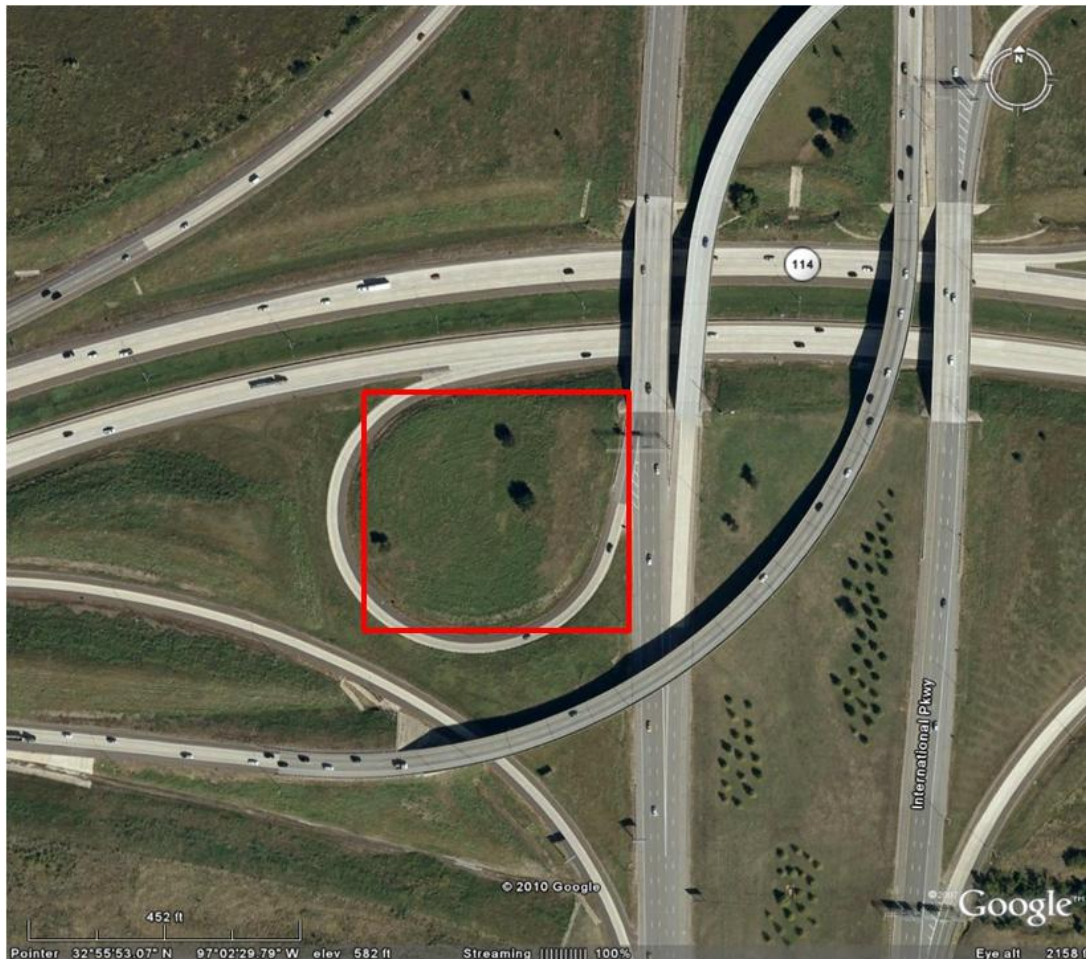


Figure 5a.7. Image Taken from Google Earth Showing the Location (Area inside Red Square) of Our First Test Site in Grapevine.



Figure 5a.8. Auger Used to Collect Soil Samples Every 1 Ft to a Depth of 4 Ft in Areas of Contrasting Conductivity.

The data from the Veris 3150 were plotted with ArcGIS software using a smart quantile classification that grouped the data into 10 classifications and plotted it (Figure 5a.9 bottom). The black lines at the top of Figure 5a.9 represent the GPS coordinates and show the trace of the Veris 3150 as data were collected. The bottom left figure shows the shallow (~2 ft) conductivity readings collected by the Veris 3150, and the bottom right figure shows the deep (~4 ft) conductivity readings. Two items to note are higher conductivity readings are present in areas where water was standing in puddles and adjacent to the drainage ditch, and the deep conductivities are higher than the shallow conductivities.

Engineering properties of the soils taken from Grapevine site 1 are shown in Table 5a.2. The first number in the sample names (11-1, 11-2, etc.) correlates with the sample numbers shown on the conductivity map. The second number represents the depth at which the sample was collected. For example, 11-1 is from point 11 on the conductivity map, and it is the sample taken from a depth of 1 ft. As shown in Table 5a.2, the moisture contents (range from 24 to over 30 percent) are very high in this site.

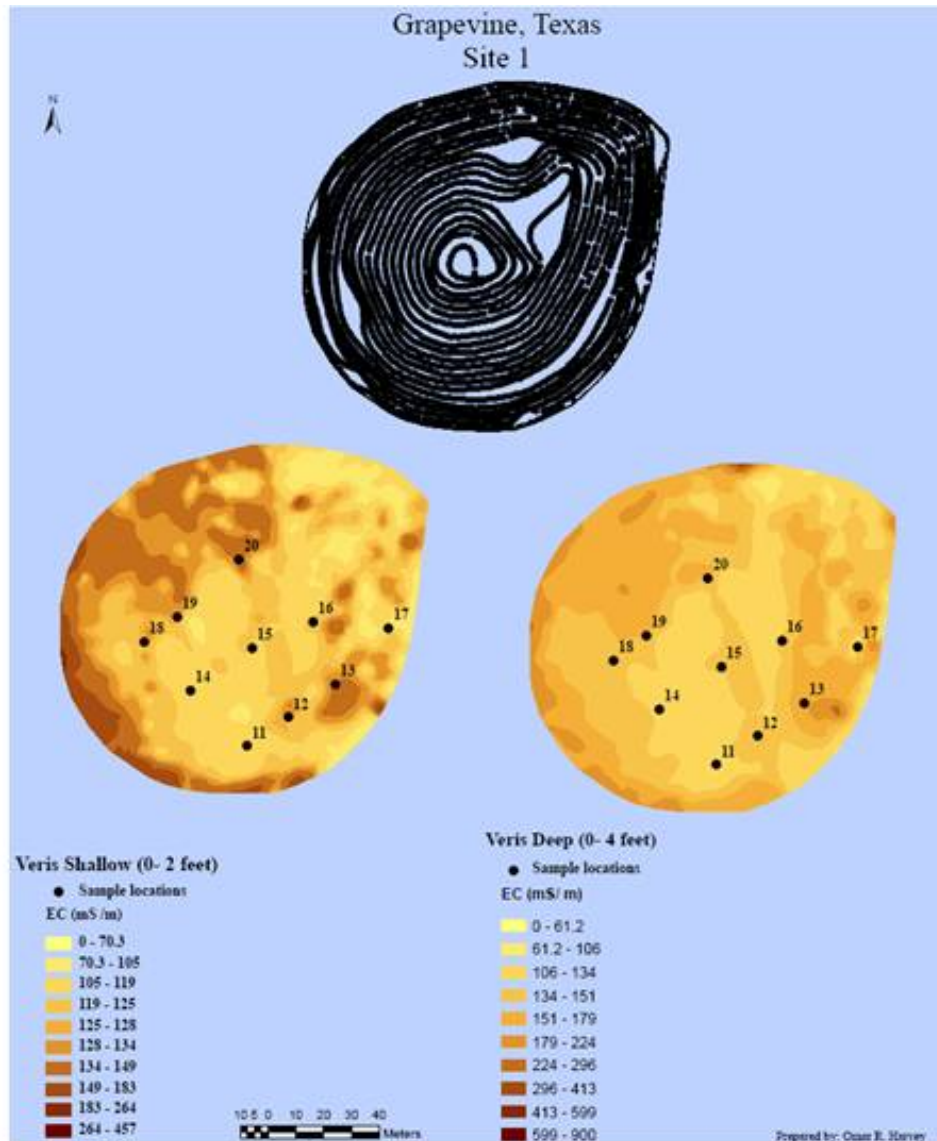


Figure 5a.9. Conductivity Data for Site 1 Collected with the Veris 3150.

Black dots labeled 11 through 20 are locations of soil samples.

The Plasticity index of these soil samples is very high, and these soils all classify as A-7-6 using the American Association of State Highway and Transportation Officials (AASHTO) designation M145. The organic matter contents do not necessarily show a decreasing trend with depth because the soil is all fill material in this section. Also, the soil maps for this area are not shown because they do not correlate with the Veris 3150 conductivity data. The lack of correlation between soils maps and conductivity data is probably because this is all fill material from the construction of the interchange.

Table 5a.2. Engineering Properties of Soil Samples from Grapevine Site 1 (Teardrop Site).

Sample Name	MC %	Sulfate (ppm)	OM (%)	PI	(%) -200
11--1	24.5	Low	0.9	34	
11--2	26.7	100	0.5		
11--3	28.0	320	0.3		
11--4	28.1	4240	0.7	62	99.5
12--1	25.6	100	2.0	31	
12--2	27.1	169	0.4		
12--3	27.7	240	0.6		
12--4	27.5	480	0.7	48	
13--1	26.9	Low	2.2	38	
13--2	25.6	140	0.7		
13--3	25.6	2360	0.4		
13--4	26.7	3600	0.3	43	
14--1	27.2	Low	2.7	28	
14--2	27.9	100	0.5		98.6
14--3	27.8	380	0.4		
14--4	27.2	9600 (8960)	0.7	60	
15--1	27.3	100	2.9	34	
15--2	27.6	380	0.5		
15--3	2.2	5600	0.7		
15--4	27.2	21,760	0.6	42	97.4
16--1	27.4	500	0.5	51	
16--2	32.2	440	0.8		
16--3	28.0	400	0.6		
16--4	28.2	280	0.4	55	
17--1	27.9	Low	1.7	34	
17--2	28.2	100	1.7		
17--3	26.2	Low	1.6		
17--4	24.2	120	2.4	33	
18--1	28.2	2400	2.7	33	
18--2	28.9	120	0.7		
18--3	29.2	200	0.6		
18--4	33.9	1580	0.6	51	
19--1	27.4	920	3.6	34	
19--2	27.5	540	0.9		
19--3	27.4	2920	1.3		
19--4	29.3	9840	0.4	55	99.1
20--1	29.3	100	1.6	34	
20--2	26.9	620	1.0		
20--3	27.1	19,200	0.5		
20--4	24.8	18,560	0.4	50	

The second site in Grapevine is located adjacent to SH 121 eastbound and the frontage road (Figure 5a.10). This site is also a fill area with steep sides (Figure 5a.11) for a bridge approach that goes over Northwest Parkway. The area outlined in red in Figure 5a.10 represents the area where the Veris 3150 was used to measure the conductivity and generate conductivity maps.

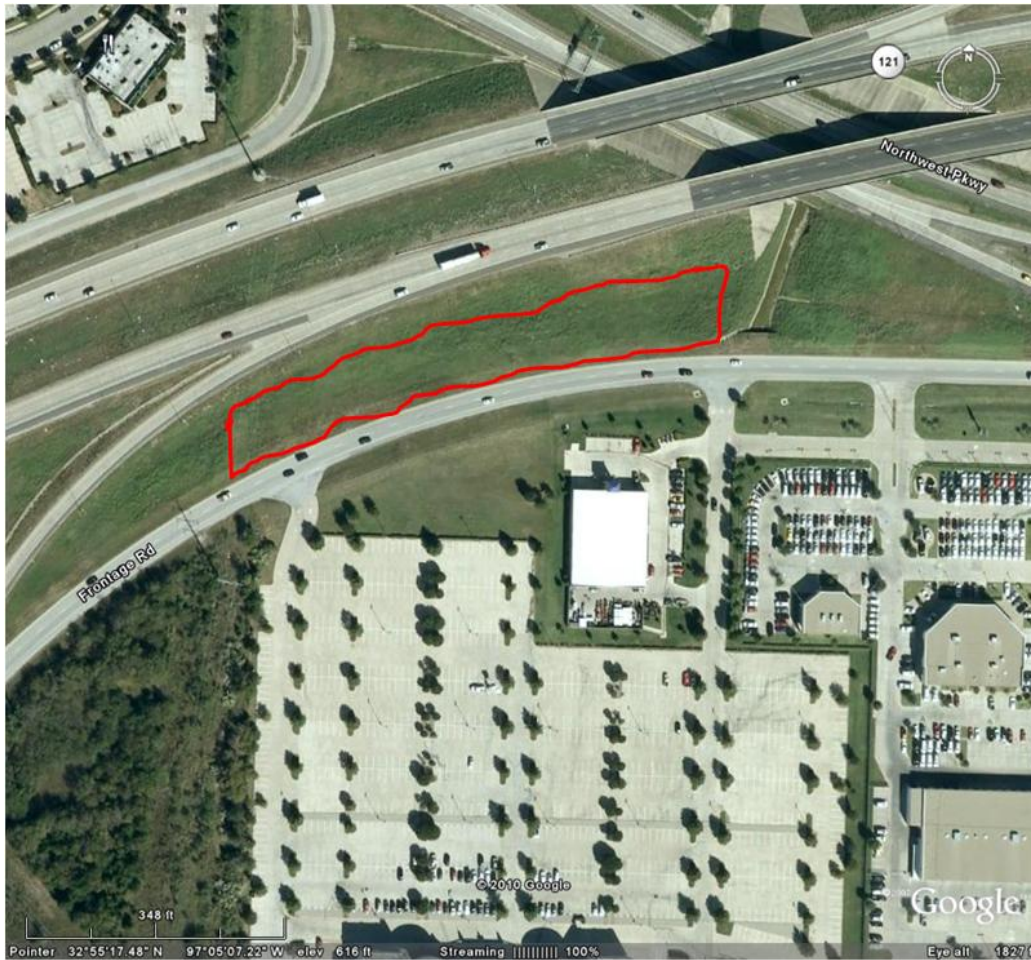


Figure 5a.10. Section Adjacent to Access Ramp for Eastbound SH 121 is the Second Location in Grapevine.

The area mapped is outlined in red.

Figure 5a.11 shows a tractor pulling the Veris 3150 and collecting conductivity data. The embankment gets steeper on the north side and as one approaches the bridge, which prevented researchers from taking samples in certain areas because the rig could not get up the steep slope. As shown in Figure 5a.12, all of the boreholes where samples were collected are on the southerly side of the site.



Figure 5a.11. Grapevine Site 2 Showing Embankment with Steep Slope that Prevented Collecting Soil Samples Using the Drilling Rig from Some Areas.

The conductivity of the shallow and deep measurements shows very similar trends with conductivity being much higher toward the east end of the project. Unfortunately, the sulfate contents for this site are very low (Table 5a.3) with the highest sulfate content being 500 ppm. A comparison of the conductivities measured at this site and the teardrop site show much higher conductivities for the teardrop site. The higher conductivities at the teardrop site may be attributed to higher sulfates, higher moisture contents, and more clay in the soils (Table 5a.2).

Table 5a.3 shows the engineering properties of the soils taken from Grapevine site 2. The first number in the sample names (1-1, 1-2, etc.) correlates with the sample numbers shown on the conductivity map. The second number represents the depth at which the sample was collected. For example, 1-1 is from point 1 on the conductivity map, and it is the sample taken from a depth of 1 ft. As shown in Table 5a.3, the moisture contents (range from 13.5 to 23.3 percent) sulfate content, and plasticity are lower than from Site 1.

According to the AASHTO Designation M145, the soils for site 2 would classify as A-6, which is a clayey soil but not as clay-rich as the soil from site 1, which classifies as A-7-6; this could help explain the lower moisture content for Site 2.

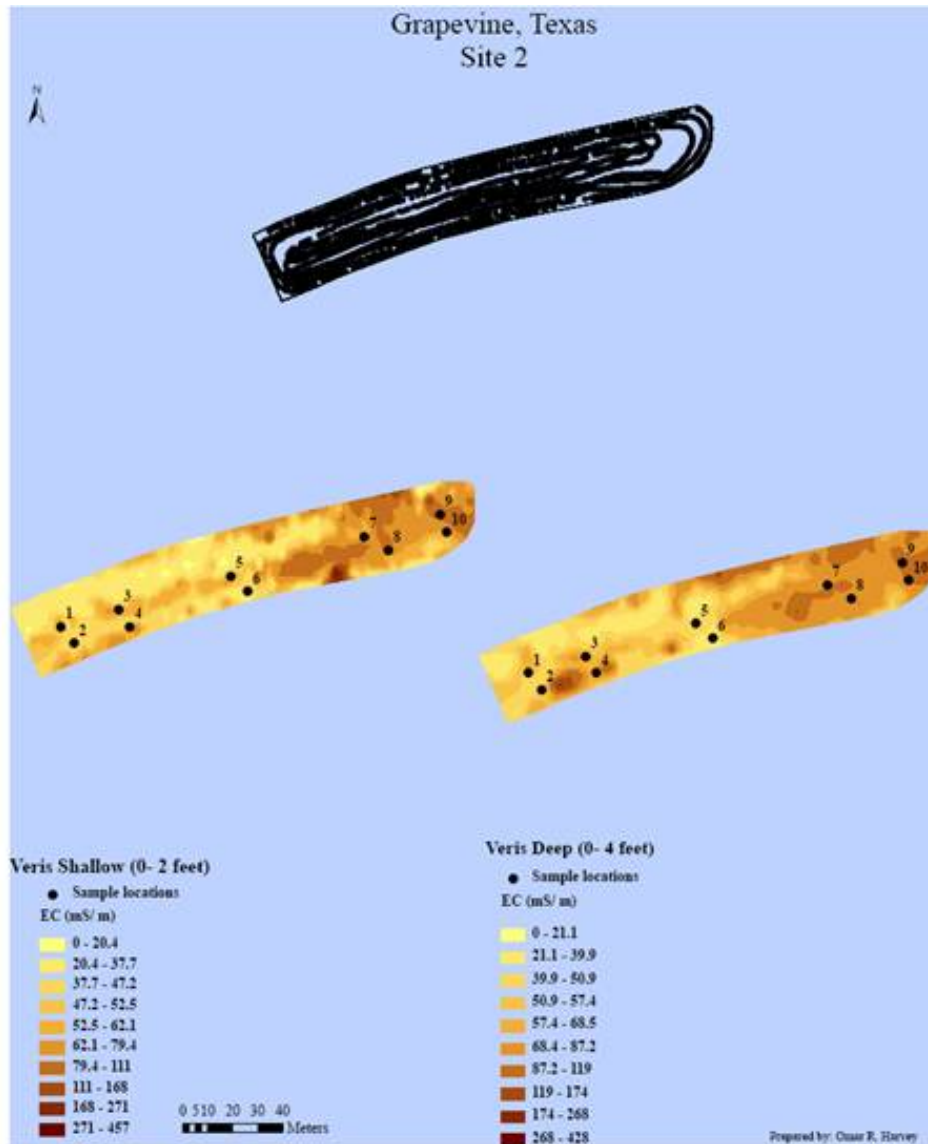


Figure 5a.12. Conductivity Data For Site 2 Collected with the Veris 3150.

Black Dots Labeled 1 through 10 Are Locations of Soil Samples. Shallow Readings Are on Left and Deep Readings Are on Right Side of Figure.

A comparison of the data does not lend itself to easy interpretation. There is not a straight-forward correlation between the conductivity (Figure 5a.12) and a single soil property (Table 5a.3). As stated previously, high conductivity in soil is caused by increased clay content, increased moisture content, and dissolved ions in solution, among others. That is why one must always collect samples where conductivities vary to determine what is causing the variability in conductivity.

Table 5a.3. Engineering Properties of Soil Samples from Grapevine Site 2.

Sample Name	MC %	Sulfate (ppm)	OM (%)	PI	(%) -200
1--1	18.0	Low	1.4	22	55.1
1--2	18.0	100	0.8		
1--3	18.2	Low	0.8		
1--4	17.5	160	0.3	21	
2--1	16.1	Low	1.6	15	
2--2	17.7	Low	1.8		
2--3	17.5	140	1.7		
2--4	20.6	220	1.2	32	
3--1	14.5	Low	0.9	14	
3--2	12.3	Low	1.0		
3--3	15.4	120	0.8		
3--4	16.5	Low	0.7	18	
4--1	23.3	Low	0.7	31	
4--2	13.5	Low	1.7		
4--3	16.8	Low	1.7		
4--4	17.1	Low	1.3	23	
5--1	17.1	Low	1.5	17	64.6
5--2	17.7	Low	1.6		
5--3	15.6	Low	1.8		
5--4	15.5	100	2.1	15	
6--1	17.5	100	1.3	22	
6--2	14.4	Low	1.7		
6--3	14.3	Low	1.6		
6--4	16.4	Low	2.0	20	
7--1	17.4	Low	1.6	23	54.0
7--2	20.7	Low	1.5		
7--3	17.9	100	1.2		
7--4	16.7	160	1.5	24	
8--1	15.2	Low	1.8	15	
8--2	14.6	Low	1.5		
8--3	13.5	Low	1.1		
8--4	14.0	120	0.6	11	
9--1	14.2	Low	0.7	12	
9--2	18.0	100	1.0		
9--3	18.1	100	1.4		
9--4	21.8	500	0.3	21	
10--1	16.9	Low	1.3	23	
10--2	18.4	100	1.1		
10--3	18.2	120	0.7		
10--4	17.7	160	0.7	20	

SHERMAN, TEXAS

The third location that researchers visited is in the Paris District where an existing road is being widened. The aerial view shown in Figure 5a.13 was taken from Google Maps; it shows a small area (outlined in red) where we made the initial data collection. The site is about 5 miles east of the city of Sherman and is adjacent to US 82; Choctaw Creek is the eastern limit of the site.



Figure 5a.13. Image Taken from Google Earth Showing the Location (Area inside Red Rectangle) of Our First Test Site in Sherman.

The section is about 800 ft long and consists of fill material in the drainage basin of Choctaw Creek (Figure 5a.14). Researchers made four passes along the right-of-way with the Veris 3150 and also made four passes with the EM-38DDRT to compare results between the instruments. After processing the Veris data, we picked 20 locations where the conductivity varied to collect soil samples at 1 ft depth intervals to a total depth of 4 ft using an auger connected to a Bobcat (Figure 5a.15).



Figure 5a.14. US 82 Site 1, East of Sherman Shows Fill Material in Choctaw Creek Drainage Basin.



Figure 5a.15. Bobcat with 9-Inch Auger Used to Collect Soil Samples to a Depth of 4 Ft.

The elongate rectangle at the top of Figure 5a.16 with four black lines shows the GPS readings taken and the four passes made by the Veris 3150 to collect data. The two color-coded plots in Figure 5a.16 show contours of the conductivity data for the shallow (left side) and deep (right side) readings taken from the Veris 3150 device. The locations for the 20 auger holes are plotted on this figure as well. One can see that the soil samples are collected from a range of conductivities. Note that the conductivity readings for the deep data are higher than the shallow data.

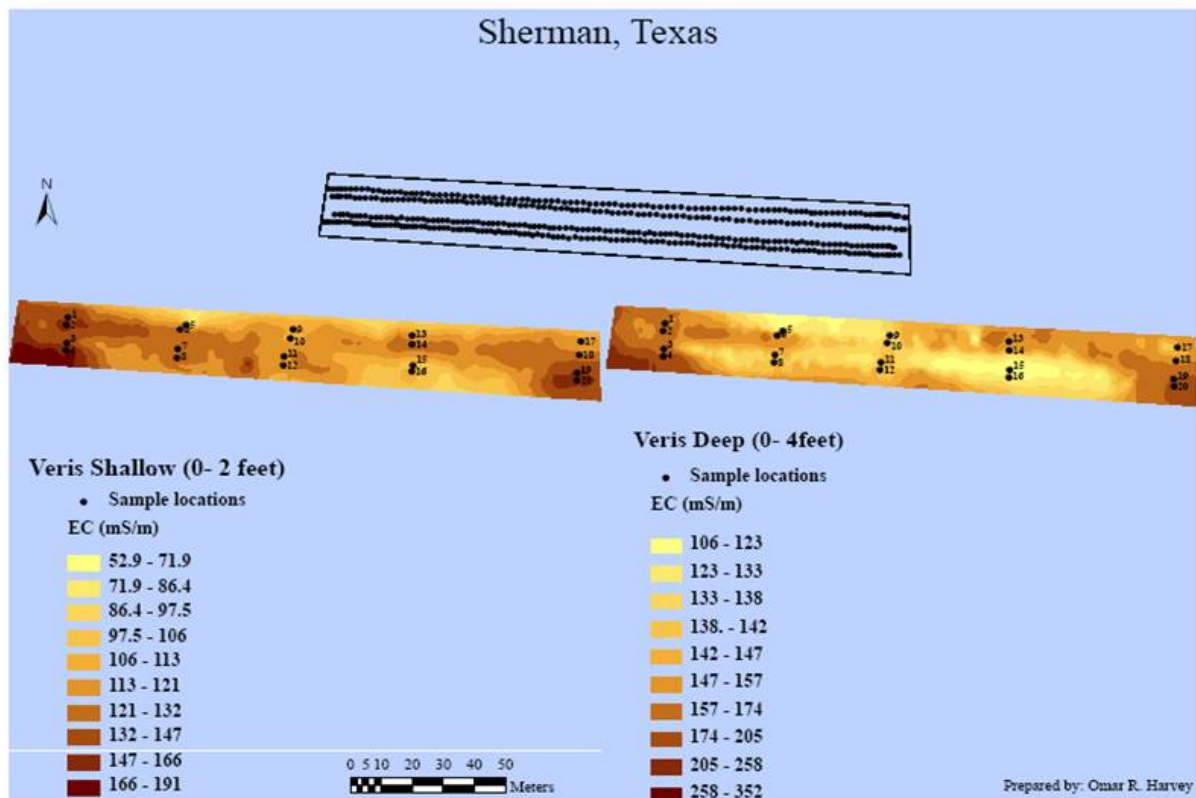


Figure 5a.16. Veris Conductivity Data for US 82 Site 1 East of Sherman.

Omar Harvey collected the EM-38DDRT data by walking and carrying the instrument at a distance of approximately 6 inches above the ground surface (Figure 5a.17). This figure follows the same format as the Veris 3150 data plotted in Figure 5a.16. The absolute conductivity numbers measured with the EM-38 are lower than the conductivity measured with the Veris 3150, but the same trends are observed with higher conductivities on the western and eastern ends of the project.

An analysis of the data generated by the Veris 3150 and the samples collected from the three sites just described is presented in the section that follows. Since these data are from natural soils and a number of variables contribute to the soil conductivity, a multivariate analysis was done to see what correlations between data could be made.

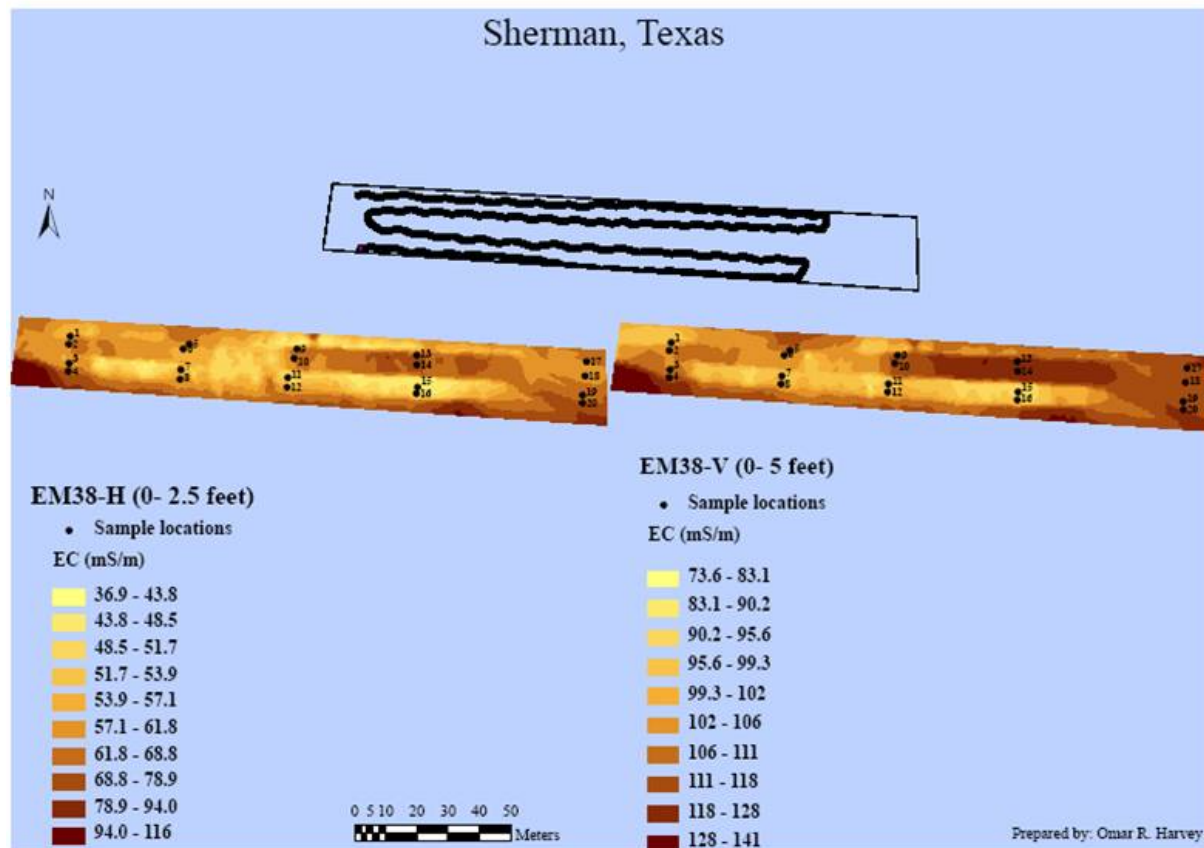


Figure 5a.17. EM-38DDRT Electromagnetic Data for US 82 Site 1 East of Sherman.

Summary of Veris EC Data Analysis

TTI collected Veris electrical conductivity data, along with spot soil samples for moisture content determination, plasticity index, and organic content at two test sites in Grapevine and one site in Sherman. The purpose of these data collection activities was to evaluate if the Veris EC can be related to soil sulfate content. This section presents key results and findings illustrating how the Veris EC device can be used to detect geospatial zones of high sulfate contents. The analysis of data show that a linear model can be used to predict the natural log of the sulfate content based on the Veris EC measurements. Although this model can be developed based upon point-specific data, a lower standard error of the estimate and improved fit can be obtained by first performing a geospatial grouping of the point sample locations in the field and then averaging data from within each geospatial population zone for input into the model development. This geospatial grouping approach improves the model because analyses of the data show that other soil parameters, in addition to sulfate content, influence the Veris EC measurement. However, conditional correlation analyses show that, when these other soil parameters are given, the Veris EC correlates directly to the sulfate content (expressed as the natural log of the sulfate concentration) without consideration of other soil parameters. The

geospatial grouping approach should group data into geospatial zones where variations in other soil parameters (such as moisture content and plasticity) are likely to be minimized, resulting in a model with lower standard error and better fit.

Multivariate Analysis of Raw Data

To begin the data review, researchers employed a cross-section multivariate analysis using raw data from all test sites. This analysis showed that the Veris EC could be successfully predicted with a linear multivariate regression. Table 5a.4 shows the data, and Table 5a.5 shows the regression output. Factors considered influencing the EC include:

- Sulfate content (ppm).
- Organic content (percent).
- Soil water content (percent gravimetric).
- Soil plasticity index (percent; used as a surrogate for clay content).

Table 5a.4. Veris EC, Sulfate, Organic Content, Water Content, and Plasticity Index for Shallow Measurements.

	VerisEC SH	SavgSH	OCavgSH	WCavgSH	PI1
Fort Worth #2	55.63	50	1.15	18.014493	22
	41.83	0	1.71	16.880999	15
	64.83	0	0.95	13.416359	14
	50.46	0	1.22	18.393091	31
	53.93	0	1.55	17.421213	17
	21.87	50	1.48	15.92051	22
	64.04	0	1.55	19.046772	23
	80.43	0	1.65	14.944242	15
	84.46	50	0.82	16.081538	12
	61.26	50	1.18	17.615592	23
Fort Worth #1	114.19	50	0.70	25.616407	34
	125.56	134.5	1.23	26.37817	31
	134.35	70	1.46	26.271879	38
	109.49	50	1.59	27.566031	28
	121.44	240	1.70	27.453916	34
	120.75	470	0.62	29.804549	51
	114.9	50	1.68	28.0817	34
	125.36	1260	1.70	28.54994	33
	129.4	730	2.24	27.433235	34
	136.95	360	1.30	28.089765	34
Sherman	139.41	440	0.65	18.334908	27
	151.99	1900	0.79	18.605957	25
	142.3	440	0.43	17.695283	25
	158.08	2460	0.37	19.857316	38
	104.41	460	0.38	17.752566	27
	123.26	650	0.48	17.838856	26
	127.87	500	0.34	17.455551	23
	128.08	4140	0.74	19.624381	28
	106.96	470	0.41	18.802719	18
	108.22	290	0.18	15.078191	17
	121.65	400	0.57	18.53866	30
	114.44	2400	0.81	18.333793	27
	116.35	1000	1.23	23.504902	25
	124.9	460	0.30	16.405852	17
	103.05	220	0.10	15.865743	15
	106.15	320	0.14	16.786992	19
	113.58	820	1.03	18.341961	24
	121.01	1025	1.29	22.096965	27
	156.7	3750	0.83	22.130391	26
	148.26	1530	0.75	23.332592	23

Table 5a.5. Multivariate Regression Output for Predicting Veris EC.

Regression Statistics				
Multiple R	0.822137376			
R Square	0.675909865			
Adjusted R Square	0.638870992			
Standard Error	20.0822575			
Observations	40			
	Coefficients	Standard Error	t Stat	P-value
Intercept	33.22509916	14.80213388	2.244616	0.031215
Sulfate (ppm)	0.013015653	0.00343193	3.792517	0.000566
Organic Carbon	-35.53851483	7.220908379	-4.92161	2.03E-05
Water Content	5.529334067	1.328676565	4.161535	0.000195
Plasticity Index	-0.476185231	0.703485583	-0.67689	0.502923

Based on the results in Table 5a.5, the regression coefficient for plasticity index is not significant at the 95 percent confidence level, so the multivariate analysis with these data show EC as a function of an intercept, sulfate content, organic content, and water content. This model predicts the Veris EC with a standard error of the estimate of 20 mS/m.

Multivariate Analysis of Transformed Data

Although the preliminary multivariate analysis showed success modeling the Veris EC as a function of an intercept, sulfate content, organic content, and water content, in practice more utility can be gained from the Veris if sulfate content can be predicted without requiring knowledge of other soil properties such as organic or moisture contents. Figure 5a.18 illustrates how the sulfate content does not appear to vary linearly with the EC. By taking the natural log of the measured sulfate contents, as Figure 5a.19 illustrates, this relationship between sulfate content (expressed as a natural log of the measured value) and measured EC now appears linear.

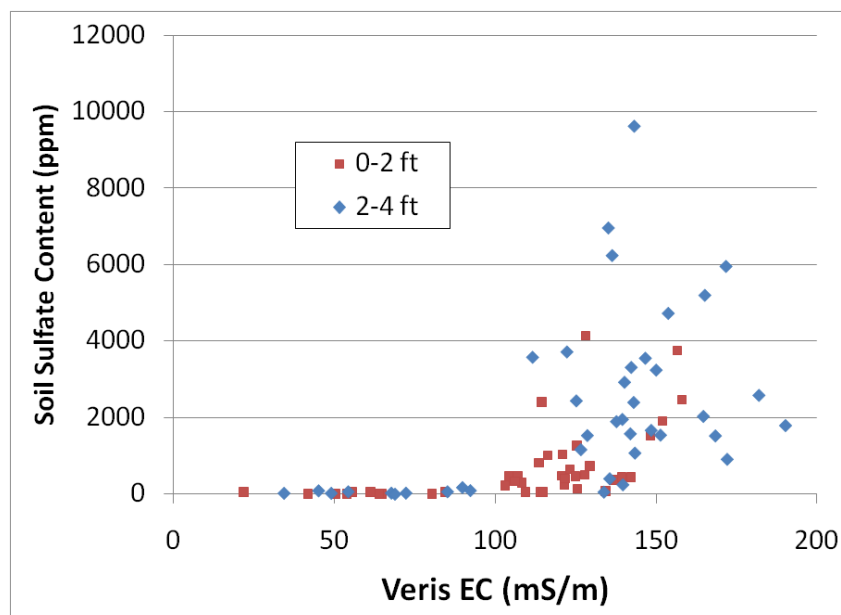


Figure 5a.18. Sulfate Content versus Veris 3150 EC.

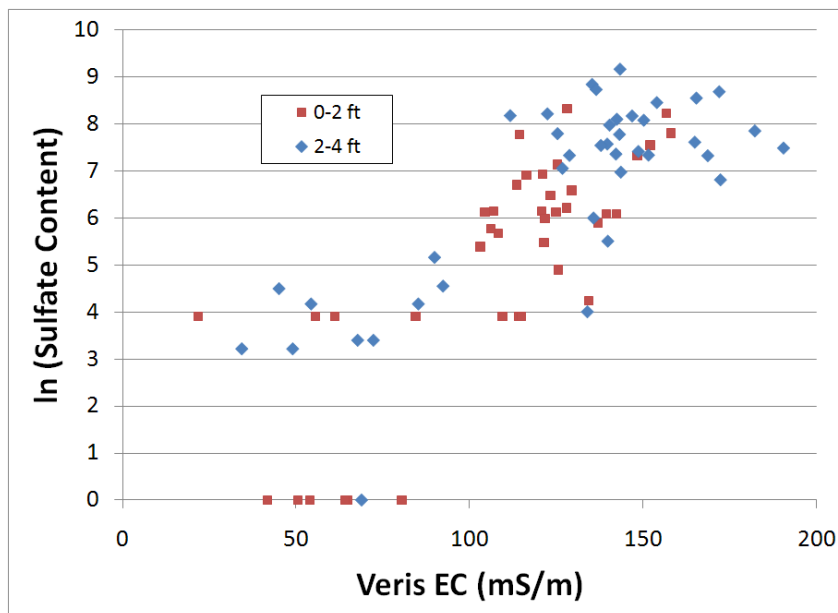


Figure 5a.19. Natural Log of Sulfate Content versus Veris 3150 EC.

Using the transformed data in Table 5a.6, where the sulfate contents are expressed as the natural log of the measured concentration, the multivariate regression shown in Table 5a.7 was developed to predict the Veris EC from sulfate content.

The results in Table 5a.7 are similar to the results in Table 5a.5 in that both models predict the Veris EC with a standard error of approximately 20 mS/m. However, in contrast to the results in Table 5a.5, when the sulfate content input is transformed using the natural log function, the multivariate regression in Table 5a.7 shows that the intercept and coefficients for organic carbon and plasticity index are not significant at the 95 percent confidence level. The model in Table 5a.7 shows the Veris EC is a function of the sulfate content (expressed as the natural log of the sulfate concentration) and the soil gravimetric water content.

Table 5a.6. Data for Predicting EC Using Natural Log of Sulfate Content, Organic Content, Water Content, and Plasticity Index.

	VerisEC_Dp	ln(SavgDp)	OCavgDp	WCavgDp	PI4
Fort Worth #2	54.29	4.17438727	0.86	17.9173104	21
	45.06	4.49980967	1.57	17.9503374	32
	67.69	3.401197382	0.84	14.6835884	18
	68.82	0	1.38	17.6847471	23
	49	3.218875825	1.76	16.4696134	15
	34.33	3.218875825	1.64	15.6385357	20
	85.16	4.17438727	1.45	18.1789446	24
	72.23	3.401197382	1.25	14.3414799	11
	89.81	5.164785974	0.81	18.0190506	21
	92.27	4.553876892	0.94	17.804045	20
Fort Worth #1	126.64	7.060476366	0.60	26.8367387	62
	139.7	5.510399971	0.94	26.9765093	48
	168.48	7.329749689	0.90	26.2013203	43
	125.22	7.799753318	1.07	27.5449651	60
	135.19	8.847934753	1.17	21.0825955	42
	135.59	6.003887067	0.58	28.9427956	55
	133.83	4.007333185	1.85	26.632297	33
	143.46	6.980075941	1.16	30.0507069	51
	146.7	8.176110342	1.55	27.9058285	55
	143.23	9.171599544	0.87	27.035493	50
Sherman	165.19	8.556413905	0.57	19.6249702	39
	164.73	7.618251098	0.58	17.5882228	18
	172.14	6.816188085	0.64	18.4753279	33
	182.04	7.857480787	1.27	21.5022312	36
	128.66	7.33628566	0.59	18.8298934	35
	151.41	7.342779189	0.53	18.3796509	42
	140.21	7.981619398	0.80	18.0819572	44
	142.3	8.106212903	0.68	19.8993952	51
	139.54	7.578145472	0.85	23.3584505	54
	143.07	7.783224016	0.43	16.8473238	30
	137.71	7.548292509	0.64	17.6181462	36
	136.37	8.738735461	0.59	18.5641956	35
	150.06	8.084870629	1.06	23.0358239	30
	142.04	7.365180126	0.39	16.3623119	29
	111.61	8.183118079	0.36	17.0617171	33
	122.32	8.221478947	0.21	17.4152945	45
	148.51	7.420578905	1.02	23.6736483	65
	153.81	8.461151801	1.00	23.3076732	54
	171.77	8.691986482	0.99	19.5526502	41
	190.28	7.492760301	1.09	20.9509966	31

Table 5a.7. Multivariate Regression Output for Predicting Veris EC where Sulfates Are Transformed Using the Natural Log.

Regression Statistics				
Multiple R	0.82102553			
R Square	0.67408292			
Adjusted R Square	0.63683525			
Standard Error	23.9637077			
Observations	40			
	Coefficients	Standard Error	t Stat	P-value
Intercept	-7.78048519	25.43915007	-0.30585	0.761533
ln(SavgDp)	13.9906181	2.670239538	5.239462	7.77E-06
Organic Carbon	-11.9636994	12.59031124	-0.95023	0.348511
Water Content	3.84430833	1.536644414	2.501755	0.017184
Plasticity Index	-0.71735974	0.524207766	-1.36846	0.179887

Zero and First-Order Conditioning Correlation Analysis

While the model in Table 5a.7 reduces the number of predictor variables down to two instead of four and eliminates an intercept value, the real desire is to investigate if the Veris EC and the soil sulfate content can be related to each other without any other explanatory variables. To study this topic, researchers used the data in Table 5a.6 and analyzed both the zero-order correlations and first-order partial correlations among the parameters measured. Table 5a.8 presents the correlation values and test statistics of the observed correlation coefficients for the data in Table 5a.6 upon zero-order conditioning. These zero-order correlations show that EC, sulfate content, organic content, moisture content, and plasticity index correlate, as Figure 5a.20 illustrates.

Table 5a.8. Correlation Matrix and Test Statistics.

Correlation Values					
	VerisEC_Dp	ln(SavgDp)	OCavgDp	WCavgDp	PI4
VerisEC_Dp	1				
ln(SavgDp)	0.791134749	1			
OCavgDp	-0.39263624	-0.514504587	1		
WCavgDp	0.441143307	0.299983928	0.15495808	1	
PI4	0.527710463	0.604200343	-0.1987177	0.75699	1
Correlation test statistics <i>t-crit=2.02 (38 d.f.; 95% conf.)</i>					
	VerisEC_Dp	ln(SavgDp)	OCavgDp	WCavgDp	PI4
VerisEC_Dp					
ln(SavgDP)	7.973427197				
OCavgDp	-2.63171522	-3.698734748			
WCavgDp	3.030174789	1.938504389	0.96690493		
PI4	3.829682896	4.67418349	-1.24990534	7.141433	

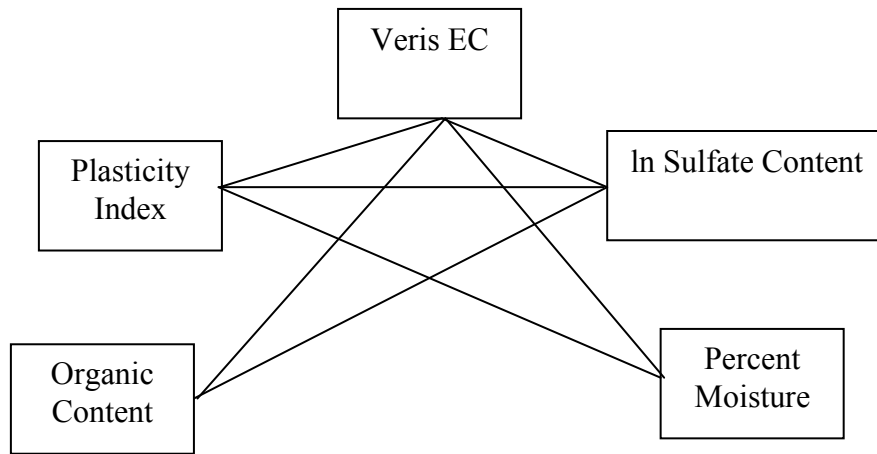


Figure 5a.20. Correlation among Soil Parameters upon Zero-Order Conditioning.

Next, since the data used to develop the correlations shown in Figure 5a.20 represent a cross section of observations where many parameters are changing, researchers investigated the conditional probabilities to assess which relationships remain under first-order conditioning. The following conditional probabilities result in the final determination of how these parameters correlate to each other:

- $\text{Corr}(\text{EC}, \text{OC} \mid \text{MC}) = -0.51$, test statistic = 0.94.
- $\text{Corr}(\text{EC}, \text{MC} \mid \text{OC}) = 0.55$, test statistic = 1.1.
- $\text{Corr}(\text{EC}, \ln\text{SO}_4 \mid \text{PI}) = 0.70$, test statistic = 2.00.
- $\text{Corr}(\text{EC}, \text{PI} \mid \ln\text{SO}_4) = 0.10$, test statistic = 0.03.
- $\text{Corr}(\ln\text{SO}_4, \text{PI} \mid \text{EC}) = 0.36$, test statistic = 0.41.
- $\text{Corr}(\text{EC}, \ln\text{SO}_4 \mid \text{OC}) = 0.75$, test statistic = 2.45.
- $\text{Corr}(\text{EC}, \text{OC} \mid \ln\text{SO}_4) = 0.03$, test statistic = 0.002.
- $\text{Corr}(\ln\text{SO}_4, \text{OC} \mid \text{EC}) = -0.36$, test statistic = 0.42.
- $\text{Corr}(\text{EC}, \ln\text{SO}_4 \mid \text{MC}) = 0.77$, test statistic = 2.69.
- $\text{Corr}(\text{EC}, \text{MC} \mid \ln\text{SO}_4) = 0.35$, test statistic = 0.39.
- $\text{Corr}(\ln\text{SO}_4, \text{MC} \mid \text{EC}) = 0.089$, test statistic = 0.024.

After examining both zero and first-order correlations, only the correlations between sulfate content and the Veris EC and the correlation between plasticity index and moisture content remain, as Figure 5a.21 illustrates. This means that, given constant plasticity, percent moisture, and organic content, the Veris EC can be described as correlating directly with the sulfate content (expressed as the natural log of the actual sulfate content). Table 5a.9 shows the regression output predicting the natural log of the sulfate content solely from the Veris EC. In Table 5a.9, the intercept is not significant. Therefore, Figure 5a.22 illustrates this model while fixing the intercept to zero.

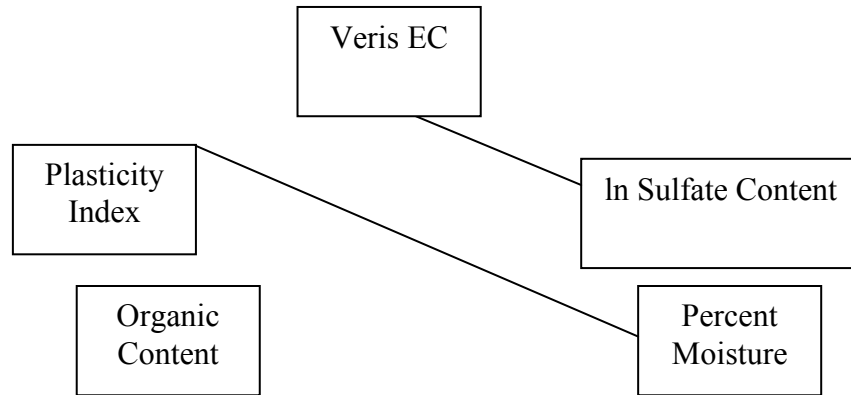


Figure 5a.21. Correlation among Soil Parameters after Zero and First-Order Conditioning.

Table 5a.9. Regression Output Predicting ln(Sulfate Concentration) from Veris EC.

Regression Statistics				
Multiple R	0.780808813			
R Square	0.609662403			
Adjusted R Square	0.599390361			
Standard Error	1.324897998			
Observations	40			
	Coefficients	Standard Error	t Stat	P-value
Intercept	1.407360404	0.705448651	1.994986	0.0532522
VerisEC_Dp	0.041102125	0.005335166	7.704001	2.801E-09

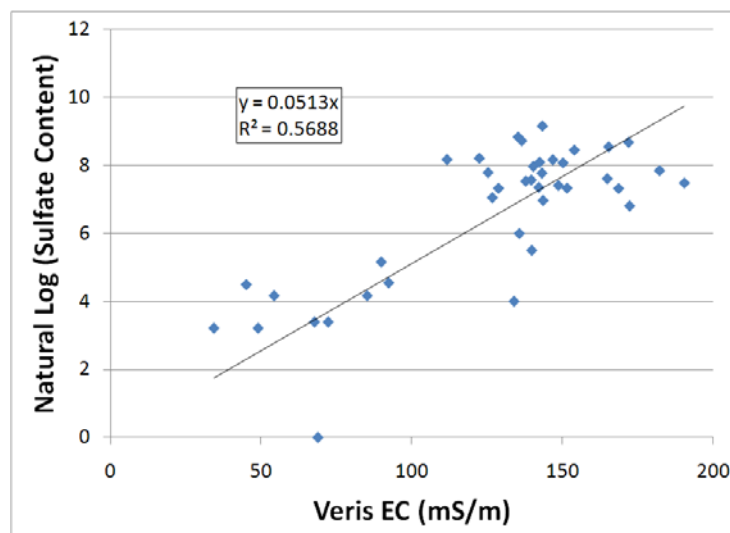


Figure 5a.22. Predicting Sulfates with Veris EC.

Note: Model developed from all “deep” data collected among three different test sites.
Intercept fixed to zero due to lack of statistical significance in Table 5a.9.

Geospatial Grouping for Model Development

The results thus far show that the Veris EC is a function of multiple soil parameters. However, if important soil parameters remain constant, the Veris EC can be related to the natural log of the sulfate content with a linear function, and researchers sought to improve upon the model represented in Figure 5a.22. The approach investigated used mapping software to group the test sites into geospatial regions. Next, each region was considered its own “population,” and any spot data collected in that region was averaged to provide an entry into the dataset used for model development. Researchers deemed this approach reasonable because they felt this geospatial grouping would smooth out the scatter in the data and help produce data where other underlying factors (such as water content) that also contribute to the conductivity measurement remain as constant as possible. Table 5a.10 presents this grouped data, and Figure 5a.24 presents the sulfate contents (transformed by the natural log function) versus the Veris EC for the grouped data, with the raw data points added for comparison. The plot in Figure 5a.23 illustrates that the general trend appears the same regardless of whether the raw or grouped data are examined.

Table 5a.10. Data Grouped According to Geospatial Regions for Input into Model Development.

	EC (mS/m)	Sulfate (ppm)	ln(SO ₄)
Deep (2-4 ft.)	56.972	42	3.73767
	34.33	25	3.218876
	84.8675	91.25	4.513603
	131.294	2205	7.698483
	148.314	3204.45	8.072296
	129.04	2638	7.877776
	144.71333	2942.465278	7.987003
	174.35833	3080.416667	8.03282
Shallow (0-2 ft.)	53.336	10	2.302585
	21.87	50	3.912023
	72.5475	25	3.218876
	116.154	172	5.147494
	130.324	510.9	6.236174
	105.758	352	5.863631
	121.23778	1253.263889	7.133507
	149.45667	1753.333334	7.469274

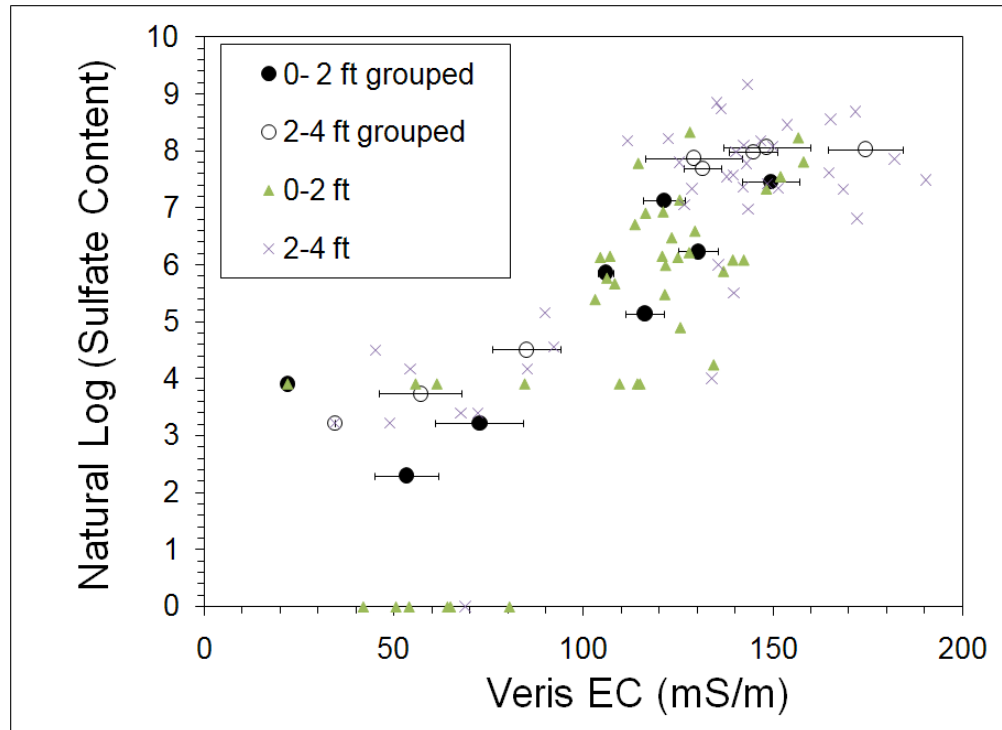


Figure 5a.23. Grouped and Ungrouped Transformed Sulfate and EC Data.

To verify if the grouped data yields similar results as the ungrouped data, researchers developed a model to predict the natural log of the sulfate content using the grouped shallow, deep, and both shallow and deep data combined. Tables 5a.11–5a.13 present the output, which shows:

- The coefficient for the Veris EC is significant in all cases.
- Considering the 95 percent confidence intervals in Tables 5a.11-5a.13 for the Veris EC coefficient, this coefficient does not statistically differ among any of the models.
- With the shallow data, the intercept is not significant, as shown by the P-value in Table 5a.11.
- The intercept is significant for the models, as shown by the P-values in Tables 5a.12 and 5a.13.

Table 5a.11. Regression Output from Shallow Grouped Data.

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	1.638011393	0.983736501	1.665092	0.146948	-0.769105104	4.045127889
Veris EC	0.03656424	0.00941801	3.882374	0.008148	0.013519199	0.059609282
Standard error of the estimate:	1.08					
R ² :	0.72					

Table 5a.12. Regression Output from Deep Grouped Data.

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	1.600312464	0.611249749	2.618099	0.039687	0.104638213	3.09598671
Veris EC	0.042412309	0.005014792	8.457441	0.000149	0.030141555	0.05468306
Standard error of the estimate:	0.65					
R ² :	0.92					

Table 5a.13. Regression Output from All Grouped Data

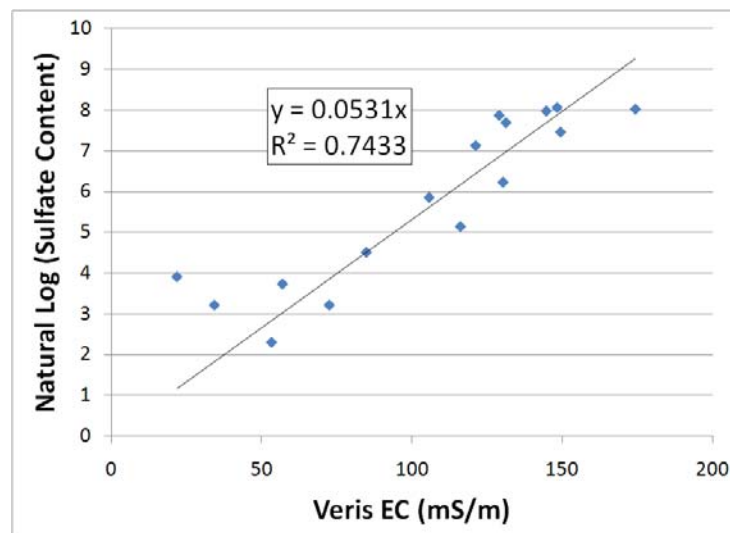
	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	1.477260599	0.571525952	2.584766	0.021608	0.25145935	2.70306185
EC	0.04107669	0.00503519	8.157923	1.09E-06	0.03027728	0.0518761
Standard error of the estimate:	0.88					
R ² :	0.83					

Conclusions

Based upon these results, researchers recommend the model shown in Figure 5a.24 be used for locating zones of high sulfates with the Veris EC device. This model uses all the grouped data and matches well with the model shown in Figure 5a.22, which used ungrouped data. The intercept has been set to zero for the following reasons:

- With the ungrouped data, this intercept was not significant.
- With the grouped shallow data, this intercept also was not significant.
- Even when significant, the observed intercept, in practice, influences the predicted sulfate content minimally if the threshold sulfate content of interest is 3000 ppm. As Figure 5a.25 shows, little divergence in the two models exists at that sulfate content level.

As a final note, referring back to Figure 5a.18, another possible approach is to simply perform additional testing at locations where the Veris EC exceeds 100 mS/m.

**Figure 5a.24. Predicting Sulfates with Grouped Data.**

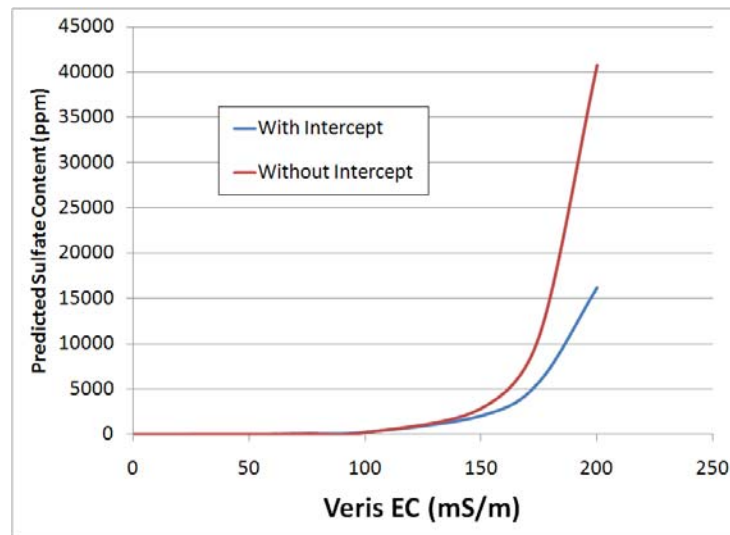


Figure 5a.25. Predicted Sulfates with and without Intercept Value.

SHERMAN, TEXAS, ROUND TWO

To further corroborate what we have learned with the Veris 3150 and EM-38DDRT conductivity devices, another trip was made to Sherman and US 82. TxDOT wanted to go to the area where the highest sulfate contents were measured during their preconstruction testing. They measured sulfate contents above 59,000 ppm at station number 1540+00. Between stations 1534+00 and 1540+00, TxDOT measured sulfate contents well above the threshold of 3000 ppm. This is the area where we wanted to focus our next round of testing.

Figure 5a.26 is a Google Earth® image of the test site. The red arrow represents the region where TxDOT measured high sulfate contents, and the blue arrow represents the length of the testing we did with the Veris 3150. We also used the EM-38DDRT to measure the conductivity for this area, but the cable connecting the unit to the GPS antenna was shorting out and rendered all data collected as useless. A ground-level view of the site is shown in Figure 5a.27. The image was taken from the southeast end of the project, looking back to the northwest. The high sulfate area is in the foreground, and the low sulfates are in the background closer to the drainage ditch.



Figure 5a.26. Aerial View of US 82 Project Shows High Sulfate Zone (Red Arrow) and Extent of Data Collection (Blue Arrow).



Figure 5a.27. View Looking from High Sulfate Area (Foreground) Back to Low Sulfate Area (Background) to the West.

Note the driveway separating the high sulfate and low sulfate areas.

Figure 5a.28 shows electrical conductivity measurements made with the Veris 3150. The EC shallow map on the left side of the figure represents shallow conductivity readings made from a 0 to 2 ft depth, and the EC deep represents deep conductivity measurements made from 0 to 4 ft depth. The lighter colors represent lower conductivity, with darker shades of gray meaning higher conductivities.

Researchers selected 20 locations to collect samples from a 0 to 4 ft depth at 1 ft intervals based on conductivity readings measured with the Veris 3150. The locations of each borehole are plotted on Figure 5a.28 in yellow. Conductivity measurements at the high sulfate site using the Veris 3150 show higher conductivity (darker areas) adjacent to the driveway access cutting across the project (Figure 5a.28). The light-colored area between boreholes 8 and 9 is the location of the driveway shown in Figures 5a.26 and 5a.27. The high sulfate area measured by TxDOT between stations 1534+00 and 1540+00 is delineated by the red arrow. Sulfate measurements made by the researchers on samples collected from the boreholes plotted in yellow are the green numbers adjacent to the EC shallow plot.

Sherman Site 2

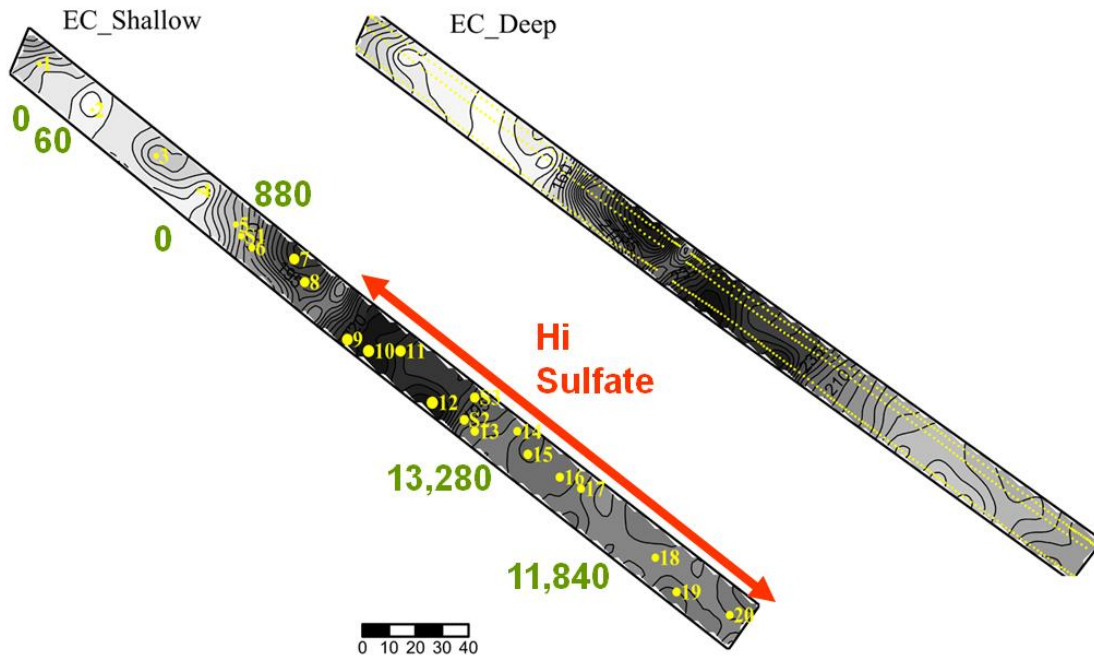


Figure 5a.28. Electrical Conductivity Measurements Taken with the Veris 3150 for US 82.

The numbers in green are actual sulfate measurements made by the researchers; the numbers in yellow represent boreholes where samples were collected for lab testing.

Table 5.14 lists the laboratory measured moisture contents, sulfate concentrations determined by Tex-146-E, the percentage of soil organic matter, and the plasticity index of samples. The first number in the sample name represents one of the 20 sites (listed in yellow) shown in Figure 5a.28. The second number represents the depth at which the sample was taken. For example, Sample Name 3-4 represents the sample at location 3 in Figure 5a.28 at a depth ranging from 3 to 4 ft.

One observation from this data set is that there are areas where the conductivity is high (dark areas in Figure 5a.28), but the measured sulfate content is low; however, the moisture contents are higher, and the soil has a higher plasticity index. Note that where samples 1–5 were taken, the conductivity is low, and the sulfates and plasticity indexes are low indicating lower clay contents.

Table 5a.14. Engineering Properties of Soil Samples from Sherman Site 2.

Sample Name	MC %dry	Sulfate (ppm)	OM (%)	PI	Sample Name	MC %dry	Sulfate (ppm)	OM (%)	PI
1--1	17.9	0	2.60	26	11--1	20.7	2140	1.90	34
1--2	14.5	0	1.20		11--2	19.5	4480	1.10	
1--3	14.2	1600	0.85		11--3	27.5	4480	1.10	
1--4	16.1	120	1.00	25	11--4	29.0	2560	1.00	42
2--1	17.2	0	1.00	24	12--1	18.1	4160	1.20	38
2--2	16.3	120	0.69		12--2	21.2	2880	1.00	
2--3	15.6	0	0.14		12--3	25.5	3840	1.10	
2--4	21.1	400	0.28	26	12--4	28.4	3520	0.79	40
3--1	16.4	0	1.10	43	13--1	14.3	1700	1.80	25
3--2	14.5	0	0.94		13--2	17.6	7360	1.20	
3--3	14.4	300	0.39		13--3	20.8	8640	0.61	
3--4	17.1	540	0.34	19	13--4	24.8	8640	0.92	41
4--1	17.2	0	2.60	25	14--1	18.0	640	2.40	27
4--2	15.5	0	0.81		14--2	26.8	2600	0.70	
4--3	16.6	0	0.50		14--3	26.8	6720	0.28	
4--4	21.9	0	0.81	25	14--4	23.2	3200	0.54	40
5--1	24.6	0	3.60	28	15--1	23.0	12480	0.43	38
5--2	24.7	0	3.60		15--2	17.3	14080	0.52	
5--3	26.4	0	2.80		15--3	23.4	20800	0.65	
5--4	23.2	560	1.70	24	15--4	23.0	13760	0.62	42
6--1	20.9	660	1.30	30	16--1	17.1	5440	0.87	33
6--2	21.7	1100	1.50		16--2	19.5	14080	0.37	
6--3	22.2	560	1.20		16--3	23.6	20160	0.88	
6--4	21.4	880	1.20	33	16--4	18.9	12480	0.35	41
7--1	19.7	1260	1.70	32	17--1	16.0	17600	0.92	33
7--2	22.9	2560	1.70		17--2	15.9	6080	0.60	
7--3	27.5	4160	1.60		17--3	19.7	17600	0.66	
7--4	32.1	35200	0.50	39	17--4	18.7	5440	0.42	41
8--1	21.4	180	1.40	36	18--1	16.4	3520	1.20	32
8--2	23.6	680	1.40		18--2	16.0	20160	0.80	
8--3	25.9	2400	1.30		18--3	15.6	27200	0.51	
8--4	32.4	29760	0.45	58	18--4	17.1	31360	0.30	45
9--1	19.2	180	1.50	28	19--1	16.0	3520	1.10	33
9--2	21.1	2880	1.70		19--2	14.2	20480	1.10	
9--3	26.0	23040	0.65		19--3	16.3	33280	0.75	
9--4	35.2	9280	0.77	38	19--4	17.8	27200	0.55	42
10--1	17.7	1700	1.90	38	20--1	15.3	2240	0.89	34
10--2	21.2	1180	1.20		20--2	15.8	2260	1.00	
10--3	28.8	2560	0.99		20--3	15.8	9280	0.67	
10--4	28.6	1360	0.86	46	20--4	16.8	26240	0.48	42

Researchers were also fortunate to collect data from Station 1528+00 to 1533+00 on July 27, 2010, when the ground was very dry and there were large cracks in the subgrade. There was a significant rain event that evening, and researchers ran the same section the following morning (July 28, 2010) after the rain event so we could compare data collected during dry and wet conditions.

Figure 5a.29 shows the deep conductivity readings from the Veris 3150 before and after the rain event. The conductivity readings are much lower and discontinuous before the rain event (with some negative conductivity readings). However, the same general trends are observed between the two data sets. As the manufacturer states, there is a certain amount of water needed to get good coverage in data.

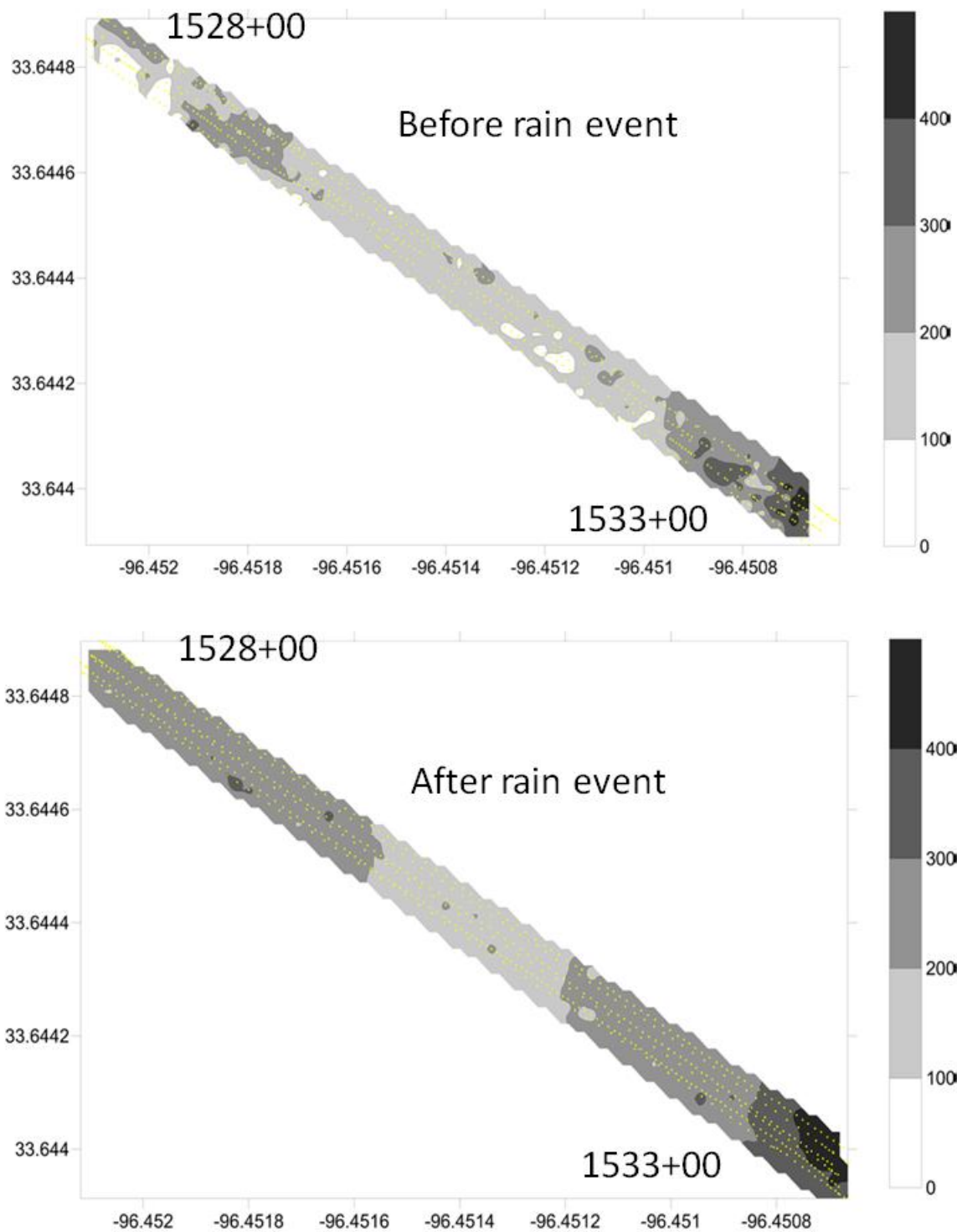


Figure 5a.29. Veris Deep Conductivity Data Plotted before and after a Rain Event from Stations 1528+00 to 1533+00 in Sherman.

Conclusions

Results of this research indicate that there is not a magic test for detecting sulfates. A combination of geological maps, the USDA/NRCS soils maps, TxDOT experience in an area, inspectors' ability to recognize sulfates in the field, and conductivity testing using techniques presented in this report backed by laboratory measurement of soil properties (i.e., plasticity index, soil organic matter, moisture contents, and sulfates) will be required to successfully identify high sulfate regions prior to construction.

This research shows that the Veris 3150 conductivity device and the EM-38DDRT electromagnetic device give comparable data with respect to soil conductivity measurements. Advantages of the Veris 3150 device are ease of use, rapid coverage of an area, and ruggedness of the instrument. The advantages observed with the EM-38DDRT device are that it can be used in confined areas, and it can be used when the soil is so wet that the Veris 3150 cannot be used due to bogging down.

We have seen that high soil conductivity may be due to elevated sulfates, other dissolved salts, high moisture contents, and high clay contents. Based on the results of this study and as illustrated in Figure 5.18, conductivity values over 100 mS/m, as measured with the Veris 3150, can be used as a threshold value for detection of sulfates. Where the conductivity is below 100 mS/m, the sulfate levels are too low to cause problems. When the conductivity is above 100 mS/m, then the elevated conductivity may be due to high sulfate levels, other salts, high clay contents, and high moisture contents.

This research shows that there needs to be enough moisture in the soil to generate a good conductivity reading. We observed high sulfates in the soil in Eagle Pass, but the conductivity was low because the moisture content in the soil was extremely low. The manufacturer recommends at least 10 percent volumetric moisture in the soil is needed to get acceptable conductivity readings with the Veris 3150. Data we collected in Sherman before and after a rain event (Figure 5a.29) show much better coverage after the rain event with no negative conductivity values, which were reported in the data before the rain event.

It is imperative that soil samples be collected based on conductivity maps constructed from the Veris 3150 data. Sampling can be better coordinated so that samples are not randomly taken every 500 to 2500 ft as they are currently done at TxDOT. Some sulfate-rich zones can be less than 500 ft wide and may be missed using the current testing program, which can have disastrous consequences.

CHAPTER 5B

EVALUATING THE NIR ATTACHMENT TO ESTIMATE SOIL ORGANIC MATTER

INTRODUCTION

The company, Veris Technologies that developed the Veris 3150 conductivity device also developed an attachment that will estimate soil organic matter using two wavelengths of light in the near infrared region. The implement attaches to the back of the Veris 3150 implement so conductivity and soil organic matter data can be collected simultaneously (Figure 5b.1). The implement and accompanying software cost approximately \$6000.



Figure 5b.1. Black Implement NIR Device that Is Attached to Veris 3150.

The attachment (Figure 5b.2) digs a small furrow into the ground about 0.5 to 1 inch deep and measures the color every 1 second, sending a reading to the data recorder. Following data collection, samples are collected where the instrument detects changes in color. The samples are then returned to the laboratory where the SOM is measured. The SOM measurements are then used to attach values to the color data spectra taken by the NIR device so a SOM map of the region can be plotted. This measurement is only a surface measurement (top 1 to 2 inches) and does not reflect what the organic matter content is at depth.



Figure 5b.2. NIR Detector (Red Arrow) Attached between the Rubber Wheels.

Kenton Dreiling, an engineer from Veris Technologies, came to Texas to demonstrate the NIR detector for the researchers and TxDOT personnel. The Austin District provided the researchers with a small test site located at the northwest corner of the intersection of US 183 and SH 29 near the town of Liberty Hill. Dreiling made two passes over the same area with the implement, and he collected five samples from the area to return to the laboratory for calibration of the data to construct the map of SOM. The researchers also collected samples from the same five locations to run independent tests for SOM. Veris Technologies sends their samples to a soils testing laboratory at Kansas State University to measure SOM. The researchers use the UV-Vis test developed in project 5540 to measure SOM.

RESULTS

Since the area that TxDOT provided for the organics testing was so small (about 500 ft long and wide enough for one pass), the vendor said that they did not have a large enough area and too little variability in SOC to generate a map of the site. The researchers at TTI collected soil samples from the site and measured the soil organic carbon content using the UV-Vis technique. Our data are reported in Table 5b.1, and all of the samples show high SOC contents. There are no low to mid-range organic contents for this site. We did not learn anything from this exercise except that we need a larger test site with more variability in SOC contents to collect useful data using the IR setup.

Table 5b.1. Organic Carbon Contents of Samples Collected from US 183 and SH 29.

Sample	Organics (%)
1N	3.53
2N	3.65
3N	4.48
4N	3.74
5N	4.35

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

This project started out as an evaluation of different techniques to detect organic matter and sulfate minerals in subgrade soils. There are several techniques to measure both of these constituents in the soil; however, most of the techniques are spot tests that require taking samples and returning them to the laboratory for analysis. These techniques leave gaps in the subgrade where high organic contents and/or sulfate minerals may be present but not detected. TxDOT currently tests subgrades for sulfate minerals from distances ranging from every 500 ft to every 2500 ft (Caroline Herrerra, personal communication, 2010). The soil organic matter is currently not even measured in most districts. There are many instances where the sulfates on a project may not be detected using the current methods because sampling is at specified intervals. If a sulfate-rich seam falls between these intervals, then it will not be detected until the damage is done.

TxDOT wanted a way to measure these constituents continuously so that there would not be any gaps in the data, and sulfate or organic-rich areas would not be overlooked. We started with a literature review to identify potential tools to measure sulfates and organic matter. We identified several techniques to detect sulfates, including: geologic maps, soil survey maps, visual observation, remote sensing with satellite images, spectroscopic techniques, X-ray fluorescence, ground penetrating radar, and conductivity techniques.

Most of these techniques were deemed impractical for TxDOT, and many were still spot tests. However, researchers identified two techniques that would provide continuous coverage over an area to a depth of 3 to 4 ft. These two techniques were using the Veris 3150 conductivity device that is used in precision agriculture and the EM-38DDRT electromagnetic device used in geophysical surveys to map conductivity variations. The project-monitoring committee advised the researchers to focus efforts on using these two techniques to detect sulfates in the subgrade. They stated that continuous measurement of organic matter was a secondary issue.

Based on extensive testing, the researchers recommend using the Veris 3150 and EM-38DDRT devices to map soil salinity, which can be related to the sulfate content. TxDOT will still be required to collect soil samples and measure the sulfate content using Tex-146-E, but they can focus on areas that exhibit high conductivity readings instead of selecting samples based on a grid that may miss potential problem areas.

We also did a brief evaluation of an infrared device that is manufactured by the same company that makes the Veris for detection of soil organic matter. The test was poorly designed and needs to be repeated on a larger project with more variability in organic matter. This test has a lot of potential to be run simultaneously with the conductivity device so organic content and conductivity can be mapped simultaneously. One would still need to collect soil samples in the field to correlate with the values given by the infrared device. A soil organic map could then be constructed similar to the conductivity maps.

The researchers recommend that TxDOT adopt the use of the Veris 3150 to map conductivity to correlate with potentially harmful sulfate contents. We also recommend using

the IR device to collect more data on projects to construct maps of soil organic matter. These two devices could help TxDOT design better stabilization strategies, which would result in savings of millions of dollars.

REFERENCES

- Bennett, D.L., R.J. George, and B. Whitfield. (2000) The Use of Ground EM Systems to Accurately Assess Salt Store and Help Define Land Management Options for Salinity Management, *Exploration Geophysics*, Vol. 31, p. 249–254.
- Bower, C.A., and R.B. Huss. (1948) *Rapid Conductometric Method for Estimating Gypsum in Soils*. U.S. Department of Agriculture.
- Bredenkamp, S., and R. Lytton. (1995) *Reduction of Sulfate Swell in Expansive Clay Subgrades in the Dallas District*. Report 1994-5. TTI, Texas Department of Transportation.
- Bui, E.N., and B.L. Henderson. (2003) Vegetation Indicators of Salinity in Northern Queensland, *Australian Ecology*, Vol. 28, p. 539–552.
- Burkart, B., G.C. Goss, and J. P. Kern. (1999) The Role of Gypsum in Production of Sulfate-Induced Deformation of Lime-Stabilized Soils. In *Environmental and Engineering Geoscience*, Vol. V, No. 2, pp. 173–187.
- Clare, K.E., and P.T. Sherwood. (1954) The Effect of Organic Matter on the Setting of Soil-Cement Mixtures. *Journal of Applied Chemistry*, Vol. 4, No. 11, p. 625–630.
- Clare, K.E., and P.T. Sherwood. (1956) Further Studies on the Effect of Organic Matter on Setting of Soil-Cement Mixtures. *Journal of Applied Chemistry*, Vol. 6, No. 8, p. 317–324.
- Dalal, R.C., and R.J. Henry. (1986) Simultaneous Determination of Moisture, Organic Carbon, and Total Nitrogen by Near Infrared Reflectance Spectrometry. *Soil Science Society of America Journal*, Vol. 50, No. 1, p. 120–123.
- Harris, J.P., S. Sebesta, and T. Scullion. (2004) Hydrated Lime Stabilization of Sulfate-Bearing Vertisols in Texas. In *Transportation Research Record 1868*, TRB, National Research Council, Washington, D.C., pp. 31–39.
- He, Y., and H. Song. (2006) Prediction of Soil Content Using Near-Infrared Spectroscopy, International Society for Optical Engineering, SPIE Newsroom.
- McNeill, J.D. (1980) *Electrical Conductivity of Soils and Rocks*. Technical Note TN-5, Geonics Limited, Ontario Canada, 22 p.
- Petry, T.M., and D.N. Little. (1992) Update on Sulfate-Induced Heave in Treated Clays: Problematic Sulfate Levels. In *Transportation Research Record 1362*, TRB, National Research Council, Washington, D.C., pp. 51–55.
- Robinson, J.W. (1970) *Undergraduate Instrumental Analysis*. 2nd ed. Marcel Dekker, Inc., New York.

Sherwood, P.T. (1962a) *The Effect of Soil Organic Matter on the Setting of Soil-Cement Mixtures*. Road Research Technical Paper #61, Her Majesty's Stationery Office, London.

Sherwood, P.T. (1962b) Rapid Method for Detecting the Presence of Deleterious Organic Matter in Soil-Cement. *Journal of Applied Chemistry*, Vol. 12, No. 6, p. 279–288.

Spies, B., and P. Woodgate. (2005) *Salinity Mapping Methods in the Australian Context*. The Department of the Environment and Heritage; and Agriculture, Fisheries, and Forestry, Commonwealth of Australia.

Sposito, G. (1989) *The Chemistry of Soils*. Oxford University Press, New York, 277 p.

The Leader in Soil Sensing Technologies. Veris Technologies, Salina, Kansas.
www.veristech.com/products/3150.aspx. Accessed July 15, 2009.