1. Report No. FWHA/TX-02/4240-1	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle LABORATORY AND FIELD PROCEDURES FOR MEASURING THE SULFATE CONTENT OF TEXAS SOILS		5. Report Date October 2002
		6. Performing Organization Code
7. Author(s) John Pat Harris, Tom Scullion and Stephen Sebesta		8. Performing Organization Report No. Report 4240-1
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 No. 0-4240
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 Research: May 2001 - May 2002
		14. Sponsoring Agency Code
15. Supplementary Notes Research performed in cooperation with the Texas Department of Transportation and the U.S. Department of Transportation, Federal Highways Administration. Research Project Title: Develop Guidelines and Procedures for Stabilization of Sulfate Soils		

16. Abstract

Project 0-4240 was initiated to provide guidelines on how to effectively stabilize sulfate rich soils. The first tasks in this project involved evaluating the various methods of measuring the sulfate content of soils both in the laboratory and in the field. In the laboratory, two test procedures were investigated, namely Texas Department of Transportation (TxDOT) Test Method Tex-620-J gravimetric approach and the Ion Chromatography approach. For this comparison, control samples with known sulfate contents were fabricated in the laboratory. The samples were treated with known amounts of fine-grained and coarse-grained sulfate crystals. A range of samples was sent to TxDOT and several private laboratories. In terms of both accuracy and repeatability, the researchers concluded that the Ion Chromatography approach is superior to TxDOT Test Method Tex-620-J. Recommendations are submitted to improve the Tex-620-J procedure. The main conclusion is that TxDOT should consider replacing Tex-620-J with the Ion Chromatography approach.

Sulfate swell problems are frequently localized, so substantial effort was placed on evaluating rapid field tests. Four potential tests were evaluated, two were found to provide good results. Both the Colorimetric/Spectrophotometric and Conductivity tests should be considered for full-scale implementation.

A survey of both automated and map systems revealed that the existing geological maps provide a good first-cut indication of locations with potentially high sulfate contents. The automated Soil Survey Geographic (SSURGO) maps hold great potential for future use, however, they are available for less than half of the Texas districts and some errors and inconsistencies were found.

17. Key Words Sulfates, Soils, Stabilization, Ion Chromatography, Field Testing, Highways, Laboratory Tests	18. Distribution Statement No restrictions. This document is available to the public through NTIS: National Technical Information Service 5285 Port Royal Road Springfield, Virginia 22161	
19. Security Classif.(of this report) Unclassified	20. Security Classif.(of this page) Unclassified	21. No. of Pages 100	22. Price

Form DOT F 1700.7 (8-72)   Reproduction of completed page authorize

LABORATORY AND FIELD PROCEDURES FOR MEASURING THE SULFATE CONTENT OF TEXAS SOILS

by

John Pat Harris
Associate Research Scientist
Texas Transportation Institute

Tom Scullion
Research Engineer
Texas Transportation Institute

and

Stephen Sebesta
Assistant Transportation Researcher
Texas Transportation Institute

Report 4240-1
Project Number 0-4240
Research Project Title: Develop Guidelines and Procedures for Stabilization of Sulfate Soils

October 2002

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 engineer in charge was Tom Scullion, P.E. (# 62683).

ACKNOWLEDGMENTS

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

Several TxDOT district offices have experienced problems with stabilizing soils containing high sulfate concentrations when treated with traditional calcium-based stabilizers. Project 0-4240 was initiated to provide guidelines to effectively treat these problem soils. This is the first report for this on-going project, and it will cover the first three tasks in the work plan. The main focus of this report is to recommend methods of measuring the sulfate content of soils both in the laboratory and in the field.

In the laboratory, two test procedures were investigated: (1) TxDOT Test Method Tex-620-J gravimetric approach, and (2) the Ion Chromatography approach. Chapter 2 of this report describes the laboratory comparisons. For this comparison control samples with known sulfate contents were fabricated in the laboratory. The samples were treated with fine-grained and coarse-grained gypsum crystals. Samples with a range of concentrations were sent to TxDOT and several private laboratories.

Many of the cases investigated in Texas revealed that sulfate problems occur in small localized areas. It is not uncommon to have one or two sulfate-induced heaves in a project, which may be several miles long. Currently there are no procedures in widespread use that can be used to locate these localized problem areas prior to application of the stabilizer. Chapter 3 of this report presents an evaluation of five rapid tests, which can be run in a matter of minutes. The development and implementation of rapid field testing procedures is one of the major objectives of this project and is the area where the researchers have spent most of their effort in the first phase of this project.

Chapter 4 describes an evaluation of the usefulness of the currently available geological and automated maps to detect high sulfate locations along Texas highways. The U.S. Department of Agriculture (USDA) maps are widely used within TxDOT, but they provide little information on the sulfate content of Texas soils. However, this project found that the existing Geological Atlas of Texas can be used to detect areas which are rich in sulfates. These maps are being used in two current forensic investigations on U.S. 82 in the Paris District and IH 40 in the Childress District. The geological maps are known and used by engineers in both the Dallas and Fort Worth Districts, however, they are not used in districts outside the metroplex that are known to have similar problem soil types.

CHAPTER 2

SULFATE CONTENT DETERMINATION – LAB TEST

BACKGROUND

Over the past 20 years, problems with sulfate-induced heave have surfaced around the world. This problem occurs when lime or cement is used to stabilize subgrade soils that bear sulfate/sulfide minerals. Hunter (1989) determined that the cause of heaving is due to the formation of two minerals that contain a large amount of water in their structure and result in a volume increase: one is called ettringite $(Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O)$ and the other is called thaumasite $(Ca_3(SO_4)(CO_3)[Si(OH)_6] \cdot 12H_2O)$. Another mechanism is created by the oxidation of iron sulfides resulting in the formation of gypsum $(CaSO_4 \cdot 2H_2O)$. This reaction forms a mineral that occupies more space than the original reduced sulfide, similar to the formation of ettringite and thaumasite, and may cause heaving due to an increase in volume as well (Dubbe et al., 1997).

It is critical to be able to accurately determine the sulfur concentrations in soils since sulfur is the key ingredient for forming these deleterious reaction products.

Studies of sulfate-bearing soils conducted by Texas Transportation Institute (TTI) in the past have yielded conflicting results for the amount of sulfate present in a soil. After reviewing TxDOT Test Method Tex-620-J some questions were raised about the technique. They are:

- How long to dry the sample in the oven?
- Does the pH of the solution need to be measured?
- Is the 1:10 dilution ratio enough for soils with high sulfate concentrations?
- Is it a valid technique to heat the sample for 24 hours since gypsum solubility is greater at lower temperatures as with lime and calcite?

Based on this information, researchers proposed to compare results of two laboratory test methods for quantitative determination of sulfate concentrations. The two methods proposed were TxDOT Test Method Tex-620-J and a procedure published by the Environmental Protection Agency (EPA) in EPA/600/2-78/054 Field and Laboratory Methods Applicable to

Overburdens and Minesoils. Once the project started, researchers discovered that commercial labs had stopped using the EPA method in favor of faster techniques involving Ion Chromatography (IC).

Therefore, a laboratory using Ion Chromatography for sulfate analysis was solicited to participate in this study. The lab initially selected was the Texas Agricultural Experiment Station-El Paso (TAMU-EP); later another lab Chemical Lime Lab in Fort Worth that uses Ion Chromatography volunteered its services.

Two labs that use Test Method Tex-620-J were also solicited for this project. The Materials and Pavement Section of the Construction Division of TxDOT agreed to test the samples as did Ana-Lab Corporation a commercial lab located in Kilgore, Texas.

GRAVIMETRIC ANALYSIS

Test Method Tex-620-J is a gravimetric technique where the element or ion of interest may be precipitated as a compound and weighed. From the weight of the precipitate, the quantity of the element or ion present can be calculated. More specifically, the Tex-620-J method involves dissolving the sulfates present in the soil with distilled water heated to near boiling. The sample is then filtered and concentrated hydrochloric acid (HCl) is added to the filtrate and heated to near boiling. A 10 percent barium chloride ($BaCl_2 \cdot 2H_2O$) solution is added to the mixture and heated again. The sulfate is removed from solution as barium sulfate ($BaSO_4$) precipitates. The precipitate is filtered and washed with water to remove chlorides, dried, and weighed. The sulfate concentration can be calculated as a percentage of the formula weight of the $BaSO_4$.

ION CHROMATOGRAPHY ANALYSIS

Ion Chromatography involves dissolving the sulfate from the soil in distilled water and adding small quantities to the IC system. The sample is injected into the system in a fixed volume and swept through the system by an inert compound like polyetheretherketone. The sample is transported into an ion exchange column where the different ions are attracted to the resin in the column and released at different times by a conductivity detector. The conductivity of each solution is measured and compared to conductivities of prepared standards to quantify

the concentration for the ion(s) of interest. These analyses may be performed rapidly and at low cost with an automated system.

TESTING PROCEDURE

To test the adequacy of the two techniques for quantifying the sulfate concentrations, samples were prepared with known concentrations of sulfate-bearing minerals. The "manufactured soils" contained 30 percent Georgia Kaolinite and a 70 percent combination of St. Peter Sand and various grain sizes of gypsum or anhydrite ($CaSO_4$).

Twenty-two samples were sent to three labs. The gypsum samples had the following concentrations of sulfate in parts per million (ppm): 0, 3000, 5000, 12,000, and 50,000. The gypsum was also submitted in two size fractions; one passing the #200 sieve and the other passing the #10 sieve and retained on the #40 sieve. These size fractions were chosen because they are representative of the more reactive sulfates found in natural soils in Texas. For example, the larger the grains, the longer it takes for them to dissolve and react (similar to powdered sugar dissolving faster than sugar cubes in a cup of coffee). Reagent grade anhydrite was also submitted in concentrations of 5000 and 12,000 ppm. All of the samples were submitted in duplicate (Table 1).

In the initial testing, 600 g of each concentration were prepared and split into 50 to 100 g subsamples and shipped to three of the four labs previously listed for analysis. The Chemical Lime Association Lab was given different samples to test at lower sulfate levels (Table 1) because it volunteered its services after the initial results had been evaluated from the other labs, and the Project Monitoring Committee (PMC) decided that lower concentrations of sulfate needed to be detected.

Because results from the initial batch received from each lab were somewhat ambiguous, another batch of samples was sent out. The second batch was mixed at lower concentrations as suggested by the PMC. Samples with concentrations of 0, 1000, 2000, and 3000 ppm gypsum were submitted in duplicate, as well as two-size fractions, for a total of 14 samples to all four labs (Table 2).

In the second round of testing, individual mixing of each sample ensured that there was not any segregation of sulfate in the samples (to ensure that all labs received the same

5

**Table 1. Manufactured Samples Used for the Initial Comparison of
Sulfate Analysis Techniques.**

Sample Name	Sulfate Concentration (ppm)	Description
EXP 1	3000	Reagent grade gypsum
EXP 2	5000	Reagent grade gypsum
EXP 3	12000	Reagent grade gypsum
EXP 4	50000	Reagent grade gypsum
EXP 5	0	Control sample
EXP 6	5000	Reagent grade anhydrite
EXP 7	12000	Reagent grade anhydrite
EXP 8	5000	Gypsum large grain (<#10>#40 sieve)
EXP 9	3000	Gypsum large grain (<#10>#40 sieve)
EXP 10	50000	Gypsum large grain (<#10>#40 sieve)
EXP 11	12000	Gypsum large grain (<#10>#40 sieve)
EXP 12	0	Control sample
EXP 13	5000	Reagent grade gypsum
EXP 14	50000	Reagent grade gypsum
EXP 15	3000	Reagent grade gypsum
EXP 16	12000	Reagent grade gypsum
EXP 17	5000	Reagent grade anhydrite
EXP 18	12000	Reagent grade anhydrite
EXP 19	50000	Gypsum large grain (<#10>#40 sieve)
EXP 20	12000	Gypsum large grain (<#10>#40 sieve)
EXP 21	5000	Gypsum large grain (<#10>#40 sieve)
EXP 22	3000	Gypsum large grain (<#10>#40 sieve)
*EX 23	12000	Reagent grade gypsum
*EX 24	5000	Reagent grade gypsum
*EX 25	2000	Reagent grade gypsum
*EX 26	1000	Reagent grade gypsum
*EX 27	500	Reagent grade gypsum
*EX 28	250	Reagent grade gypsum
*EX 29	100	Reagent grade gypsum
*EX 30	50	Reagent grade gypsum
*EX 31	0	Control sample
*EX 32	0	Control sample
*EX 33	12000	Gypsum large grain (<#10>#40 sieve)
*EX 34	5000	Gypsum large grain (<#10>#40 sieve)
*EX 35	2000	Gypsum large grain (<#10>#40 sieve)
*EX 36	1000	Gypsum large grain (<#10>#40 sieve)

*All labs except the Chemical Lime Association Lab received samples EXP 1 to EXP 22. The Chemical Lime
Association lab received the samples labeled EX 23 to EX 36 and denoted with an asterisk(*).*

Table 2. Description of Samples Used for the Follow-Up Sulfate Analysis Techniques.

Sample Name	Sulfate Concentration (ppm)	Description
1a	2000	Reagent grade gypsum
2a	3000	Gypsum large grain (<#10>#40sieve)
3a	1000	Reagent grade gypsum
4a	0	Control sample
5a	3000	Reagent grade gypsum
6a	1000	Gypsum large grain (<#10>#40sieve)
7a	2000	Reagent grade gypsum
8a	3000	Gypsum large grain (<#10>#40sieve)
9a	0	Control sample
10a	1000	Reagent grade gypsum
11a	2000	Gypsum large grain (<#10>#40sieve)
12a	1000	Gypsum large grain (<#10>#40sieve)
13a	3000	Reagent grade gypsum
14a	2000	Gypsum large grain (<#10>#40sieve)

concentrations of sulfates). For instance, the initial set of samples were mixed as a single batch and divided into three equal fractions to send to each of the three labs. It is possible that a 1000 ppm sulfate sample sent to one lab could have higher or lower sulfates than the 1000 ppm sulfate sample sent to another lab due to heterogeneous mixing.

Following testing of the "manufactured soils," in-situ soil samples were selected from an area on U.S. 82 in the Paris District. This area is currently under construction and has experienced problems with sulfate heave. Researchers selected 12 samples from the westbound lane of this new construction project. Selection was based on conductivity readings taken in the field. Samples with high and low conductivities were delivered to the four laboratories to see how the various techniques compared using real soils.

RESULTS WITH MANUFACTURED SOILS

Table 3 and Figures 1 and 2 are the results of the initial round of testing where samples were submitted only to the Texas Department of Transportation, Texas Agricultural Experiment Station-El Paso and Ana-Lab Corporation. Because the Chemical Lime Lab did not participate in the initial testing, its results are presented separately in Figure 3. A second round

Table 3. Data Received from the First Round of Testing with TxDOT, TAMU (El Paso) and Ana-Lab.

Lab Sulfate Content Data (all in PPM)

Description	Known Concentration	TxDOT	Error	% Error	TAMU	Error	% Error	AnaLab	Error	% Error
Anhydrite	5000	6845	1845	36.9	4431	-569	-11.38	3600	-1400	-28
Anhydrite	5000	4623	-377	-7.54	4451	-549	-10.98	3000	-2000	-40
Anhydrite	12000	17626	5626	46.88333	9643	-2357	-19.64167	9400	-2600	-21.66667
Anhydrite	12000	11865	-135	-1.125	9022	-2978	-24.81667	9300	-2700	-22.5
Coarse-Grained	3000	1783	-1217	-40.56667	1372	-1628	-54.26667	2400	-600	-20
Coarse-Grained	3000	1742	-1258	-41.93333	2812	-188	-6.266667	900	-2100	-70
Coarse-Grained	5000	2291	-2709	-54.18	2703	-2297	-45.94	4900	-100	-2
Coarse-Grained	5000	2922	-2078	-41.56	4654	-346	-6.92	3800	-1200	-24
Coarse-Grained	12000	4033	-7967	-66.39167	6238	-5762	-48.01667	9100	-2900	-24.16667
Coarse-Grained	12000	3923	-8077	-67.30833	5965	-6035	-50.29167	15000	3000	25
Coarse-Grained	50000	7421	-42579	-85.158	7367	-42633	-85.266	37000	-13000	-26
Coarse-Grained	50000	6598	-43402	-86.804	6925	-43075	-86.15	47000	-3000	-6
Fine-Grained	0	412	412		48	48		0	0	
Fine-Grained	0	604	604		41.9	41.9		0	0	
Fine-Grained	3000	4019	1019	33.96667	2791	-209	-6.966667	2000	-1000	-33.33333
Fine-Grained	3000	3402	402	13.4	2876	-124	-4.133333	2000	-1000	-33.33333
Fine-Grained	5000	8285	3285	65.7	4932	-68	-1.36	4100	-900	-18
Fine-Grained	5000	3964	-1036	-20.72	4774	-226	-4.52	3300	-1700	-34
Fine-Grained	12000	16570	4570	38.08333	7172	-4828	-40.23333	8500	-3500	-29.16667
Fine-Grained	12000	10672	-1328	-11.06667	6982	-5018	-41.81667	9800	-2200	-18.33333
Fine-Grained	50000	24333	-25667	-51.334	7019	-42981	-85.962	47200	-2800	-5.6
Fine-Grained	50000	23222	-26778	-53.556	6881	-43119	-86.238	40000	-10000	-20

Mean Values

Description	Known Concentration	TxDOT	Error	% Error	TAMU	Error	% Error	AnaLab	Error	% Error
Anhydrite	5000	5734	734	14.68	4441	-559	-11.18	3300	-1700	-34
Anhydrite	12000	14745.5	2745.5	22.87917	9332.5	-2667.5	-22.22917	9350	-2650	-22.08333
Coarse-Grained	3000	1762.5	-1237.5	-41.25	2092	-908	-30.26667	1650	-1350	-45
Coarse-Grained	5000	2606.5	-2393.5	-47.87	3678.5	-1321.5	-26.43	4350	-650	-13
Coarse-Grained	12000	3978	-8022	-66.85	6101.5	-5898.5	-49.15417	12050	50	0.416667
Coarse-Grained	50000	7009.5	-42990.5	-85.981	7146	-42854	-85.708	42000	-8000	-16
Fine-Grained	0	508	508		44.95	44.95		0	0	
Fine-Grained	3000	3710.5	710.5	23.68333	2833.5	-166.5	-5.55	2000	-1000	-33.33333
Fine-Grained	5000	6124.5	1124.5	22.49	4853	-147	-2.94	3700	-1300	-26
Fine-Grained	12000	13621	1621	13.50833	7077	-4923	-41.025	9150	-2850	-23.75
Fine-Grained	50000	23777.5	-26222.5	-52.445	6950	-43050	-86.1	43600	-6400	-12.8

This data show how the lab-determined concentrations compare with the actual concentrations. All of the concentrations are in ppm. Samples labeled Anhydrite are $CaSO_4$. Samples labeled Coarse-Grained are gypsum ($CaSO_4 \cdot 2H_2O$) that passes #10 and are retained on the #40 sieve. Fine-Grained samples are gypsum ($CaSO_4 \cdot 2H_2O$) and all pass the #200 sieve. These data were used to generate the graphs in Figures 1 and 2.

of testing was performed by all four labs, and results are presented in Table 4 and Figure 4. Figure 5 is a plot of all sulfate measurements up to 12,000 ppm comparing the Texas Department of Transportation Test Method Tex-620-J to the Ion Chromatography technique.

In Figures 1, 3, and 4, the sulfate content of the manufactured samples determined by each lab has been divided into coarse-grained gypsum (<#10>#40 sieve) and fine-grained gypsum (<#200 sieve) because the labs (except Ana-Lab) generally had more difficulty detecting all of the coarse-grained sulfates. This difficulty is shown by a larger deviation from the known

Lab Sulfate Content of Soils Treated with Coarse-Grained Gypsum
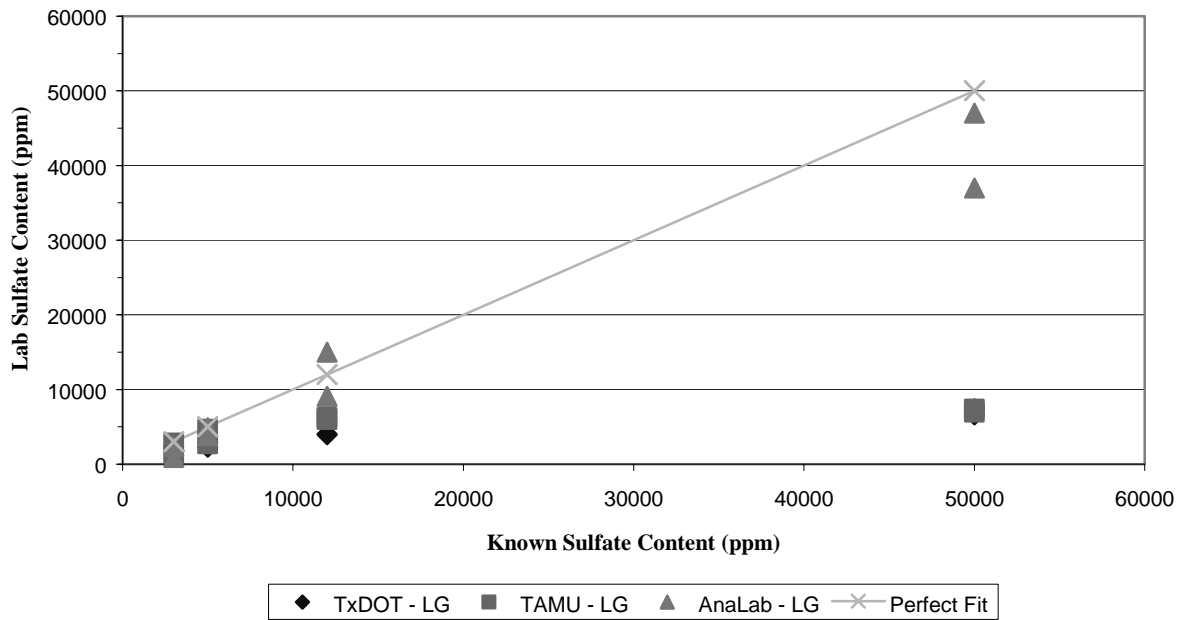
Lab Sulfate Content of Soils Treated with Fine-Grained Gypsum
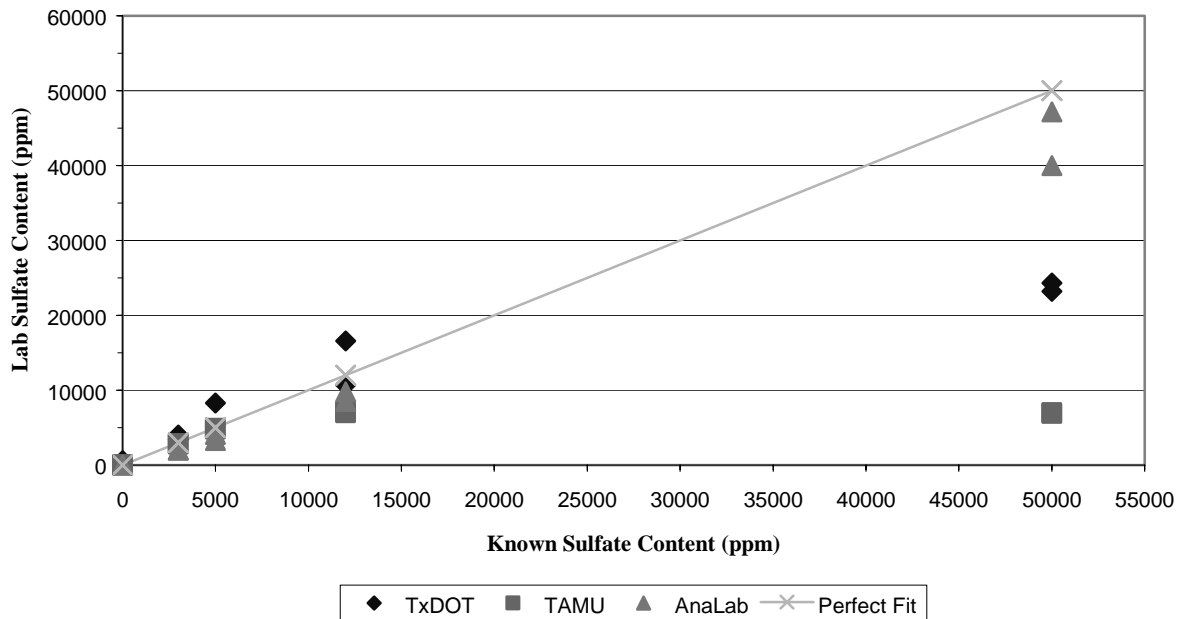
Figure 1. Known Sulfate Content vs. Laboratory Determined Sulfate Content.

Mean Errors in Sulfate Content Determination for Soils Treated with Coarse-Grained and Fine-Grained Gypsum
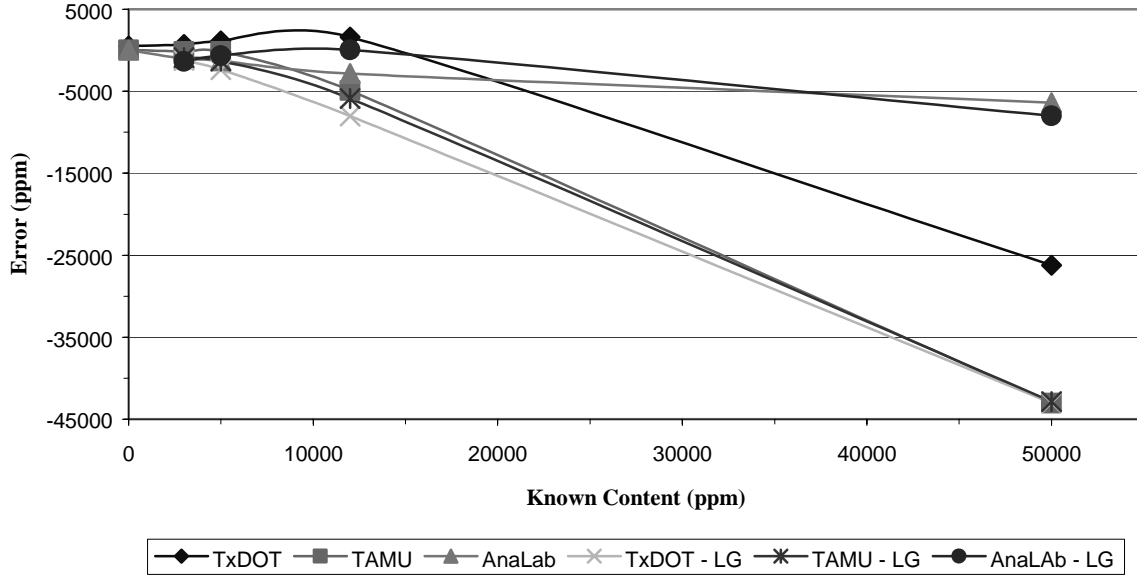
Mean % Error for Coarse and Fine-Grained Sulfates

Figure 2. Mean Error and Percent Error for Three Laboratories.

Chemical Lime Lab Sulfate Content Results

Chemical Lime Lab Measurement Error

Figure 3. Chemical Lime Lab Results.

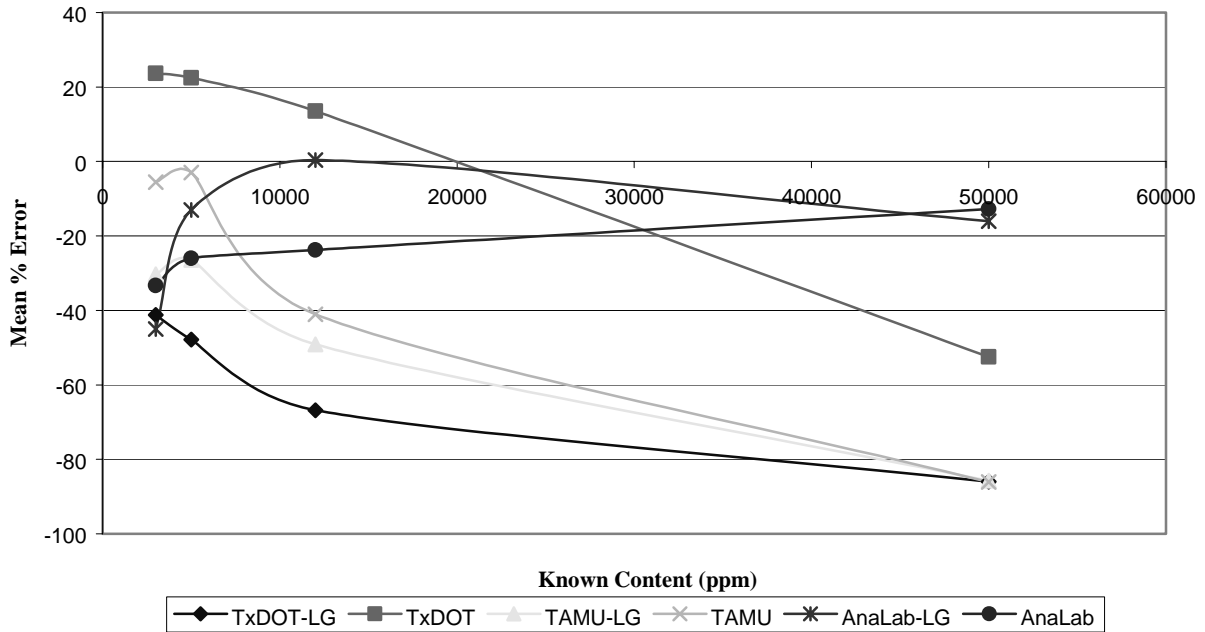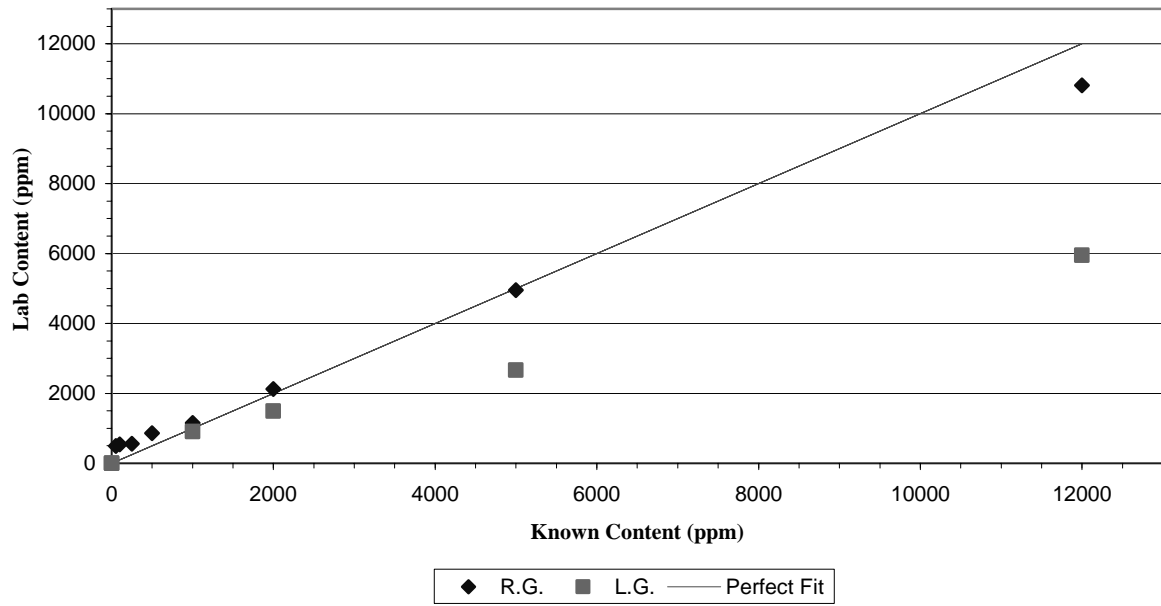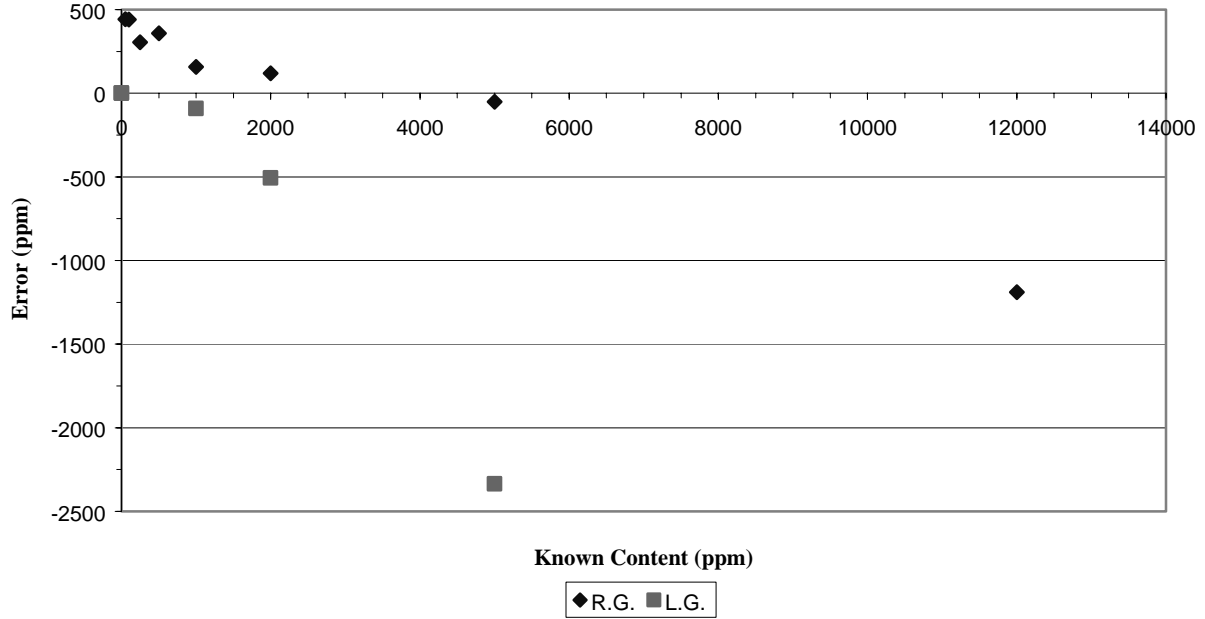## Table 4. Sulfate Content and Error for Four Laboratories (Second Round Samples).

Sample #	Description	Known Concentration	TxDOT	Error	% Error	TAMU	Error	% Error	AnaLab	Error	% Error	Chem Lime	Error	% Error
12a	LG	1000	2881	1881	188.1	1227	227	22.7	1060	60	6	1141	141	14.1
6a	LG	1000	2675	1675	167.5	893	-107	-10.7	1080	80	8	1258	258	25.8
11a	LG	2000	2702	702	35.1	1496	-504	-25.2	2500	500	25	1660	-340	-17
14a	LG	2000	3237	1237	61.85	1689	-311	-15.55	2150	150	7.5	1585	-415	-20.75
2a	LG	3000	3964	964	32.13333	1602	-1398	-46.6	3830	830	27.66667	2497	-503	-16.76667
8a	LG	3000	3031	31	1.033333	1752	-1248	-41.6	2150	-850	-28.33333	2409	-591	-19.7
10a	RG	1000	1852	852	85.2	1407	407	40.7	670	-330	-33	1482	482	48.2
3a	RG	1000	1920	920	92	1608	608	60.8	860	-140	-14	1535	535	53.5
1a	RG	2000	2414	414	20.7	2370	370	18.5	1750	-250	-12.5	2241	241	12.05
7a	RG	2000	3442	1442	72.1	3099	1099	54.95	910	-1090	-54.5	2385	385	19.25
13a	RG	3000	3566	566	18.86667	3517	517	17.23333	2330	-670	-22.33333	3228	228	7.6
5a	RG	3000	3374	374	12.46667	1606	-1394	-46.46667	2500	-500	-16.66667	3269	269	8.966667
4a		0	7133	7133		228	228		210	210		700	700	
9a		0	5500	5500		314	314		180	180		658	658	

Mean Values

Description	Known Concentration	TxDOT	Error	% Error	TAMU	Error	% Error	AnaLab	Error	% Error	Chem Lime	Error	% Error
LG	1000	2778	1778	177.8	1060	60	6	1070	70	7	1199.5	199.5	19.95
LG	2000	2969.5	969.5	48.475	1592.5	-407.5	-20.375	2325	325	16.25	1622.5	-377.5	-18.875
LG	3000	3497.5	497.5	16.58333	1677	-1323	-44.1	2990	-10	-0.333333	2453	-547	-18.23333
RG	1000	1886	886	88.6	1507.5	507.5	50.75	765	-235	-23.5	1508.5	508.5	50.85
RG	2000	2928	928	46.4	2734.5	734.5	36.725	1330	-670	-33.5	2313	313	15.65
RG	3000	3470	470	15.66667	2561.5	-438.5	-14.61667	2415	-585	-19.5	3248.5	248.5	8.283333
	0	6316.5	6316.5		271	271		195	195		679	679	

Since duplicate samples were sent to each lab, the lower part of the table lists averages of the two samples analyzed by each lab and represented in the graphs in Figure 4. LG is the coarse-grained gypsum, and RG is the fine-grained gypsum.

concentration (Figures 1, 3, and 4). Figure 2 illustrates that both coarse (LG) and fine-grained (RG) size samples have more error at higher concentrations.

Results of samples sent to the Chemical Lime lab are given in Figure 3. The fine-grained sulfate gave good results, but the coarse-grained material was not fully dissolved.

Based on project experience, the Dallas/Ft. Worth Districts of the Texas Department of Transportation currently do not use calcium-based stabilizers (lime, cement) for sulfate concentrations above 2000 ppm; therefore, the second round of testing was focused on the ability of the labs to detect lower concentrations of sulfate in the manufactured samples.

Average Sulfate Results, Coarse-Grained Gypsum
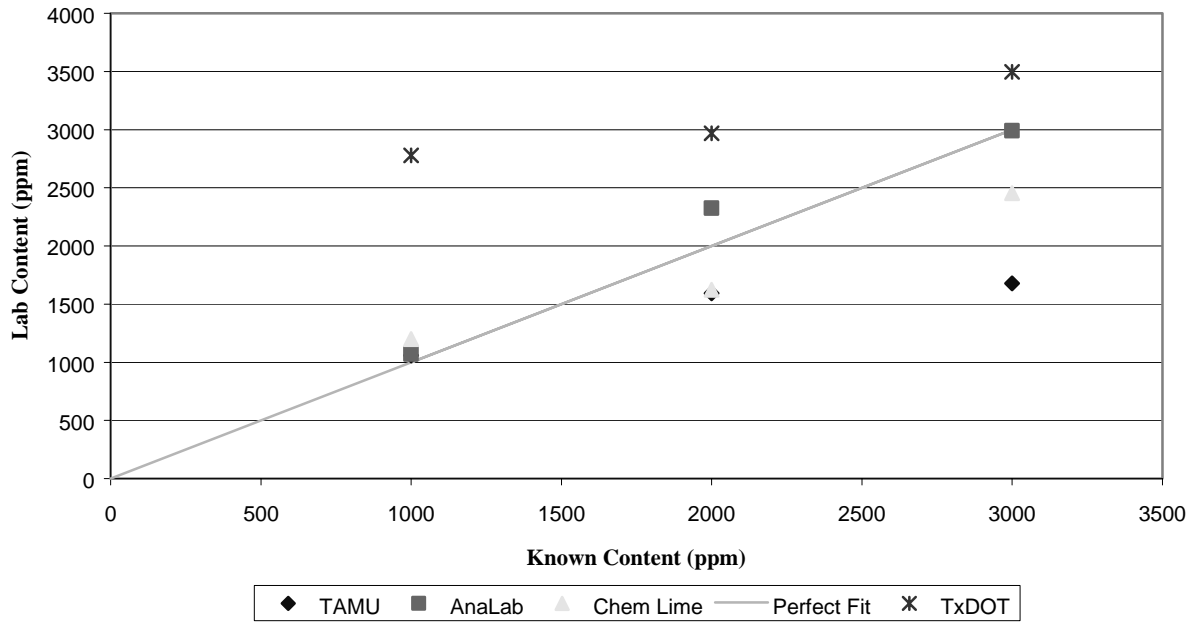
Average Sulfate Results, Fine-Grained Gypsum
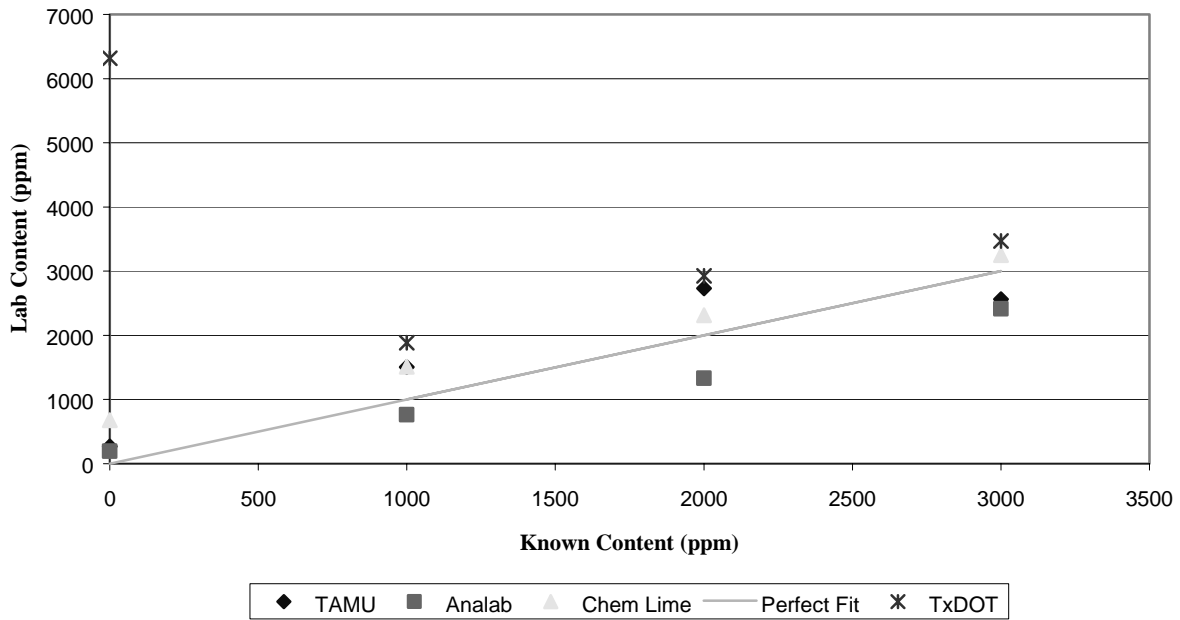
Figure 4. Lab Determined Sulfate Concentrations for Four Laboratories.

Tex-620-J Results
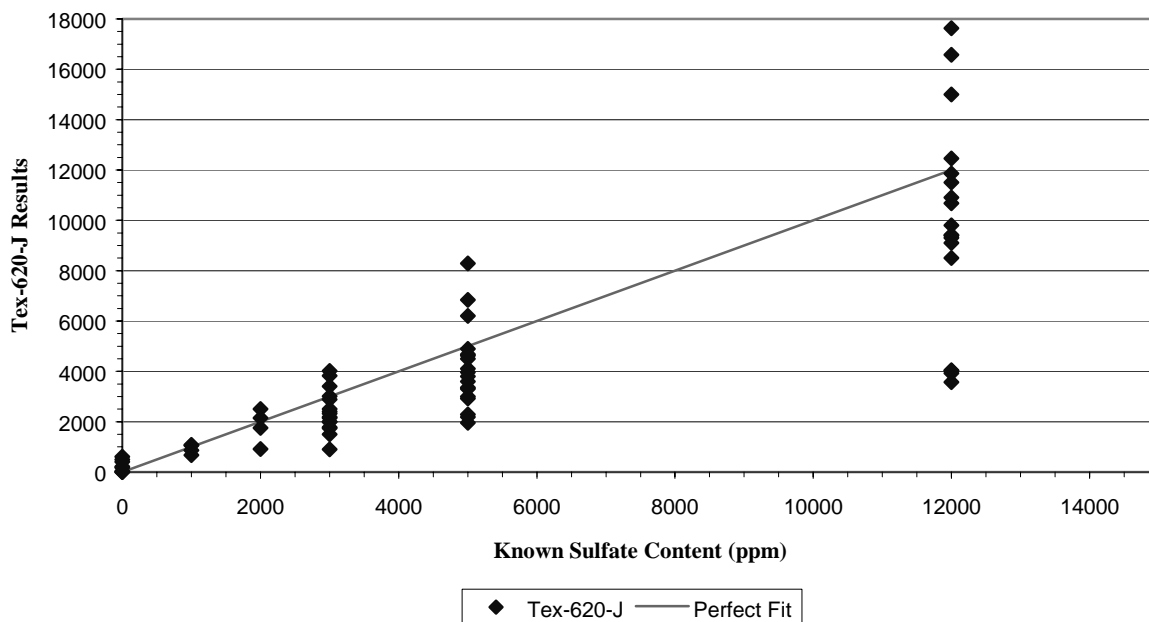
Ion Chromatography Results

Note: Excludes TAMU
above 5000 ppm as dilution
ratio was not sufficient
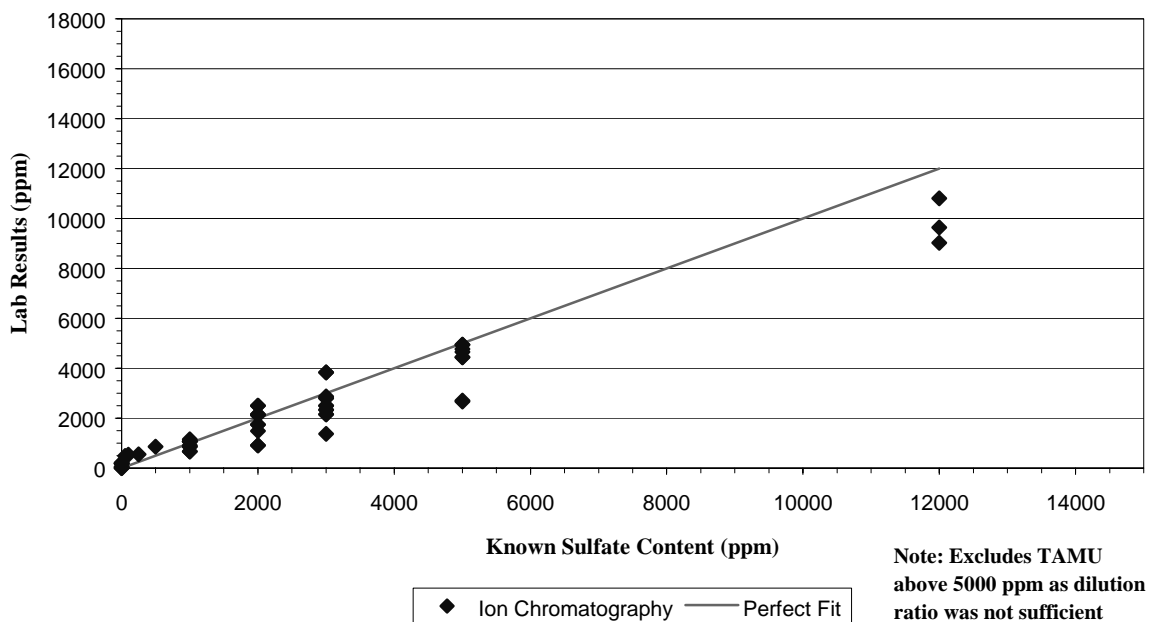
Figure 5. Sulfate Measurements Using Tex-620-J and I.C.

RESULTS FROM FIELD SAMPLES

Twelve samples from U.S. 82 in the Paris District, east of Sherman, Texas, were sent to all four labs for sulfate measurements. Results are given in Table 5, which shows good correlation between low- and high-sulfate levels. Since these are field samples where the true sulfate content is unknown, one can only speculate on the true concentration.

Table 5. Sulfate Content from U.S. 82 Field Samples.

Sample Name	TxDOT Sulfates (ppm)	Ana-Lab Sulfates (ppm)	Chemical Lime Sulfates (ppm)	TAMU Sulfates (ppm)
1596R	82	330	0	33
1597R	160	90	0	28
1603L	130	260	0	30
1612R	172	150	515	61
1612L	103	150	502	60
1613R	1299	1100	2125	2079
1613L	5797	1900	1669	693
1614R	526	380	807	358
1614L	398	470	704	190
1615R	151	100	529	84
1615L	302	230	638	174
1635L	503	810	1025	559

DISCUSSION

Figures 1, 3, and 4 illustrate the difficulty in measuring all of the coarse-grained gypsum. TAMU-EP uses a standard method endorsed by the EPA that calls for passing the sample through a 2 mm sieve. Sulfates in that coarse-size range will not all be dissolved, which results in low, measured concentrations. In the fine-grained sulfates with concentrations of 3000 and 5000 ppm, TAMU-EP obtained very good results. At higher concentrations, TAMU-EP results were extremely low because a 1:5 soil to distilled water dilution ratio was used. Using the solubility data given by Burkart et al. (1999), a saturated solution of gypsum at 25 ºC and a 1:5 dilution ratio would contain 9100 ppm sulfate. This means that 9100 ppm is the maximum

amount of sulfate they could detect, so at concentrations of 12,000 and 50,000 ppm, their numbers were far too low, consistent with a saturated sulfate solution (Table 1).

The Texas Department of Transportation and Chemical Lime labs also had difficulty detecting all of the coarse-grained sulfates due to inefficient pulverization. Ana-Lab obtained better results with the coarse-grained sulfates by using the traditional mortar and pestle to pulverize samples; the samples were then passed through a #50 to #100 sieve, which resulted in more consistent fine-grained particles. This process allowed better dissolution of the sulfate minerals.

As observed in Figure 5, there is a larger spread in the data with the Tex-620-J method than with the Ion Chromatography technique. There are numerous factors that can contribute to such a large variation in results, some of which are listed below:

- Use only (ultrapure) trace metal grade HCl because a sulfate contribution from the acid may result if anything less pure is used.
- Obtaining a finely ground representative sample is critical.
- Remove all of the particulates from the solution to avoid extra weight added from clays and other minerals remaining in the system (Mohamed, May 2002).
- The rate at which barium chloride is added to the solution is critical. It needs to be added slowly to ensure formation of fewer, larger barium sulfate crystals and to reduce the coprecipitation of chloride (de la Camp and Seely, 2002).
- The sample digestion period needs to be at a consistent temperature and length of time to ensure that barium sulfate has ample time to precipitate into crystals large enough to not pass through the filter paper.
- When washing out the salts that form, use only ice cold water because hot water may result in peptization (part of the precipitate reverts to the colloidal form) of the barium sulfate (TAMU, 2002).
- Care must be taken to not lose sample when burning off filter paper with the meeker burner (Coward, 2002).
- It is critical that the analytical balances are calibrated frequently. Some private labs calibrate balances on a daily basis (Mohamed, 2002).

- Foreign anions (nitrate and chlorate) and cations (ferric iron, calcium, and alkali metals) can be coprecipitated either with the barium sulfate or as substitutional impurities within the barium sulfate crystals. This can cause a substantial error if these ions are present in large concentrations (de la Camp and Seely, 2002).

TAMU-EP switched from gravimetric analysis to Ion Chromatography due to the time requirement and all of the possible sources of error with gravimetric analysis; Dr. Abdul-Mehdi (2002) stated that there are no interferences with the Ion Chromatography technique for sulfates.

Evaluation of the Texas Department of Transportation Test Method Tex-620-J for determining soluble sulfates has been a challenge.

Both the Tex-620-J and Ion Chromatography technique appear to give reasonably accurate results with repeated testing; i.e., the average of multiple test results is reasonably close to the true sulfate content (see previous Figure 5). For Tex-620-J, the average test result was approximately 8 percent below the true value for 1000 and 2000 ppm and approximately 18 percent below the true value for 3000, 5000, and 12,000 ppm. Being a gravimetric technique, the errors in Tex-620-J could be due to incomplete dissolution of the gypsum, loss of sample during the test procedure, and other operational or equipment errors covered above.

Average results from Ion Chromatography were low by approximately 6 percent, 9 percent, 12 percent, 16 percent, and 18 percent for 1000, 2000, 3000, 5000, and 12,000 ppm, respectively. This is a decent improvement in accuracy when compared to Tex-620-J. To thoroughly evaluate Ion Chromatography versus Tex-620-J, two quantitative analyses were performed: 1) What is the required sample size to define, within a specified range, the 95 percent confidence interval for the true sulfate content, and 2) What are the precision (repeatability and reproducibility) statistics for each test method?

The appropriate sample size needed to define the 95 percent confidence interval for the true sulfate content within plus or minus a certain range, E, is defined as (Jarrell, 1994):

$$n = \left(\frac{1.96S}{E} \right)^2$$

where n = sample size (number of tests required)

S = standard deviation of test results

E = specified acceptable maximum error

This means if n tests are conducted, there is 95 percent confidence that the true sulfate content is within $\pm E$ ppm of the average test result. The appropriate sample size is dependent on the expected dispersion of test outcomes and the specified allowable range, E. For this analysis, the standard deviation of test results was the estimator for S, and values of ± 10 percent, ± 20 percent, and ± 30 percent were used for E. Table 6 shows the needed number of samples. In general, fewer tests are required with Ion Chromatography, and in no cases were fewer tests required with Tex-620-J. At lower levels of allowable error (i.e., greater desired accuracy), Ion Chromatography offers a sizeable reduction in testing requirements. At higher levels of allowable error of ± 30 percent, there is not much of an appreciable difference in testing requirements between the two techniques until the true sulfate content is at least 5000 ppm.

Table 6. Number of Tests Needed for Tex-620-J vs Ion Chromatography for Specified Allowable Error.

True SO$_4$ Content (ppm)	$\pm 10\%$ Tex-620-J	$\pm 10\%$ Ion Chrom.	$\pm 20\%$ Tex-620-J	$\pm 20\%$ Ion Chrom.	$\pm 30\%$ Tex-620-J	$\pm 30\%$ Ion Chrom.
1000	15	12	4	3	2	2
2000	45	33	11	9	5	4
3000	31	21	8	6	4	3
5000	43	14	11	4	5	2
12,000	46	7*	12	2*	6	1*

Estimate based on historic coefficient of variation due to lack of sample size at this concentration.

The two sulfate test methods can also be compared by evaluating their precision. Precision is defined as within lab repeatability and between lab reproducibility. The data collected were processed using American Society of Testing and Materials (ASTM) E 691 to develop the precision statistics. A more precise test method is indicated by a lower precision statistic. The precision statistics presented in Table 7 (for Test Method Tex-620-J) and Table 8 (for Ion Chromatography) indicate that Ion Chromatography is a more precise test. For material that is 3000 ppm, the only sulfate content at which sufficient data were available for comparison between the test methods, the repeatability and reproducibility limits for Ion Chromatography were approximately half that of Tex-620-J. For a given homogeneous material, the absolute value of the difference between any two test results at the same lab is expected to be less than the

repeatability limit, *r*, for 95 percent of all observations. Similarly, the absolute value of the difference between any two sulfate content results from different labs, on the same material, is expected to be less than the reproducibility limit, *R*, for 95 percent of all observations. Further discussion of precision is available in ASTM E 177.

Table 7. Precision Statistics for Test Method Tex-620-J.

Material	$\overline{X}$	S_r	S_R	*r*	*R*
3000 ppm	2687	828	828	2318	2318
5000 ppm	4195	1617	1617	4528	4528
12,000 ppm	9700	2985	3667	8358	10,268

Note: This procedure calls for results from six labs using the same technique, however, this analysis is from only 2 labs since very few perform this test.

Table 8. Precision Statistics for Ion Chromatography.

Material	$\overline{X}$	S_r	S_R	*r*	*R*
1000 ppm	1319	145	169	406	473
2000 ppm	2065	273	365	764	1022
3000 ppm	2485	392	468	1098	1310

Note: This procedure calls for results from six labs using the same technique, however, this analysis is from only 2 labs.

CONCLUSIONS

- Overall, Ion Chromatography is superior to the TxDOT Test Method Tex-620-J gravimetric technique. It is more accurate and repeatable, requires less time, personnel are not exposed to toxic chemicals, there is less interference from other constituents in the soil, and the method is not as sensitive to individual operator biases; however, the initial cost of the equipment is substantial.

- Detection of coarse-grained sulfates is dependent upon efficiency of pulverization.

- At higher concentrations of sulfate, a larger number of tests is required to obtain an accurate sulfate concentration in the soil.

- The TxDOT Test Method TEX-620-J is valid, but there are multiple steps in the analysis where error may be introduced by operation interpretation creating a large standard deviation of test results. See recommendations in Chapter 5.

CHAPTER 3

SULFATE CONTENT DETERMINATION – RAPID FIELD TEST

INTRODUCTION

The purpose of this portion of the project was to pinpoint a technique that could be used in the field to identify potential problems due to soils with high sulfate/sulfide concentrations. Since many of the sulfate problems are localized within a small zone (Figure 6) a rapid field test needs to be developed to identify these potential problem areas before or during construction. The ink pen (Figure 6) is parallel to one of the filled fractures and shows how localized these sulfate seams can be. To be useful in the field, the technique should be simple to run and yield rapid results while the equipment should be portable and durable enough to withstand field-operating conditions.

Figure 6. Gypsum Filled Fractures in the Eagle Ford Formation on U.S. 82.

An extensive literature review was performed to identify ways that sulfate and sulfide testing is performed in soil environments. There are numerous techniques available to locate sulfates, but most of them generally involve expensive and cumbersome equipment that is not practical in a field environment. The researchers identified four techniques that hold promise for working in the field. There is a scarcity of information available for sulfide determination useful in a field environment, so only one technique was evaluated.

BACKGROUND

Bower and Huss (1948) published a paper using conductivity to measure sulfate content in soils. Their procedure was to mix 10 to 20 g of air-dried soil with distilled water and agitate continuously for 30 minutes, which dissolved the gypsum. Acetone was then added to re-precipitate the gypsum. The re-precipitated gypsum was washed to remove salts (NaCl, etc.) and then was re-dissolved in 40 ml of distilled water. The conductivity was then measured and compared to a calibration curve to determine gypsum concentration in the soil.

The test, developed by Bower and Huss (1948), was adapted by the USDA as a qualitative field test. It involved adding distilled water to 10 to 20 g of air-dried soil in an 8 oz bottle and hand shaking six times at 15-minute intervals. The extract was filtered through medium porosity filter paper. A test tube contained 5 ml of the extract, and an equal volume of acetone was added to the extract. If a milky precipitate formed, then gypsum was present in the soil.

The Department of Soil and Crop Sciences at Texas A&M University is also home to many scientists with the Texas Agricultural Experiment Station (TAES), so the researchers contacted them about how TAES analyzes for sulfates in soils. They use the Bower and Huss (1948) technique to measure gypsum content in soils. One limitation of the technique is that it only measures gypsum, not total sulfate, which may yield optimistic results. However, if calcite is present in the soil then the gypsum content measured may be too high because calcite will dissolve in distilled water and calcium ions will be available to react with other dissolved sulfates to form gypsum. For our purposes, this limitation is actually an advantage because we are interested in total sulfate and not just gypsum. The Eagle Ford Formation, which causes many of Texas' sulfate heave problems (Burkart et al., 1999), possesses abundant calcite in the form of limestone. Therefore, other sulfate minerals that may be in the soil can form gypsum

when acetone is added because of the excess calcium that is supplied by the dissolution of limestone. This will provide a better estimate of total sulfate.

Bredenkamp and Lytton (1995) proposed a simple field test to detect sulfates which involved mixing the soil with distilled water and measuring the conductivity of the solution. They hypothesized that high electrical conductivity would be due to the presence of soluble sulfates. Researchers at TTI noted the following limitations:

- The current protocol calls for measurements to be taken immediately after mixing; this could lead to *underestimation* of the problem with soils containing large sulfate crystals which take longer to dissolve.
- Other salts may be present in the soil. This increases the conductivity in addition to the sulfates, which will lead to *overestimation* of the problem.
- As discussed previously, sulfide minerals may be present in the soil and not be detected by this technique, which will lead to an *underestimation* of the problem.
- A pH and temperature at which the test should be performed was not specified. Gypsum is more soluble at low pH and low temperatures.

Two other rapid field techniques (colorimetric and barium chloride test) for sulfates were identified by perusing the environmental testing and water quality sales literature. These tests were designed for measuring sulfate concentrations in natural waters, but they may be adapted to soils by dilution and filtration. They operate on the principles of colorimetry (measure degree of absorption of light transmitted through the sample by human eye) or spectrophotometry (when an instrument measures the light transmitted).

The lone sulfide technique is a "spot test" where one of the reactants is used in the form of a solution. McClellan et al. (1998) identified one simple field test for sulfide sulfur from the general chemical literature. The test involves adding solutions composed of sodium azide and iodine to a sample that contains sulfides/pyrite. The sulfides do not participate in the reaction, but they catalyze a reaction between sodium azide (NaN_3) and iodine (I_2) which evolves nitrogen (N_2) gas. The gas evolution can be observed as bubbles forming on the soil sample containing sulfides (Feigl, 1958).

CONDUCTIVITY THEORY

The conductivity of a solution 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 with which each ion travels through the solution (Robinson, 1970).

Robinson (1970) lists six factors which influence the speed of the ion:

- the solvent (water or organic),
- the applied voltage,
- size of ion (larger ions less mobile),
- nature of the ion (if it becomes hydrated, then the effective size is increased),
- viscosity of solvent, and
- temperature of solvent.

The conductivity of a solution is the sum of the conductivities of the ions present; therefore, it cannot distinguish between different types of ions. At higher concentrations the ions may form some un-ionized molecules which will reduce the conductivity (Robinson, 1970).

COLORIMETRY THEORY

The theory behind colorimetry hinges on Beer's law:

$$A = abc = \log (I_o/I_1)$$

A = absorbance

a = absorptivity of the sample

b = optical path length

c = concentration

I_o = intensity of light entering solution

I_1 = intensity of light emerging from solution

There is a linear relationship between absorbance and concentration of a solution if the optical path length and wavelength of radiation remain constant. By measuring the ratio I_1/I_o absorbance can be measured, therefore concentration can be calculated. Beer's law usually holds at low concentrations, but deviations are common at concentrations above ≈ 200 ppm (Robinson, 1970).

TESTING PROCEDURE

Based on the criteria of being quick, portable, and easy to perform, four rapid field sulfate tests were identified, and one rapid field sulfide test was targeted for inclusion in this phase of the project. The four sulfate tests include the conductivity test proposed by Bredenkamp and Lytton (1995), the modified Bower and Huss (1948) Acetone Test proposed by the USDA, the Barium Chloride Test, and the Colorimetry Test. The lone sulfide test that was evaluated was one proposed by McClellan et al. (1998).

CONDUCTIVITY TEST

This phase of the research focused on answering questions regarding the conductivity test proposed by Bredenkamp and Lytton (1995). Specific questions included:

- Was distilled water an efficient solvent for sulfates?
- Did sulfate grain size impact conductivity measurements?
- How could the test be sped up (i.e., pulverization, different solvents)?
- Was the test applicable to natural soil environments?

To answer these questions researchers developed a series of experiments using the same lab-created "manufactured soils" samples that were shipped to the four laboratories for quantitative sulfate analysis (Chapter 1, Tables 1 and 2).

All of the conductivity measurements performed in the lab and reported in the results section were performed on an Accumet™ AR50 pH/Conductivity meter (Figure 7) equipped with an Accumet (13-620-155) glass-bodied conductivity cell with a cell constant of 1.0 cm^{-1}; an external temperature probe was used for temperature compensation. Measurements of pH were made using an Accu-pHast™ (13-620-296) glass-bodied combination electrode. The pH electrode is on the left, and the conductivity cell and external temperature probe are located on the right side of the instrument. Conductivity measurements made in the field were performed with an Omega PHH-80™, pH/Conductivity meter.

Figure 7. Accumet Model AR50 pH/Conductivity Meter.

All of the samples were evaluated under identical conditions to ensure that the results being compared were not due to procedural differences. The procedures are outlined below:

(1)   Calibrate conductivity and pH meter per manufacturer's instructions. Estimate conductivity and pH, and calibrate with standards close to those estimates. For example, a carbonate rich sample will be basic, so standardize pH with a pH 10 standard in addition to the pH 7 standard.

(2)   Measure 5 g to the nearest 0.1 g of air-dried soil into a 125 ml (HDPE) Nalgene brand bottle.

(3)   Measure 100 g to the nearest 0.1 g of double-distilled water into the bottle.

(4)   Place the samples onto a Burrell™ Model 75 wrist-action shaker (Figure 8) and shake on the maximum setting for 1 minute.

(5) Remove the samples from the shaker and immediately take conductivity and pH measurements with the Accumet Model AR50 pH/Conductivity Meter.

(6) After 50 minutes the samples were put on the wrist-action shaker for a period of 10 minutes, and were shaken at the maximum setting.

(7) Remove the samples from the shaker and immediately take conductivity and pH measurements.

(8) This procedure was followed every hour up to 8 hours.

(9) The next day samples were placed on the wrist action shaker and shaken at the maximum setting for 10 minutes, one time in the morning and one time in the afternoon.

(10) Conductivity and pH were measured immediately after shaking.

(11) This procedure continued up to several days until conductivity had stabilized at a constant value.

Figure 8. Burrell Model 75 Shaker for Conductivity Measurements.
Note: The 125 ml Nalgene Bottles Attached to the Shaker.

ACETONE TEST

This test was originally a quantitative technique developed by Bower and Huss (1948) and modified by the USDA to be a rapid and inexpensive field technique for detecting sulfates in soil (Figure 9). The procedures used for this technique are as follows:

(1) Add 10 g of air dry soil to a 250 ml (HDPE) Nalgene centrifuge bottle.

(2) Add 100 ml of double-distilled water to the 250 ml centrifuge bottle for a 1:10 ratio of soil to solvent.

(3) Shake the sample for 15 minutes with the Burrell Model 75 wrist-action shaker at the maximum shaking intensity.

(4) Filter the extract with a Whatman™ #42, 5-inch diameter filter paper into 250 ml beakers. Centrifugation may be necessary with fine-grained soils to remove all particulates from suspension.

(5) Place approximately 5 ml extract into 40 ml glass centrifuge tube (Figure 10).

(6) Add approximately 5 ml acetone to the solution in the centrifuge tube and agitate. After 5 to 10 minutes a cloudy suspension or a white precipitate will be observed if gypsum is present (Figure 10). This test will indicate the presence of gypsum, but it is not quantitative. The sample on the right contains sulfate, and the sample on the left does not.

Figure 9. Equipment Required for Acetone Field Test Kit.
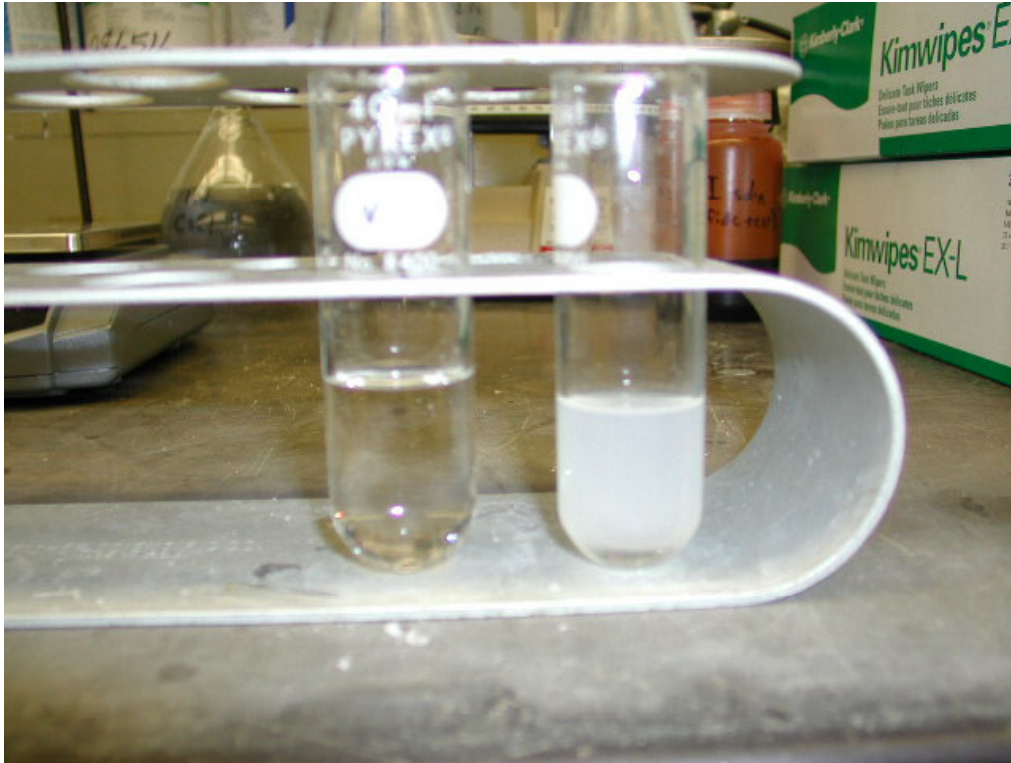
Figure 10. Filtrate of Samples from U.S. 82, Sherman, Texas.

BARIUM CHLORIDE TEST

The barium chloride test is a true colorimetric technique because judgment of sulfate concentration is based upon comparison with a chart (Figure 11). This test is somewhat subjective since the human eye is used to judge the concentration. The procedure for this test is written with respect to the equipment provided with the soil testing kit and is included in the kit. The test procedure is as follows:

(1) Fill test tube to mark with Universal Extracting Solution (3 percent acetic acid and 10 percent sodium acetate with distilled water).

(2) Use orange soil measure to add one level measure of soil to test tube.

(3) Cap test tube and shake for one minute.

(4) Put filter paper in funnel and pour extract solution in funnel and collect filtrate.

(5) Use transfer pipette to add five drops of clear filtrate to turbidity vial.

(6) Add one drop of Sulfate Test Solution (0.2 percent HCl and 5 percent $BaCl_2 \cdot 2H_2O$), and gently swirl to mix.

(7) Lay sulfate color chart under neutral light. Hold turbidity vial 0.5 inch above black strip in middle of chart. Look down through the turbid sample. Match sample turbidity to a turbidity standard.

(8) Record as ppm sulfate.

Figure 11. Equipment Required for Barium Chloride Field Test Kit.

COLORIMETRY/SPECTROPHOTOMETRY

This technique employs an AQUAfast™ II Colorimeter/Spectrophotometer to measure the amount of light transmitted through the sample (Figure 12). This particular unit is equipped with a light-emitting diode (LED) for the light source because the LED's wavelength ($\lambda = 528$ nm) is stable with shifting temperatures. The procedures outlined in the manual, with a modification for soils, are as follows:

(1) Measure 5 g of air-dried soil into a 125 ml Nalgene bottle, and add 100 ml double-distilled water.

(2) Shake on the Burrell wrist-action shaker for 15 minutes at maximum speed.

(3) Remove from shaker and filter with Whatman #42, 9.0 cm, filter paper into a 250 ml beaker. Centrifugation may be necessary with fine-grained soils to remove all particulates from suspension.

(4) Put on latex gloves.

(5) Fill sample vial with filtrate to the 10 ml mark and wipe vial clean with Kimwipes or equivalent delicate task wipe.

(6) Switch the unit to "ON."

(7) Press the MODE key until the desired method is displayed.

(8) Place the sample vial into the sample chamber with the Δ vial mark aligned with the Δ housing mark.

(9) Press the ZERO/TEST key. The method symbol flashes for approximately 3 seconds and confirms zero calibration.

(10) After zero calibration, remove the vial from the sample chamber.

(11) Add sulfate tablet to vial without touching the tablet with hands and crush immediately with white plastic rod provided. Always be consistent with the time and amount of crushing.

(12) Cap vial and immediately place in the sample chamber with the Δ marks aligned.

(13) Press the ZERO/TEST key. The method symbol flashes for approximately 3 seconds, and the result appears in the display. Take a minimum of three readings and average.

- This test will only read concentrations from 5-200 mg/l. If (÷**Err**) message appears, then the measuring range has been exceeded or there is excessive turbidity. This will require diluting the sample with more double-distilled water and measuring until the message disappears and there is a numerical answer. If (-**Err**) message appears, then result is below the measuring range.

Figure 12. Equipment Required for Colorimetry/Spectrophotometry Field Test Kit.

SULFIDE TEST

As discussed previously, sulfides weather to produce sulfate in near-surface environments. A simple field test was described by McClellan et al. (1998), which requires observation of gas bubbles evolved from the soil sample (Figure 13). A solution is prepared by dissolving 3 g of NaN_3 (sodium trinitride/sodium azide) in 100 ml of 0.1 N iodine solution. Sodium azide and iodine do not react with each other under normal conditions, but when sulfide is added to the solution, nitrogen gas is evolved. The following reaction theoretically takes place:

$$2NaN_3 + I_2 \rightarrow 2NaI + 3N_2$$
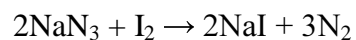
The sulfide catalyzes the above reaction. The sodium azide reacts with the iodine, in the presence of sulfide, to form sodium iodide and nitrogen gas (Feigl, 1958). The nitrogen gas is observed as bubbles. This reaction does not occur if sulfides are not present in the sample. Procedures for running this test are very simple and listed below:

(1) Mix sodium azide and iodine solution in the proportions listed above.

(2) Put a small sample of air-dried soil or crushed stone into porcelain spot plate (Figure 13).

(3) Add 1 to 2 ml of iodine mix to spot plate with a disposable pipet.

(4) Look for bubbles forming in sample. If bubbles form, then there is sulfide in the sample. A pocket magnifier or binocular microscope may be required to see the bubbles. (**Note:** It takes some experience observing the evolution of N_2 gas.)

Figure 13. Equipment Required for the Sulfide Test Kit.

RESULTS WITH MANUFACTURED SOILS

Results were obtained from each of the four sulfate tests and the lone sulfide test using "manufactured soil" samples, which emphasize evaluation of the conductivity test proposed by Bredenkamp and Lytton (1995). Table 9 shows conductivity measurements performed on the manufactured samples by TTI and TxDOT (Jim Kern, Dallas District Lab).

**Table 9. Comparison of Conductivity Results from TxDOT and TTI.**

Sample	Comment	Known Sulfate Concentration (ppm)	TxDOT Conductivity (microsiemens)	TTI Initial Conductivity (microsiemens)	TTI Final Conductivity (microsiemens)
EX 5		0	11	29	123.4
EX 12		0	13	26.7	112
EX 9	coarse-grained	3000	38	81	409
EX 22	coarse-grained	3000	44	25.4	217
EX 1	fine-grained	3000	317	328	421
EX 15	fine-grained	3000	325	318	375
EX 6	anhydrite	5000	505	382	593
EX 17	anhydrite	5000	503	517	577
EX 21	coarse-grained	5000	49	48.6	499
EX 8	coarse-grained	5000	38	76.8	528
EX 2	fine-grained	5000	452	474	576
EX 13	fine-grained	5000	459	599	666
EX 7	anhydrite	12000	1029	958	
EX 18	anhydrite	12000	941	579	1031
EX 11	coarse-grained	12000	72	140	1064
EX 20	coarse-grained	12000	92	111	966
EX 3	fine-grained	12000	1070	1036	1109
EX 16	fine-grained	12000	950	1033	1036
EX 10	coarse-grained	50000	412	338.7	2258
EX 19	coarse-grained	50000	284	352	2222
EX 4	fine-grained	50000	2200	2183	2311
EX 14	fine-grained	50000	2400	2187	2279

Note: All measurements were made on unpulverized samples. Samples labeled coarse-grained are gypsum that passes #10 and are retained on the #40 sieve. Fine-grained samples are all gypsum and all pass the #200 sieve.

These samples were analyzed as received and not pulverized to evaluate the effect of gypsum grain size on conductivity measurements. TxDOT results compared very well with TTI initial conductivity measurements, however, there is no specification on the amount of shaking in the procedure that TxDOT follows. The same procedure was followed at TTI, with the exception of specifying a shaking time of 1 minute on a mechanical shaker before taking the initial conductivity measurements. TxDOT made only initial measurements whereas TTI performed measurements over time (Figure 14). The last column in Table 9 consists of final conductivity measurements taken at TTI. Conductivity and pH measurements were taken every hour for the first 8 hours followed by measurements two times a day up to 500 hours.

The top graph in Figure 14 shows how conductivity increases over time with the samples containing coarse-grained gypsum. Note that the samples with lower concentrations of gypsum result in lower conductivity values than samples bearing higher concentrations of gypsum. The bottom graph illustrates how the samples bearing fine-grained gypsum reach equilibrium much more rapidly than the coarse-grained samples.

Conductivity vs. Time for Samples Treated with Coarse-Grained Gypsum
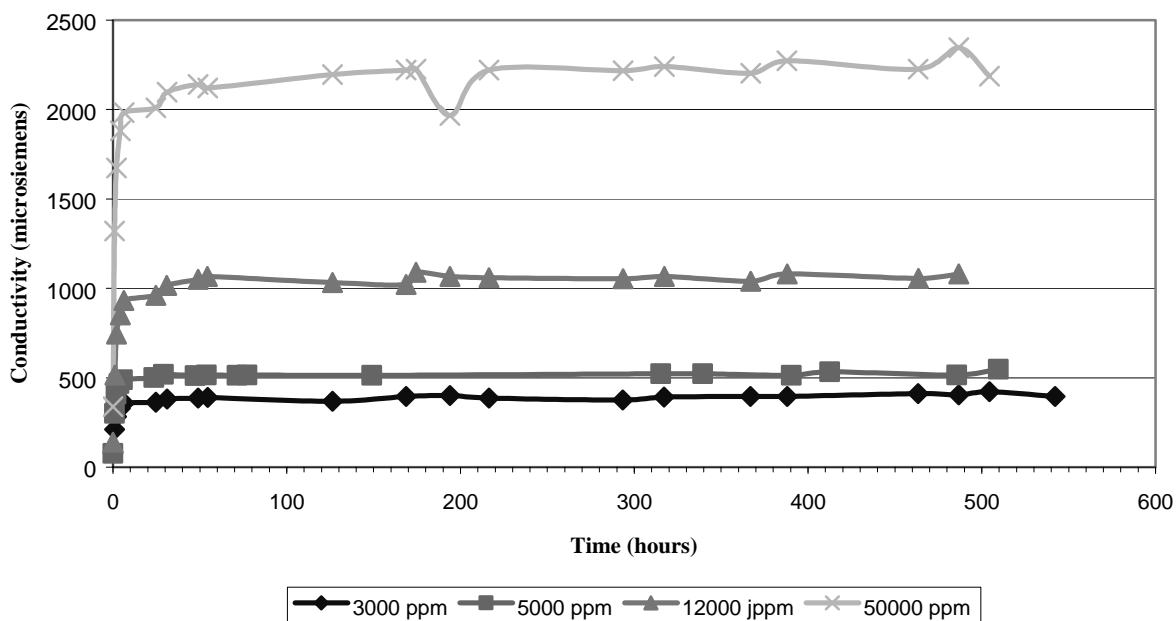
Conductivity vs. Time for Samples Treated with Fine-Grained and Coarse-Grained Gypsum
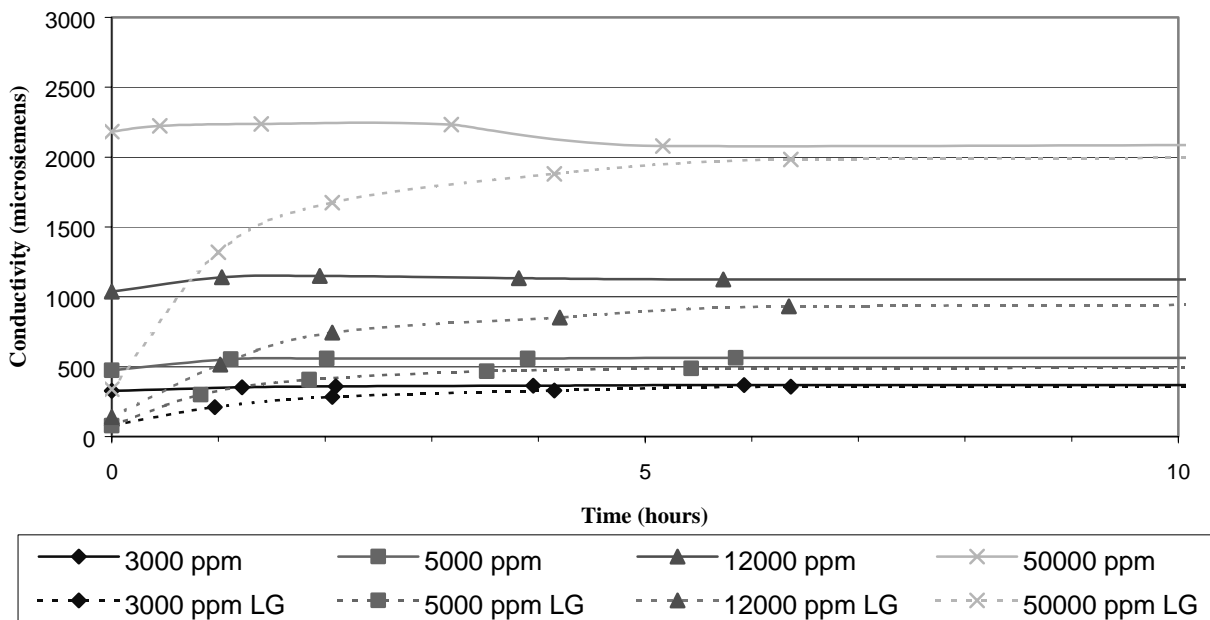
Figure 14. Conductivity vs Time Measurements for Treated Samples.

To measure the effectiveness of pulverization on reducing the time required for conductivity to reach the maximum/equilibrium value, samples of coarse-grained gypsum at various concentrations were pulverized with a mortar and pestle and passed through a #200 sieve before mixing with double-distilled water and measuring conductivity. Figure 15 is a graph of these data for gypsum retained on the #40 sieve and passing the #10 sieve at a concentration of 3000 ppm sulfate. The triangles represent the pulverized gypsum that was passed through the #200 sieve. It is clear from this graph that pulverization helps achieve equilibrium much more rapidly.

Coarse-Grained Gypsum (3000 ppm)
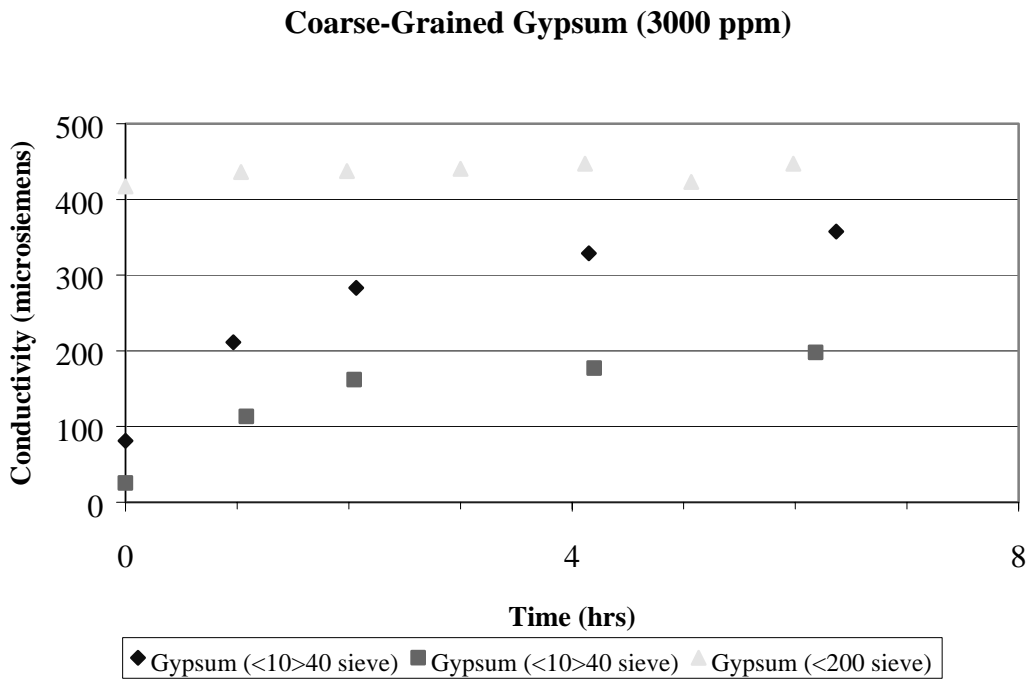
Figure 15. Effect of Pulverizing Coarse-Grained Gypsum.

To determine the effect of pH on conductivity measurements, manufactured soils with concentrations of 0, 1000, 2000, 3000, 5000, and 12,000 ppm sulfate were treated with pyrite (pH=4.0-4.5) or calcite (pH=8.5-9.0). Pyrite (FeS_2) and calcite ($CaCO_3$) were used because they are natural minerals that occur in rocks from Texas and naturally make the soil more acidic and basic, respectively. Sposito (1989) listed the pH range for naturally occurring soils between a pH of 3 to 9.5 so our lab-generated specimens fall within the upper and lower limits of this range. As illustrated in Figure 16, both high and low pH increase the conductivity over the neutral pH sample (triangles). Figure 17 shows that both low and high pH samples follow the same trend observed with the neutral pH samples. When the sulfate content increases, the conductivity increases as well.

Calcite vs. Pyrite at 12,000 ppm Sulfate Content
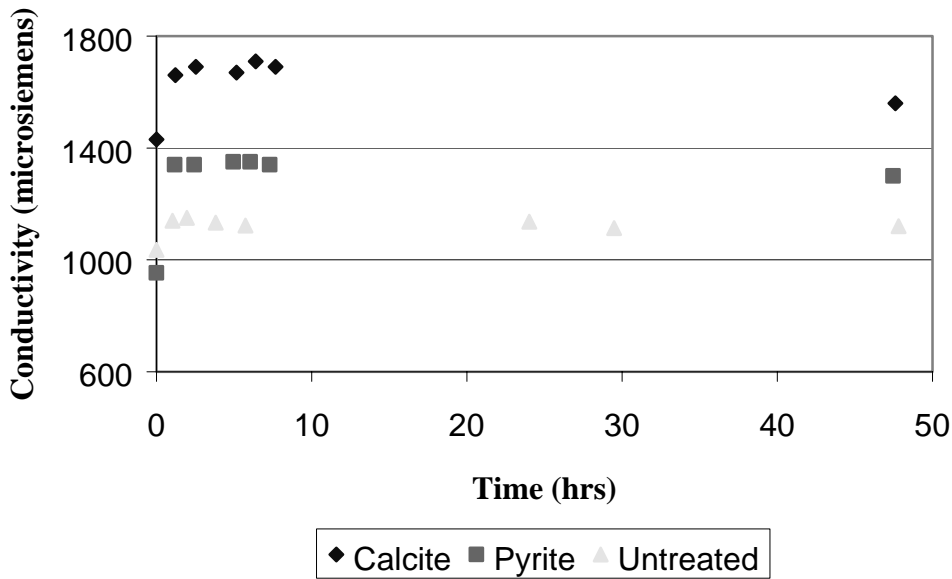
Figure 16. Effect of pH on Conductivity Measurements.

Pyrite (low pH)

0 ppm Sulfate ■ 1000 ppm ▲ 2000 ppm ✕ 3000 ppm ✱ 5000 ppm ● 12,000 ppm

Calcite (high pH)

◆ 0 ppm Sulfate ■ 1000 ppm ▲ 2000 ppm ✕ 3000 ppm ✱ 5000 ppm ● 12,000 ppm
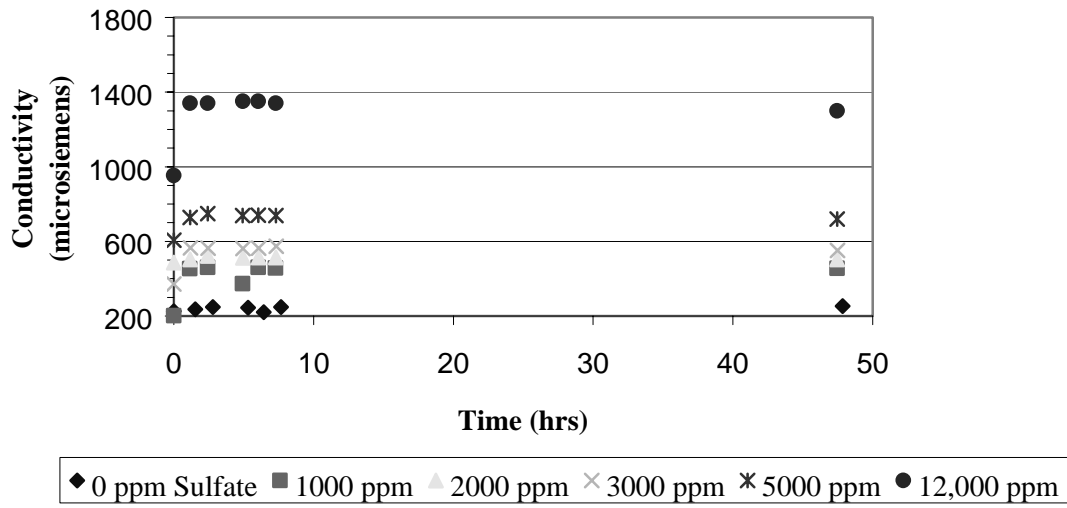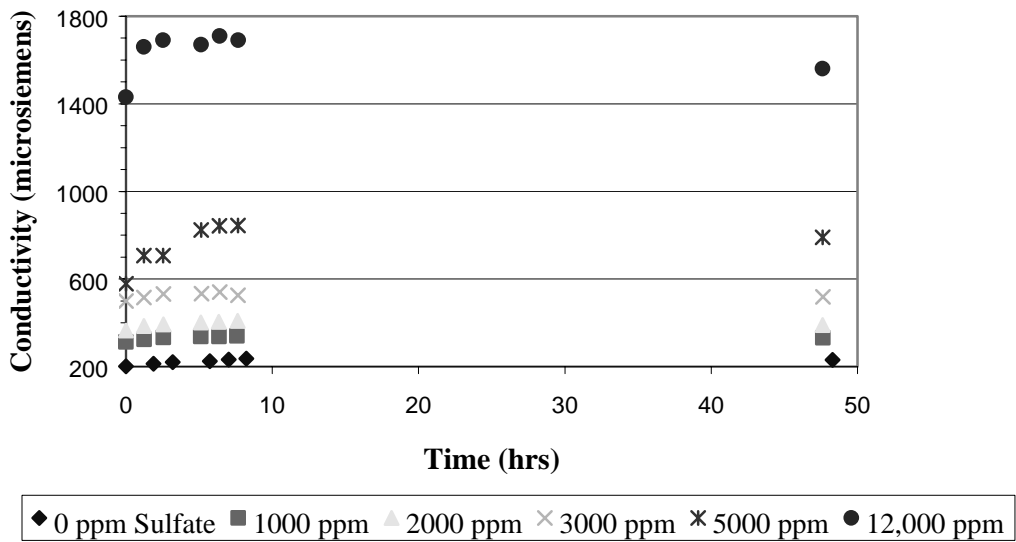
Figure 17. Trends Observed Using Low and High pH Samples.

Conductivity of sulfate standards was measured to generate a calibration curve (Figure 18) showing conductivity versus sulfate content. The manufactured soils correlated well with the calibration curve.

**Lab Soils Conductivity with
Conductivity Calibration Curve**
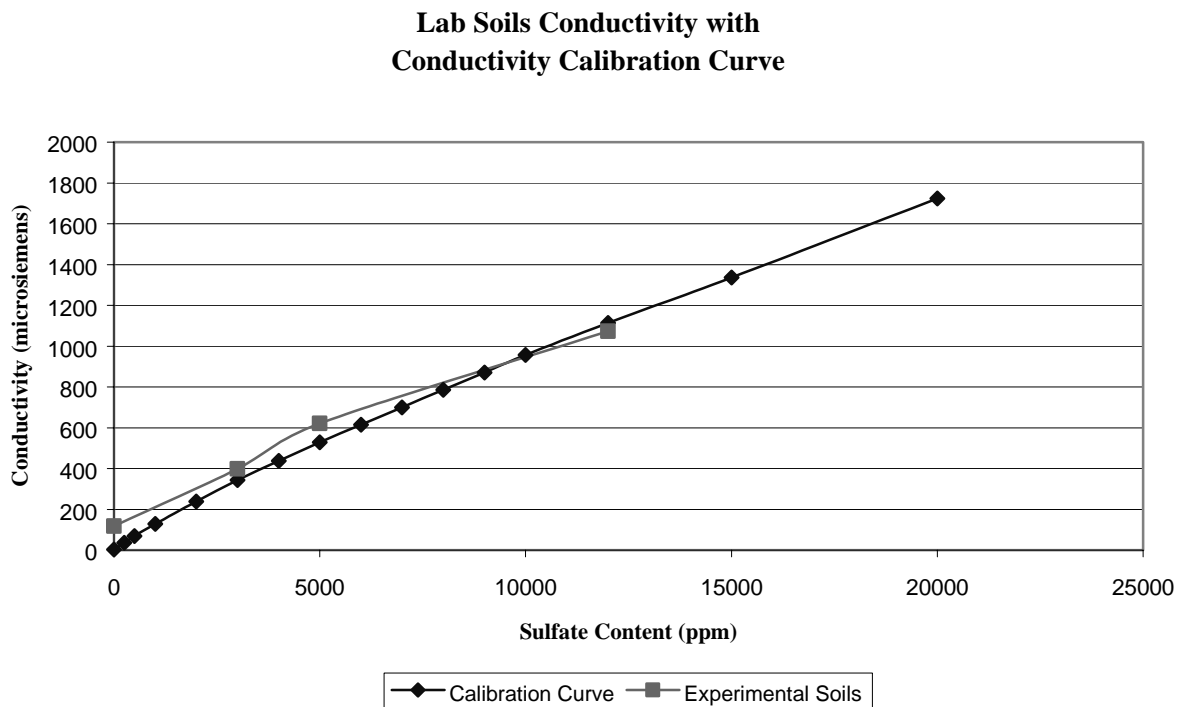
Figure 18. Correlation of Experimental Soil Concentrations to a Calibration Curve.

RESULTS WITH FIELD SAMPLES

Tests on natural soils from three projects in different parts of the state judged the adequacy of the rapid field techniques. The first sample was from the Childress District on IH40, east of Shamrock, Texas, where they experienced a heave problem (Table 10). The second sample was from the Fort Worth District. The third sample was from a new construction project in the Paris District on U.S. 82, east of Sherman, Texas.

Table 10. Rapid Field Test Results of Soils from U.S. 82.

Sample Name	Acetone (1:20 dilution)	Sulfide	Colorimeter SO_4^{-2} (ppm)	Initial Conductivity (µS)	Final Conductivity (µS)
1596R	N.D.	Very Little	0	40	55
1597R	N.D.	Very Little	0	40	59
1603L	N.D.	N.D.	160	40	53
1612R	N.D.	N.D.	0	40	94
1612L	N.D.	N.D.	180	40	61
1613R	N.D.	N.D.	1800	50	219
1613L	Precipitate	N.D.	3960	20	178
1614R	N.D.	N.D.	780	30	217
1614L	N.D.	N.D.	760	20	152
1615R	N.D.	Minor	100	30	76
1615L	N.D.	Minor	100	30	68
1635L	Precipitate	N.D.	>4000	50	403

Note: Barium chloride test was not performed on these samples due to the high carbonate content of these soils. N.D. = not detected; Precipitate = precipitate of sulfates in the sample.

DISCUSSION

Conductivity measurements are easily performed and consistent results can be obtained between different laboratories (Table 9). Comparison of initial conductivity measurements made by TTI with TxDOT results show a very good correlation. As discussed in the background, all of the factors affecting conductivity can be easily controlled so the possible error is minimal, as long as the same techniques are followed. Conductivity measurements are affected by a dirty or improperly calibrated cell, or a malfunctioning meter. Temperature fluctuations can also cause conductivity measurements to drift and not yield a stable reading; therefore, this is a good reason for not cooling the sample below ambient temperature to increase gypsum solubility. The conductivity reading will not stabilize until the temperature of the solution reaches equilibrium with the air temperature.

In a search for obtaining faster dissolution of sulfates for conductivity measurements, an acidic and basic solvent was tried to enhance the sulfate solubility. A lime-saturated solution (pH ≈ 12.4) and a 3 percent, by volume, acetic acid solution was used as the high pH and low

pH solvents, respectively. High ionic concentrations due to the presence of hydrogen and hydroxyl ions resulted in lower conductivity measurements because complexes formed in the solution which decreased the conductivity. Therefore, using a high pH or low pH solvent to enhance sulfate solubility is not advisable. The acidic and basic solvent could also lead to solubilizing other minerals, which would contribute to the conductivity. For instance, at high pH's (above ≈ 10.5) both silica and alumina-bearing phases are soluble (Krauskopf, 1967). These ions are liberated from the soil and become a part of the solution which will add to the conductivity until the concentration is high enough for molecules to start forming, thus reducing the conductivity. The lesson learned here is that use of high and low pH solvents only complicates conductivity determinations.

The researchers believe that conductivity measurements with double-distilled water over an extended time period are beneficial for determining relative grain size of the soluble sulfates. Figure 19 is a group of hypothetical curves generated showing conductivity profiles for three different sulfate scenarios, which illustrate how conductivity measurements over time without pulverization may give an indication of sulfate grain size and could affect how the soil is stabilized. The upper curve is composed of fine-grained sulfates that dissolve in distilled water rapidly and would react very quickly with lime/cement stabilization to form deleterious products. The middle curve is representative of coarse-grained sulfates that take longer to dissolve. These sulfates would still react with traditional stabilizers, but may take longer to show up. The lower curve indicates that sulfates are not a problem for stabilization.
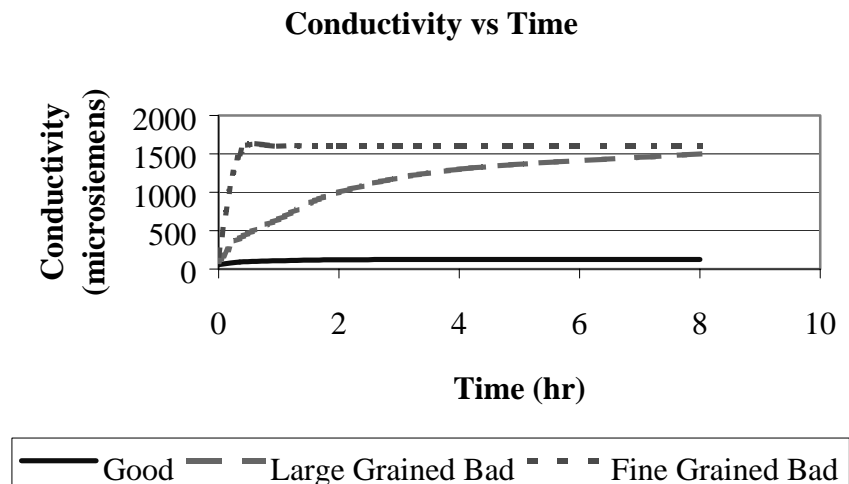
Figure 19. Hypothetical Curves.

41

The colorimetric technique (light transmitted through a sample) should be run under exact conditions to obtain good results and avoid some of the following sources of error:

- constant solution temperature,
- acidity of solution,
- size of $BaCl_2$ crystals,
- amount of $BaCl_2$ added,
- time of stirring,
- rate of stirring,
- time that the suspension stands before a measurement is taken, and
- condition of glass vials as clean and free of oils from human hands to ensure consistent light transmittance through the sample.

In contrast to Tex-620-J and Ion Chromatography, the colorimetric technique proved to be highly repeatable. To define the 95 percent confidence interval for true sulfate content to within ±10 percent of the true known value for concentrations of sulfates up through 3000 ppm, only two samples are required with this technique. At 5000 ppm and above, only one sample is needed. This is in sharp contrast to Tex-620-J, which requires a minimum of 15 tests, and Ion Chromatography, which requires at least seven tests to define the 95 percent confidence interval to within ±10 percent (see previous Table 6). At lower desired accuracy levels such as ±20 percent or ±30 percent, only one test is needed with the colorimeter. The higher precision of the spectrophotometer is graphically illustrated in Figure 20. It is clear that, with repeat testing, the Colorimeter results are much less dispersed than Tex-620-J.

The Colorimetric technique is subject to some of the same interferences as the Tex-620-J gravimetric technique, but since the distilled water is not boiled, only the more soluble soil minerals will go into the solution, resulting in less interference from ferric iron, calcium, and alkali metals.
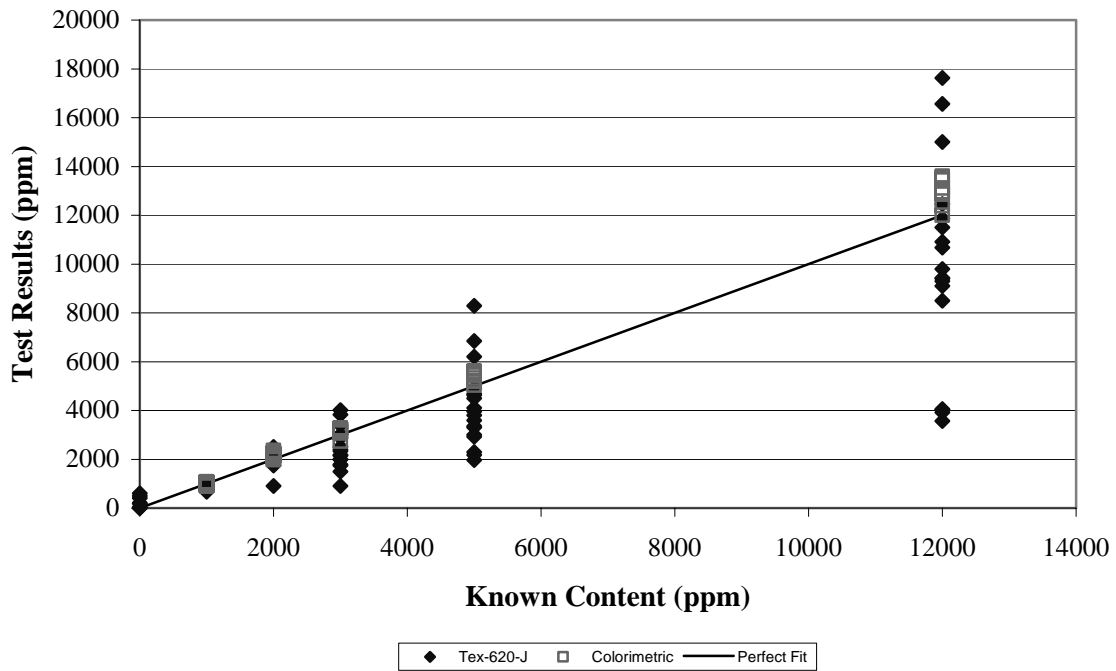
Figure 20. Colorimetric Results with Tex-620-J Results.

The Acetone Field Test is a very good qualitative technique that can only verify the presence of sulfates. It can be used as a screening tool by changing the dilution ratio. At the 1:20 soil-to-water dilution ratio used in this project, concentrations below approximately 2000ppm will not be detected with this technique. To detect lower concentrations one simply reduces the dilution ratio. For example, to detect concentrations down to approximately 1000 ppm, a 1:10 soil-to-water dilution ratio would be used.

The Barium Chloride Test is somewhat more cumbersome than the Colorimetric technique, and the results are not as good. This test, in its present form, is also difficult to run in soils with high concentrations of carbonates because the acetic acid solvent reacts vigorously with the carbonates, resulting in loss of much of the solution making the results erroneous.

The Sulfide Field Test is a good technique for identifying the presence of pyrite (FeS_2) but it is not quantitative. It will only detect the presence of sulfides and not actual concentrations. It is a very quick test to perform, but a pocket magnifier or binocular microscope (Figure 13) is useful for seeing the N_2 bubbles being evolved. Using the reagent mixture specified in the test procedure, this test only works on concentrations down to 5000 ppm.

CONCLUSIONS

- The Conductivity Test is a good technique for identifying possible sulfate-rich soils, however, it will give high values in a soil containing other salts. This test is very easy and may be rapidly performed in the field. This technique is recommended as a good screening tool. If there are high conductivities, then perform lab testing to determine if sulfates are causing the high conductivity readings.

- Soil pH deviations from neutral (pH = 7) cause conductivity to increase.

- Pulverizing samples prior to conductivity analysis gives a faster estimate of soluble salt concentrations in soils.

- Performing conductivity measurements on unpulverized samples at selected intervals for an extended period of time will give an idea about relative grain sizes of soluble salt crystals. The researchers believe this to be invaluable in selecting a stabilization plan.

- The Barium Chloride Test for detecting sulfates works reasonably well at detecting sulfates at low concentrations, but is difficult to perform in carbonate rich soils and requires a longer time to run due to filtration. Barium chloride is highly toxic as well. Therefore, this test is not recommended for use with Texas soils because many of the sulfate-bearing soils also contain a large percentage of carbonate minerals.

- The Acetone Test works well at detecting sulfates. The time for analysis is an issue with this test as well because the sample has to be filtered prior to analysis. The dilution ratio can be modified to detect different concentrations of sulfates. This test is not quantitative but will detect actual sulfates.

- The Colorimetric/Spectrophotometric Test could be used in a laboratory or field office setting. As a lab test, it can provide accuracy approaching that of IC. Results from Colorimetric/Spectrophotometric Testing indicate that this technique could either augment or replace Test Method Tex-620-J. The equipment needed to perform this test costs about $400.00 and testing can be performed in about 25 to 30 minutes.

CHAPTER 4
MAPS OF SULFATE SOILS

TxDOT engineers have, for many years, made use of the USDA soil series maps. The majority of the state has been surveyed and detailed maps have been produced identifying several key soil parameters. The TxDOT road network is superimposed on the paper copies of these maps. Most of the data in these maps are related to crop growth potential. However, there are several key engineering properties used by TxDOT engineers that include: the plasticity index (PI) of the soils, Unified Soils Classification, percentage clay, etc. One example of the utility of these maps is identifying sections of high PI soils on future full-depth recycling projects. The Bryan District (Goehl, 2001) uses the soil maps to identify locations with potential edge-cracking problems.

The USDA started providing automated versions of these maps in the mid 1980s. These automated maps are called the State Soil Geographic Database (STATSGO) maps and are available in Arc-info format. Recognizing the potential benefit to TxDOT, Mr. Bryan Stampley and Mr. Craig Cox of the Pavement Management Information System (PMIS) section began using these maps as the background layer for PMIS data maps. They used the Unified Soils Classification information as a background and overlaid the existing highway system, which they color-coded for various levels of pavement condition.

The USDA soil series maps are well known within Texas. However, another set of geological maps is also available from the Bureau of Economic Geology at the University of Texas in Austin (www.beg.utexas.edu). The entire state has been surveyed, and the results are stored on 38 detailed paper maps. The information within these maps is being digitized by the Natural Resources Conservation Service (NRCS). The digital information is stored with the Soil Survey Geographic Maps (SSURGO) database, and they are intended as updated replacements for the USDA maps (www.tnris.state.tx.us/stratmap/soil.htm). These maps provide a host of more detailed information about variations in soil conditions around Texas. The elements stored within the database are provided in Appendix A of this report. From the highway engineering perspective, the key engineering properties stored for each soil type are as follows:

- American Association of State Highway and Transportation Officials (AASHTO) group classification,

- cation exchange capacity (max and min),
- clay content,
- corrosion potential of uncoated steel,
- potential frost action,
- gypsum content (max and min),
- liquid limit,
- percent passing (max and min for 4, 10, 40 and 200 sieves),
- organic matter (max and min),
- permeability rate,
- soil pH (max and min),
- plasticity index,
- depth to bedrock (max and min),
- shrink-swell potential, and
- water table depth (max and min, seasonal variations).

For this project, clearly the item of interest is the gypsum content. However, one limitation of the digitized maps is that they are available only for about 110 of Texas' 254 counties. Several are under review and will be released in the near future.

In this section of the report, examples of existing paper maps (Geologic Atlas of Texas) and how they could be used within TxDOT are correlated with the digital maps along with results from the field soil investigation. Mr. Craig Cox from TxDOT PMIS section in Austin developed the integrated digital maps presented in this section.

USE OF EXISTING GEOLOGICAL ATLAS OF TEXAS

Lime-induced sulfate heave problems have been reported extensively in the Dallas/Ft. Worth Districts. As described by Burkart et al. (1999) the majority of these problems have been associated with one geologic formation, namely the Eagle Ford Formation. Burkart et al. (1999) described 15 failures of highway projects attributable to sudden and large increases in pavement roughness. The swells were associated with sulfate heave in the lime-treated layer. The Eagle Ford Formation is known to contain large percentages of gypsum near the surface. Two recent failures on TxDOT projects are shown in Figure 21. The upper photograph shows regularly

spaced swells that occurred in 1999 on a section of U.S. 67 shortly after compaction and sealing of the granular base. The swells were associated with large horizontal expansions in the lime-treated subgrade, leading to blow-ups in the overlying pavement. The expansions were noted just after the area experienced a heavy rainfall. The swells continued to grow and reached maximum heights between 9 and 12 inches. To repair the section the base was removed and the lime-stabilized layer was replaced with a select material containing little or no sulfates. The lower figure shows a problem on U.S. 82 in the Paris District. This section was completed in early 2002, and shortly after placement of the chip seal surface several large swells were noted in the completed section. The cause of these swells and remedial actions are now the subject of a forensic study being conducted by Dr. D. Chen of TxDOT.

A portion of the geological atlas of the Dallas/Fort Worth area is shown in Figure 22a. The locations of the failed section on U.S. 67 and the major sulfate heave failure on recreational roads near Joe Pool Lake are annotated on this map. Both failures occurred on the "blue" area, which is the Eagle Ford Formation. The Eagle Ford Formation is described in the map notes as "shale, medium grey, selenitic, and calcareous concretions." While not commonly known to highway engineers, selenite is a form of gypsum which is observed as plate-like glassy crystals. Both the Dallas and Fort Worth districts use these maps on a regular basis to identify potential problems in new projects. However, these maps are not widely used elsewhere in Texas. The map in Figure 22b shows that the failure on U.S. 82, east of Sherman, also occurred in soils developed on the Eagle Ford Formation.

It is clear that these maps should be more widely used within TxDOT. The whole state is covered by 38 maps, and each map costs around $6. It may be advisable to review the information contained in the geologic atlas and to classify each formation according to the three levels of risk as follows:

- **Priority A**: Geologic formations producing certain types of soils rich in sulfates or sulfides, which are known to be problematic, must be both field and laboratory tested. The techniques developed in this study should be implemented.

a. U.S. 67 near Dallas/Fort Worth District Line (1999).

b. U.S. 82 Newly Constructed Section, West of Sherman (2002).

Figure 21. Recent Sulfate Heave Problems on Texas Highways.
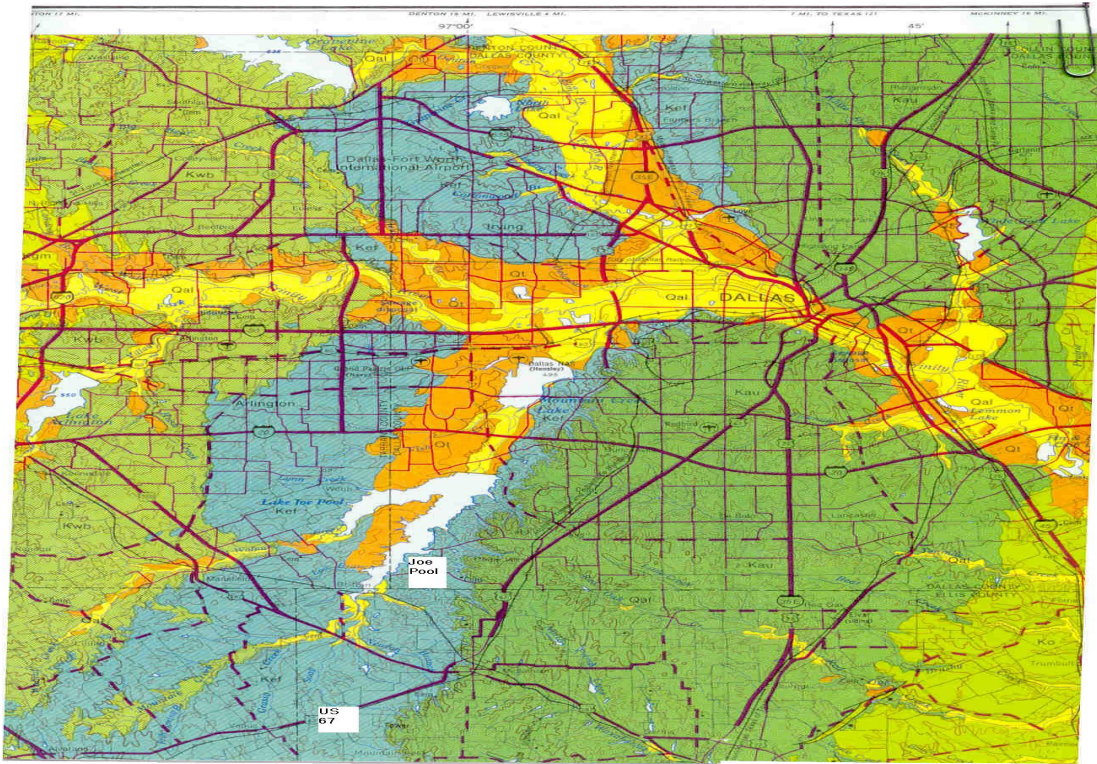
a. Dallas District: Location of US 67 and Joe Pool Lake Failures.
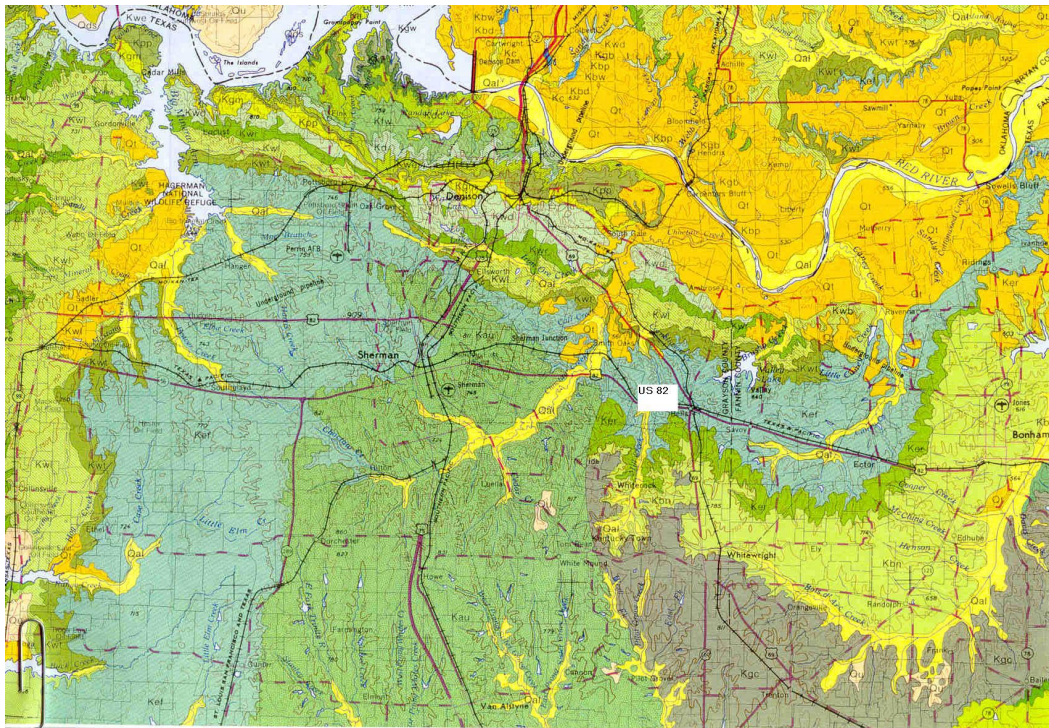
b. Paris District: Location of Recent Failure on U.S. 82.

Figure 22. Excerpts from the Geological Atlas of Texas, Showing Locations of Failures on the Eagle Ford Formation. Scale: 1:250,000.

- **Priority B**: Geologic formations producing certain types of soils, which may be <u>potentially</u> problematic, must be field tested on a limited basis before application of any calcium-based stabilizer.

- **Priority C**: Geologic formations producing certain types of soils <u>do not</u> produce sulfate or sulfides and <u>do not</u> need to be tested.

Each district should be provided with the appropriate maps and a classification of problematic formations. If this approach is shown to be effective, then it could be expanded to cover other problems such as soils rich in organics.

However, it must be emphasized that this approach will not eliminate the sulfate problem. Many projects include extensive cut and fill operations, where soils are hauled to the job site. Nevertheless, it is important to distribute this information to the districts so they are aware of these potential problems.

EXTENT OF EAGLE FORD FORMATION IN TEXAS

From the above discussion, soils developed on the Eagle Ford Formation in the Dallas and Fort Worth area and recent problems in the Paris District confirm that soils developed on this formation should not be treated with lime until a thorough field and/or lab evaluation has been performed. The extent of this formation in Texas is shown in Figure 23. This figure shows that these rocks stretch the entire length of Texas, from Oklahoma to the Mexican border. It should be stressed that this map only shows the rocks exposed at the surface; it may be that in some areas the problems are buried beneath the surface deposits, which may be a problem if cut and fill work is specified.

DIGITIZED SOILS MAPS FOR DALLAS AND FORT WORTH

The soils information from both Tarrant and Dallas Counties was downloaded from the SSURGO databases and subsequently loaded into ARC-info by Mr. Craig Cox of TxDOT. The standard Texas road map was then placed directly over the background soils information.

An interesting feature of this database is that any item in the database can be selected as the background map. In addition, the information for each soil in the county is provided to different depths below the surface. For example, for each soil type the average level of sulfates

Figure 23. Shaded Area Showing the Approximate Distribution of the High Sulfate Eagle Ford Formation in Texas.

is given in 2 ft intervals down to 10 ft. Therefore, it is possible to create both a surface map showing sulfate levels in the top 48 inches and then construct a map showing sulfate levels at greater depths. This would be beneficial in identifying potential problems in jobs where deep cuts are made. It is well established in several soils that sulfate concentration increases with depth.

The results for Tarrant County are shown in Figures 24 and 25, and the results for Dallas County are shown in Figures 26 and 27. For Tarrant County the problem areas are in the east where the soils are developed on the Eagle Ford Formation. One particular area of interest is in the southeast location along State Highway (SH) 303. For large lengths of this highway the near surface soils are shown to have sulfate levels in the 1 to 3 percent range (Figure 26) where in the deep soils the sulfate levels are predicted to increase to over 10 percent in some areas (Figure 27). Clearly this would be a highway where careful consideration will be required prior to any chemical stabilization with lime or cement.

The use of the SSURGO maps is now under investigation by both Dr. Andrew Wimsatt, P.E., and Dr. Dar-Hao Chen, P.E., from TxDOT. Both have identified potential inconsistencies in the available data. This usually occurs where in one county a particular soil series is identified to be high in sulfates and just across the county line the sulfate values for the same soil drop to zero. Dr. Chen evaluated these maps in investigating the failure on U.S. 82, east of Sherman. This sulfate heave problem is shown in the geological atlas to be sitting on the Eagle Ford Formation (Figure 22b). This is correctly reported on the Dallas map to be a high-risk area. However, with the data provided for Grayson County, soils developed on the Eagle Ford Formation were reported to be low in sulfates; this is clearly not the case as measurements made on the U.S. 82 failure found sulfate levels to be in excess of 20,000 ppm.

The new maps clearly offer potential, but there appears to be some data consistency issues. Most of the state has not been released for general use, and many of the districts have only provisional distribution as the data consistency is still being checked. Nevertheless, these maps offer good potential to address not only sulfate but perhaps other issues such as organics and depth to bedrock. TxDOT and the research community should continue work in this area.
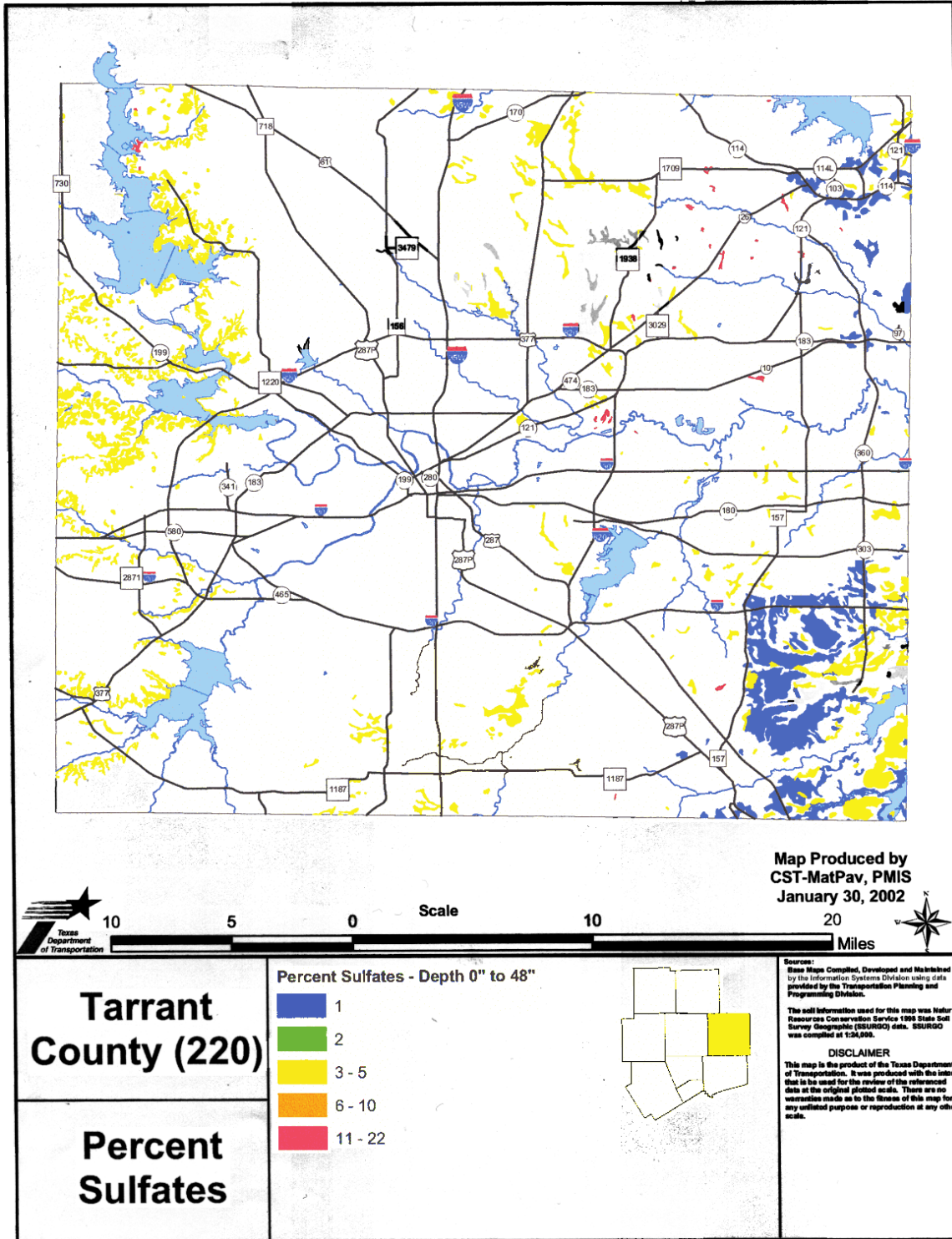
Figure 24. Near Surface (0-4 feet) Sulfate Levels in Tarrant County (Cox, 2002).

**Map Produced by
CST-MatPav, PMIS
January 30, 2002**

Scale

10   5   0   10   20

Miles

Texas Department of Transportation

Tarrant County (220)

Percent Sulfates

Percent Sulfates - Depth More Than 48"

- 1
- 2
- 3 - 5
- 6 - 10
- 11 - 22
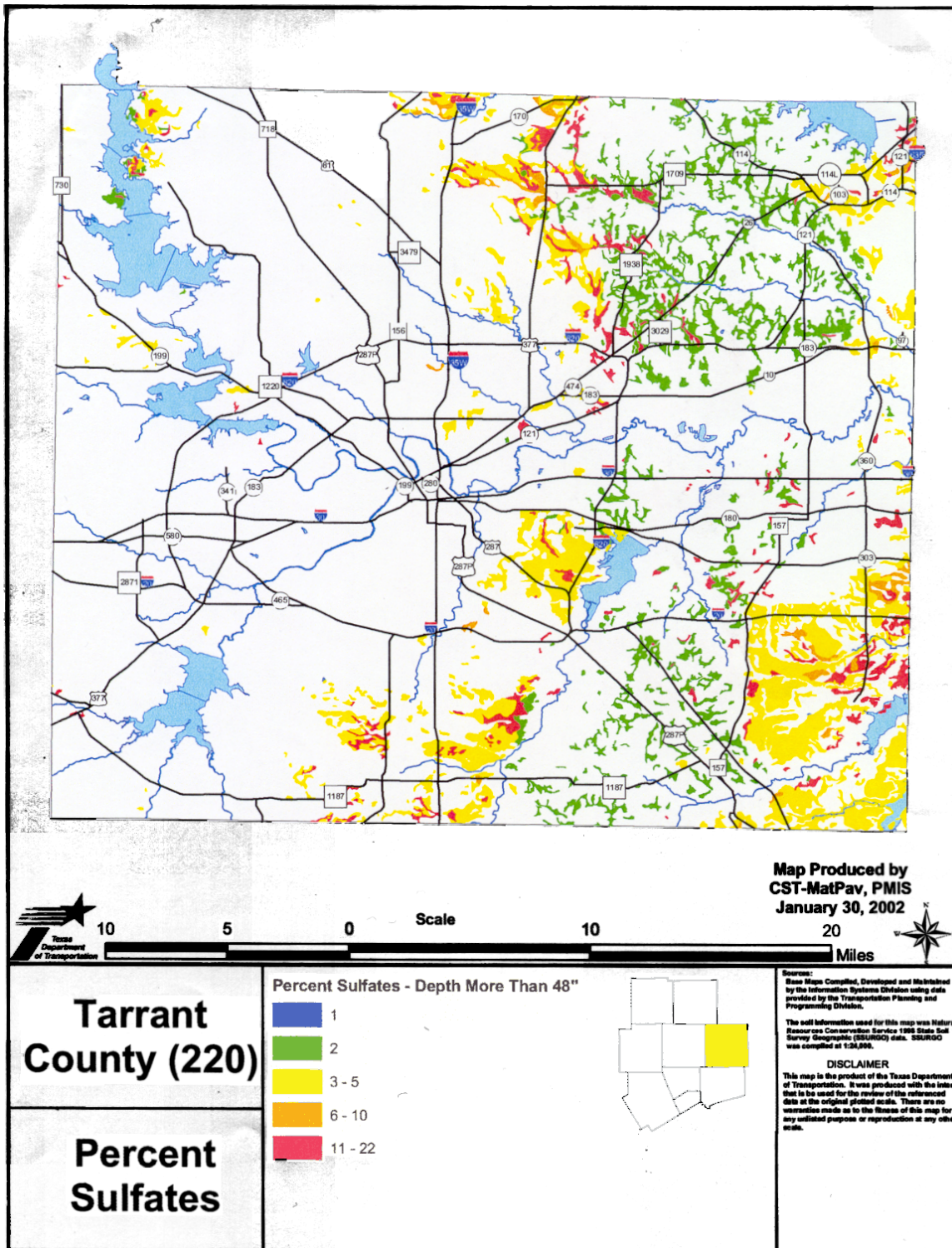
**Figure 25. Sulfate Levels at Depths Greater than 4 feet in Tarrant County
(Cox, 2002).**
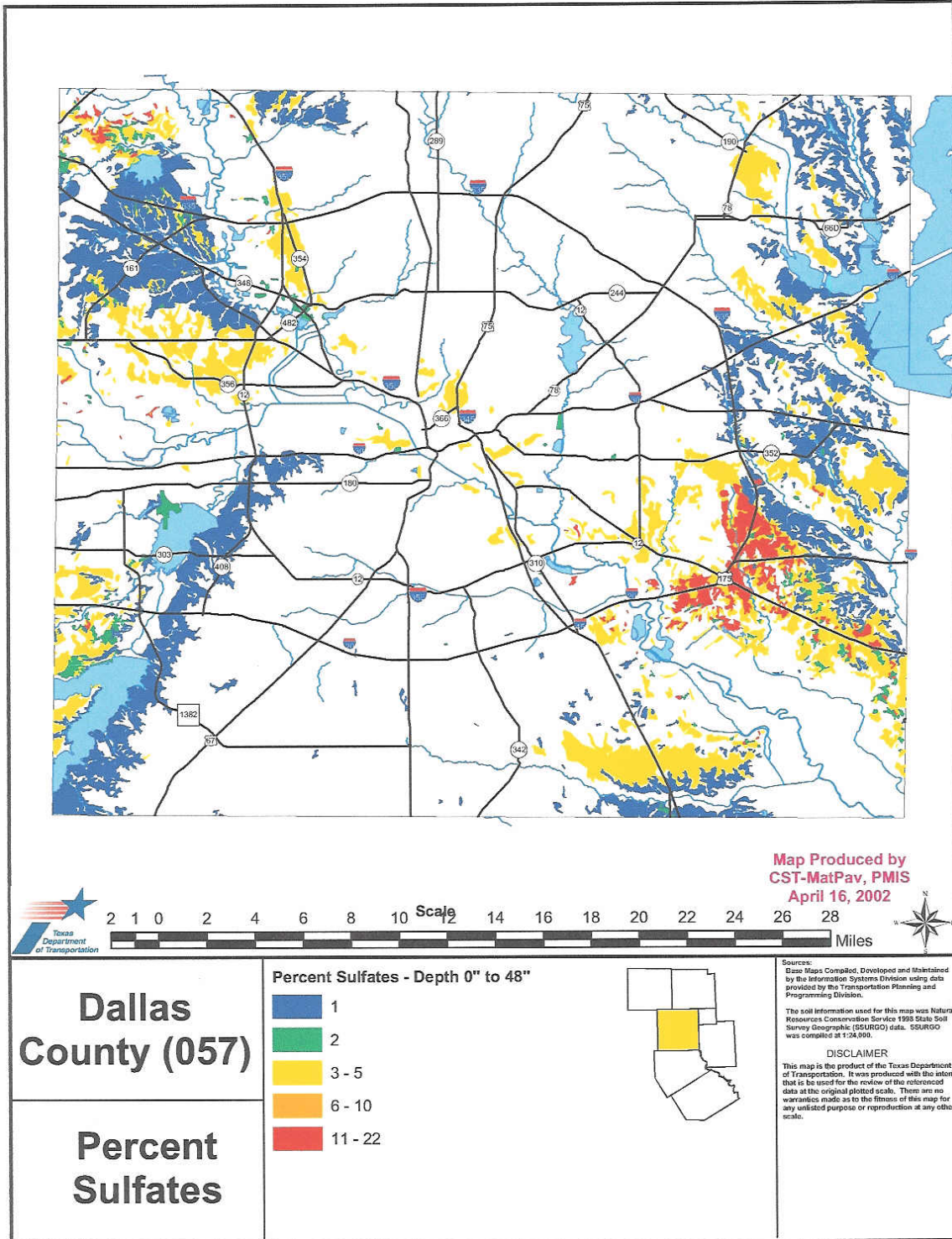
Figure 26. Near Surface (0-4 feet) Sulfate Levels in Dallas County (Cox, 2002).
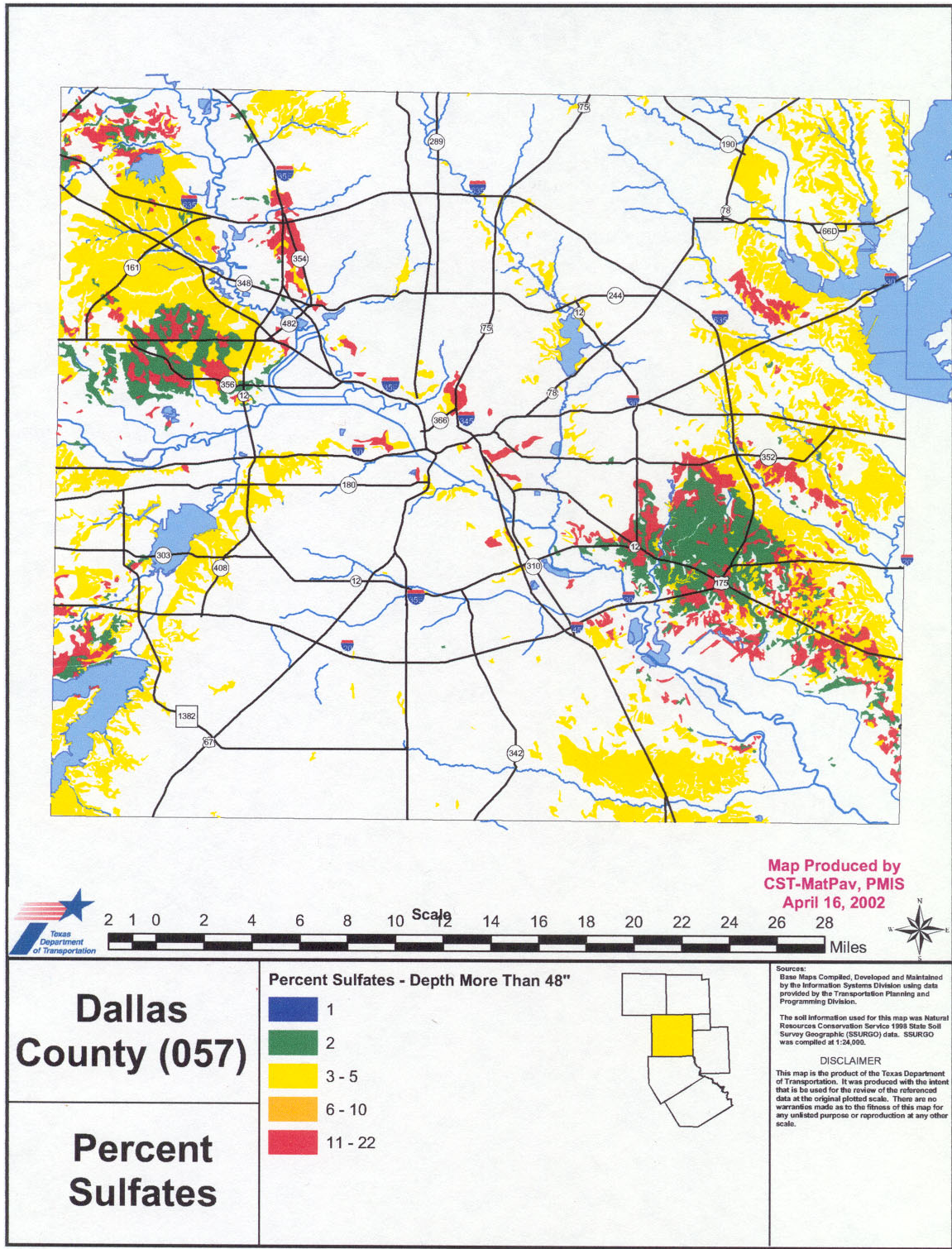
Figure 27. Sulfate Levels at Depths Greater than 4 feet in Dallas County (Cox, 2002).

FIELD EVALUATION OF EXISTING DIGITIZED MAPS

The Fort Worth District under the direction of Materials Engineer Richard Williammee and Pavement Engineer Andrew Wimsatt is in the process of evaluating the existing SSURGO data. The district performs an extensive soil investigation prior to the finalization of the pavement design. Samples are taken at approximately 0.5 mile intervals to a depth of 20 ft. Standard tests, such as Atterburg limits, are run for the entire soil profile so that the potential vertical rise can be calculated. However, as part of this testing the district coordinates with the district geometric design office to locate areas where cuts are proposed. In those locations the sulfate concentration at the level of the proposed cuts is measured in the laboratory using TxDOT Test Method Tex-620-J. If cuts are not planned, then the sulfate content for each hole from 0 to 4 ft depth is measured. This process is used to identify potential problem areas requiring alternatives to calcium stabilization and will require more detailed testing during construction prior to lime or cement stabilization.

To evaluate the usefulness of the SSURGO data, the district now produces a digitized map showing the potential problem areas prior to drilling. One of the first tests was conducted on a section of Farm to Market Road (FM) 8 from the Erath County Line to Lingleville. One potential sulfate area confirmed by field drilling and lab testing (Wimsatt, 2002) is shown on Figure 28. The gypsum data were summarized and areas where gypsum could be present at depths down to 10 ft were identified. As shown in Figure 28, only one section near the west side of the project was identified as a potential gypsum location; this area is color coded in green. These data are in reasonable agreement with the soluble sulfate levels found in the TxDOT soils report produced by TEAM Consultants Inc., who sampled soils at 22 locations. Most of the testing was restricted to the top 5 ft because cuts were not planned for these locations.
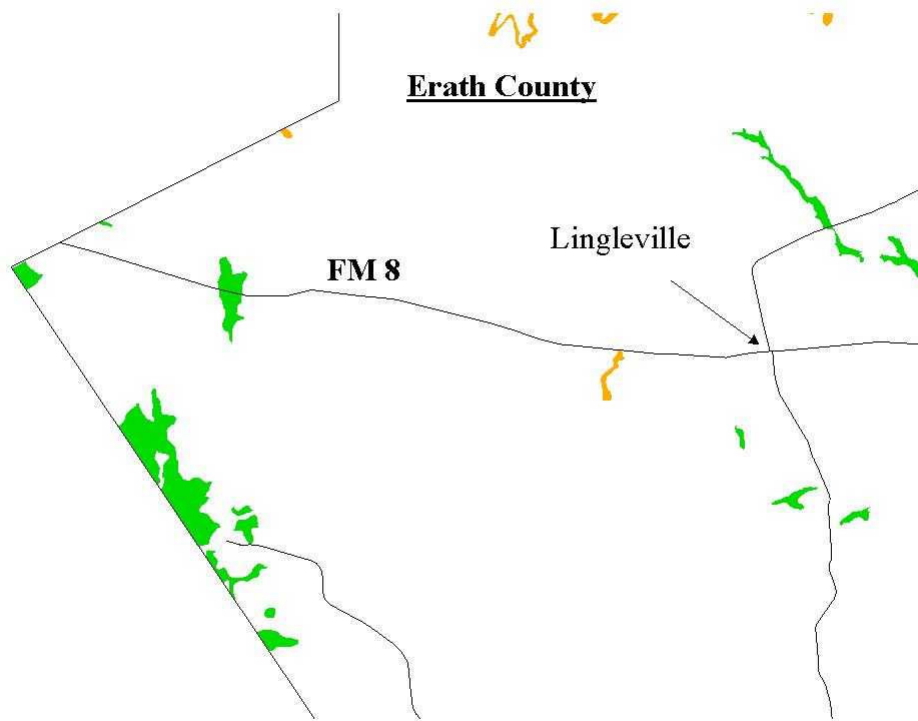
Figure 28. TxDOT Developed Digital Map of FM 8 Project.

CONCLUSIONS

Based on the evaluation of existing maps and automated databases the following conclusions are made:

- The Geologic Atlas of Texas works well for identifying formations which are high in sulfates and/or sulfides.
- The SSURGO database has incorrect sulfate contents in some regions of the state as shown on U.S. 82, East of Sherman, Texas.
- Much of the TxDOT data are incompatible with the NRCS database.

CHAPTER 5

RECOMMENDATIONS

Based on conclusions drawn in preceding chapters, detailed recommendations are presented below.

LABORATORY TESTS (Chapter 2)

- TxDOT should consider adopting the Ion Chromatography test technique as the laboratory standard.
- If TxDOT is to continue use of Test Method Tex-620-J, it is recommended that procedural changes be made as follows:

1. Pulverize the sample with a mortar and pestle to pass the #50 sieve (300 μm).
2. Weigh out 10 to 50 g of soil and dilute in 1:10 ratio with deionized water; this requires some knowledge of the origin of the samples because higher sulfates will require less sample than lower sulfate contents.
3. Heat deionized water and soil mixture to near boiling for only six to eight hours.
4. After filtering with Whatman #42 filter paper, some particulate matter may remain in the filtrate, so centrifuge it at 5000 revolutions per minute for 20 to 30 minutes.
5. Ensure the concentrated HCl is trace metal grade (ultrapure) because there can be sulfate introduced into the sample from the HCl.
6. When the 25 ml of $BaCl_2$ solution is added to the hot filtrate, ensure that it is added a few drops at a time and continuously stirred to maximize crystallite size.
7. Digest the solution for a minimum of one hour instead of 10 minutes.
8. Remove from the hot plate and let cool for 24 hours instead of 15 minutes and keep it covered.
9. Filter through a desiccated filter paper with a known mass and wash with *cold* water instead of hot water to remove chlorides adsorbed to the barite.
10. Place filter paper and precipitate in weighed platinum crucible and dry in 100 ℃ oven for a minimum of one hour.
11. Do not char the filter paper with a meeker burner.

12. Place crucible in muffle furnace for one hour to burn off filter paper.

13. Cool in a desiccator and weigh.

14. Subtract weight of crucible from the total weight to get the R value used to calculate sulfate content.

15. When running the test use a methods blank and a series of standards ($MgSO_4$) in the same concentration range suspected for the soil.

16. USE GREAT CAUTION BECAUSE BARIUM CHLORIDE IS HIGHLY TOXIC. AVOID GETTING THIS MATERIAL ON YOUR SKIN!

RAPID FIELD TESTS (Chapter 3)

- It is recommended that the Conductivity Test be considered for full-scale implementation. This test on unpulverized samples can provide information on both the total sulfate content and the grain size distribution. As fine-grained gypsum crystals are highly reactive, this may be a critical piece of information when deciding upon treatment and construction options. A Conductivity Test Kit can be assembled for around $500.

- TxDOT should purchase the Colorimeter Test equipment to run parallel with its laboratory test. If this is shown to be highly accurate on a range of soils, then it is feasible to equip each district lab dealing with sulfate soils with both a Conductivity Test Kit and a lab Colorimeter Test Kit for less than $1000.

MAPS (Chapter 4)

- TxDOT should purchase the entire Geologic Atlas of Texas and identify formations (in addition to the Eagle Ford Formation) that are high in either sulfates or sulfides. The three levels of risk for each formation should be implemented.

- These maps should be distributed to TxDOT districts as the first step in identifying locations where field sulfate tests should be conducted.

- The methods used by Mr. Cox and Dr. Wimsatt to develop automated SSURGO maps should be documented, and training should be provided to other district materials/pavement engineers on how to use this new resource.

- Communication channels should be opened between TxDOT and the NRCS to identify concerns about data within the existing databases.

- In addition to the statewide SSURGO efforts the Materials and Pavements Section of the Construction Division should work with the PMIS group to develop the Geologic Atlas of Texas as a base map. They should overlay all roads that have experienced sulfate heave problems onto the base map. This will help identify formations that have the potential for sulfate heave.

REFERENCES

A. Abdul-Mehdi. (2002) Personal Communication with TAMU (El Paso). Ion Chromatography, April.

S. Bredenkamp, and R. Lytton. (1995) *Reduction of Sulfate Swell in Expansive Clay Subgrades in the Dallas District.* Report 1994-5. TTI, Texas Department of Transportation.

C.A. Bower, and R.B. Huss. (1948) Rapid Conductometric Method for Estimating Gypsum in Soils.

B. Burkart, G.C. Goss, and J.P. Kern. (1999) The role of Gypsum in Production of Sulfate-Induced Deformation of Lime Stabilized Soils. *Environmental and Engineering Geoscience*, Vol. 5, No. 2, pp. 173-187.

U. de la Camp, and O. Seely. (2002) Gravimetric Sulfate Determination. http://chemistry.csudh.edu/oliver/che230/labmanual/gravsulf.htm

C. Coward. (2002) Personal Communication with TxDOT's Materials and Pavements Section, March.

C. Cox. (2002) Personal Communication with TxDOT's PMIS section. Maps generated using SSURGO data.

D.D. Dubbe, M.A. Usmen, and L.K. Moulton. (1997) Expansive Pyritic Shales. In *Transportation Research Record 993*, TRB, National Research Council, Washington, D.C., pp. 19-27.

F. Feigl. (1958) *Spot Tests in Inorganic Analysis*. Elsevier Publishing Company, New York.

D, Goehl. (2001) Presentation at the 2001 Short Course, College Station, Texas, September.

D. Hunter. (1989) *The Geochemistry of Lime-Induced Heave in Sulfate Bearing Clay Soils.* (Dissertation) University of Nevada, Reno.

S.B. Jarrell. (1994) *Basic Statistics*. Wm. C. Brown Publishers, Dubuque, Iowa.

K.B. Krauskopf. (1967) *Introduction to Geochemistry*. McGraw-Hill Book Company, New York.

G.H. McClellan, J.L. Eades, and N.A. Johnson. (1998) Simple Field Method to Detect Sulfide Sulfur in Rocks. *Environmental and Engineering Geoscience*, Vol. 4, No. 1, pp. 115-116.

M. Mohamed. (2002) Personal Communication with Ana-Lab chemist. May.

J.W. Robinson. (1970) *Undergraduate Instrumental Analysis*. 2nd ed. Marcel Dekker, Inc., New York.

Soil Survey Report, TxDOT, FM 8, Erath County, prepared by Team Consultants, Inc, for the Forth Worth District, Jan 3, 2002.

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

TAMU Chemistry Department. (2002) Gravimetric Analysis.
http://www.chem.tamu.edu/class/majors/tutorialnotefiles/gravimetric.htm

G.N. White. (2002) Personal Communication with Soil & Crop Sciences Dept. Texas A&M University.

A. Wimsatt. (2002) Personal Communication with TxDOT's Fort Worth District. Maps generated using SSURGO data.

APPENDIX A

DEFINITION OF SOIL DATA ELEMENTS

Element	Tables	Long name	Description
aashind	layer	AASHTO Group Index	AASHTO (American Assoc. of State Highway and Transportation Officials) group index. A modification to AASHTO group classification of a soil.
aashto	layer	AASHTO Group Classification	AASHTO (American Assoc. of State Highway and Transportation Officials) group classification. A code for AASHTO group classification for a soil.
anflobeg	comp	Annual Flooding Month Begin	Month in which annual flooding (flooding likely to occur during the year) begins in a normal year.
anflodur	comp	Flood Duration Class	The duration of annual flooding in a normal year.
anfloend	comp	Annual Flooding Month End	Month in which annual flooding (flooding likely to occur during the year) ends in a normal year.
anflood	comp	Annual Flooding Frequency	Descriptive term used to describe the frequency of annual flooding (flooding likely to occur during the year) that is likely to occur. Frequent (FREQ) – > 50% chance of flooding; Occasional (OCCAS) – 5 to 50% chance of flooding; Rare (RARE) – 0 to 5% chance of flooding.
awch	layer	Available Water Capacity	Maximum value for the range of available water capacity for the soil layer or horizon, expressed as inches/inch.
awcl	layer	Available Water Capacity	Minimum value for the range of available water capacity for the soil layer or horizon, expressed as inches/inch.
bdh	layer	Bulk Density	Maximum value for the range in moist bulk density of the soil layer or horizon, expressed as grams per cubic centimeter.
bdl	layer	Bulk Density	Minimum value for the range in moist bulk density of the soil layer or horizon, expressed as grams per cubic centimeter.
caco3h	layer	Carbonate as $CaCO_3$	Maximum value for the range of calcium carbonate ($CaCO_3$) in the soil layer or horizon, expressed as a percent.

Element	Tables	Long name	Description
caco3l	layer	Carbonate as CaCO$_3$	Minimum value for the range of calcium carbonate (CaCO$_3$) in the soil layer or horizon, expressed as a percent.
cech	layer	Cation Exchange Capacity	Maximum value for the range in cation exchange capacity for the soil layer or horizon.
cecl	layer	Cation Exchange Capacity	Minimum value for the range in cation exchange capacity for the soil layer or horizon.
clascode	comp taxclass	Taxonomic Classification code	Code for the taxonomic classification for the soil. Definition of codes are in the taxclass table.
class clayh	taxclass layer	Taxonomic Classification Clay	The taxonomic classification (name) of the soil. Maximum value for the range in clay content of the soil layer or horizon, expressed as a percentage of the material less than 2 mm in size.
clayl	layer	Clay	Minimum value for the range in clay content of the soil layer or horizon, expressed as a percentage of the material less than 2 mm in size.
clirr	comp	Irrigated Capability Class	A rating of the soil for irrigated agricultural use. The number indicates progressively greater limitations and narrower choices for use.
clnirr	comp	Nonirrigated Capability Class	A rating of the soil for nonirrigated agricultural use. The number indicates progressively greater limitations and narrower choices for use.
cntycode	mucoacre ssacoac	County (FIPS) Code	FIPS code identifying an individual county within a soil survey area.
cntyname	ssacoac	County Name	The name of a county within a soil survey area.
code	codes	Data base code	A listing of codes used in the specified data base.
codedesc	codes	Code Description or Meaning	Narrative description or explanation of codes used in the data base.
codename	codes	Code Name	The long name (unabbreviated) for the code.
col	element tblelt valrange	Column Name	Column name used in data base.
comment	ssarea	Survey Area Comments	Remarks used to clarify or document information for a soil survey area. A list of sources, and other information for the survey area.
comname	plantnm	Plant Common Name	The common name for the plant most widely used by the state.

Element	Tables	Long name	Description
compacre	comp	Component Acres	The acreage of the component of a soil map unit. Component acres are normalized to 100 percent to exclude inclusions. The sum of all the component acres for a map unit will equal 100 percent.
compkind	comp	Kind of Component	Code identifying the kind of component of the map unit. Example: Series (S); Family (F); Variant (V); Taxadjunct (T); Taxon above family (G); Miscellaneous area (M).
compname	comp	Component Name	The name of the component (series, taxonomic unit or miscellaneous area) of the map unit.
comppct	comp	Component Percent	The percentage of the component of the map unit.
corcon	comp	Corrosion - Concrete	An interpretation rating of the susceptibility of concrete to corrosion when in contact with the soil.
cordate	ssarea	Correlation Date	The date of final correlation of the soil survey.
corsteel	comp	Corrosion - Uncoated Steel	An interpretation rating of the susceptibility of uncoated steel to corrosion when in contact with the soil.
cropname	compyld muyld yldunits	Crop Name	The common name for the crop for which a yield is given.
domid	codes element	Domain ID	A code identifying the domain for the data element. Domain contains the broad definition and codes used for all data elements within its domain.
drainage	comp	Soil Drainage Class	Code identifying the natural drainage condition of the soil and refers to the frequency and duration of periods when the soil is free of saturation. Example: Well Drained (W); Excessive (E); Moderately Well (MW); Poorly (P); Somewhat Excessively (SE); Somewhat Poorly (SP).
eddate	ssarea	Date SSA Data Edited	The date as mouth/day/year the data for the soil survey area was certified by the state soil scientist as edited and available for public use.
edstat	ssarea	SSA Editing Status	Code identifying the status of editing or certification level for the soil survey.
eldesc	element	Element Description	The characteristics or properties that define or describe an element.
ellable	element	Element Label - long name	The long name assigned to an element.
frostact	comp	Potential Frost Action	An interpretation rating of the susceptibility of the soil to frost heaving.

71

Element	Tables	Long name	Description
inclsoil	inclusn	Included Soil	Name of soil or miscellaneous land type included in the map unit.
irryld	compyld muyld	Irrigated Crop Yield	The expected yield of the specific crop with irrigation. Defined as the yield expected in an average year under a high level of management.
kfact	layer	Soil Erodibility Factor, includes rock fragments	An erodibility factor which is adjusted for the effect of rock fragments.
kffact	layer	Soil Erodibility Factor, rock fragments free	An erodibility factor which quantifies the susceptibility of soil particles to detachment and movement by water. This factor is used in the Universal Soil Loss Equation to calculate soil loss by water.
laydeph	layer	Layer Depth	The depth to the lower boundary of the soil layer or horizon, expressed in inches.
laydepl	layer	Layer Depth	Depth to the upper boundary of the soil layer or horizon, expressed in inches.
layerid	layer	Layer Identification Number	A convention to identify the original layers on the SOI-5 record. Example: layerid 11 for the first surface of a multisurface record, 12 for the second surface layer, 2 thru 9 for subsurface layers.
layernum	layer	Layer Number	The sequence number identifying layers in the soil profile. A layer number of 1 would indicate the layer is the surface layer.
len	valrange	Column Length	The maximum length of a column.
llh	layer	Liquid Limit	The maximum value for the range in liquid limit of the soil layer or horizon, expressed as percent moisture by weight.
lll	layer	Liquid Limit	The minimum value for the range in liquid limit of the soil layer or horizon, expressed as percent moisture by weight.
minalogy	taxclass	Mineralogy	Code for the MINERALOGY class of the Family category of taxonomic classification.
mlra	mapunit	Major Land Resource Area	The code used to identify the dominant Major Land Resource Area (MLRA) within which the soil map unit is mapped.
muacres	mapunit	Mapunit Acres	The acreage of the soil map unit in the soil survey area.

Element	Tables	Long name	Description
grpcode	interp	Interpretative Group Code	Code identifying the interpretative group or category for the interpretation specified. Examples of interpretative groups are septic tank absorption fields and shallow excavations.
grtgroup	taxclass	Great Group	Code for the taxonomic GREAT GROUP category.
gsflobeg	comp	Growing Season Flooding Begins	Month in which growing season (season for common field crops in the area) flooding begins in a normal year.
gsflodur	comp	Growing Season Duration	The duration of flooding during the growing season (season for common field crops in the area).
gsfloend	comp	Growing Season Flooding Ends	Month in which growing season (season for common field crops in the area) flooding ends in a normal year.
gsflood	comp	Growing Season Flooding Frequency	Descriptive term describing the frequency of flooding during the growing season (season for the common field crops in the area). Frequent (FREQ); Occasional (OCCAS); Rare (RARE).
gypsumh	layer	Gypsum	Maximum value for the range in sulfates reported as gypsum ($CaSO_4$) in the soil layer or horizon, expressed as a percent.
gypsuml	layer	Gypsum	Minimum value for the range in sulfates reported as gypsum ($CaSO_4$) in the soil layer or horizon, expressed as a percent.
hydgrp	comp	Hydrologic Group	The hydrologic group for the soil. Example: A, A/D.
hydric	comp inclusn	Hydric Soil Rating	The symbol (Y/N) identifying hydric soils.
inch10h	layer	Weight Percent Greater than 10 inches	The maximum value for the range in percent by weight of the rock fragments greater than 10 inches in the soil layer or horizon.
inch10l	layer	Weight Percent Greater than 10 inches	The minimum value for the range in percent by weight of the rock fragments greater than 10 inches in the soil layer or horizon.
inch3h	layer	Weight Percent 3 to 10 inches	The maximum value for the range in percent by weight of the rock fragments 3 to 10 inches in the soil layer or horizon.
inch3l	layer	Weight Percent 3 to 10 inches	The minimum value for the range in percent by weight of the rock fragments 3 to 10 inches in the soil layer or horizon.
inclpct	inclusn	Included Soil Percent	The percentage of the map unit occupied by the specified included soil.

Element	Tables	Long name	Description
muid	comp compyld forest hydcomp helclass inclusn interp layer mapunit mucoacre muyld plantcom rsprod windbrk wlhabit woodland woodmgt	Mapunit Identification Symbol	A symbol created by concatenation of the soil survey area symbol (ssaid) and mapunit symbol (musym). It uniquely identifies a mapunit within a state. For example, ssaid 061 and musym 1 is stored as muid 061001. The muid is used as a key for linking information in the MUIR tables.
mukind	mapunit	Mapunit Kind	Code identifying the kind of map unit: Consociation (C); Association (A); Undifferentiated Group (U); Complex (X).
muname	mapunit	Mapunit Name	Correlated name of the map unit (recommended name or field name for surveys in progress).
musym	mapunit comp	Mapunit Symbol	The symbol used to identify the soil map unit on the soil map.
muwathel	helclass mapunit.	Mapunit HEL Class (water)	The highly erodible lands rating for the soil map unit. The rating is based an evaluation of the water erosion hazard of the components of the map unit. If all components are of a single class that class applies, if not then a 2 (Potential Highly Erodible) is assigned.
muwndhel	helclass	Mapunit HEL Class (wind)	The highly erodible lands rating for the soil map unit. The rating is based an evaluation of the wind erosion hazard of the components of the map unit. If all components are of a single class that class applies, if not then a 2 (Potential Highly Erodible) is assigned.
nirryld	compyld muyld	Nonirrigated Crop Yield	The expected yield of the specific crop without supplemental irrigation. Defined as the yield expected in an average year under a high level of management.
no10h	layer	Percent Passing Sieve Number 10	The maximum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 10 sieve.
no10l	layer	Percent Passing Sieve Number 10	The minimum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 10 sieve.

Element	Tables	Long name	Description
no200h	layer	Percent Passing Sieve Number 200	The maximum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 200 sieve.
no200l	layer	Percent Passing Sieve Number 200	The minimum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 200 sieve.
no40h	layer	Percent Passing Sieve Number 40	The maximum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 40 sieve.
no40l	layer	Percent Passing Sieve Number 40	The minimum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 40 sieve.
no4h	layer	Percent Passing Sieve Number 4	The maximum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 4 sieve.
no4l	layer	Percent Passing Sieve Number 4	The minimum value for the range in percent by weight of the soil material in a layer or horizon which is less than 3 inches and passes a No. 4 sieve.
omh	layer	Organic Matter	The maximum value for the range in organic matter content of the soil layer or horizon, expressed in percent by weight.
oml	layer	Organic Matter	The minimum value for the range in organic matter content of the soil layer or horizon, expressed in percent by weight.
order	taxclass	Order	Code for the taxonomic ORDER category of the record.
ordsym	woodmgt	Ordination Symbol	The ordination symbol is the class and subclass part of the woodland suitability group. The first element in ordination symbol is the productivity class. This is a number that denotes potential productivity in cubic meters of wood per hectare per year for an indicator tree (1 m³/ha is equal to 14.3 ft³/ac). The second part of the ordination is the subclass, a capital letter symbol which indicates certain soil or physiographic characteristics that contribute to important hazards or limitations in management. Example: W - Excessive wetness. Subclasses are listed in ranked order.
otherfam	taxclass	Other Family	This field consists of OTHER FAMILY codes for soil depth class, slope class, consistence class, classes of coatings and classes of cracks of the Family category of taxonomic classification.

Element	Tables	Long name	Description
otherph	comp	Class-Determining Phase Criteria	Class-determining phase criteria, other than slope and texture, recorded on the SOI-6 and used to select appropriate interpretation and rating from the SOI-5 Record.
pandeph	comp	Depth to Cemented Pan	Maximum value for the range in depth to the upper boundary of a cemented pan, expressed in inches.
pandepl	comp	Depth to Cemented Pan	Minimum value for the range in depth to the upper boundary of a cemented pan, expressed in inches.
panhard	comp	Cemented Pan Thickness	The degree of induration and thickness of the cemented pan. A pan is rated as "THICK" if it is more than 3 inches thick and continually indurated or more than 18 inches thick and discontinuous or fractured. Pans not meeting these criteria are rated THIN.
partsize	taxclass	Particle Size	Code for the PARTICLE-SIZE class of the Family category of taxonomic classification.
permh	layer	Permeability Rate	The maximum value for the range in permeability rate for the soil layer or horizon, expressed as inches/hour.
perml	layer	Permeability Rate	The minimum value for the range in permeability rate for the soil layer or horizon, expressed as inches/hour.
phh	layer	Soil Reaction (pH)	The maximum value for the range in soil reaction (pH) for the soil layer or horizon.
phl	layer	Soil Reaction (pH)	The minimum value for the range in soil reaction (pH) for the soil layer or horizon.
pih	layer	Plasticity Index	The maximum value for the range in plasticity index for the soil layer or horizon, expressed as percent of moisture by weight.
pil	layer	Plasticity Index	The minimum value for the range in plasticity index for the soil layer or horizon, expressed as percent of moisture by weight.
plantcov	forest	Plant Ground Cover	The percentage of the ground covered by the plant (forest understory).
plantpct	plantcom	Plant Production Percentage	The percentage of total site production attributed to the specified plant, expressed as percent of air dry plant material weight.
plantsym	forest plantcom plantnm windbrk woodland	Plant Symbol	Symbol used to identify a specific plant.

Element	Tables	Long name	Description
pndbeg	comp	Ponding Begin	Month in which soil surface ponding begins in a normal year.
pnddeph	comp	Ponding Depth	The maximum value for the range in depth of surface water ponding on the soil.
pnddepl	comp	Ponding Depth	The minimum value for the range in depth of surface water ponding on the soil.
pnddur	comp	Ponding Duration	The duration of surface water ponding.
pndend	comp	Ponding End	Month in which surface water ponding ends in a normal year.
prec	valrange	Precision Value	The number of digits to the right of a decimal.
primfml	mapunit	Prime Farmland Classification	The prime farmland classification of the map unit. State codes have been developed for some states.
prodfav	rsprod	Range Production Favorable	The estimated annual potential production of range forage for the soil in a year with favorable or above average growing conditions. Round to nearest 100 pounds.
prodnorm	rsprod	Range Production Normal	The estimated annual potential production of range forage for the soil in a year with normal or average growing conditions. Round to nearest 100 pounds.
produnfv	rsprod	Range Production Unfavorable	The estimated annual potential production of range forage for the soil in a year with unfavorable or below average growing conditions. Round to nearest 100 pounds.
rangeh	valrange	High Range	The maximum value for the range in values of an element.
rangel	valrange	Low Range	The minimum value for the range in values of an element.
rating	interp	Soil Interpretative Rating	Rating of soil for specified use. Suitability ratings are good, fair, and poor. Limitation ratings are slight, moderate, and severe.
reaction	taxclass	Reaction	Code for the REACTION class of the Family category of taxonomic classification.
restct1	interp	Rating Limitation Restrictions	Restrictive feature code, 1st.
restct2	interp	Rating Limitation Restrictions	Restrictive feature code, 2nd.
restct3	interp	Rating Limitation Restrictions	Restrictive feature code, 3rd.

Element	Tables	Long name	Description
rockdeph	comp	Depth to Bedrock	The maximum value for the range in depth to bedrock, expressed in inches.
rockdepl	comp	Depth to Bedrock	The minimum value for the range in depth to bedrock, expressed in inches.
rockhard	comp	bedrock hardness	The degree of hardness of the underlying rock. Rated as: HARD - Excavation requires blasting or special equipment or SOFT - Excavation can be made with trenching machines, backhoes, or small rippers.
rsid	rsprod rangenm	Range Site Identification	Code used to identify the NRCS range site.
rsname	rsprod rangenm	Range Site Name	Name for the NRCS range site.
s5id	comp layer	Soil Interpretations Record Number	The Soil Interpretations Record (SOI-5) identification number assigned to the particular SOI-5. Example: CO0034.
salinh	layer	Salinity	The maximum value for the range in soil salinity of the soil layer or horizon measured as electrical conductivity of the soil in a saturated paste. Values are expressed in mmhos/cm.
salinl	layer	Salinity	The minimum value for the range in soil salinity of the soil layer or horizon measured as electrical conductivity of the soil in a saturated paste. Values are expressed in mmhos/cm.
sarh	layer	Sodium Absorption Ratio	The maximum value for the range in Sodium Absorption Ratio (SAR) for the soil layer or horizon.
sarl	layer	Sodium Absorption Ratio	The minimum value for the range in Sodium Absorption Ratio (SAR) for the soil layer or horizon.
sciname	plantnm	Scientific Plant Name	The scientific name of a plant.
sclirr	comp	Irrigated Capability Subclass	Irrigated Capability Subclass. Concatenation of capability class and subclass codes: Example: class 2 and subclass e are combined and entered as 2E.
sclnirr	comp	Nonirrigated Capability Subclass	Nonirrigated Capability Subclass.Concatenation of capability class and subclass codes. Example: class 2 and subclass e are combined and entered as 2E.

Element	Tables	Long name	Description
seqnum	comp	Sequence Number compyld	A number identifing the sequence of components in a map unit. The first component of a multitaxa map forest unit has a seqnum of 1, the second component 2, and interp so on.
	layer plantcom rsprod windbrk wlhabit woodland woodmgt		
shrinksw	layer	Shrink-Swell Potential	An interpretation rating of the soil layer or horizons behavior of changing volume (shrinking and swelling) upon wetting and drying.
sitind	woodland	Site Index	The height in feet of the larger trees at some given age, normally 100 years in the western United States, and 50 years in the east. The pinyon-juniper forest type is an exception, where the site index is determined by basal area.
slopeh	comp	Soil Slope	The maximum value for the range of slope of a soil component within a map unit.
slopel	comp	Slope of Soil	The minimum value for the range of slope of a soil component within a map unit.
soiltemp	taxclass	Soil Temperature	Code for the SOIL TEMPERATURE class of the Family category of taxonomic classification.
ssaacres	ssarea	Soil Survey Area Acres	The acreage of the soil survey area.
ssacoac	ssacoac	SSA County Acres	The acres of a county within a soil survey area.
ssaid	mapunit ssarea	Soil Survey Symbol	Three character numeric code which identifies the soil survey area. For survey areas covering a single county the ssaid is the county FIPS code. For multicounty survey areas the ssaid is identified in the Soil Survey Schedule. Example: 617,...012.
ssaname	ssarea	Soil Survey Area Name	The name given to the survey.
ssanum	ssarea	State/SSA Identification Number	A five character identification number for the soil survey area. Created by combining the numeric state code and the soil survey area symbol (ssaid). Example: 08617.
state	ssarea	State Code (Alpha)	FIPS alpha code for the state. Example: CO,...AR.

Element	Tables	Long name	Description
stssaid	comp compyld forest hydcomp helclass inclusn interp layer mapunit mucoacre muyld plantcom rsprod ssacoac ssarea windbrk wlhabit woodland woodmgt	State Soil Survey Area ID	A concatenation of FIPS alpha code for a state and the soil survey area symbol (ssaid). Example: CO017.
subgroup	taxclass	Subgroup	Code for the taxonomic SUBGROUP category of the record.
subinith	comp	Initial Subsidence	Maximum value for the range in initial subsidence that can be expected when drained. expressed in inches (organic soils only).
subinitl	comp	Initial Subsidence	Minimum value for the range in initial subsidence that can be expected when drained, expressed in inches (organic soils only).
suborder	taxclass	Suborder	Code for the taxonomic SUBORDER category of the record.
subtoth	comp	Total Subsidence	Maximum value for the range in total subsidence that can be expected when drained, expressed in inches (organic soils only).
subtotl	comp	Total Subsidence	Minimum value for the range in total subsidence that can be expected when drained, expressed in inches (organic soils only).
suitcode	woodland	Woodland Tree Suitability	Code indicating if the tree is common to the site; Existing (E), or a tree which could be planted as a tree crop; Potential (P). Trees which are both existing and have a potential for planting are giving a dual code (EP).
surftex	comp	Surface Soil Texture	Code for the USDA texture for the surface layer or horizon. Example: Loam (L); Sandy loam (SL). Also includes terms used to modify texture and terms used in lieu of texture.
table	table element tblelt	Table Name	Data dictionary - Identifies the short name used to identify the table in the data base.

Element	Tables	Long name	Description
texture	layer	Soil Texture Class	Code for the USDA texture for the specified layer or horizon of the soil. Example: Sandy Loam (SL); Loam (L). Also includes terms used to modify texture and terms used in lieu of texture.
tfact	layer	T Factor	Soil loss tolerance factor. The maximum rate of soil erosion that will permit a high level of crop production.
unified	layer	Unified Soil Classification	The Unified soil classification. An engineering classification of soils.
wdequip	woodmgt	Woodland Equipment	Woodland limitation rating for the use of equipment, year round or seasonal.
wderosn	woodmgt	Woodland Erosion	Woodland limitation rating identifying the probability that damage may occur as a result of site preparation and following cutting operations where soil is exposed.
wdplant	woodmgt	Woodland Plant Competition	Woodland limitation rating for the likelihood of the invasion or growth of undesirable species when openings are made in the canopy.
wdseed	woodmgt	Woodland Seeding Mortality	Woodland limitation rating identifying the probability of death of naturally occurring or planted tree seedlings as influenced by kinds of soil or topographic conditions.
wdwind	woodmgt	Woodland Windthrow Hazard	Woodland limitation rating identifying the windthrow hazard. Windthrow is the likelihood of trees being uprooted by wind as a result of insufficient depth of the soil to give adequate root anchorage.
weg	layer	Wind Erodibility Group	The wind erodibility group (weg) assigned to the soil layer or horizon.
wei	layer	Wind erodibility index	The wind erodibility index assigned to the soil layer or horizon.
wlconif	wlhabit	Wildlife Habitat Element (coniferous trees)	Suitability of the soil to produce the wildlife habitat element coniferous trees.
wlgrain	wlhabit	Wildlife Habitat Element (grain)	Suitability of the soil to produce the wildlife habitat element grain.
wlgrass	wlhabit	Wildlife Habitat Element (grass)	Suitability of the soil to produce the wildlife habitat element grass.
wlhard	wlhabit	Wildlife Habitat Element (hardwood trees)	Suitability of the soil to produce the wildlife habitat element hardwood trees.
wlherb	wlhabit	Wildlife Habitat Element (herbaceous plants)	Suitability of the soil to produce the wildlife habitat element herbaceous plants.

Element	Tables	Long name	Description
wlopen	wlhabit	Wildlife Habitat Potential (openland)	Suitability of the soil to produce the habitat requirements for openland wildlife.
wlrange	wlhabit	Wildlife Habitat Potential (rangeland)	Suitability of the soil to produce the habitat requirements for rangeland wildlife.
wlshlwat	wlhabit	Wildlife Habitat Element (shallow water)	Suitability of the soil to produce the habitat element shallow water.
wlshrub	wlhabit	Wildlife Habitat Element (shrubs)	Suitability of the soil to produce the wildlife habitat element shrubs.
wlwet	wlhabit	Wildlife Habitat Potential (wetland)	Suitability of the soil to produce the habitat requirements for wetland wildlife.
wlwetplt	wlhabit	Wildlife Habitat Element (wetland plants)	Suitability of the soil to produce the wildlife habitat element wetland plants.
wlwood	wlhabit	Wildlife Habitat Potential (woodland)	Suitability of the soil to produce the habitat requirements for woodland wildlife.
wndbrkht	windbrk	Windbreak Tree Height	Windbreak tree height in feet at age in 20 years.
woodprod	woodland	Production Class	Production class information for a specific tree measured in cubic meters per hectare per year (1 m³/ha = 14.3 ft³/ac).
wtbeg	comp	Water Table Begins	Month in which seasonal water table occurs at the depth specified in a normal year.
wtdeph	comp	Water Table Depth	Maximum value for the range in depth to the seasonally high water table during the months specified.
wtdepl	comp	Water Table Depth	Minimum value for the range in depth to the seasonally high water table during the months specified.
wtend	comp	Water Table Ends	Month in which seasonal water table subsides below the depth specified in a normal year.
wtkind	comp	Water Table Kind	The type of water table: Apparent (APPAR); Artesian (ARTES); Perched (PERCH).
yldunits	yldunits	Yield Units	The units used to record the yield for the specified crop.

APPENDIX B

RECOMMENDED TEST PROCEDURE FOR SULFATE SOILS

Objectives: To delineate sulfate-bearing subgrade soils prior to stabilization for road construction.

1.   **Collect Background**.

1.1 *Scope*: To obtain as much information of the composition of the subgrade soils as possible prior to construction to determine the most effective stabilization methods.

1.2 *Equipment*: Geologic Atlas of Texas, USDA Soil Survey for specific county where construction is to commence and any other geologic maps or literature available for the construction site.

1.3 *Procedure*: Look at the maps to determine the geologic formation and soil types on which construction will be performed. Look for key words in geologic and soil descriptions such as: anhydrite, gypsum (also called alabaster, satin spar, selenite/selenitic), pyrite/pyritic, and marcasite. These are all sulfur-bearing minerals that can cause sulfate heave.

2.   **Field Test**.

2.1 *Scope*: To identify soils that may have sulfate concentrations too high for traditional calcium-based stabilizers.

2.2 *Equipment*: Portable pH/conductivity meter, conductivity standards, 125 ml HDPE Nalgene bottles (one for every sample taken in the field), portable scale, double-distilled water, stopwatch, graduated cylinder (100 ml), 500 ml wash bottle for double-distilled water.

2.3 *Sample Preparation*: Collect soil samples from subgrade to be stabilized at surface and near base of material to be stabilized. For instance, if one is stabilizing 6 inches of subgrade, then collect a sample from the surface to 6 inches down. Let the sample air dry before weighing.

2.4 *Procedure:* Calibrate conductivity and pH meter per manufacturers' instructions. Estimate conductivity and pH and calibrate with standards close to those estimates. For example, a carbonate rich sample will be basic so standardize pH with a pH 10 standard in addition to the pH 7 standard.

- Measure 5 g to the nearest 0.1 g of air-dried soil into a 125 ml (HDPE) Nalgene brand bottle.

- Measure 100 g to the nearest 0.1 g of double-distilled water into the bottle.

- Shake the sample by hand for 1 minute.

- After shaking the sample, immediately take conductivity and pH measurements with the pH/conductivity meter.

- After a minimum of 12 hours has elapsed, agitate the sample again for 1 minute.

- After shaking the sample, immediately take conductivity and pH measurements.

2.5 *Interpretation of Data*:   Upon completion of this test, there should be two conductivity measurements approximately 12 hours apart.   Absolute conductivity values are difficult to determine because every soil will be different, so the district personnel will have to experiment with different soils to see what should be typical conductivities for their soils.   However, if the second reading is more than 100 μS greater than the first, then this should raise a red flag to test the sample for sulfates following the procedure outlined below.   Also, if conductivity is greater than 200 μS test for sulfates following the procedure outlined below.

3. **Field Office Test/Lab Test.**

3.1 *Scope*: To determine the actual sulfate concentration (parts per million) in subgrade soils.

3.2 *Equipment*: 2.5 cm Whatman #42 filter paper or 12.5 cm Fisher Q2 filter paper, 80 ml Nalgene funnel, 250 ml beaker, portable colorimeter/spectrophotometer (for sulfate analysis), sulfate reagent tablets, latex gloves, graduated cylinder (10 ml), spatula, 2 ml disposable pipettes (one for each sample), 2 pipette bulbs (an extra for breakage or loss).

3.3 *Sample Preparation*:   The sample produced in the field test section will be used for this analysis.

3.4 Procedure: Shake the sample by hand for 1 minute.

- Filter with Whatman #42 or equivalent, 12.5 cm, filter paper into a 250 ml beaker. Centrifugation may be necessary with fine-grained soils to remove all particulates from suspension.

- Put on latex gloves.

- Fill sample vial with filtrate to the 10 ml mark; a 2 ml pipette may be necessary to fill vial.

- Switch the unit to "ON."

- Press the MODE key until the desired method is displayed.

- Wipe the sample vial with a lint-free cloth (Kimwipes) to remove any oils from the vial to ensure repeatable results.

- Place the sample vial into the sample chamber with the Δ vial mark aligned with the Δ housing mark.

- Press the ZERO/TEST key. The method symbol flashes for approximately 3 seconds and confirms zero calibration.

- After zero calibration, remove the vial from the sample chamber.

- Add sulfate tablet to vial without touching the tablet with hands and lightly tamp the tablet with white plastic rod until tablet starts to crumble. Tamp vigorously 20 to 30 times in a random circular motion until tablet is dispersed throughout the sample vial.

- Cap vial and immediately place in the sample chamber with the Δ marks aligned.

- Press the ZERO/TEST key. The method symbol flashes for approximately 3 seconds and the result appears in the display. Take three readings and average.

- This test will only read concentrations from 5-200 mg/l. If (÷**Err**) message appears, then the measuring range has been exceeded or there is excessive turbidity. This will require diluting the sample with more double-distilled water and measuring until the message disappears and there is a numerical answer. If (**-Err**) message appears, then result is below the measuring range.

- If the measuring range has been exceeded, then measure out 5 ml of the filtrate into a 10 ml graduated cylinder and add exactly 5 ml of double-distilled water with a 2 ml pipette. This will change the dilution ratio to 1:40 and increase the measurable range to 8000 mg/l, or ppm.

- Calculate sulfate concentration in parts per million (ppm) by multiplying the average of the three readings by the dilution ratio used. For example, the sample was mixed in a 1:20 ratio of soil to water in the conductivity test. If one obtains an average sulfate reading of 50 mg/kg, then just take 50 X 20 = 1000 ppm sulfate in the soil. If a 1:40 dilution ratio was used, then multiply 50 by 40 to obtain 2000 ppm.