QUANTIFICATION AND GEOSTATISTICAL MAPPING OF SOLUBLE SULFATES IN SOILS ALONG A PIPE LINE ALIGNMENT

by

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ABSTRACT

QUANTIFICATION AND GEOSTASTICAL MAPPING OF

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Sulfate induced heave is a growing concern for civil engineering projects utilizing calcium based soil stabilizers in clay soils. Therefore, addressing sulfate concentrations in soils is vital to the success of a civil engineering project. Pavement, foundations, tunnels, embankments, pipe lines, etc. are all susceptible to sulfate induced heave if the appropriate conditions and constituents are present. The work presented here within focuses on the soluble sulfate testing completed on six (6) different geologic formations within the Integrated Pipe Line Project (IPL) alignment of North Texas. The testing and analyses were conducted to identify problematic zones and quantify sulfate values in order to prevent damage to the pipeline due to sulfate induced volumetric expansion. By identifying problematic zones within the alignment, maintenance costs due to pipeline damage caused by sulfate induced heave can be reduced or prevented. In addition to testing, mapping of the soluble sulfate concentrations using Surfer 9 geostatistical software was implored in hopes to further identify and visualize sulfate concentrations along the pipe line alignment. Following the contour mapping, geostatistical validation was conducted to determine the validity of the contour map projections. Explanations of the interpretation of the test data, map results, as well as recommendations are made. Due to the random manifestation of sulfates in the soil, all analyses and test results presented in this

work are strictly dependent on the data set analyzed and further analysis of sulfate values can change the interpretation and results of the work presented in this thesis.

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CHAPTER 1

INTRODUCTION

1.1 General

The Dallas/ Fort Worth (DFW) Metroplex is experiencing a large population growth, specifically over 120,000 new residents from July 2010 to July of 2011 (U.S. Census Bureau). This rapid growth has placed high demands on water resources and the estimated continued growth will further strain our water supplies in the years to come. The solution to the problem, The Integrated Pipeline (IPL) project, is a joint effort between the Tarrant Regional Water District (TRWD) and Dallas Water Utilities (DWU) that will bring additional water supplies to the DFW area. This project involves design and installation of approximately 150 mile pipeline that collects and transfers water from lakes such as Richland Chambers, Cedar Creek and Lake Palestine to the Metroplex. As a part of the pipeline layout and construction, large amounts of soil might be chemically stabilized to ensure proper strength and reduce volumetric expansion. It has been understood that improper stabilization of soils containing soluble sulfates can cause unwanted expansion that results in poor performance and damage to infrastructure.

Both lime and cement based stabilizers contain calcium, which when in contact with soluble sulfates and reactive alumina of soils results in the formation of Ettringite. This sulfate mineral undergoes large volumetric expansion when hydrated or through crystal growth and can ultimately fail the structure resting on the treated soil through strength or serviceability loss; therefore a comprehensive understanding of the concentration of such soluble sulfates can reduce the negative effects of calcium-based stabilization. Threshold studies (Harris 2004; Puppala 2005; Adams 2008) have been conducted to determine what the limiting sulfate concentration is, that induces heave. However, the results of the those studies are not conclusive and indicate that soils containing 2000 ppm to 5000 ppm of sulfates or higher are considered problematic (Harris 2004; Puppala 2005; Adams 2008). When soils with such quantities of sulfates are treated with calcium based stabilizers, sulfate induced heaving occurs.

The relevance of soluble sulfate concentration measurement to this study is to assess any heave related problems that can cause damage to the pipeline or its related structures if soils treated with calcium based stabilizers are used as pipe bedding or backfill materials. By identifying the zones along the pipeline alignment that contain soluble sulfates and avoiding calcium based chemical treatments in these zones, a reduction in infrastructure damage and there by long-term project cost savings can be achieved. This research focuses on establishing these zones of concerns by testing soils from different geological formations along the pipeline alignment and using geostatistics to develop contour maps that will help in visualizing the problem zones along the pipeline alignment.

1.2 Research Objective and Scope

As mentioned above the main objective of this research was to establish problematic sulfate zones along the IPL alignment and develop a contour map showing soluble sulfate concentrations along the alignment. For this purpose the research was undertaken in two phases. The first phase of the research project focused on identification and quantification of soluble sulfate concentrations through chemical testing on soils from select borings from 6 different geologic formations along the alignment of the pipeline. The six geologic formations selected for this study were Eagle Ford, Ozan, Wolfe City Sand, Neylandville, Kemp Clay, and Wills Point. The selection of geological formations is based on the previous experience of UTA and Fugro Consultants Inc. with regard to soluble sulfate concentrations and the ability to create valid geostatistical maps. The soluble sulfate content concentrations in various soil samples from these formations were determined using the “Modified UTA Method” as outlined in Puppala (2002). The second phase focused on the use of geostatistics as a tool to aid in further identification of soluble sulfates in locations that were not tested in these 6 formations and develop usable geostatistical maps to aid the design engineers. A validation of the geostatistical maps was then carried out using a Kriging error technique by establishing a 45° comparison line for estimate, Kriged values, versus true test, input values. This thesis summarizes both phases of the soluble sulfate study and the results of the geotechnical investigations conducted to identify, quantify, and map soluble sulfate concentrations along the pipeline alignment.

Figure 1.1 shows the Integrated Pipe Line alignment highlighting the reservoirs and lakes to be connected and the counties the IPL lies in. Figure 1.2 shows the IPL alignment overlain on a geological map and highlights the formations utilized for testing in this project.

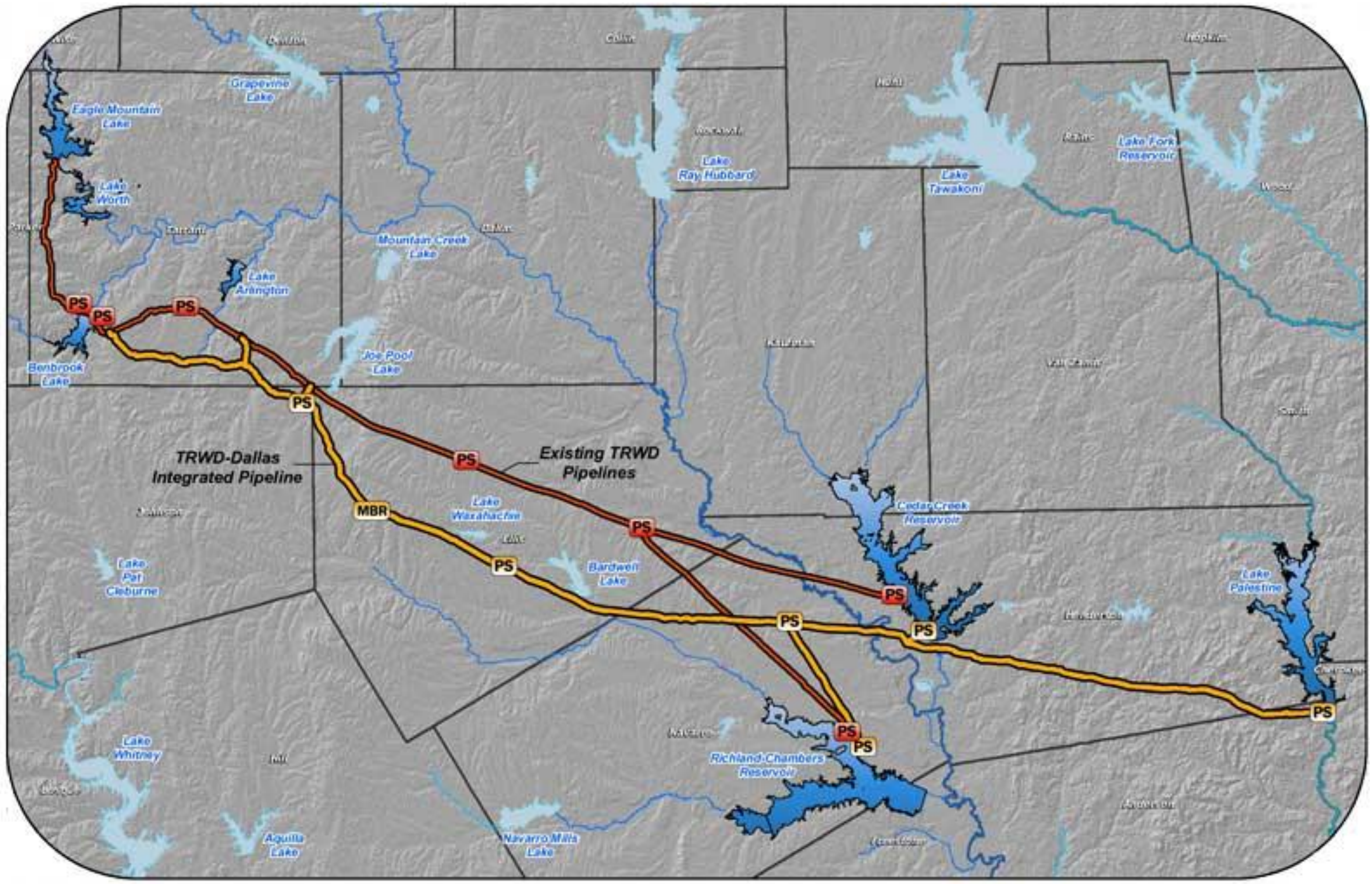

Figure 1.1 IPL Project Alignment Map Showing Existing Pipeline and New Pipeline Source: TRWD

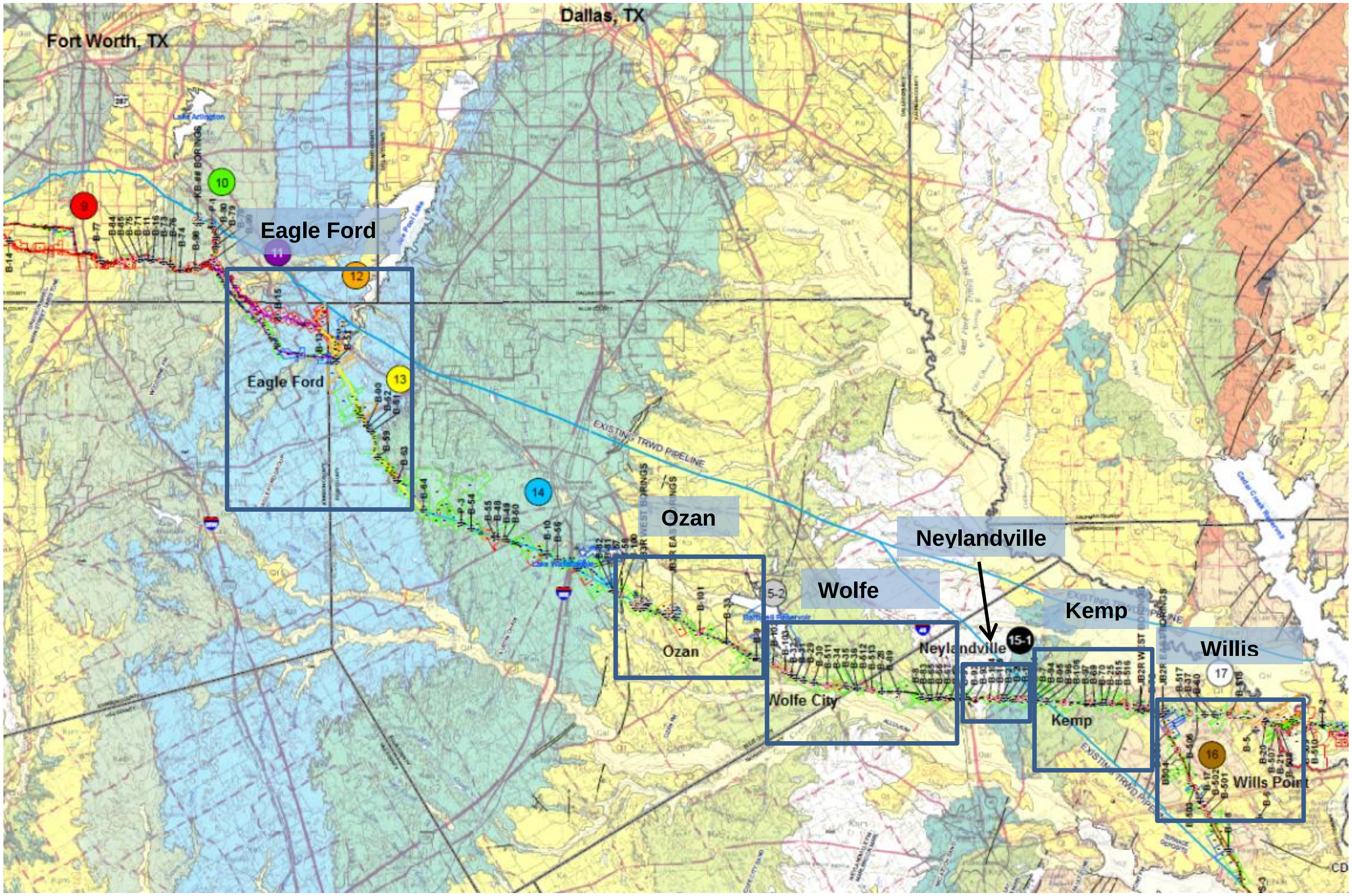

Figure 1.2 Geophysical Map of the IPL Alignment Highlighting Test Formations Source: Fugro Consultants Inc.

1.3 Thesis Organization

This thesis consists of five chapters: Chapter 1: Introduction, Chapter 2: Literature Review of Sulfates in Soil and Geostatistics, Chapter 3: Experimental Program and Results, Chapter 4: Geostatistical Analysis and Mapping and Chapter 5: Conclusions, recommendations, and future research needs/implications.

Chapter 1 provides the introduction to the sulfate study, the research objectives, and thesis organization.

Chapter 2 provides a comprehensive summary of studies that include: Sulfate induced heave in soils, the origins on sulfates in soils, the heaving mechanisms, case studies on sulfate induced heave, sulfate attack on civil engineering projects, mitigation methods for high sulfate soils, and a comparative evaluation of sulfate testing methods. Following sulfate heave review, a brief review of the history of geostatistics and use of geostatistics in geotechnical projects is discussed. All literature utilized for the compilation of this chapter is presented in text as well as in the References section at the end of the paper.

Chapter 3 presents the three main test methods considered for the sulfate testing, the procedures and equipment associated with those methods, the soil selection and sampling, the results of sulfate testing, and the deterministic analysis of the sulfate test results.

Chapter 4 provides the geostatistical analysis and mapping results as well as the validation of the geostatistical mapping.

Chapter 5 provides an overall summary of test results, geostatistical mapping, and geostatistical validation. Conclusions and connections are also provided on the above mentioned topics in this chapter. Lastly, implications on newer studies concerning the IPL project as well as recommendations for further testing and analysis are provided in this chapter. Following chapter five, all references and programming used for this study are cited.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter is intended to be a comprehensive compilation of sulfate heave related information as it applies to soils and a compilation of fundamentals and applications of geostatistics in geotechnical engineering. The literature review presented in this chapter was collected from conventional library resources such and databases for the University of Texas at Arlington Library, electronic search engines, and various reports and technical papers accumulated by Dr. Puppala's graduate geotechnical research team. The organization of the literature review begins with an introduction to sulfate heave in soils first, and is followed by the documentation on the manifestations of sulfates in soils. Heave mechanisms and theories of heave involved in sulfate soils are discussed, followed by threshold of problematic sulfate levels in soils. Various case histories involving sulfate heaving and the mitigation methods to control these heaves are also detailed. A comparative evaluation of sulfate testing methods is also presented as the justification for selecting the method for sulfate testing. After establishing the literature review for sulfate bearing soils, geostatistics is discussed in two main sections. The first section explains the inception of geostatistics, its natural evolution, and a basic understanding of geostatistical approaches. The second section of the geostatistics portion of the literature will document case studies and technical papers pertaining to the use of geostatistics in geotechnical engineering applications.

2.2 Sulfate Heave

Often in geotechnical engineering projects, especially those involving expansive soils are required to have chemical stabilization to reduce the volumetric expansion due to moisture and salt influx, increase durability, or to achieve proper strength specifications for the particular design (Mitchell 1992; Dermatas 1995; Kota 1996; Azam 2003). Chemical stabilization typically takes place in the form of lime or cement, both of which are cost effective and contain appreciable amounts of calcium. Lime induced sulfate heave has been documented by Mitchell (1986), and Mitchell (1992), among others. Stabilization with lime creates a chemical reaction that forms an interlocking gel between the clay particles and ultimately increases strength, reduces plasticity, increases workability, and reduces in swell behavior (Dempsey and Thompson 1968; Bell 1989). Additionally, Kota (1996) and Ksaibati (1996) have shown that cement based stabilizers also induce sulfate heave. Stabilization with cement creates a poizzalonic reaction that lowers the plasticity of the soil and also produces gels that increase the strength of the soil and reduce swelling potential (Bugge and Bartlesmeyer 1966; Nelson and Miller 1992).

Clayey soils generally consist of three minerals with one generally dominating: Kaolinite, Illite, and Montmorillonite and all three are susceptible to sulfate induced heave (Wang 2004). All three of these minerals naturally contain alumina (Aluminum Oxide, $\mathrm{Al_2O_3}$). The introduction of calcium in the stabilizers in the presence of free alumina in the soil and soluble sulfate minerals can create a calcium – alumina – sulfate hydrate compound known as Ettringite $(\mathrm{Ca_6(AlOH_6)_2(SO_4)_3(OH_{12})} * 26\mathrm{H_2O}$ or a calcium – silica – hydroxide – sulfate – hydrate compound known as Thaumasite $(\mathrm{Ca_3Si(OH)_6(CO_3(SO_4)} * 12\mathrm{H_2O})$ (Sherwood 1962; Mehta and Klein 1966; Mehta and Wang 1982; Mitchell 1986; Hunter 1988; Perrin 1992; Petry 1994; Burkhart et al 1999). Sulfate heave in soils was introduce to geotechnical engineering in 1986 by Mitchell and since that time both minerals have been well documented to cause heaving in soil (Dermatas 1995; Little 2005; Puppala 2007). The heave associated with these reactions creates distress and eventually poor performance of the infrastructure and can ultimately reduce the life time of the structures (Puppala 2002). Various case studies of sulfate induced heave failures were reported across the United States (Mitchell, 1986; Hunter, 1988; Kota et al., 1996). In some cases, the cost of repairs exceeded the cost of stabilization (Hunter, 1988).

Sulfate – bearing soils are found in many regions in the United States, particularly in the Western and Southwestern United States (Puppala et al. 2002). These states include Texas, Nevada, Louisiana, Kansas, Colorado and Oklahoma (Solanki et al. 2010). Studies conducted by Burkhart et al. (1999) identified certain geologic formations that possess high sulfates and that gypsum was the most common sulfate mineral in Dallas area soils. Therefore, in these regions it is very important to identify and quantify sulfate bearing soils in order to prevent damage to infrastructure due to sulfate induced heave. Sulfate related heave and failures have gained notoriety over the years as more and more design professionals become aware of the effects of sulfate related heave on civil engineering projects. One of the most severe cases of sulfate induced heave in the DFW area was associated with the Eagle Ford formation, where sulfate concentrations were found ranging from 4,000 ppm to 27,800 ppm (Chen et al., 2005). High sulfate concentrations of 35,540 ppm have been reported in the Childress district located in the Pan Handle area of North Texas (Zhiming 2008). Gaily (2012) conducted further studies on Texas soils containing sulfates to identify other factors such as soil type, lime dosage, and mellowing period on sulfate related heave and Figure 2.1 on the following page identifies the location and sulfate concentrations of these test soils.

Sulfate induced heave or chemical swelling is a long term scenario which often worsens as time passes (Hunter 1988). Unlike physical heaving, the rate of chemical heaving often stays constant or increases with time (Ferris et al. 1991). Figure 2.2 on the following page depicts specific counties within the state of Texas that have sulfate bearing soils as well as highlights the Eagle Ford formation, which has been previously established to be highly problematic for sulfate induced heave (Harris et al. 2004; Chen et al. 2005).

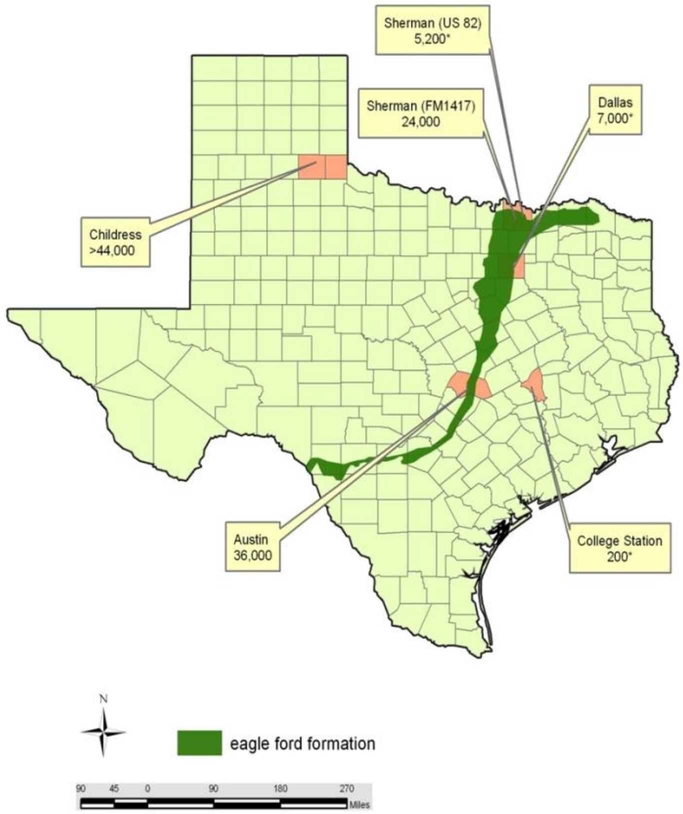


Figure 2.1 Sulfate Concentrations and Locations of Test Soils (Gaily 2012)

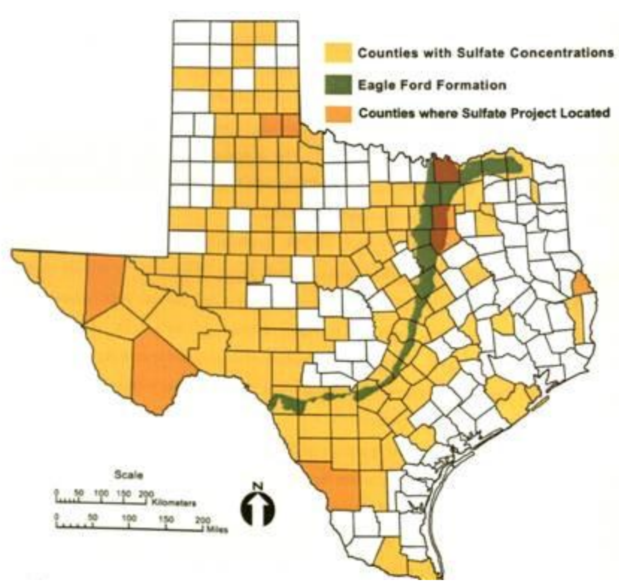


Figure 2.2 Location Map Showing Sulfate-bearing Soils in Texas (Harris et al., 2004)

2.3 Sulfate Compounds in Soil

A variety of sulfate compounds exist in both nature and in soil and can manifest as a solid, liquid, or a vapor (Chikyala 2007). Soils and rocks are examples of solid state sulfates, while air and water examples in their respective states (Hawkins et al 1998). Sulfates in soils can manifest in two different forms. The first form is primary sulfate sources and the other is secondary sulfate sources. Primary sulfate sources come from compounds that exist or are formed naturally, while secondary sources are the products of other chemical reactions, such as oxidation, or from engineering practices. Both primary and secondary sulfate sources are discussed in the sections below as both sources can contribute to sulfate heave.

2.3.1 Primary Sources of Sulfate Sources in Soil

Most sulfate minerals are created and deposited through evaporation of salt water followed by the precipitation of salts (Zanbeck et al. 1986). Typically Anhydrite $(\mathrm{CaSO_4})$, Gypsum or Calcium Sulfate Dihydrate $(\mathrm{CaSO_4} * 2\mathrm{H_2O})$, Halite (NaCl), and Dolomite $(\mathrm{CaMg(CO_3)_2})$ are formed through this process (Zanbeck et al. 1986). Halite is more commonly known as sea salt and is not a sulfate mineral nor is Dolomite, which is carbonate mineral. These minerals were included in the discussion for the sake of completeness. Evaporation minerals are more commonly found in the upper crust and manifest in as evaporate, clay, or fine grain sediment deposits (Zanbeck et al. 1986). Texas soils are primarily made of clay or fine grain sedimentary deposits; therefore the most common primary sulfate source of sulfate in Texas soil is in the form of gypsum. In addition to being formed naturally through the evaporation and precipitation method described previously, gypsum can also be formed as a result of the breaking down of anhydrite into an aqueous solution that then recrystallizes to form gypsum (Indiana Geological Survey).

In addition to gypsum, other primary sources of sulfates in soil are Thenordite or Sodium Sulfate $(\mathrm{Na_2SO_4})$, and Epsomite or Magnesioum Sulfate $(\mathrm{MgSO_4} * 7\mathrm{H_2}0)$. Sodium sulfate and Magnesium sulfate tend to manifest in more arid regions (Hilgard 1906; FAO 2001;

Bing et al. 2007). Figure 2.3 shows other regions in the United States that have soils that contain gypsum. Figures 2.4.a through 2.4.e display soil samples containing gypsum utilized in this research project. Figure 2.5 shows a sodium sulfate sample from the National Museum of Natural History and Figure 2.6 shows a sample of epsomite from the Dr. Geier Mine in Germany.

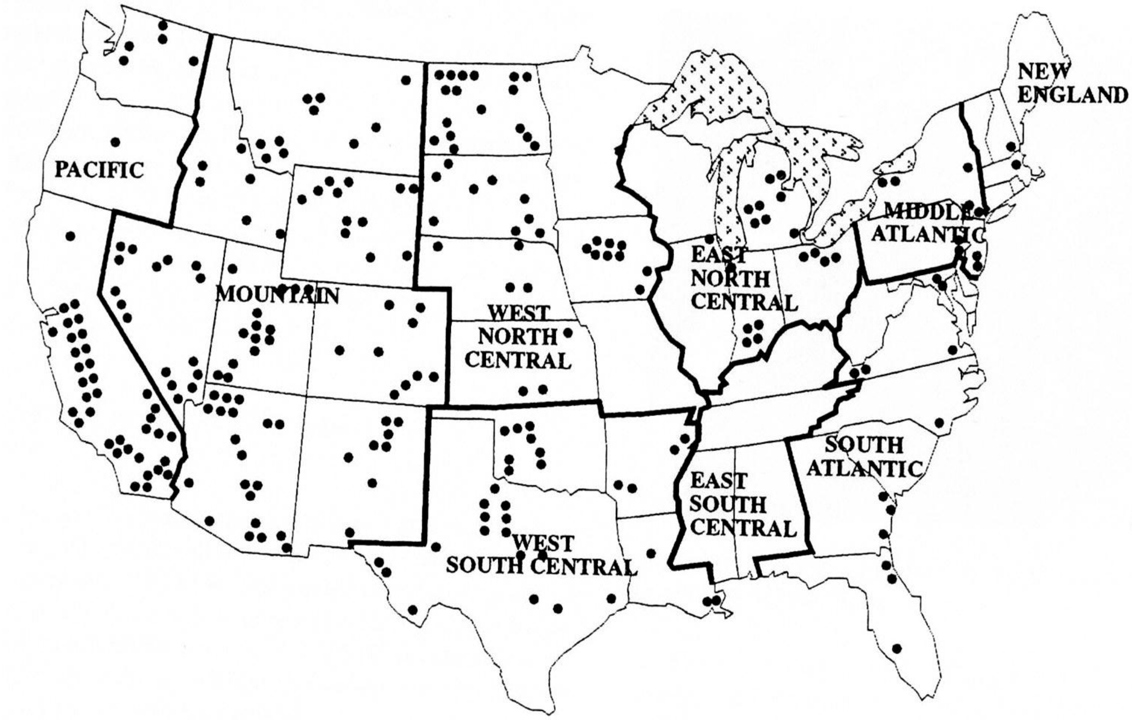
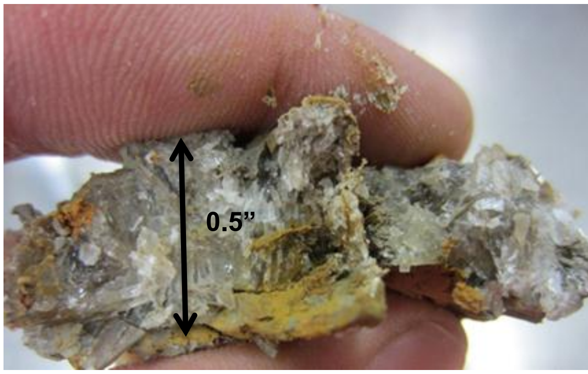
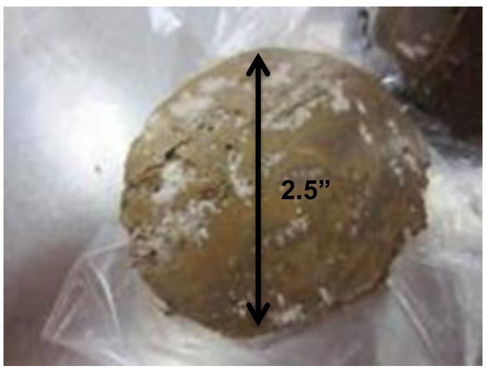


Figure 2.3 Location of soils containing Gypsum in United State
(Kota et al., 1996)

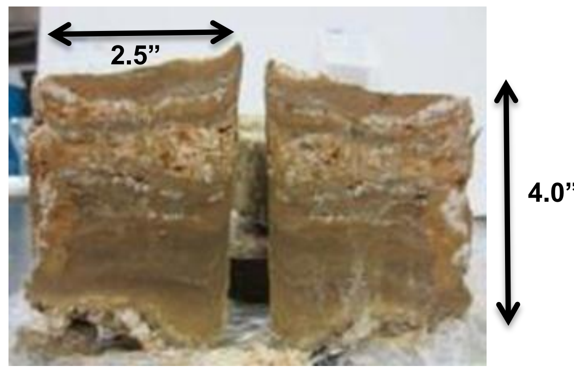


(a)

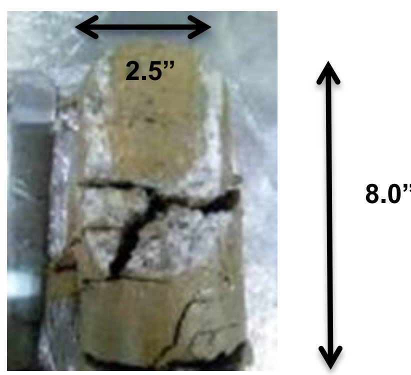
(b)



(c)



(d)



(e)

Figure 2.4.a through 2.4.e Various Types of Gypsum Manifestation in Test Soils

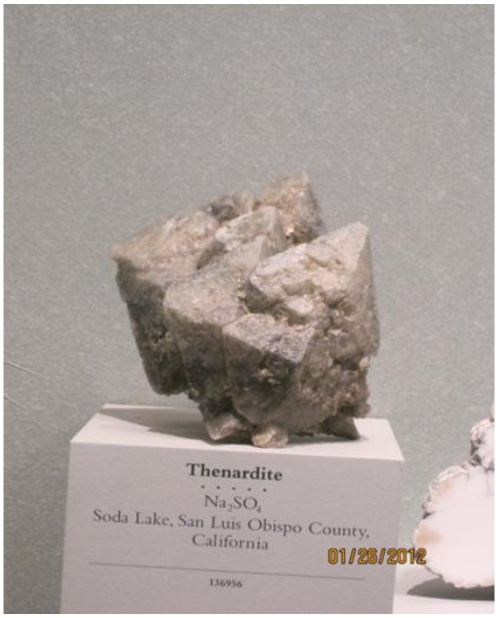


Figure 2.5 Thenardite Sample form U.S. Natural History Museum

Figure 2.6 Epsomite from Dr. Grier Mine, Germany

2.3.2 Secondary Sources of Sulfate in Soil

Secondary sulfate sources can manifest in many different ways. One secondary source of sulfates in soils comes from the decomposition of pyrites (FeS_2). Pyrites break down when oxidized and can convert into sulfate minerals, like gypsum, that may then be dissolved and transported by ground water (Steger and Desjardins 1977; Dubbe et al 1997, Bryant et al. 2003; Harris et al. 2004). In addition to pyrite oxidation, the use of Chromite Ore Processing Residue (COPR), a byproduct of the chromite ore lime – based roasting process used to isolate and extract soluble chromate (CrO_4^{2-}), in structural fills can react under certain soil conditions to from gypsum (Dermatas 2006). Other studies that discuss soluble sulfates and their ability to be transported and redistributed by groundwater flows were conducted by Skousen et al. (1996), and Puppala (2005). An example of transportation of sulfates through ground water flows would be during the wet season when soluble sulfates in the top layers of the soil dissolve and move downward by gravity into stabilized layers, and conversely during the dry season dissolved sulfates move upward due to evaporation or capillary rise into the top layers increasing their concentration (Dermatas, 1995; Natarajan 2004). Additionally, soluble sulfates may come from water sources used in construction. For example, Rollings et al. (1999) conducted a forensic investigation on the failure of Bush Road in Georgia. Five months after construction, distress cracks and bumps from soil heave were observed. The investigation concluded that the failure was due to the formation of Ettringite in the subgrade material. However, initial geotechnical testing observed no sulfates in the subgrade soil and the subgrade soil was treated with cement. It was determined at the conclusion of the study that water from a nearby well used for mixing concrete and for field compaction purposes contained sulfates and the introduction of this sulfate water to the subgrade soil combined with the cement stabilization induced sulfate heave. Similarly, the Yongam Dam, China was also contaminated with sulfate bearing water used in construction which ultimately led to the formation of Thaumasite and the eventual degradation of the concrete used in the dam construction (Mingyu et al. 2006)

2.4 Sulfate Heave Mechanisms

Sulfate attack in Portland cement concrete was established in the early 19th century (ACI1982; DePuy 1994). At that time it was proven that calcium rich cement mixed with sulfate and free alumina could create Ettringite and Thaumasite minerals (ACI 1982). Additionally, the formation of Ettringite and its subsequent growth in concrete was explained by Cohen (1983). Cohen established two different growth mechanisms for Ettringite and Thaumasite, crystal growth and hydration. Similarly, in soil the same two expansion mechanisms were established (Dermatas 1995). The first mechanism causes expansion due to the formation and/or oriented crystal growth of Ettringite (Ogawa and Roy, 1982). The second is a thorough solution mechanism where expansion is related to swelling due to hydration Ettringite (Mehta 1973; Mehta and Wang 1982).

2.4.1 Heaving due to Crystal Growth

Crystal growth theory dictates that aluminum, calcium, and sulfates can concentrate around Ettringite nucleation sites and combine to form additional Ettringite, essentially building onto the internal lattice structure of the Ettringite molecule (Ogawa and Roy 1982). As per the theory, this crystal growth occurs at the beginning stages of cement hydration and as water is introduced to the system, the crystals form their needle like shape. As the crystals build they begin to come in contact with one another, exerted pressures on each other and causing the whole system to swell. The pressure generated by the crystal growth is then exerted on the surrounding soil inducing strains and pressures. Once the load applied through crystal growth exceeds that of the capacity of the restraining medium, heave occurs. Crystal growth of Ettringite is favored in high pH ranges. As a solution becomes more basic alkaline earth metals, such as calcium, and alumina dissolve more readily. The dissolution of these constituents drives the reaction and the formation of Ettringite. Soil is a more plastic substance than concrete and allows for the accommodation of some Ettringite growth. However, at some point as the reaction continues the soil can no longer absorb the strains and pressures exerted by the growth of the Ettringite crystals and heaves, transferring the stress to the next path of least resistance.

2.4.2 Heaving due to Hydration

Swelling due to hydration was proposed by Mehta (1973). The theory suggests that formation of Ettringite follows a thorough solution mechanism. In the presence of saturated calcium hydroxide $(Ca(OH)_2)$, the rate of hydration of aluminum decreases significantly, causing the Ettringite to form gel – like and colloidal crystals. The resulting colloidal crystal gels are hydroscopic in nature and can adsorb large quantities of water molecules due to their high surface area and net negative charge. As more water molecules are absorbed into the system, the gel begins to swell exerting pressures and strains on the surrounding medium. Mehta and Wang (1982) observed that the amount of expansion of the Ettringite gel is directly related to the amount of water adsorbed and the size of the crystals. Coarser colloidal crystal gels were found to expand more than finer colloidal crystal gels. The factor influencing the type of crystals is related to the hydroxyl levels. High hydroxyl levels develop colloidal crystal gels, whereas low hydroxyl levels create rod like crystals that expand much less.

2.5 Threshold Sulfate Levels

Problematic threshold levels for sulfates in soils are a difficult concept to establish. Some cases report these levels as being between 1,500 ppm to 5,000 ppm and in other cases levels as high as 10,000 ppm are reported as threshold levels (Harris et al 2004; Puppala et al. 2005; Adams et al. 2008). Unfortunately, soil conditions such as plasticity, density, and void ratio coupled with stabilization techniques and environmental factors largely affect these threshold levels (Puppala 2005). Therefore, setting threshold levels “across the board” is nearly impossible (Adams et al. 2008). There are studies hoping to determine sulfate threshold levels based on mineralogy and geological depositional environments, but this will only address some of the issues associated with sulfate heave thresholds (Adams et al. 2008). With this in mind, it is the opinion of the author that until such studies can conducted and confirmed, threshold levels for sulfate soils should be conducted on a case by case basis, considering all potential factors related to sulfate heave.

2.6 Case Studies

There are many case studies from all over the United States to the United Kingdom and China that document sulfate induced heave (Hawkins 1987; Little 1989; Wimsatt 1999; Chen et al., 2005; Mingyu 2006; Rollings et al. 2006; Zhiming, 2008; Adams 2008; Bagley et al. 2009; Puppala et al. 2010). Primarily these case studies were under taken as the direct result of infrastructure failures and were conducted to determine the causes of failure. In the cases presented in the following sections, the infrastructure failures are due to the growth of Ettringite or Thaumasite and are primarily due to the improper stabilization or identification of sulfate soils. The details of these studies, conclusions drawn, and innovations in sulfate heave forensics are described

2.6.1 Forensic Investigations to Evaluate Sulfate Induced Heave Attack on a Tunnel Shotcrete Liner. Dallas, Texas

Considering the similarity of a pipe line with a tunnel structure, the Puppala et al. 2010 study is the most relevant to this research project. As such, this study will be discussed in detail, first. In this study, cracking and water leakage was discovered in the shotcrete liner of a tunnel in Dallas, Texas. The tunnel was built in the Eagle Ford Formation, one of the test formations in this sulfate pipeline study. The surrounding tunnel material was limestone. As part of the forensic investigation coring was done on the liner at key areas of leakage and distress. Upon completion a white powder like material and gel like substance were found both within and behind the shotcrete liner. Samples of the powder and gel material as well as the rock core samples from distress locations were collected and tested at the University of Texas at Arlington (UTA) geotechnical laboratories. The samples were first subjected to mineralogical and micromorpholoical testing by XRPD and EDAX methods. Rock core samples were subjected to strength tests, such as, unconfined compressive strength (UCS), indirect tensile strength (ITS) and unconsolidated undrained (UU) tests. Figures 2.7, 2.8., and 2.9 show some of the distressed regions where samples were taken.

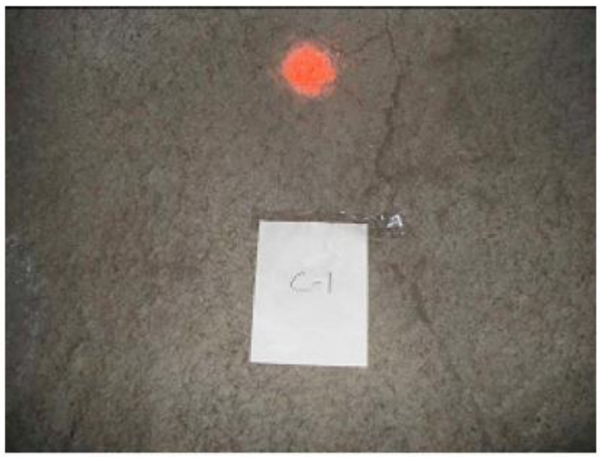


Figure 2.7 Distress Cracks form Sulfate Attack (Puppala 2010)

Figure 2.8 White Powder and Gel from Sulfate Reaction (Puppala 2010)

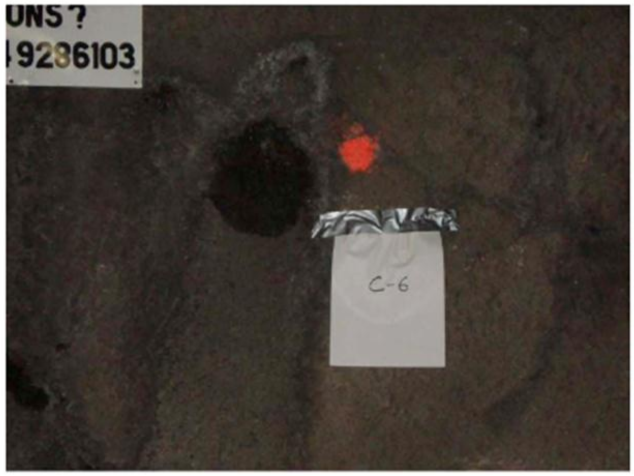


Figure 2.9 Core Hole from Forensic Investigation (Puppala 2010)

The results of the study discovered that the sulfate attack was due to the growth of both Ettringite and Thaumasite. Additionally, high moisture contents were related to higher levels of distress. UCS, UU, and ITS testing indicated a decrease in strength values with increasing distress magnitude. It was also determined that sulfate levels in the distressed areas ranged from 1,500 ppm and above. It was determined that sulfates percolated through the limestone and accumulated at the shotcrete liner, ultimately leading to the sulfate reaction that caused the distress in the tunnel liner. The results of this work indicated that the limestone had significant strength loss and that the tunnel was a potential safety hazard that needed to be monitored continuously.

2.6.2 Forensic Investigation of a Sulfate Heaved Project in Texas, U.S. 82

Considering the location of the IPL project, the discussion of case studies within the state of Texas are considered very relevant. This particular case study involved U.S. Highway 82 and was a Texas Department of Transportation (TXDOT) study conducted by Chen et al., 2005. The construction of U.S. 82 began in 2001 which made this study center on a relatively new pavement construction. The pavement was a 50 mm asphalt concrete over 300 mm flexible base, followed by 200 mm lime treated subgrade. As with the tunnel study previously discussed, the soil in question for this project was part of the Eagle Ford Formation. The project was initiated because heaving on the East side of the project area was observed and severely affected the performance of the pavement structure. During the investigation it was noted that within the sub grade soil, sparkling gypsum fragments were present, and this gypsum was likely the culprit in the heave distress to the pavement. Figure 2.10 depicts some of the gypsum crystals that were located at the site with their relative sizes. Figure 2.11, on the following page, shows the heave associated with the east side of the project.

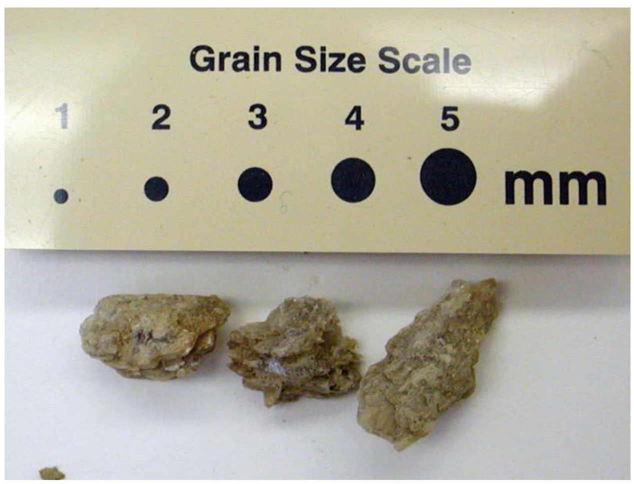


Figure 2.10 Gypsum Crystals Retrieved from the Investigation Site (Chen et al. 2005)

The study involved the coring of the pavement to obtain samples of the lime treated subgrade and underlying, untreated subgrade. Scanning Electron Microscope (SEM) analysis was conducted on the samples collected from the field. The SEM results on the treated subgrade indicated that crystals had formed and were confirmed to be Ettringite. Interestingly, no crystals were found in the untreated subgrades, either indicating that no sulfates were present at those depths or that stabilizer had not leached into the lower sub grade. Further testing included chemical analysis, conductivity, and pH for both the east and west side of the project. Conductivity testing indicated that as the depth increased conductivity increased significantly. Further investigation determined this increase in conductivity was due to soluble gypsum that seeped into the lower sub grade. Therefore, it was concluded from the study that conductivity measurements are a good indicator of soils with high amounts of soluble sulfates. Specifically, soils from the east side of the project were determined to have anywhere from 4,000-27,800 ppm of soluble sulfates, while the west side contained very low sulfate values.

Figure 2.11 Heaved Area on East side of Project Area (Chen et al. 2005)

2.6.3 Forensic Investigation of Premature Pavement Failure Due to Soil Sulfate Induced Heave, Childress, County U.S. 287 Texas

TXDOT's Materials and Pavement Division investigated large cracks and swells in pavement near Bear Creek Bridge on U.S. 287 in Childress County Texas (Zhiming 2008). Both North and South bound lanes had been previously reconstructed around 2001. Approximately 2 years after reconstruction the section of pavement near Baylor Creek began experiencing extensive fatigue cracking and swelling. At that point, restoration in the form of milling and inlays were under taken. Figure 2.13 shows the distress to U.S. 287 after heave distress set in. The culprit the heave associated with the premature failure of this pavement was due to the interaction of gypsum interbeds with the 9 in sub grade that was treated with lime at 3% by dry weight of soil. Figure 2.12 on the following page shows the gypsum interbeds as they appear adjacent to the pavement structure.

Subgrade soil samples from North and South bound lanes were collected and sent out for laboratory testing. Sulfate testing determined that soils in the North bound lane contained sulfate concentrations above 35,000 ppm, which was confirmed by the fact that the North bound lane experienced much worse distress and heave. Testing determined that the sulfate levels in the South bound lane were low in concentration. Further testing identified North bound soils as being fine grained whereas south bound soils were coarser in nature. This also helps to explain why the heave was greater in the North bound lane.

Figure 2.12 Gypsum Interbed near Baylor Creek U.S. 287 (Zhiming 2008)

After extensive lab testing for Atterberg limits, linear shrinkage, conductivity, moisture susceptibility tests, resonant column, and UCS, four basic remedy methods were suggested for proper stabilization of the sub grade soil. The first suggestion was complete removal of the sulfate soil and replacement with select fill. The second recommendation was the reworking of the subgrade with a lime and fly ash. The third suggestion was the use of geosynthetics such as a geogrid to mechanically reinforce the subgrade. The fourth option was to apply an over lay, mill and apply composites to the shoulder area of the pavement.

2.6.4 FM 201 Sabine County Pineland, Texas Sulfate Heave

FM 201 is located just east of U.S. 96, in East Texas near the city of Lufkin. The particular road in question was a TXDOT project and it was determined that large, "roller coaster", type bumps in the road were due to improper stabilization of expansive soils. The project was constructed on the Eocene Yazoo Formation which is clay, sandy; with inter beds of silt and glauconitic sand with marine fossils. Gypsum beds were seen the drainage wash surrounding the pavement. Cores of the pavement and the sub grade were taken from areas of high expansion and examined. Ettringite was found in these samples and Figure 2.11 below shows some of the gypsum crystal formations detected within the drainage washes from the study. The solution for this construction project was blanket removal of the top two feet of subgrade and reconstruction with cement stabilized select fill (Harris et al. 2006). Figure 2.13 depicts gypsum crystals discovered in the drainage wash adjacent to the roadway.

Figure 2.13 Gypsum Crystals Located in Drainage Wash Adjacent to the Roadway (Harris et al. 2006)

2.6.5 U.S. 67 Ellis County, Texas Sulfate Heave

In a study conducted by Wimsatt 1999 in conjunction with TXDOT on U.S. 67 it was determined that sulfate induced heave was responsible for the deterioration of a pavement structure. The project was initiated as a widening project for U.S. 67. The location of the project and the geologic formation associated with the project is indicated by Figure 2.14. The subgrade was stabilized with 10% and 11% lime by dry weight of soil. The stabilized subgrade was then sealed to cure. During curing a large rain event occurred and upon arriving at the site the following day, construction crews notice many evenly spaced heave ridges as shown in Figure 2.15. The research was initiated to determine exactly what the caused the distress heaves.

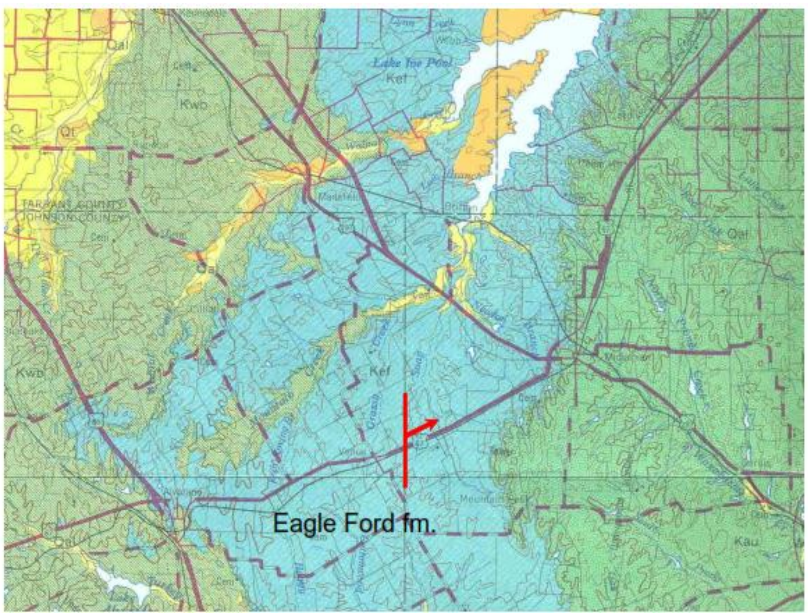


Figure 2.14 Geologic Map Identifying the Eagle Ford Formation and U.S. 67 Project Location (Wimsatt 1999)

As with the U.S. 82 project, the Eagle Ford Formation was a part of a research study involved with distress heave. The Eagle Ford is referred to as a selenetic formation. Selenite is simply another name for gypsum. In this particular case study it was determined that gypsum present in the soil interacted with the lime stabilization to form Ettringite. The Ettringite growth induced heave in the pavement structure ultimately causing its failure before the completion of construction. The use of X – ray diffraction and SEM analysis confirmed that the expansive mineral was indeed Ettringite. Open full completion of laboratory testing it was determined that U.S. 67 soils had sulfate concentrations ranging from 11,000 to 32,000 ppm. Ultimately, the best option for this project was to remove the sub grade and replace it with select fill that did not need to be stabilized. This was chosen because there still remained a fair amount of unreacted gypsum in the soil that could cause further expansion. One conclusion of this study was the more proper identification of sulfates in the field prior to construction through the use of the Geologic Atlas of Texas, conductivity tests, and sulfate concentration testing.

Figure 2.15 Heave Associated with US 67 near Midlothian, TX (Wimsatt 1999)

2.6.7 Other Heave Cases in Texas

Other cases of heave in Texas include the SH 161 and SH183 interchange in the Dallas County. In that case, sulfate concentrations as high as 27,000 ppm were discovered. Based on this discovery, the soil was treated with a combination of lime and Ground Granulated Blast Furnace Slag (GGBFS) prior to construction. Another case was on FM 3338 in Webb, County in 2005. In this study sulfates in the order of 40,000 ppm were discovered and had to be mitigated through the use of a combination of lime, GGBFS, and clay star product. Also, SH 118 in Brewster County in far West Texas near the city of El Paso experienced sulfate related heave. In the more arid regions of Texas lime is not typically used for stabilization, however cement is typically used to prevent erosion of sub grade material. In this case large amounts of evaporated gypsum were discovered at the top of the sub grade. Currently, studies are still ongoing to determine the best approach for the rehabilitation of the pavement. Suggestions such as fly ash have been suggested, but no method has been chosen yet Yet another example of sulfate related issues was observed in Culberson County in TXDOT's El Paso District on SH 54. In this case head walls were cracked and culverts were deforming due to sulfate heave. It was determined that gypsum in the soil reacted with the cement stabilizer to induce sulfate heave. The solution was to replace the culverts and use an untreated backfill. All of the cases presented were collected from Harris (2006), the USFHWA Report FHWATX-06/0-4240-4. It can be seen from these cases that sulfate related heave in Texas is primarily due to stabilizer reactions with gypsum. In addition the location of the project in Texas, climate, soil type, and stabilizer type vary from project to project while still experiencing sulfate heave.

2.7 Heave Mitigation Methods

The mitigation of sulfate induced heave is a complex and difficult subject to address due to the various components of the reaction, climatic factors, and soil properties. Various techniques have been generated over the years from adjustment and substitution of stabilizers to extending mellowing and even complete removal of the sulfate soil. It has even been suggested that lime or cement treatment of sulfate laden soils should be avoided completely (Mitchell, 1986). However, in most cases the soil must be stabilized to achieve the proper strength and behavioral characteristics needed for the particular design specifications. Therefore, this section will address the possible solutions to achieve proper stabilization without inducing detrimental sulfate heave. First, the adjustment and substitution of typical stabilizers are discussed and then other topics such as the use of geosynthetics, other materials, and changes in engineering practice for mitigation of sulfate induced heave are addressed.

2.7.1 Substitution with Ground Granulated Blast Furnace Slag (GGBFS)

Substitution of materials typically used in construction with byproducts of industrial processes that would normally be discarded can be a cost effective and sustainable option. GGBFS is the result of the ferro – silica industry and is the material formed when molten iron blast furnace slag is rapidly chilled (quenched) by immersion in water. It is a coarse glassy product that is then grinded in a granular product with very limited crystal formation, is highly cementitious in nature and, and hydrates like Portland cement (PC) (USFHWA). The use of GGBFS in soil applications have been discussed by Tasong et al. (1999), Wild et al.(1999), Puppala et al. (2003), Wang et al. (2003), Chavva et al. (2005), Higgins (2005), Deepti et al. (2007), McCarthy et al (2009), Jegandan et al. (2010), and Wilkinson et al. (2010). In those studies a variety of blends were tested to observe the effects of GGBFS and its viability for soil stabilization as well as the mitigation of sulfate induced heave.

Studies conducted by Jaganden et al. (1999) concluded that GGBFS in conjunction with other binders such as PC and Lime can increase Unconfined Compressive Strength (UCS) of the soil, making it a viable option for increasing strength of the soil, which is one of the main goals in chemical stabilization of soils. In most cases, the use of GGBFS as a replacement for some PC or Lime content resulted in an increase in UCS beyond the use of PC or Lime alone as well as the rate of increase in strength(Jaganden et al. 1999; Wilkinson et al. 2010). In addition, GGBFS helped to reduce the linear expansion of soil samples during wetting, which is one of the other main goals of chemical stabilization of soils (Higgins 2005). Lastly, the plasticity of the soil is reduced and the undrained shear strength was increased when GGBFS is used (Wilkinson et al. 2010).

In studies conducted by Tasong et al. (1999), Wild et al. (1999), Puppala. et al. (2003) all focused on the use of GGBS to specifically mitigate the sulfate expansion. Tasong et al. (1999) determined that the use of GGBFS caused a progressive change in the microstructure of the Ettringite molecule and the change was the result of reducing the amount of available calcium needed for the Ettringite reaction. Wild et al. (1999) determined that for kaolinite clays contacting gypsum that anywhere from 60% - 80% of chemical stabilizer should be in the form of GGBFS in order to sufficiently reduce the effects of sulfate induced expansion from Lime treatment. In other words, 60%-80% of the determined Lime content from the Eades and Grim Test should be substituted with GGBFS. The study conducted by Puppala et al. (2003) determined that when only GGBFS is used, 20% GGBFS from dry weight of soil was the most effective at reducing liquid limit of the sulfate soil and therefore the plasticity as well as reducing the free swell characteristics.

2.7.2 Use of Fly Ash

Like GGBFS, Fly Ash is a byproduct of industrial processes. Fly ashes are finely divided residue resulting from the combustion of ground or powdered coal. They are generally finer than cement and consist mainly of glassy-spherical particles as well as residues of hematite and magnetite, char, and some crystalline phases formed during cooling (USFHWA). The use of fly ash in concrete started in the United States in the1930's. One of the first large scale uses of fly ash in concrete was the construction of Hungry Horse Dam in Montana in 1948, which used 120,000 metric tons of fly ash. The uses of byproducts such as fly ash have been become an established practice for changing the engineering and behavioral properties of soil (Wilkinson et al. 2010). Fly ash, like cement, creates a poizzalonic bond within the soil particles, increasing strength and workability while reducing plasticity.

Fly ash has a history of suppressing sulfate heave in concrete, and while the compositions and constituents of sulfate reaction in concrete differ from that of soil, they are similar (McCarthy et al. 2009). Therefore it is a logical step to introduce fly ash into sulfate bearing soils to reduce the negative effects of sulfate heave. Studies have been conducted by Puppala et al. (2003), Wang et al. (2003), Chavva et al. (2005), Punthutaecha etal. (2006), Deepti et al. (2007), McCarthy et al. (2009), Solanki et al. (2010), and Wilkinson et al. (2010) to assess the effect of fly ash on the suppression of sulfate induced heave.

The study conducted by Puppala et al. (2003) utilized class F fly ash, as this type is low in calcium and would yield the best result for the mitigation of sulfate heave. Class C fly ash contains higher amounts of calcium and yields better strength properties; however the introduction of the calcium is known to increase the probability of Ettringite formation. Three mix designs were used in the study, first a control soil with no stabilizer, next 10% fly ash by dry weight of soil, and last 20% of fly ash by dry weight of soil. It was determined that the class F fly ash improved the physical and engineering properties of the soil by plasticity of the soil, increasing UCS, and reducing the free swell and shrinkage characteristics of the test soils. When compared to other methods, such as sulfate resistant cements and GGBFS, the strength gain using class F fly ash was not as high, but the cost effectiveness of this method exceeds that of the other mentioned methods.

The McCarthy etal. (2009) study focused on the use of fly ash in conjunction with lime. This study also utilized class F fly ash so as to reduce the amount of calcium present in the stabilizer. For this study four mixes were created. All four mixes contained 3% Lime by dry weight of soil and the addition of fly ash was done by 3%, 6%, 9%, and 12% by dry weight of soil. The results of the study indicated that swelling decreased with the reduction of Lime and the increase of fly ash. Surprisingly, the study found nearly the same amount of Ettringite growth in control samples and fly ash samples that contained the same amount of Lime leading the researchers to believe that mitigation of Ettringite in soils and concrete do not follow the same mechanisms. In addition, the mellowing period was determined to have an effect on swelling due to Ettringite growth. It was found that in increase in mellowing time before reintroduction of Lime and fly ash significantly decreased volumetric swell. The final results of the study determined the optimum Lime to fly ash mix to contain 3% Lime and10%-15% fly ash by weight and a mellowing period of at least one day. Overall, the use of class F fly was determined to be a viable and cost effective means of stabilizing soil while mitigating swell associated with Ettringite growth.

2.7.3 Extended Lime Mellowing and Double Lime Application

As the McCarthy study showed in regards to fly ash and lime combination, mellowing periods have a large effect on swelling of soils. Therefore a study was conducted by Talluri et al. (2013) to determine the effects of extended lime mellowing in stabilized soils. For this research initiative, six natural expansive soils from the state of Texas were used. Additional sulfates were added to the soils with lower sulfate concentrations to observe the effects of mellowing on high sulfate soils The study examined three different mellowing periods of 0, 3 and 7 days, and basic classification and chemical tests were performed to establish the clay mineralogy of the soils. After each specified mellowing period, the samples were subjected to three dimensional (3-D) volumetric swell, shrinkage and UCS tests. The results of study showed that soils with 0 days of mellowing exhibited the observed largest swell. As the mellowing period increased the early formation of ettringite began. The remixing of the samples after mellowing and before final compaction seemed to break the ettringite minerals apart and separate them from the sulfate minerals needed to continue further growth. Extended mellowing can also force the formation of other deleterious compounds before remixing and final compaction (Berger et al., 2001; Harris et al., 2004). As with the Talluri et al. (2013) study, mellowing before remixing and final compaction of periods from 1 – 7 days was recommended to achieve heave arrest.

The double application of lime has also been shown to mitigate swell behavior due to Ettringite growth in sulfate soils; however it is limited to soils with sulfate values lower than 7,000 ppm and soils that do not contain pyrites (Kota et al. 1996; Harris et al. 2004). The initial application of lime allows for the beginning stages of Ettringite growth while the second application, which is applied after a mellowing period, creates the poizzalonic bonds needed to improve the strength of the soil. After the initial lime treatment water must be added to the soil in order to dissolve the sulfate compounds and encourage the initial growth of Ettringite. Then, the mixing and reapplication of lime can break up the Ettringite and disperse it within the soil. As long as all of the soluble sulfates have been eradicated through the wetting and initial Ettringite growth processes after the initial Lime application, there will be no sulfates left to continue the reaction and future heave will be arrested.

2.7.4 Combination of Lime and Cement

Srivitmaitrie et al. (2008) conducted a study to see the effects of Lime and Cement combined on sulfate induced heave in soils with low to medium sulfate levels. In this study, soils were treated with 12% lime only and a combination of 6% lime and 6% cement. The UCS, free swell, and linear shrinkage properties were then monitored and evaluated. Test sections selected for the study were constructed in the Arlington, Texas area. The test sections were pavements built on Southmoor and International Street Prior to construction laboratory tests were conducted to establish the properties of the stabilization mixes. The initial Atterberg Limit Tests concluded that cement and lime combination created a lower Plasticity Index than lime alone. In addition, lime and cement mixed soils exhibited higher UCS values than lime alone. The swell and shrinkage characteristics were also examined and the lime cement combination showed nearly no swelling or shrinkage and showed less water absorbing capability. After the laboratory tests were conducted, the sub grades for the test roads were stabilized and the roads constructed. Field cores were then obtained and compared to the laboratory testing done prior to construction. The results of the field samples versus the laboratory samples were very similar with the field samples showing slightly less strength characteristics, indicating good field performance prediction and high quality results.

2.7.5 Sulfate Resistant Cement

Portland cement has been used in various aspects of civil engineering projects. PC is primarily used as the binder in concrete but has also been used in soil applications. There are five basic types of cement that are used to meet the various requirements of different civil engineering projects. The first, Type I cement is the most commonly used cement and is widely used in reinforced concrete applications. Type II is used when low to moderate sulfate ions are assumed to present before, during or after construction. Type III cement is used for projects that require high early strength values but gain little strength over time. Type IV cement is known as a low heat of hydration cement and is ideal for concrete applications that are in constant contact with water, such as dam structures. Lastly, Type V cement is known as high sulfate resistant cement and is used for projects where high sulfate are encountered before, during, or after construction. Sulfates are highly corrosive to concrete; therefore using Type II and Type IV cement is ideal for reducing the negative effects of sulfate and concrete interaction. With this knowledge known, the next logical step is to attempt to use sulfate resistant cements in soil stabilization applications.

In non-sulfate resistant cements (Type I/III/IV), tricalcium aluminate (C_3A) is formed during the mixing and hydration process. The tricalcium aluminate provides the free alumina required to form Ettringite (Rollings et al., 1999). However, sulfate resistant cement inhibits the formation of Ettringite by limiting the availability of reactive alumina, because tricalcium aluminate concentrations in sulfate resistant cements are low. The reduction of the needed alumina for Ettringite formation inhibits the ability for the reaction to occur. On this principal, sulfate resistant cements have been used in soil stabilization situations where sulfates are present in the soil.

One experimental study on stabilization of soil using sulfate resistant cement was conducted by Puppala et al. (2004). In the study, the effects of sulfate resistant cement on UCS, plasticity, free swell and linear shrinkage. Control soils were compared to treated soils to observe the absolute difference between all stabilization mixes. Two basic mixes were utilized for the study. First, Type I/II cement was used in 4 soils at a dosage of 5% and 10% by dry weight of soil. Next, Type V cement was used on the same 4 test soils at dosage of 5% and 10%. The 4 test soils had varying ranges of sulfate levels: Soil1 <1,000 ppm, Soil 2 = 1,000 – 2,000 ppm, Soil 3 = 2,000 – 5,000 ppm, and Soil 4 > 5,000 ppm. Both cement treatments showed a reduction in soil plasticity, which is to be expected with cement treatment. In addition UCS strengths were increased with cement treatment, again an effect that is expected. It is interesting to note, however, that the Type V cement generated a larger UCS strength than Type I/II cement. Free swell of the treated samples was reduced to nearly no expansion in both cement treated samples, and linear shrinkage was reduced by the addition of cement. The Type V cement treatment showed lower linear shrinkage percentages during 14 day curing than Type I/II cement, but at 90 days the shrinkage strains were equal. Overall the sulfate resistant cement treatment showed good results in increasing strength, reducing plasticity, and reducing swell and shrinkage. However, many practitioners are still skeptical of this method due to the large amounts of alumina present in clay soils. This natural alumina content can counteract the low alumina content of sulfate resistant cements, therefore, further studies must be conducted on the viability of sulfate resistant cements in soil stabilization.

2.7.6 Other Methods

Harris (2006) suggests a myriad of techniques that can be utilized to achieve proper stabilization of problematic soils in Texas while inhibiting sulfate heave. Other researchers such as, Zhiming (2008) suggest, that geogrids can be used as an alternative to chemical stabilization of high sulfate soils, and geogrids were successfully used on stabilization of high sulfate soils in that study. Cement kiln dust (CKD) has also been used in various studies as a cement substitute. Like fly ash and GGBFS, the CKD reduces the amount of calcium introduced to the sulfate soil and reduces the amount of swell due to Ettringite growth (Solanki et al. 2009). Barium hydroxide ($BaOH$) has also been used in a study by Walker (1992). In the study barium hydroxide was added to lime in hopes to form new, less soluble compounds with the sulfate minerals in order to prevent sulfate heave. Amorphous silica has also been used to prevent sulfate heave by deterring the formation of Ettringite (McKennon et al. 1993). Nylon fibers have been used to mitigate sulfate heave by reducing swell pressures in a study conducted by Punthutaecha etal. (2006). This study, however did not observe the effects of fiber stabilization on sulfate bearing soils. Aihong et al. (2009) utilized a heavy cover technique to reduce swelling of soil. In this technique large overburden pressures are induced to prevent swelling by overriding the pressures induced by swelling, however this method may not be very practical in most cases and has not been applied to sulfate heave. Other suggested heave mitigation techniques arise in the form of modifying engineering practices such as compaction. An investigation of soils in Las Vegas, Nevada by Hunter (1989) identified Ettringite/Thaumasite is treated soil and suggested that the heave was worsened because the soil was so heavily compacted. Other researchers have noted that decreased density may reduce sulfate heave because less dense soils contain more void space to allow for the accommodation of expansive minerals (Mitchell and Dermatas, 1992; Wild et al., 1999; Kota et al., 1996; Harris et al., 2004).

2.8 Comparison of Sulfate Measurement Methods

Comparison of the AASHTO, TXDOT and UTA method was conducted by comparing the inherent advantages and disadvantages of the methods. The advantages and disadvantages are discussed as they relate to speed of testing, accuracy of testing, and need for certain hazardous or expensive reagents. After comparisons were made the UTA method was deemed to be the most useful testing procedure given the categories of advantages and disadvantages. The results of this comparison are presented in this section.

Talluri et al. 2012 conducted an experiment to verify the accuracy of the UTA method, the gravimetric AASHTO T 290 – 95, and TXDOT Tex – 145 – E. The colorimetric AASAHTO method is less accurate than the gravimetric as stated by AASHTO T – 290 – 95 and therefore was not used for comparison in this study. Known concentrations of sulfates were added to two control soils from Fort Worth, Texas and from College Station Texas (Riverside).The control soils contained no sulfates. Table below displays some soil characteristics of the two control soils prior to addition of sulfates. The soils with known concentration of sulfates were then test using all three methods to compare their accuracy. It was determined that the UTA method resulted in the most accurate readings of sulfate concentrations for both test soils (See Table 2.2 and Figures 2.16 and 2.17). It can be seen that the AASHTO method achieved good accuracy for the Fort Worth soil, but was less accurate when used on the Riverside soil. It can also be seen that the AASHTO and UTA method were more accurate at higher sulfate levels. The TXDOT method results were not consistent and either over or under predicted sulfate values. Given the nature of the findings for accuracy, UTA was most accurate at all sulfate levels, while AASHTO was only slightly less accurate, and TXDOT was inconsistent and the least accurate of all three methods.

Table 2.1 Soil Classification Information (Talluri et al. 2012)

Soil Property	Soil Locations	
	Fort Worth	Riverside
Liquid Limit (LL, %)	61	35
Plastic Limit (PL. %)	24	11
Plastic Index (PI, %)	37	24
Specific Gravity	2.7	2.56
USCS Classification	CH	CL
Soluble sulfates, ppm	0	0

Table 2.2 Sulfate Measurement Techniques Comparison (Talluri et al. 2012)

Soil Location	Sulfate content, ppm*	Modified UTA			T 290-95			Tex-145-E		
		Trial 1	Trial 2	Average	Trial 1	Trial 2	Average	Trial 1	Trial 2	Average
Riverside	10000	9141	9724	9432.5	8552	8589	8570.5	13920	13600	13760
	5000	4970	5128	5049	3972	4428	4200	5120	4800	4960
	3000	2860	2667	2763.5	2342	2725	2533.5	2460	3040	2750
	1500	1457	1525	1491	1560	1440	1500	1440	1440	1440
Fort Worth	10000	9174	10091	9632.5	6877	6959	6918	12800	11200	12000
	5000	4638	4441	4539.5	4190	4173	4181.5	4480	4320	4400
	3000	2935	2710	2822.5	2235	2660	2447.5	2507	2880	2693.5
	1500	1432	1476	1454	1366	1375	1370.5	1440	1280	1360

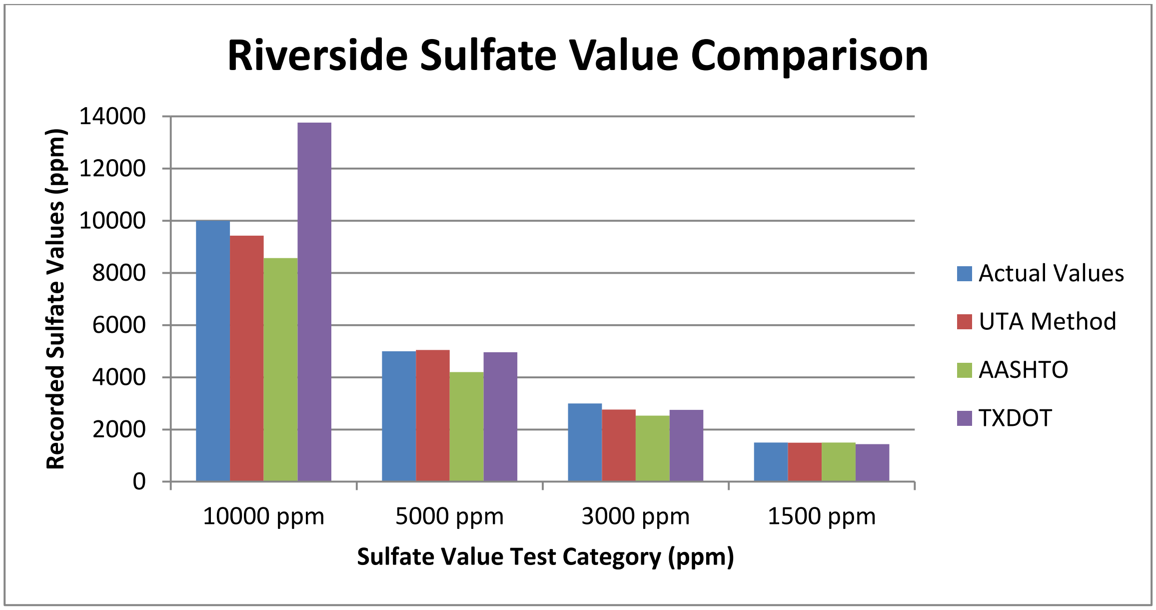


Figure 2.16 Riverside Soil Sulfate Testing Results Comparison (Talluri et al. 2012)

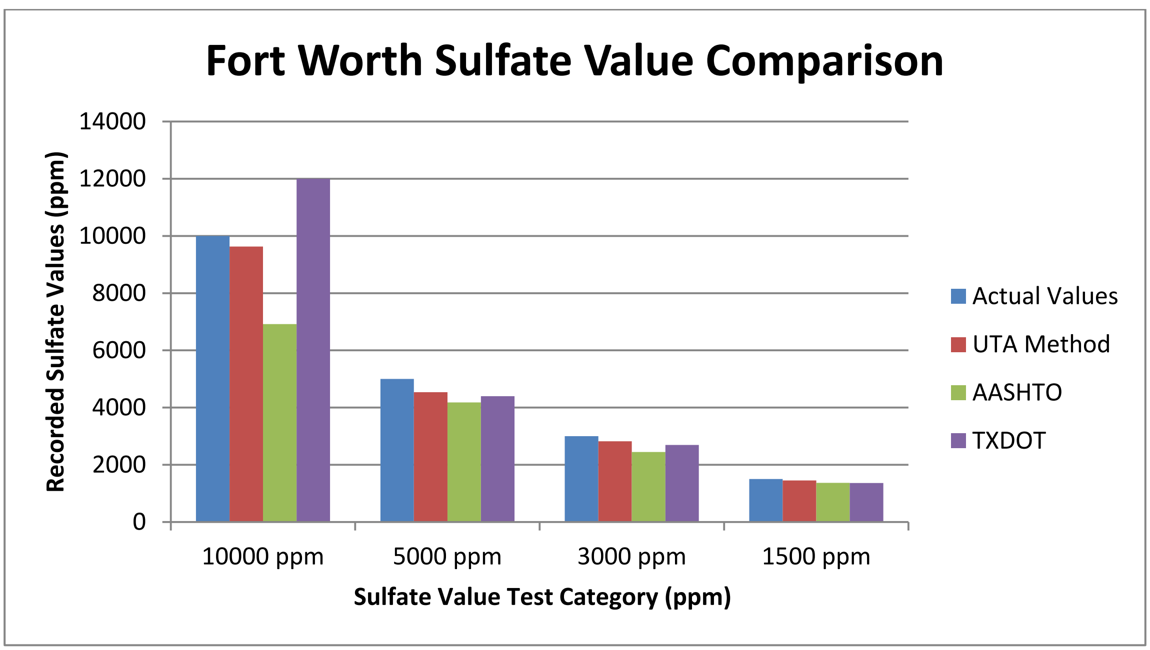


Figure 2.17 Fort Worth Soil Sulfate Testing Results Comparison (Talluri et al. 2012)

The last two comparisons deal with the need for dangerous or expensive reagents and the speed of testing. The UTA method only requires the use of Barium Chloride as precipitation reaction inducer as well as diluted Hydrochloric Acid (HCl) to adjust the pH level of the sample to range of 5.0 to 7.0 allowing for the most efficient precipitation reaction. The AASHTO method requires the use of Hydrofluoric Acid (HF), which is a very strong and dangerous acid. The AASHTO method also requires the use of Sulfuric Acid $(\mathrm{H_2SO_4})$ and Silver Nitrate $(\mathrm{AgNO_3})$. In addition, the gravimetric AASHTO method requires the combustion of the final precipitate at 800°C which is more dangerous than the simple weighing of the precipitate at the end of testing as in the UTA Method. The TXDOT method requires the use of sulfate indicator tablets which are hazardous, but on nearly the same level as barium chloride which is used in both the AASHTO and the UTA method. Speed was also a factor in the decision on what method to use as speed was a major constraint on the project. The TXDOT method can be done in about two days but requires the guessing of sulfate values. In order to find the correct sulfate value, the tester must start at the 1:20 dilution which can only detect sulfates up to 4,000 ppm. If the sample contains more than 4,000 ppm an error message is received from the calorimeter and the dilution must then ben increased and the test re – conducted. This is not satisfactory when testing over 300 in situ soils of which no concentrations are known. The AASHTO method typically only takes 2 to 3 days to complete which is quite comparable to the UTA method which takes 3 to 4 days to complete, however the volatility of the experiment as well as the dangerous reagents makes the UTA method the most useful for accuracy, safety and speed. Based on the accuracy study by Talluri et al. 2012, the speed of testing, and the examination of the methods for safety, the UTA method is the best choice.

2.8 Geostatistics Introduction

The introduction of geostatistics as a tool to aid in mining and petroleum operations was brought about in as early as the 1940’s. Since that time the use of geostatistics has been proven to be a valuable tool for visualizing geologic phenomenon and for predicting quantities values of geologic data beyond testing points. Within the realm of geostatistics, there are three basic approaches, linear, nonlinear, and multivariate geostatistics. Linear geostatistics was developed first and is the most widely used and non – linear geostatistics is the second kind and is used much less due to its complex mathematical nature (Rivoirard 1994). Lastly multivariate geostatistics attempts to use two fields of data that are related to each other in one analysis. Linear geostatistics is discussed in more detail in this literature review as it was the approach taken in this research study. Multivariate and non – linear techniques will only be briefly addresses for completeness of the discussion. In addition to the discussion of the fundamentals of geostatistics, various case studies pertaining more to geotechnical applications are discussed.

2.9 General History of Geostatistics

One of the first pioneers to attempt quantification of geologic related data using statistical approaches was Sichel (1947) in South African mining operations. Previous to Sichel many frequency distributions and statistical modeling had been attempted, but with little success (Krige 2000). Additionally, Vistelius (1949) attempted to use Markov chain analysis to aid in quantification geologic related data, but this analysis only allowed for analysis in one direction (Dubrule 1998). Daniel G. Krige furthered Sichel's work by submitting two very impactful papers in 1951 and 1952. This eventually led to the recognition of his contributions to the evaluations of mineral deposits through the coining of the term 'Kriging' that is used to describe a spatial mineral evaluation .These papers eventually made their way to France and were studied by Dr. Allais and his students. One of his students was G. Matheron who eventually became a professor and developed the theory of Regionilsed Variables (Krige 2000). Around 1960, G. Matheron propounded linear geostatistics, which embodied the use of the variogram and Kriging (Rivoirard 1994). Geostatistics offers a way of describing the special continuity that is an essential feature of many natural phenomena and provides adaptations of classical regression techniques to take advantage of this continuity (Isaaks and Srivastava 1989). Initially, over 40 years ago, geostatistics proved its superiority as a method for estimating reserves in most types of mines and as recent as the 1990's has been applied to the petroleum industry, particularly for contour mapping and for modeling of internal heterogeneity (Armstrong 1998). Contour maps of Kriging may be used to spot areas needing more sampling, if possible (Hohn 1999). This proves to be a useful tool to reduce the cost of drilling and sampling for a variety of projects. In the recent past geostatistics has evolved and moved from the oil fields and mines in to the geotechnical world to study phenomena such as, hydrocarbon deposition in soil, erosion of soil near coastlines, improving site investigations, and many others (Parsons and Frost 2002; Lloyd and Atkinson 2006; Kim et al. 2008; Vamerali et al. 2008; Li et et al. 2010; Fasona et al. 2011; Ren et al. 2012).

2.9.1 Uses of Geostatistics within Geologic Applications

Armstrong (1998) describes in great detail several applications for geostatistics as they apply to geologic data. First, the estimations on total reserves for mining and petroleum operations are an ability of geostatistics. Using this type of approach allows for an accurate estimate of the total tonnage or other quantity being examined as well as the grade, or quality, of the deposit helping the investigator determine if further investment in the project is warranted. Next, is error estimates. The quantification of error generated in the prediction associated with geostatistics is essential to adding validity to the analysis. Kriging variance can be used to identify the error involved with geostatistical analysis. Acceptable error in Kriging can also be used from the onset of the analysis to determine the most effective sample spacing. Gridding and contour mapping can also be done with geostatistics. Most recently petroleum geologists and environmental scientists have been using contouring techniques. The options presented are just some of the possibilities when using geostatistics, other options include: estimating block reserves, simulating deposits to evaluate an exploration plan, and estimating recovery.

These options apply more to mining and petroleum geology and as such were not discussed in further detail.

2.10 Linear Geostatistics: The Variogram

All geostatistical analyses begin with the evaluation of the data set acquired from testing or site investigations. The variogram also known as the semivariogram is the tool by which the variance of the data set is analyzed. The variogram is the basic geostatistical tool for measuring spatial autocorrelation of a regionalized variable (Isaaks and Srivastava 1989; Hohn 1999). The variogram is simply the cumulative variance of the data set over a lag distance (Cressie 1993). The total lag is the distance linearly that describes the special extent of the data. Therefore, data must be examined from four directions if the analysis it to be considered in 2D.This total lag is then broken up into sub lags. For example, if the longest distance in the x or y direction of the measurements for a particular data set is 1000m, then the total lag is 1000m. The total lag can be broken into as many sub lags as the user wants. Therefore, for this particular example one could break the total lag into 10 sub lags of 100m. In doing this, the cumulative variance will be calculated for all data points with the first 100m of lag. This becomes the first point on the semivariogram. The next point on the semivariogram considers all points within the first 200m, extending the lag by 100m each time. Eventually, ten points are calculated on the semivariogram. Figure 2.18 below shows a typical semivariogram plot created by Surfer 9 Software.

Equation 2.10.1 Semivariogram Definition

$$\gamma^{*}(\mathrm{h}) = \frac{\sum[\mathrm{z}(\mathrm{x}) - \mathrm{z}(\mathrm{x}+\mathrm{h})]^2}{2\mathrm{n}}$$

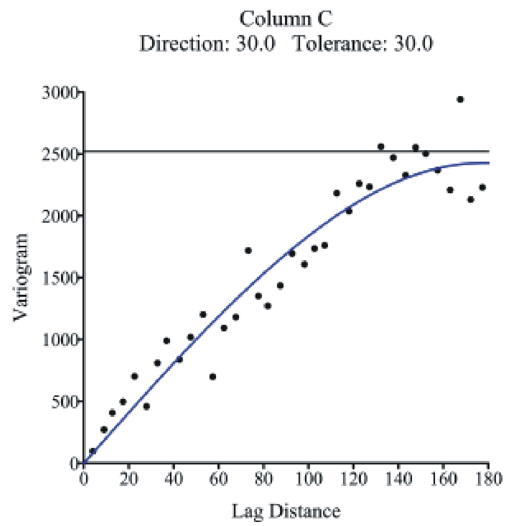


Figure 2.18 Semivariogram Example Plot (Surfer 9)

Equation 2.10.1 is the calculation by which the semivariogram derived. This expression is a straightforward way of measuring how a variable z changes in value between site x and another site h. The quantity $\gamma^*(h)$ is the semivariogram quantity that belongs in the y axis (Armstrong 1998; Hohn 1999). Note that this calculated variance is not the same as simple variance used in regular stochastic models. Unlike the simple variance about a mean value, the semivariogram measures the variance between two samples. Upon completion of the semivariogram plot a mathematical function is then applied to fit the data. A properly fitted mathematical model then allows for a computer program to calculate linear estimates that reflect the spatial extent and orientation of autocorrelation in the variable to be mapped (Hohn 1999). All variograms that have mathematical functions fitted to them have a range and a sill by which predictions are no longer valid due to a lack of correlation between data points (Armstrong 1998; Hohn 1999). This sill is the limiting value of the mathematical model and the range is the lag distance from the origin of the variogram to sill. Figure 2.17 shows and example of a range and sill on a variogram plot. Not all models reach a sill. In some cases the sill continues to increase with distance. Figure 2.18 is an example of a variogram that does not reach a sill and therefore has unlimited range. Variograms that reach a sill and have a finite range are named as bounded and variograms that do not reach a sill are named as unbounded (Armstrong 1998). In many cases mathematical model fitting can be a difficult task to achieve. In those cases it may be necessary to change the lag intervals by increasing or shortening them to achieve a more distinct trend (Armstrong 1984).

2.11 Linear Geostatistics Mapping Techniques: Kriging

A myriad of mapping techniques can be used in conjunction with a semivariogram. The most common mapping technique and the one utilized, and the one utilized for the soluble sulfate study, is Kriging. Kriging can be done using linear geostatistical approaches, non – linear approaches, and multivariate approaches depending on the wanted outcome of the analysis. Kriging is deterministic interpolation method that focuses on estimation that gives the best unbiased (minimum variance) linear estimate of point values or block averages (Armstrong 1998; Dubrele 1998; Olea 2009). Sampling only provides information at discrete data points, Kriging is the tool used to describe what is happening at the intermediate points between sampling data. The method was created by Dr. D.G. Krige, for the South African mine fields and was improved by Professor G. Matheron into the techniques used today (Dubrule 1998; Stein 1999). The accuracy of Kriging projections depends on several factors. The number of samples and the quality of the data at each point, the positions and spacing of the samples, the distance between samples, the spatial continuity and inherent variability of the data are all extremely important when developing sound and accurate Kriging models (Armstrong 1998; Hohn 1999). Kriging approaches in linear geostatistics can be done in many different ways. Simple Kriging, Ordinary Kriging, Universal Kriging, and are the most common approaches (Olea 2009). The following sections will discuss in detail the different forms of Kriging. It is important to note that within all forms of Kriging, two basic approaches can be made. The first is Point Kriging were the data points are known to be discrete and random and cannot be generalized in a block of certain larger dimensions than the test point. The second is Block Kriging, where the data point can be attributed to a larger area called a block. Once the Kriging technique has been used to “fill in the gaps” of the data, simple 2D contouring can be done to connect the points and then build a 3D model.

2.11.1 Simple Kriging

Simple Kriging involves the use of weights, such that they minimize the error variance. Such weights turn out to be the solution to a system of equations called, the normal system of equations. Equation 2.11.1 illustrates the typical form of this system of equations and the definitions of its constituents. The assumptions made in Simple Kriging are: 1) the sample is the partial realization of a random function Z(s), where (s) denotes the spatial location; 2) Z(s) is second order stationary; 3) the mean is not only constant but known. These assumptions make simple Kriging the most restrictive form and the least accurate (Olea 2009). Simple Kriging is rarely used in day to day applications because the mean must be known and in most cases the mean is not known. There are some cases where the mean is known such as mine fields that have been mined for decades and in that case the mean value of samples can be known (Armstrong 1998).

2.11.2 Ordinary Kriging

Ordinary Kriging is the original form of Kriging, is the most widely used, and the most robust form (Haining et al. 2010). Ordinary Kriging is usually referenced with the acronym B.L.U.E. meaning best linear unbiased estimator. Ordinary Kriging is “linear” because its estimates are weighted linear combinations of the data used in analysis; it is “unbiased” since it tries to have the mean residual error equal zero; is “best” because it aims at minimizing the variance of the errors associated with the analysis (Isaaks and Srivastava 1989). The whole idea behind Ordinary Kriging is to find the optimal weights that minimize the mean square estimation error (Olea 2009).The assumptions made in Ordinary Kriging are: 1) the sample is the partial realization of a random function Z(s), where (s) denotes the spatial location; 2) Z(s) is second order stationary or can honor the intrinsic hypothesis; 3) the mean is constant but unknown (no trend) (Olea 2009).

2.11.3 Universal Kriging

Universal Kriging is a form of Ordinary Kriging that allows for the accommodation of a trend (Isaaks and Srivastava 1989). Matheron (1969) introduced this to help examine data that had a strong deterministic component. Examples of this type of data could be ocean depth near the shore line or temperature changes in the upper atmosphere (Olea 2009). Therefore, this form of Kriging analysis is not well suited for random phenomenon with little to no predictable trend. Universal Kriging removes the both the requirement that the data must contain a constant mean and that the user must know that mean (Olea 2009). The assumptions of Universal Kriging are: 1) the residuals Y(s) are a partial realization of a random function $\mathrm{Y}(s) = \mathrm{Z}(s) - \mathrm{m_z}(s)$, where $\mathrm{m_z}(s)$is the trend or drift of the function Z(s); 2) Z(s) is second order stationary or can honor the intrinsic hypothesis; 3) The mean is now neither know nor constant; 4) the trend in the mean is considered a deterministic component amenable to analytical modeling, usually polynomials (Hohn 1999; Olea 2009).

2.12 Multivariate and Non – Linear Geostatistics

Multivariate geostatistics is used when one wants to study and exploit the covariance between two or more regionalized variables (Hohn 1999; Olea 2009). In other words, this technique may be used when two unrelated phenomenon a share some sort of correlation with each other. For example, density of a certain soil may have a direct link with permeability and multivariate techniques can allow for the analysis and prediction of both qualities simultaneously. One advantage of this technique is that one attribute may be more expensive to examine than the other and by using the correlation of the more expensive attribute to one that is less expensive to sample Kriging can still be done accurately. Matheron (1965) developed this method called Co Kriging. Multivariate geostatistics is considered to be a form of linear geostatistics. Other forms of multivariate geostatistics, which are less well known include: regression Kriging, Kriging with an external drift, simple Kriging with locally varying means, collocated Kriging, and multivariate factorial Kriging (Goovaerts 1993; Hohn 1999; Haining et al. 2010). Non – linear geostatistics is less well known and used due to the complicated mathematical nature of the analysis (Rivoirard 1994). Linear geostatistics deals only with linear combinations of one variable under study Y(x), but sometimes we need to estimate, not Y(x) itself, but one or more functions f[Y(x)] of Y(x). To do this, only non-linear methods will suffice (Rivoirard 1994). The two main non – linear approaches are Disjunctive Kriging, Log Normal Kriging, and Indicator Kriging (Rivoirard 1994; Hohn 1999; Haining et al. 2010). Disjunctive Kriging was developed by Matheron (1973) and aims normalizing the raw data, creating a variogram based on the normalized raw data, calculating coefficients and finally estimation local averages and frequency distributions (Hohn 1999; Haining et al. 2010). Lognormal Kriging involves the transformation of the initial raw data by the use of logarithms, which when completed makes the data appear to be normally distributed. Next, the variogram is modeled from the transformed data, then the data is Kriged, and finally back transformed into the original units (Hohn 1999).

2.13 Case Studies of Geostatistics in Geotechnical Applications

In this portion of the literature review, various case studies involving the use of geostatistics and geotechnical applications are discussed in detail. Tradition dictates that most case studies involve the use of geostatistics in mining and petroleum operations, however over the last two decades the use of geostatistics has become more prevalent in other forms of engineering. The case studies presented over a wide variety of applications for geostatistics in soil applications.

2.13.1 Evaluating Site Investigation Quality Using GIS and Geostatistics

The current practice of geotechnical site investigations relies heavily on the characterization of sub surface conditions and usually involves the interpretation of data from laboratory and in situ testing. The purpose of the study conducted by Parsons and Frost (2002) was to create a method of evaluation for the quality of the deterministic results of laboratory and in situ testing. The paper aims at the development and implementation of a performance – based investigation and monitoring approach for assessing the quality, or thoroughness, of site investigation and monitoring activities in a quantitative, spatially sensitive manner. A quality geotechnical investigation ultimately reduces the cost of a project by making more accurate information available, which ultimately leads to a more optimized design. Figure 2.19 below illustrates the comparison of a poor and quality investigation as it pertains to potential cost.

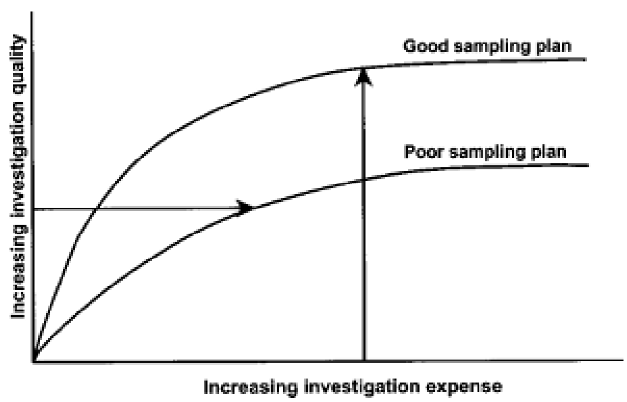


Figure 2.19 Investigation Quality vs. Expense of Two Different Sampling Plans (Parsons and Frost 2002)

The approach for developing a quality measurement on site investigation was based on the principle of the best linear unbiased estimator (B.L.U.E.). For the study linear geostatistics in the form of ordinary block Kriging was used to help define quality for systems with valid enough data that a geostatistical approach can be made. The particular case study used involved the evaluation of SPT blow counts on Treasure Island, an artificial island, in the San Francisco Bay. The evaluation of the SPT considered the effects of the change in SPT method and depth. The analysis focused on the possibility of liquefaction as it relates to the corrected SPT for granular soils $N_{1(60)}$. The soils used to build Treasure consist of a fill layer composed of the Yerba Buena Shoals and an underlying layer of Young Bay Mud followed by Old Bay Mud. The Yerba Buena Shoals consist of dense sand; the Young Bay Mud is a normally consolidated, medium plastic to medium stiff, to stiff silty clay that may experience strength loss under dynamic loading; the Old Bay Mud is sandy, silty, peaty clay that is dense but is impacted very little by dynamic loads due to its depth and strength level. The primary cause of engineering concern is the Yerba Buena Shoals which is susceptible to liquefaction, later spreading, and settlement under dynamic loading. Figure 2.18 shows the experimental variogram created for the Yerba Buena Shoals fill layer. The geostatistical analysis was done using GIS – ASSESS software. A spherical model with a nugget effect was chosen as the model for the variogram. The nugget effect represents short scale, test variability, and the range and sill indicate the limits of the models prediction capability. With the variogram block Kriging techniques were implored using 100 m^2 x 100 m^2 blocks. Figure 2.20 depicts the experimental variogram utilized in the study.

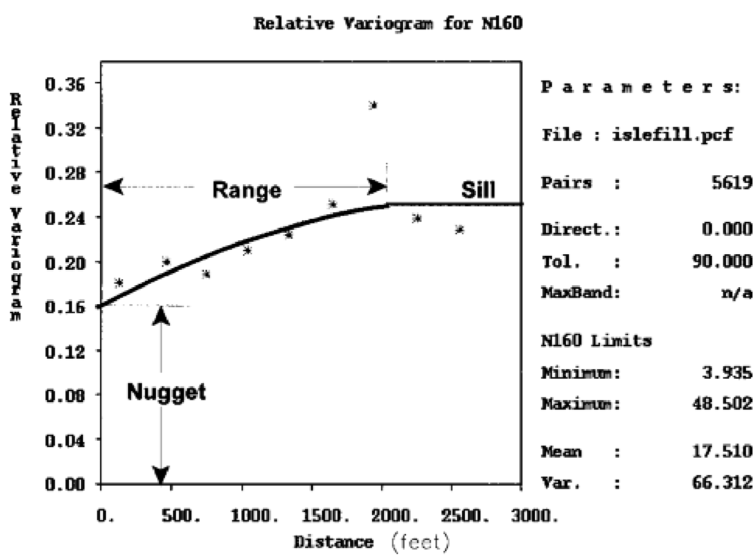


Figure 2.20 Experimental Variogram for Treasure Island Yerba Buena Shoals (Parsons and Frost 2002)

The thoroughness of the data was then examined by Kriging error. Additionally, the potential of liquefaction based on the SPT values was Kriged, and lastly the amount of data points needed for a thorough investigation was determined through Kriging in order to determine if a less involved site investigation is needed. The results of the study indicated areas of potential liquefaction and areas of inadequate thoroughness when it comes to the necessary information required for a quality geostatistical evaluation as well as the fact that additional sampling does increase the accuracy of the investigation, but the rate of increase continuously declines as more samples are added. This begs the question and offers a new portion of the study to determine what the proper threshold for additional data points might be.

2.13.2 Spatial and Temporal Variations in Grain Size of Surface Sediments in the Littoral Area of the Yellow River Delta

This study was conducted by Ren et al. 2012 in China and focused on the mapping of particular grain sizes of sediments and their pathways as well as the grain size response to the drastic decrease in riverine sediment discharge in the Littoral area of the Yellow River Delta from 2000 to 2007. For this study the grain sizes were determined using a Coulter SL100Q laser particle sizer and categorized into sands, silts, and clays using the Folk’s Triangular Diagram. Ordinary point Kriging techniques were used in conjunction with the semivariogram to create the maps used for studying the sediments. Figure 2.21 shows the results of the geostatistical study on sediment deposits and their transport change characteristics from 2000 to 2007.

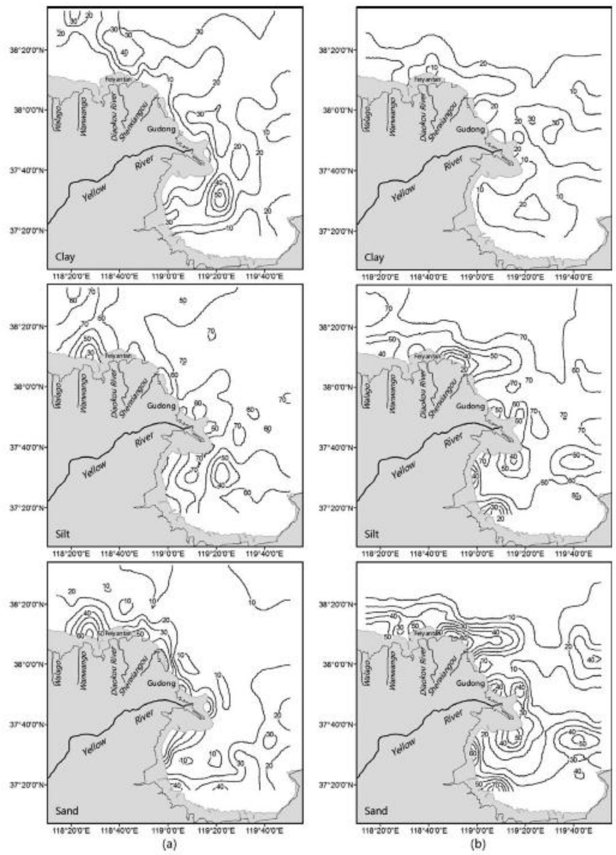


Figure 2.21 Multivariate Kriging Output for Clay, Silt and Sand (Ren et al. 2012)

2.13.4 Other Cases

Other cases where geostatistics was used to map the spatial correlation of soil related phenomena are becoming more common in the 2000's. One example is the monitoring of beach erosion in Lagos, Nigeria by Fasona (2011). In this study, remote sensing and geostatistics were used to monitor land degradation on the Mud Beach Coast of Southwest Nigeria. This case study used a new form of geostatistics called binary logistic regression, which utilizes the natural log function. The study actually used geostatistics to predict the future degradation of the coast for the next 100 years. Another case deals with the identification of root structures, which can be a very important issue for foundation related issues as roots can be a primary source of natural dewatering of a sub grade. This study was conducted by Vamerali et al (2008) and used Ordinary Kriging to evaluate root length density for maize fields in Italy to improve crop performance. While this application lends itself more to agriculture, the same concept could be applied to larger rooting structures that could severely impact the performance of shallow foundations. A study conducted by Li et al. (2010) involves the use multivariate geostatistics to evaluate the deposition of toxic hydrocarbons into the soil in an industrial area of China. These concepts could also be applied to leeching of contaminated groundwater in a land fill structure. Similarly, a study conducted by Hooshmand et al. (2011) used Kriging and CoKriging to evaluate groundwater quality parameters in Iran. Kim et al.(2008) used ordinary Kriging to evaluate soil properties along coastal dunes in Korea. This study was done in conjunction with Texas A&M for the Journal of Coastal Research. Lastly, Lloyd and Atkinson (2006) use ordinary Kriging in conjunction with LiDAR readings to create surface elevation maps in the United Kingdom. Many other studies are now turning to the use of geostatistics as an analysis tool and it appears the applications are unlimited. Carroll and Oliver (2005) utilized electrical conductivity readings for geostatistical analysis on soil properties. Kilic et al. (2003) use SPT resistances in a geostatistical analysis in hopes of reducing the amount of SPT in a geotechnical investigation. Recently Peterson et al. (2007) implemented geostatistical analysis in intelligent compaction procedures in order to reduce the number of test beds required to calibrate the intelligent compaction machinery.

2.14 Summary

It can be seen from the literature review provided in this chapter that sulfate bearing soils are a major concern in civil engineering projects due to the heave created upon chemical stabilization with calcium based stabilizers. The knowledge of this phenomenon stretches back to the 1980’s when Mitchell brought the issue to the forefront of geotechnical engineering. Sulfates in soils can be derived from many sources from natural primary sources to more complex secondary reactions or even through engineering practice. The heave mechanisms associated with soil sulfate heave are comparable to those established in concrete. There is currently no set threshold on problematic sulfate levels as this depends on many other factors related to the soil and environment. Currently, many mitigation practices have been established and can successfully be carried out to achieve proper stabilizations without contributing to detrimental sulfate related heave. Many case studies are available to describe the nature and extent of the damage that can be caused by sulfate heave. Many of those cases were the direct result of improper stabilization as a result of poor site investigation in relation to soluble sulfates

It can also be seen from the second portion of the literature review that geostatistics, while complex and heavily rooted in stochastic and mathematics, can be a very useful tool in geological and geotechnical arenas. From the inception of geostatistics by Sichel and Krige for the South African gold mines to the use of geostatistics on geotechnical site investigation, it can be seen that possibilities with geostatistics are endless. The varying forms and analysis approaches allow the user to evaluate many different types of data sets to evaluate the spatial correlation of data and increase the accuracy and efficiency of engineering practices.

CHAPTER 3

EXPERIMENTAL PROGRAM AND RESULTS

3.1 Introduction

The following chapter contains the soil sampling and selection, descriptions of the testing procedures and equipment for sulfate testing, the results of sulfate testing, and a deterministic evaluation of sulfate results. In addition to the presentation of results, discussions are presented to explain the nature and results of the deterministic analyses. The chapter begins with the presentation of three different sulfate testing procedures. Next, deterministic Bar Plots are presented to show the distribution of sulfate vales along a straight line distance through all formations. This is a useful tool in highlighting the variability of the deterministic sulfate values along all formations of the testing.

3.2 Soil Selection and Sampling

Soil sampling locations were selected by Fugro Consultants, Inc. A total of 302 samples were collected from six different formations along the pipeline alignment. Table 3.1 specifies the six chosen formations for soluble sulfate testing, as well as the distribution of the samples within the given formations. Regular, 5ft depth intervals were chosen for study (i.e. 5ft, 10ft, 15ft, and 20ft). Considering the placement of the proposed pipe at depth and the contribution of overburden pressure to nullify any expansive nature of the soil, the maximum depth chosen to test was 20 ft. Representative samples were collected from the field by the program – wide geotechnical consultants (Fugro Consultants Inc.) under the contract with TRWD and these materials were transported to UTA for soluble sulfate studies. These soils were then prepared and tested using the Modified UTA Method for Soluble Sulfate Determination in Soils. This method is preferred due to its accuracy and speed (Talluri et al. 2012). Summary of test procedures and test results are presented in the following sections. In Figure 3.1, parts a) and b) depict samples after receiving from Fugro Consultants, Inc., while part c) of the figure shows a pulverized soil sample after drying.

Table 3.1 Sulfate Testing Breakdown by Geologic Formation

Geologic Formation	Total # of Borings	Total # of Samples
Eagle Ford	20	80
Ozan	15	60
Wolfe City Sand	10	46
Neylandville - Marlbrook	4	16
Kemp Clay	10	40
Wills Point	15	60
Totals	74	302

a)

c)

b)

Figure 3.1 a) & b) Samples Receipt and Preparation Prior to Testing c) Pulverized Sample after Drying

3.3 Soluble Sulfate Testing

A myriad of soluble sulfate testing methods are available for use in soils. Specifically, the AASHTO Method, TXDOT Method, and the Modified UTA Method for Determination of

Soluble Sulfates in Soils will be discussed and compared for their inherent advantages and disadvantages given the project constraints. Ultimately, the Modified UTA Method was determined to be the most accurate and could be conducted with the greatest speed, therefore it was chosen as the test methodology for this research project. The details of the method are presented in later sections. For the sake of comparison and completeness the AASHTO Method and the TXDOT Method will be discussed in detail as well.

3.3.1 AASHTO T290 – 95 Determining Water – Soluble Sulfate Ion Concentration in Soil

The American Association of State Highway and Transportation Officials (AASHTO) created two different methods for determining soluble sulfate concentrations in soil in 2007, Method A and Method B. Method A is a gravimetric approach, while Method B is colorimetric. Both procedures will be discussed in detail so as to create a comparison with The UTA Method for Soluble Sulfate Determination in Soil, which is gravimetric, and the TXDOT 145 – E method, which is colorimetric. Method A is noted as being more time consuming than Method B, but it yields more accurate results. Both methods begin by taking the field sample and oven drying the sample at no higher than 60°C (140°F), this temperature is considered "air drying" and is not sufficient to evaporate gypsum. Then the aggregated sample is then pulverized in a mechanism that does destroy the natural particle size. Sieving is then carried out on the pulverized sample through a 2.0 mm (U.S. No. 10) sieve. All material retained on higher sieves must be re-pulverized until they pass a No. 10 sieve. A representative sample is then taken for testing by Method A or Method B.

Method A, the gravimetric method, begins by adding 100g of the soil sample and 300g of deionized water (DI) into a 500mL Erlenmeyer flask. The flask is then stoppered and shaken. Next the soil is extracted from the sample by centrifugation. The resulting solution is then filtered through a 0.45 μm filter membrane. A drop of concentrated Nitric Acid may be added at this time to precipitate finely suspended particles. Next the sulfate ion solution is added to a 250 mL beaker and the volume of the solution is adjusted to 200mL by adding DI water or through evaporation by boiling. Next, the solution's acidity is adjusted to the methyl orange endpoint and 10 mL of hydrochloric acid is added to the solution. The solution is then heated to boiling and 5 mL of 10% w/w barium chloride solution is added. The solution is then removed from heat and allowed to sit for a minimum of 2 hours. Filter the now barium sulfate solution is filtered onto a fine, ash less filter paper and is washed with warm water to remove free chloride ions. The solution is checked with 10% w/w silver nitrate solution to ensure nearly all chloride ions have been washed away. The contents of the filter paper are then placed on a tarred, platinum crucible (Figure 3.2). The sample is then charred without flaming at 800 °C for one hour. The sample is then removed from heat and a few drops of hydrofluoric acid and sulfuric acid are added and the evaporation is released in a fume hood. The sample is then reignited at 800 °C and is cooled in a desiccator. The mass of the barium sulfate is then determined through weighing.

Figure 3.2 Platinum Crucible Used in AASHTO T 290 – 95 Method A (Gravimetric) Photo Source : VRS Laboratory

Method B, the calorimetric method is less accurate than Method A, but is faster. Method B begins by adding 100g of the soil sample and 300g of deionized water (DI) into a 500mL

Erlenmeyer flask. The flask is then stoppered and shaken. Next the soil is extracted from the sample by centrifugation. The resulting solution is then filtered through a 0.45 μm filter membrane. A drop of concentrated Nitric Acid may be added at this time to precipitate finely suspended particles. Next, 50mL of the sample is taken and added it to a beaker. 10 mL of 1:1 glycerin to water solution is added to the beaker. A sodium chloride solution is prepared using 240 g of sodium chloride 20mL of hydrochloric acid and the solution is diluted to 1L. 50 mL of the sodium chloride solution is also added to the beaker. A 40 mm colorimeter vial is then filled with the solution and is zeroed out in the colorimeter. The sample is then transferred from the vial to another free and clean beaker and 0.3 of barium chloride dehydrate is added and stirred. That solution is then poured back into the vial and is tested in the colorimeter for the sulfate content.

3.3.2 TXDOT Tex – 145 – E Determining Sulfate Content in Soil Colorimetric Method

The TXDOT method was created in 2005 by the Texas Department of Transportation. This method is a turbidity based technique. The sample preparation for this technique begins with drying of 300 g of collected field sample at 60°C (140°F). Once dry the soil is pulverized to pass a 0.422 mm (U.S. No. 40) sieve. Duplicates must be run on this particular method and the resulting concentrations are averaged to obtain the final sulfate value. Next, 10g of sample is placed in a flask and 200 mL of DI water is added. The flask is then stoppered and shaken vigorously by hand for approximately 1 minute. The sample is then allowed to sit for a minimum of 12 hours. After sitting for 12 hours, the sample is agitated by shaking for 1 min and is then filtered through a Whatman 42® (fine porosity), or equivalent, filter paper. The filtrate is then collected and a pipette is used to transfer 10 ml of filtrate into the glass vile used in the specific colorimetric device. Kimwipes® , or equivalent, are then used to clean the glass vile to remove dirt or fingerprints that may cause erroneous readings. The glass vile is then placed in the colorimetric device (Figure 3.3) and the device is zeroed, this ensures the device is calibrated.

The vial is then removed and one sulfate tablet is added and completely crushed with a plastic rod. If sulfates are present the solution will become hazy or milky colored. The vile is then placed back in the colorimeter and is tested. A minimum of three readings are required when using this method and the readings are then averaged. The resulting average reading number is multiplied by the initial dilution ratio to determine the ppm of the sample (Ex: 1:20 dilution with average reading of 100 = 2000 ppm).

Figure 3.3 Photograph of a Colorimeter and Conductivity Meter

3.3.3 Modified UTA Method for Soluble Sulfates Determination in Soil

This test is performed to assess the concentration of soluble sulfates in the soil. The method is a modified procedure from the standard gravimetric method by Clesceri (1989). The overall goal of the test is to dissolve the soluble sulfate and then precipitate the sulfate by using barium chloride. Barium (Ba^{2+}) is insoluble in water and is a divalent cation; therefore it will attach itself to the sulfate molecule (SO_4^{2-}) and will be precipitated out as barium sulfate ($BaSO_4$). The resulting barium sulfate precipitate is then weighed and stoichiometry techniques are utilized to isolate the weight of the sulfate ion from entire molecule. This resulting weight of sulfate is then transferred into a ppm value which is the unit typically used to describe soluble sulfates.

The test begins with preparation of the soil sample. Soils received from Fugro Consultants Inc. were visually inspected and then approximately 100g to 1.0 kg of soil was removed from the sample and oven dried at temperatures no higher than 60°C (140°F), as gypsum readily evaporates at temperatures higher than 60°C. The oven dried sample was then pulverized to a particle size less than 4.75mm (U.S. No. 4) to ensure proper dissolution of the sulfate mineral. Next, 10g of the prepared soil is placed in flask and 100g of DI is added. The soil and DI are allowed to react, covered for 24 hrs. ensuring the maximum dissolution of the sulfate mineral. After 24 hrs., the sample is shaken in an Eiberbach shaker for 30 min to break any further crystals remaining in the solution. Then, the soil is extracted from the dissolved solution through centrifugation and is discarded. If necessary, hydrochloric acid is added to the solution in order to keep the pH values within the range of 5 to 7. The dissolved solution, now free of the majority of soil particles, is filtered through a 0.1μm filter paper to remove smaller particles not extracted through centrifugation. The resulting filtrate is then heated, covered to near boiling and 40 mL of barium chloride 10% w/w solution is added. The final solution is then removed from heat and allowed to cool and is then transferred to an oven for an incubation period of at least 12hrs. This allows for the reaction to continue and facilitates maximum precipitation. Next, the solution is filtered through a 0.1μm filter paper and is oven dried for 18 to 24 hrs. until constant weight is achieved. Finally, the resulting barium sulfate precipitate is weighed and the portion of sulfate in the precipitate is calculated. Figure 3.4 presents a flow chart of this method while Figure 3.5 presents an, illustration of the testing procedure.

Figure 3.4 UTA Soluble Sulfate Determination for Soils Flow Chart (Puppala et al 2002)

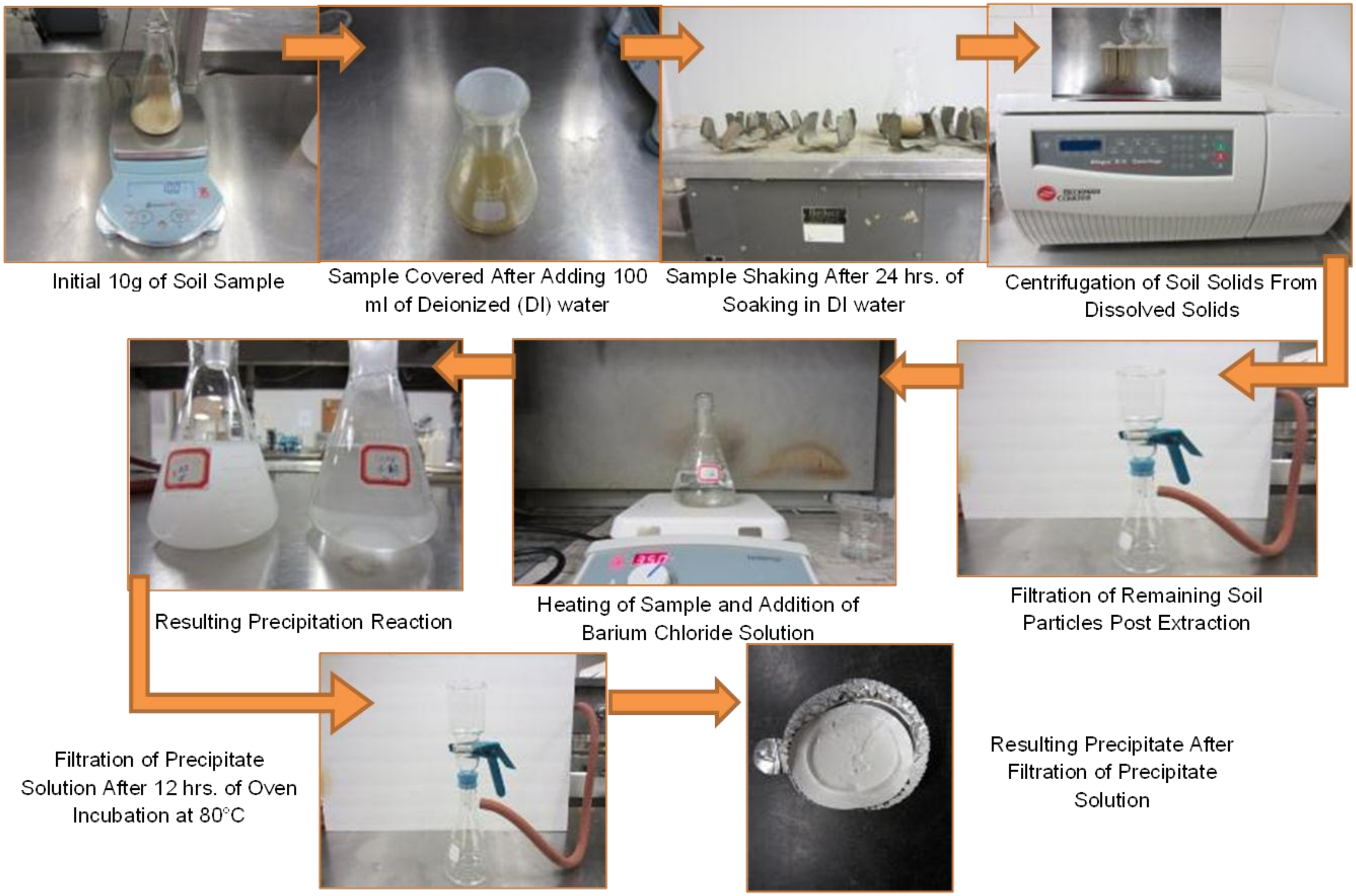


Figure 3.5 Illustrated UTA Method for Soluble Sulfates Determination in Soil

3.4 Sulfate Testing Introduction

The following tables present the results of the sulfate testing program that was conducted as per Modified UTA Method for Determining Soluble Sulfates in Soil presented in the previous chapter. Tables 3.2 through 3.7 present the soluble sulfate concentrations of the several soil samples from Eagle Ford, Ozan, Wolfe City Sand, Neylandville Marl, Kemp Clay and Wills Point formation, respectively. Geostatistical analysis was performed on this data to develop sulfate contour maps for each of these formations. The details of the geostatistical analysis are presented in the following sections. Note that all cells with the notation N/A indicate that samples were not available at those depths and locations. Following the testing results, Bar Chart Plots of the entire testing data set are presented for all depth intervals.

Table 3.2 Eagle Ford Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-013	13	Eagle Ford	490	500	N/A	N/A
B-015	11	Eagle Ford	N/A	1320	1270	N/A
B-051	13	Eagle Ford	2005	525	N/A	540
B-059	13	Eagle Ford	40	185	300	185
B-061	13	Eagle Ford	450	200	140	130
B-062	13	Eagle Ford	8450	2250	1915	8080
B-063	13	Eagle Ford	12,000	18,300	715	750
B-090	13	Eagle Ford	12,700	1620	1160	18,450
B-093	13	Eagle Ford	14,400	3620	16,160	19,620
B-164	11	Eagle Ford	960	560	640	15,260
B-168	12	Eagle Ford	1810	890	15,670	9360
B-084	12	Eagle Ford	4600	1050	3800	4200
B-198	12	Eagle Ford	580	15,100	17,500	7000
B-201	12	Eagle Ford	3710	15,200	1560	5530
B-202	12	Eagle Ford	420	6300	530	14,050
B-196	12	Eagle Ford	60	370	60	75
B-199	12	Eagle Ford	400	180	1030	2200
B-200	12	Eagle Ford	325	105	275	3340
B-209	13	Eagle Ford	17,960	11,725	17,100	16,000
B-318	11	Eagle Ford	735	280	290	1320
B-319	11	Eagle Ford	485	N/A	N/A	N/A
B-326	12	Eagle Ford	11,625	1090	1095	1005

Table 3.3 Ozan Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-009	15-2	Ozan	16,700	2820	1500	1130
B-033	15-2	Ozan	1190	1055	1100	16,000
B-057	14	Ozan	12,600	275	275	280
B-082	14	Ozan	60	200	205	290
B-118	15-2	Ozan	15,150	1235	1255	17,400
B-120	15-2	Ozan	18,705	1435	1315	16,760
B-122	15-2	Ozan	15,000	755	690	950
B-224	14	Ozan/A - Chalk	345	110	100	115
B-116	15-2	Ozan	6700	2345	1510	17,110
B-119	15-2	Ozan	1500	15,620	15,600	16,700
B-227	15-2	Ozan	1090	1050	975	1100
B-100	14	Ozan	3630	415	620	570
B-123	15-2	Ozan	2800	1750	11,310	17,300
B-124	15-2	Ozan	15,500	1270	15,940	15,479
B-126	15-2	Ozan	10	25	20	1,015

Table 3.4 Wolfe City Sand Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-008	15-1	Wolfe	1200	750	90	60
B-029	15-2	Wolfe	50	785	700	620
B-030	15-2	Wolfe	310	800	700	625
B-031	15-2	Wolfe	20	1050	1000	775
B-032	15-2	Wolfe	125	200	N/A	335
B-034	15-2	Wolfe	670	100	480	N/A
B-035	15-2	Wolfe	1065	10,600	1255	1460
B-036	15-2	Wolfe	25	115	135	330
B-065	15-1	Wolfe	20	120	45	130
B-066	15-1	Wolfe	65	45	85	75
B-067	15-1	Wolfe	80	115	105	350
B-089	15-1	Wolfe	390	310	470	14,100

Table 3.5 Neylandville Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-026	15-1	Neylandville	975	1500	605	710
B-027	15-1	Neylandville	535	690	790	450
B-174	15-1	Neylandville	90	135	110	315
B-194	15-1	Neylandville	460	180	210	840

Table 3.6 Kemp Clay Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-007	15-1	Kemp	180	280	325	100
B-025	15-1	Kemp	1880	17,000	1100	2400
B-068	15-1	Kemp	1270	255	215	470
B-069	15-1	Kemp	11,600	16,100	1360	2430
B-070	15-1	Kemp	2120	8420	10,000	18,080
B-094	15-1	Kemp	260	255	90	250
B-095	15-1	Kemp	115	300	220	200
B-096	15-1	Kemp	1065	690	700	620
B-097	15-1	Kemp	1400	840	850	1170
B-180	15-1	Kemp	3215	1070	970	15,120

Table 3.7 Wills Point Sulfate Concentrations

Boring	Section	Formation	Soluble Sulfate at Different Depth Interval			
			5 ft. (ppm)	10 ft. (ppm)	15 ft. (ppm)	20 ft. (ppm)
B-005	16	Wills Point	260	150	200	510
B-017	16	Wills Point	105	55	30	20
B-018	16	Wills Point	55	390	185	200
B-020	17	Wills Point	650	670	575	675
B-021	17	Wills Point	90	325	365	510
B-037	17-1	Wills Point	45	20	30	20
B-060	17-1	Wills Point	350	300	650	400
P-002	18	Wills Point	N/A	N/A	35	N/A
B-145	16	Wills Point	315	380	220	350
B-146	16	Wills Point	750	230	20	5
B-147	16	Wills Point	240	100	270	200
B-148	16	Wills Point	300	150	25	15
B-150	16	Wills Point	280	40	60	40
B-151	16	Wills Point	135	200	70	250
B-152	16	Wills Point	200	10	115	230
B-153	16	Wills Point	415	90	70	20

3.5 Deterministic Sulfate Concentration Bar Plots

Figures 3.6 – 3.9 show the bar chart plots generated to illustrate the variation of sulfate concentration values for all depth intervals utilized for testing and geostatistical mapping. This is helpful in highlighting the variability between data points with respect to distance. The datum for the bar plots was chosen to be B – 014 which lies in the Mainstreet Limestone/Grayson Marl formation. All distances on the bar chart plots are straight line distances in relation to this boring, which was not a boring from the testing scheme but is the west most boring as identified on the geological map for the pipeline alignment (see Figure 1.2). The distance between borings is the straight line distance as calculated by simple distance formula as this approach yields the best visual results. Using the approach of straight line distance from the datum, certain data points can be seen rather than be obscured from view. Distances between data points can be taken as the numerical difference between the distances labeled on the plot that corresponds to the borings in question. Only some of the borings have been listed so as to make the plots easier to visualize. Additionally, the formations have been added to the plots by using brackets and can be seen above the formations they represent. It can be seen from both the bar plots in Figures 3.6 through 3.9 and the above Tables 3.2 through 3.7 that sulfate values are quite random with respect to distance. Considering the Eagle Ford formation show in Figures 3.6 through 3.9, it can be seen that sulfate values tend to spike and fall randomly in close proximity to one another. Looking at the Ozan in Figures 3.6 through 3.9 we see the same pattern. Again we see the same pattern with regards to Wolfe City Sand and Kemp Clay in Figures 3.6 through 3.9. The rising and falling of sulfate values does not appear in the Neylandville and Wills Point Formations. The Neylandville and Wills Point Formations exhibited consistently low sulfate values throughout the entire testing length and depth. These patterns of rising and falling sulfate levels carry over into all depth intervals tested for the Eagle Ford, Ozan, Wolfe City Sand and Kemp Clay as indicated by Figures 3.6 through 3.9. This information reiterates the random manifestation of sulfates in soil (See Figures 2.3.a – 2.3.e). The random manifestation of such sulfates might possibly be due to ground water transportation of the sulfate minerals or due to the complex nature of the initial formation and deposition of sulfates through the evaporation and precipitation reactions of salt water. Never the less, the prediction and mapping of sulfate values using a deterministic approach is impossible, therefore the use of geostatistics must be implored. The next chapter focuses on the development and analysis of the geostatistical portion of the research.

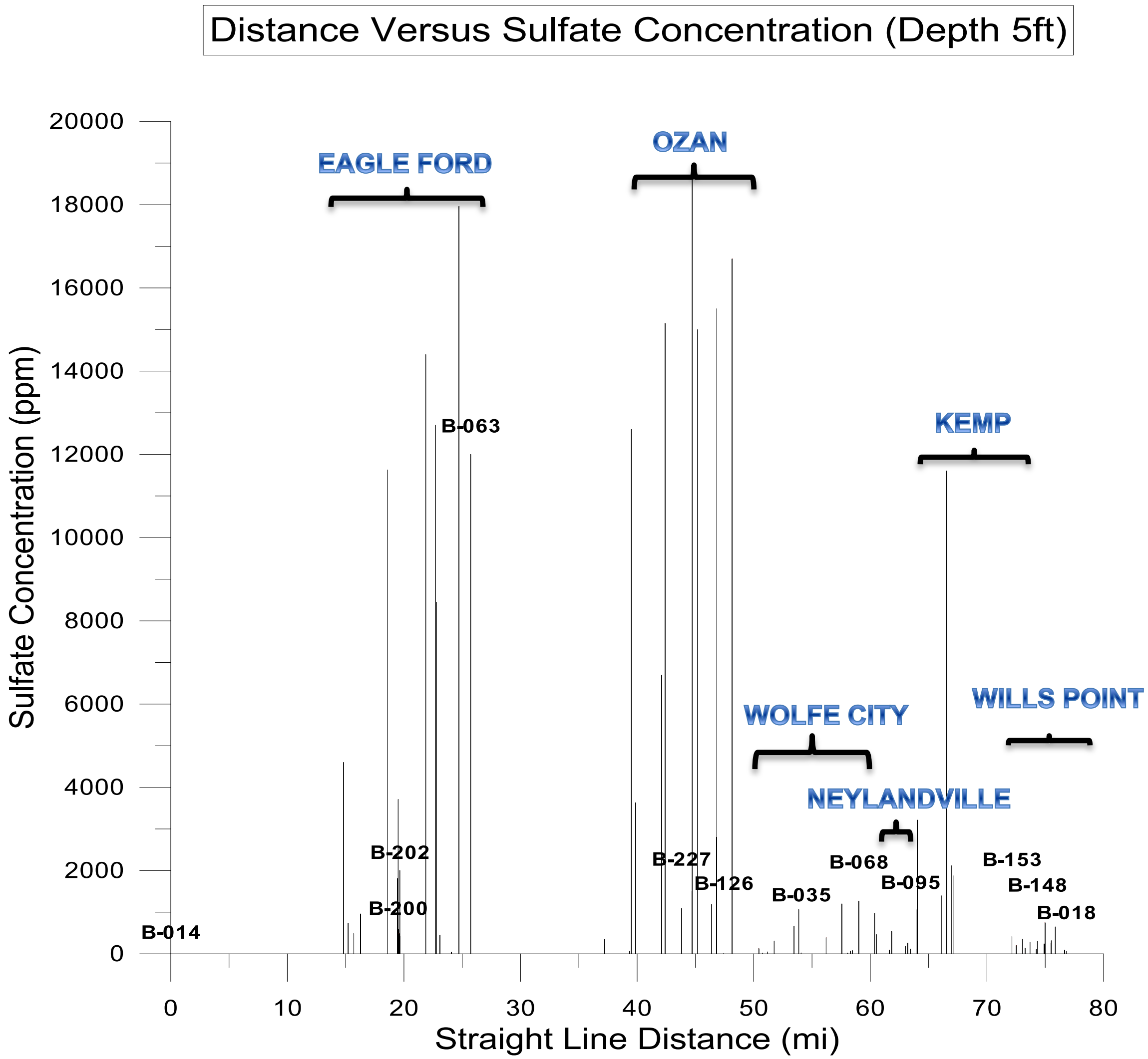


Figure 3.6 Straight Line Distance vs. Sulfate Concentration (Depth 5ft)

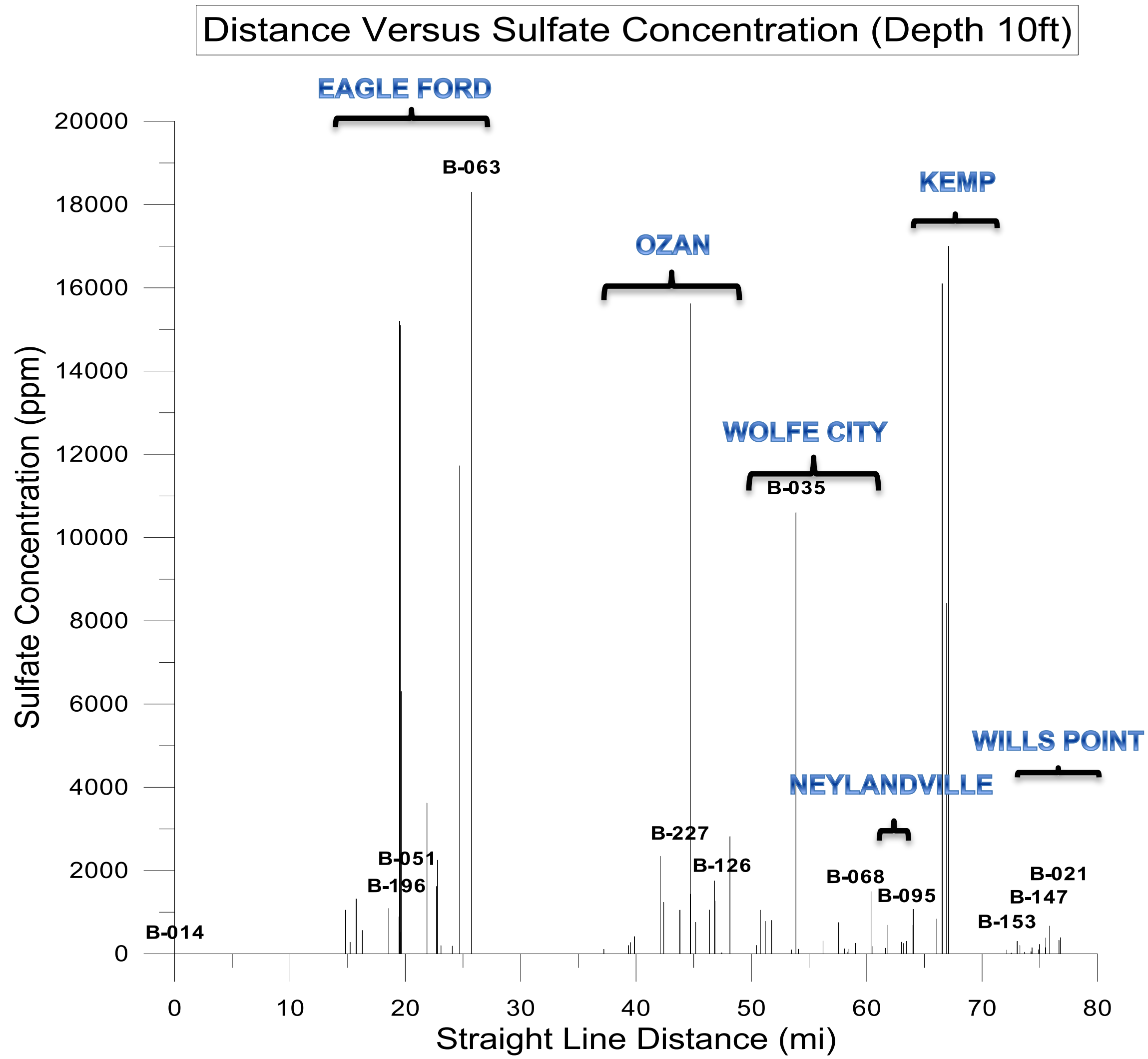


Figure 3.7 Straight Line Distance vs. Sulfate Concentration (Depth 10ft)

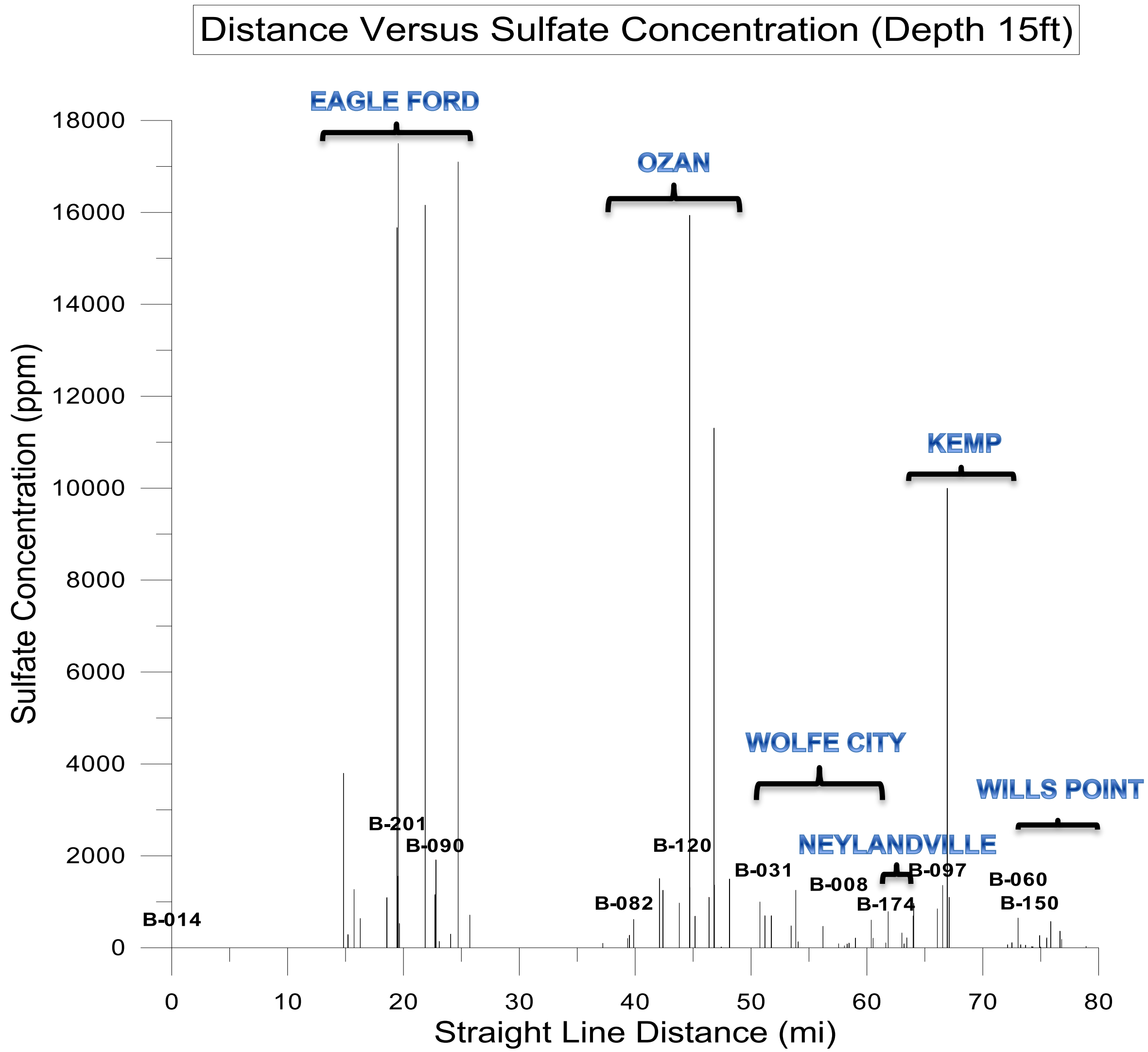


Figure 3.8 Straight Line Distance vs. Sulfate Concentration (Depth 15ft)

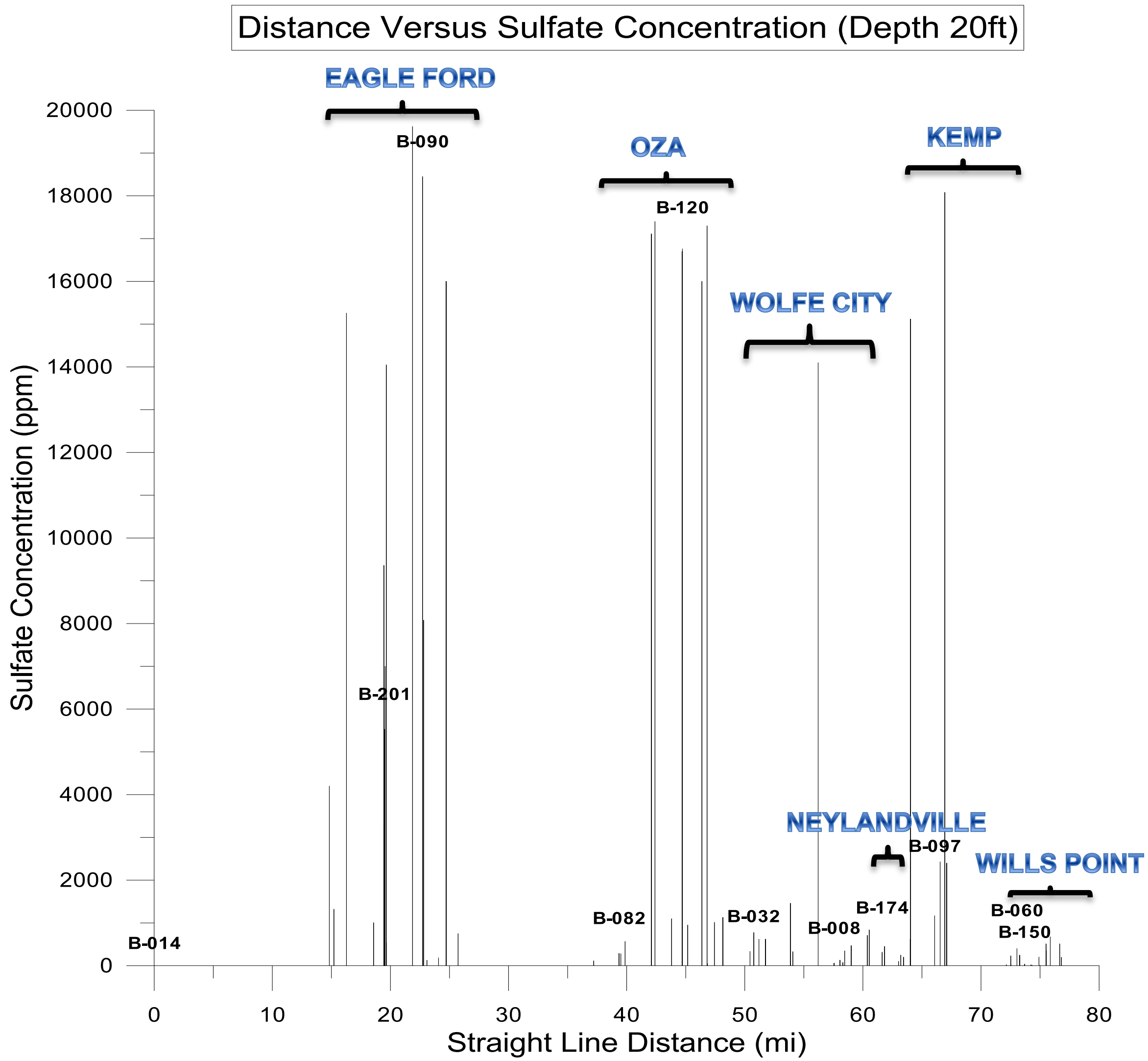


Figure 3.9 Straight Line Distance vs. Sulfate Concentration (Depth 20ft)

CHAPTER 4

GEOSTATISTICAL ANALYSIS AND MAPPING

4.1 Introduction

The following chapter contains various aspects of the geostatistical analysis of sulfate values. In addition to the presentation of results, discussions are presented to explain the nature and results of the analyses. The chapter begins with the presentation of individual formation contour maps. Next, the overall mapping of all formations collectively is presented. This is a useful tool in comparing the individual formation maps with the overall formation maps. Lastly, the Kriging error associated with the geostatistical analysis is presented in order to bring validity to the mapping. This is done by plotting the most variable formation maps, least variable formation maps, as well as all formations collectively.

The geostatistical analysis was conducted using Surfer 9 Software manufactured by Golden Software Inc. ©. 2-D Aerial contours were generated for each formation individually as well as all formations collectively at the unique depth interval specified. The 2-D analysis was not conducted using sulfate values other than the values determined at the specific depth interval. In other words, only 5ft. depth sulfate values were used to generate the 5ft. depth maps. Point Kriging techniques were utilized in conjunction with experimental variograms. Ordinary Kriging principles and techniques have been developed specifically for data points that are discreet and without trend (Armstrong 1998). Therefore, Ordinary Point Kriging was chosen to analyze the discrete non-continuous data set that describes the soluble sulfate concentrations for the pipeline alignment. Due to the irregularity in the data a linear model was used for the variograms. Sulfate concentrations are inherently random and in the current data set only the linear trend model proved useful for geostatistical analysis.

A total of 4 maps (at 5 ft., 10 ft., 15 ft. and 15 ft. depths) were developed for each formation and also for all formations together. This generated a total of 32 maps for the 6 different formations that were tested in this project. In order to reduce the number of maps, Wills Point and Kemp Clay were combined in mapping as well as the Wolfe City Sand and

Neylandville formations based on their proximity to each other and the number of data points available to make successful predictions. Eagle Ford and Ozan maps were not combined due to the gap in data associated with the Austin Chalk formation, which was not tested as part of this research project. After combining formations, the number of maps was reduced to 16 individual formation maps and 4 collective formation maps. The collective formation maps are also shown overlain onto the geologic formation map supplied by Fugro Consultants Inc., totaling 24 maps. Each of the formation's maps corresponds to the unique selected depth interval for testing (i.e. 5ft., 10ft., 15ft., & 20ft.). Therefore, the maps can be read regardless of elevation changes. In other words, the generated maps will indicate possible sulfate values at the depth specified by the map in any location within that particular formation. Each map was designed to predict sulfate values at the corresponding depth only. Interpretation of sulfate values in between depth intervals would be done at the risk of the interpreter.

4.2 Eagle Ford Formation

The Eagle Ford formation results indicate that it is one of the more problematic formations when it comes to high soluble sulfate values and sulfate values greater than 2000 ppm. The range of sulfate values in the Eagle Ford ranges from 40 ppm to 19,620 ppm. Figure 4.1 indicates several zones of concern where sulfate values range from 5000 ppm to 14,000 ppm. In addition, Figure 4.1 indicates there are zones of sulfates lower than 2000 ppm. Specifically, from B-51 to B-196 values of sulfate are below typical problematic threshold levels. Also, from B-164 to B-84 a zone of low sulfate values is present. However, in the 10ft map, Figure 4.2 shows that in the same zone of low sulfate values for Figure 4.1, there is a pocket of high sulfates ranging from 4000 ppm to 14,000 ppm. This is due to the random manifestation of sulfates in the soil).

Also, it can be seen in Figure 4.3 that a large portion of the formation appears to have sulfate values in the acceptable range. Looking at Figure 4.4, 3 major zones of concern with sulfate values higher than 5000 ppm and ranging to 15,000 ppm are present. These high sulfate zones are located near B-209, B-198, and B-93. Figure 4.8, the 20ft. map, shows the highest concentrations of sulfate values for the Eagle Ford. Sulfate values range from 75 ppm to 19,620 ppm. The specific zones of interest are located near B-164, B-202, B-93, and B-209. Looking at all depths it can see that certain zones appear to be below problematic threshold values. Specifically, B-84 and B-318 appear to have very low sulfate values. In addition, B-61 and B-59 are observed to have very low sulfate ranges throughout all depths. Conversely, zones around B-93 and B-198 have consistently high sulfate values throughout all depths tested. Overall the Eagle Ford is very problematic and must be treated with caution.

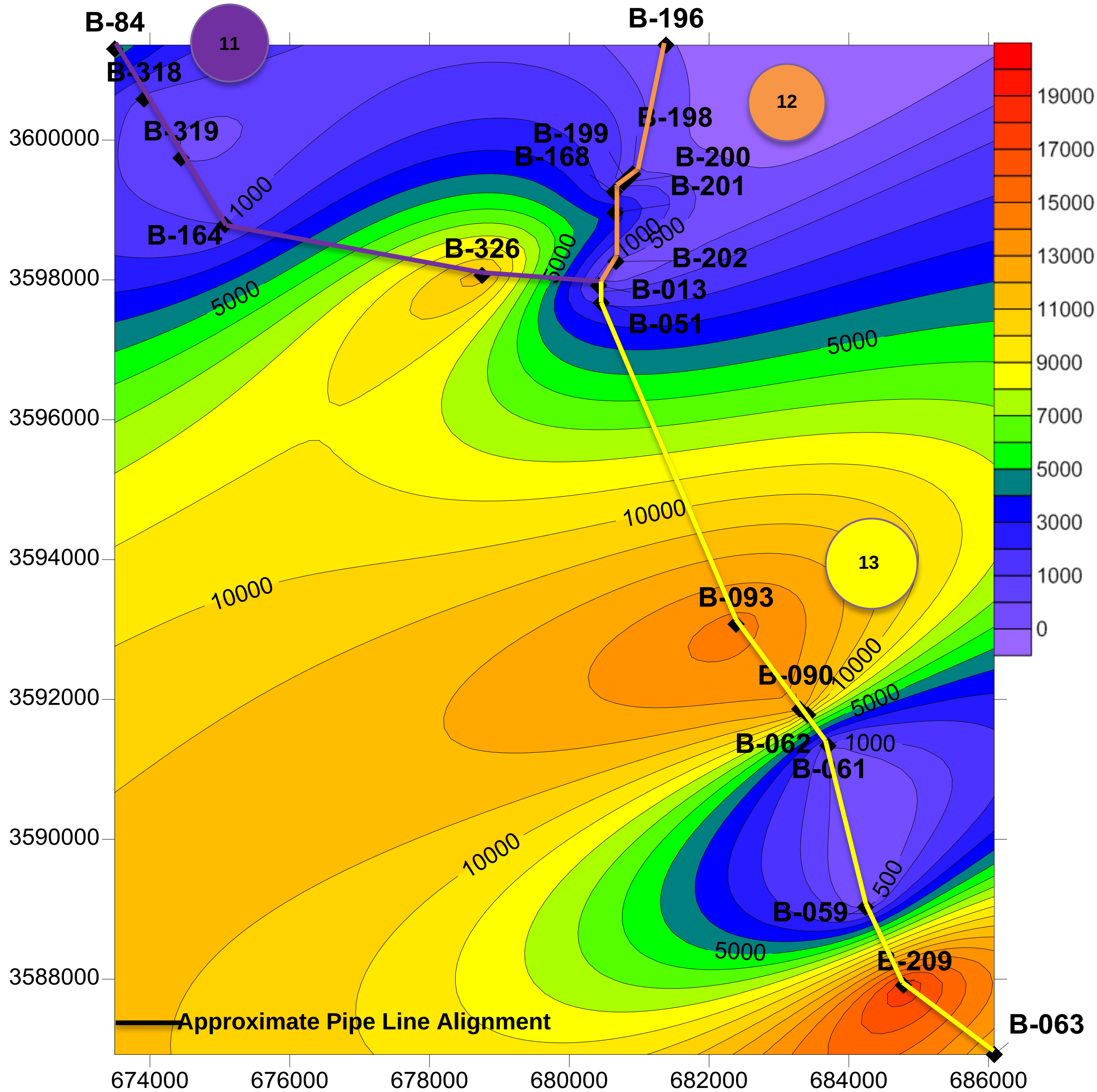


Figure 4.1 Eagle Ford Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 5ft)

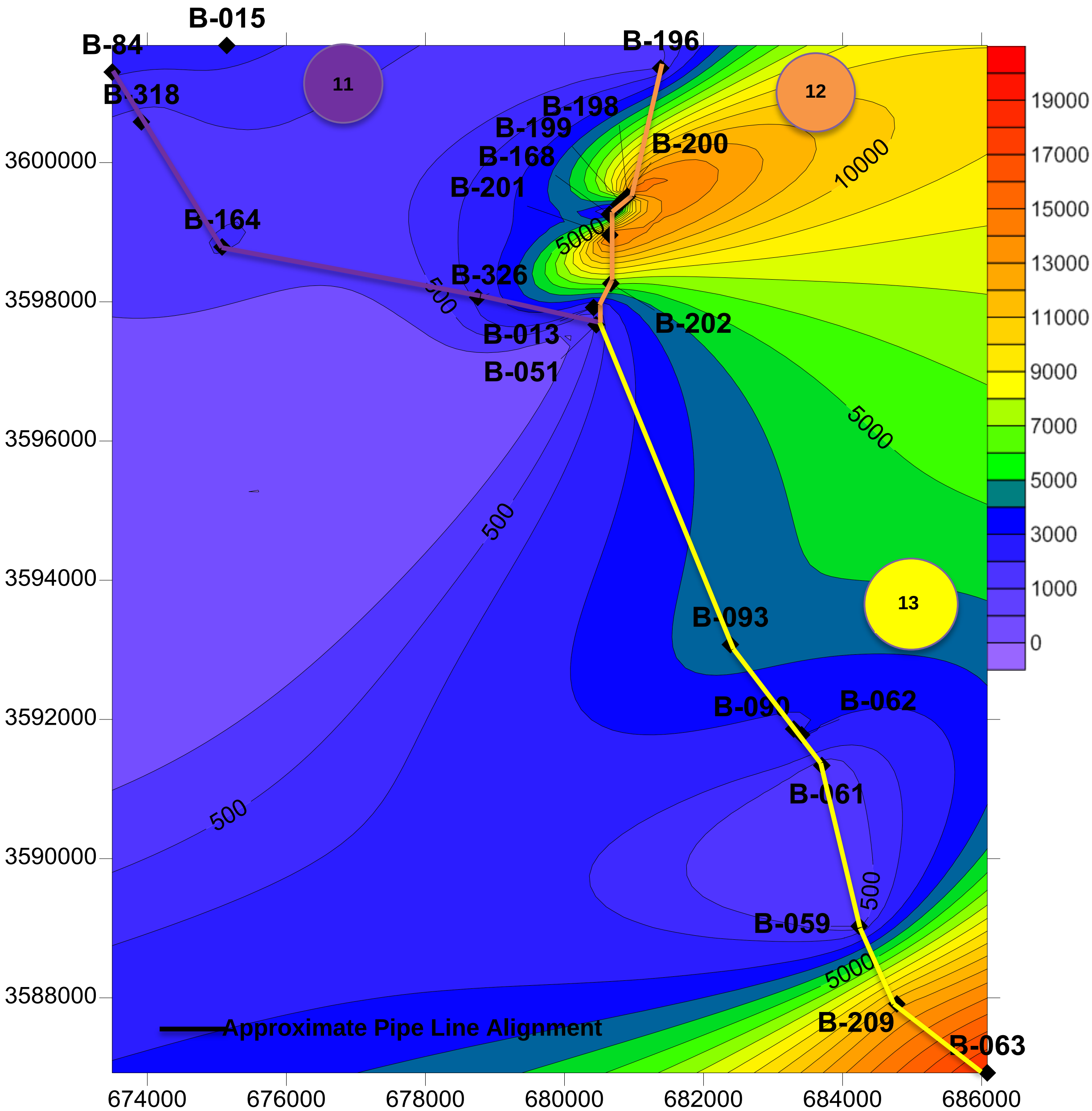


Figure 4.2 Eagle Ford Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 10ft)

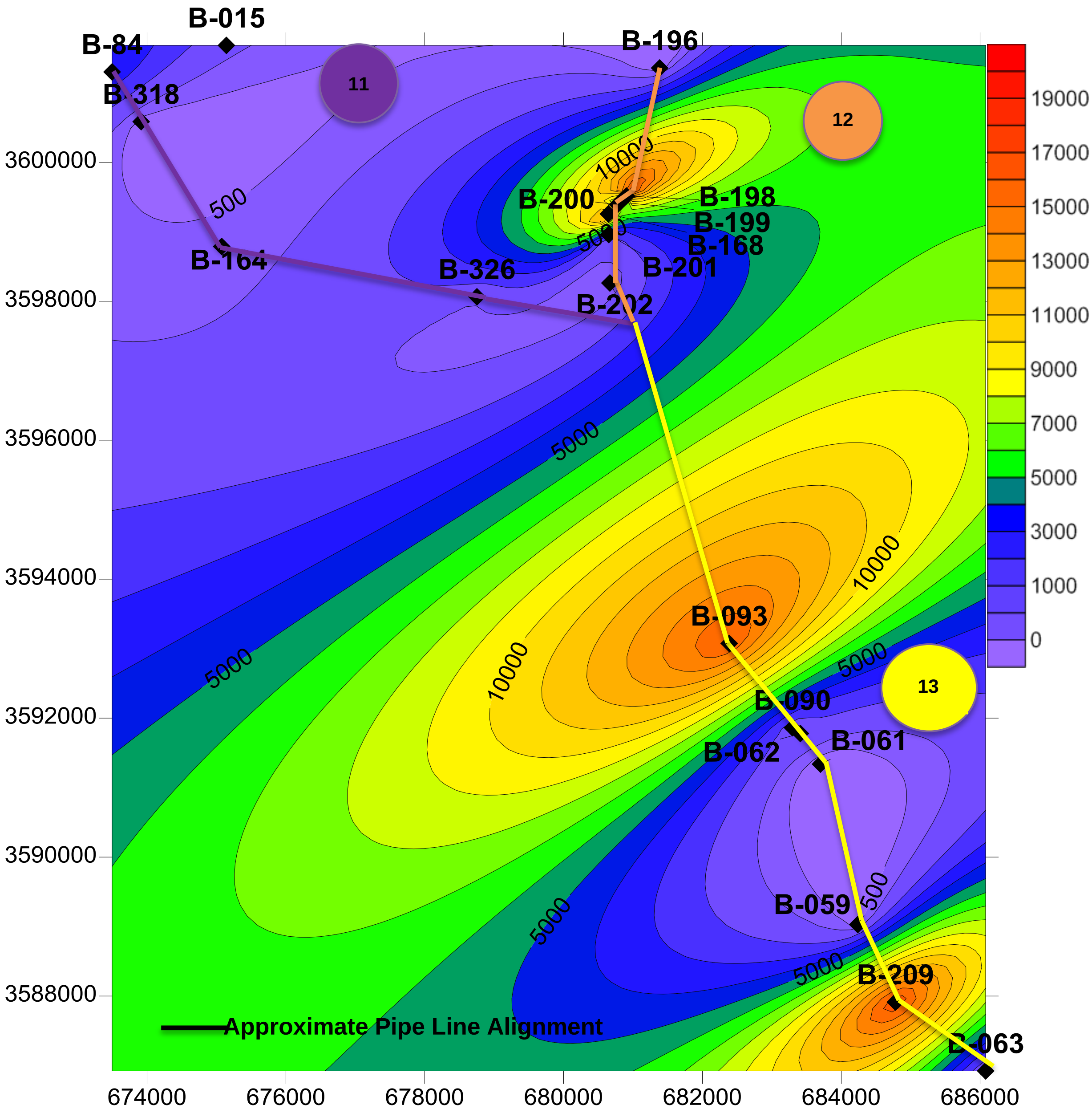


Figure 4.3 Eagle Ford Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 15ft)

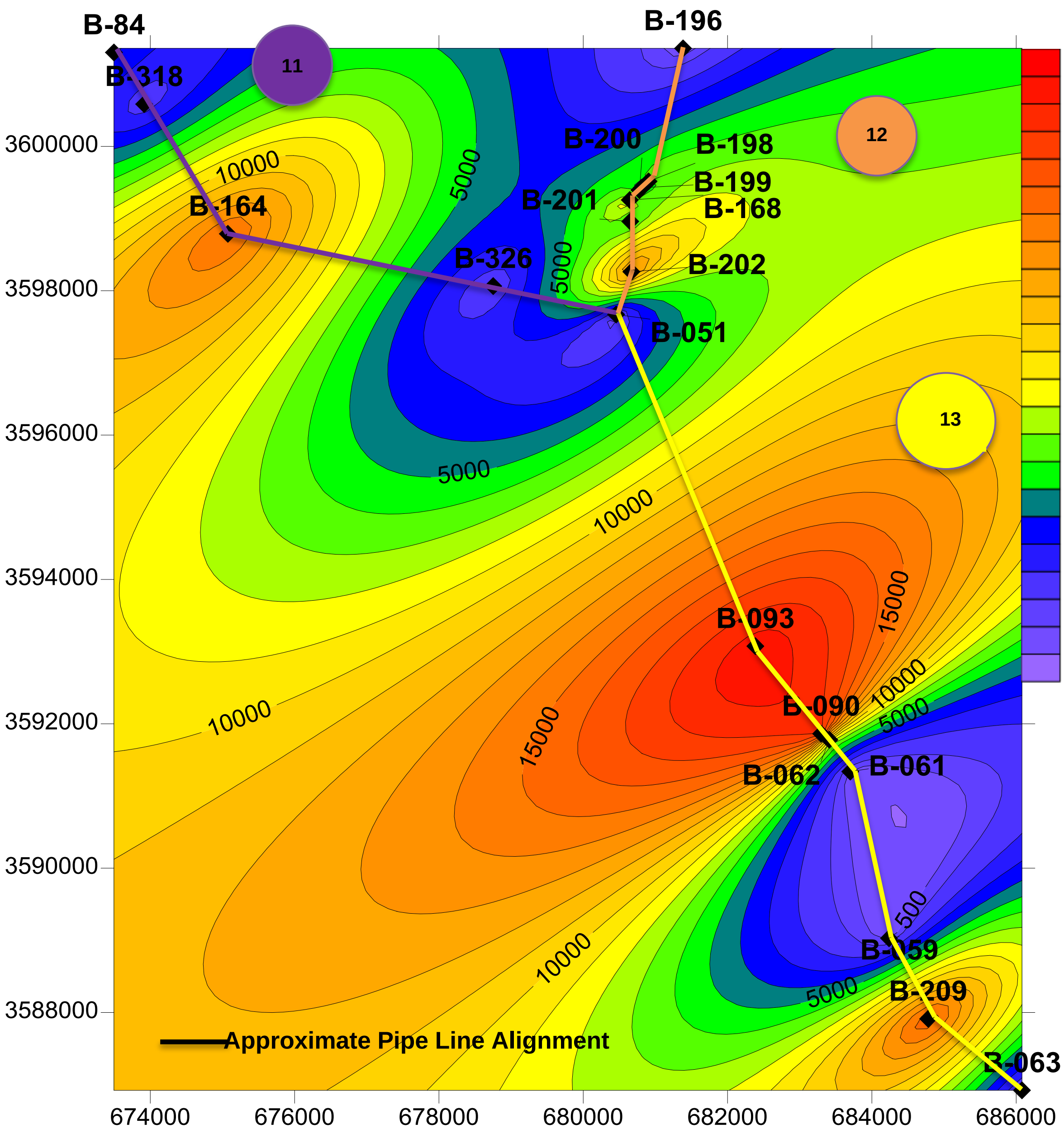


Figure 4.4 Eagle Ford Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 20ft)

4.3 Ozan Formation

The Ozan Formation, much like the Eagle Ford, is also very problematic in regards to high sulfate values. From Figure 4.5, the 5ft. depth map, it can see that there are several problem areas with sulfate values higher than 5000 ppm and there appear to be a few zones of sulfate values less than 2000 ppm. However, Figure 4.6 indicates that much of the formation has sulfate levels less than 2000 ppm with only one zone of high sulfate near B-119 and B-120. Sulfate values for B-119 and B-120 are as high as 16,760 ppm. Moving to Figure 4.7, the 15ft. map, it can see that much of the formation seems to be below typical threshold levels of sulfate, but there are zones of high sulfates near B-119, B-120, B-123 and B-124. In these locations the sulfate values range from 1300 ppm to 16,000 ppm which far exceeded problematic levels of sulfates. Lastly, in the Figure 4.8, the 20ft. map, the most problematic sulfate conditions are observed. Similar to the 5ft. map, Figure 4.8, those zones near B-116 and B-118 have high sulfate pockets. In addition, zones near B-119, B-120, B-124, and B-33 have significantly high sulfate values. Looking at all maps combined a trend of low sulfate areas near B-224 and B-87 is observed, located in the northwest corner of the formation. In addition B-227 appears to have sulfate values in below problematic threshold ranges for all depths. Overall, the Ozan has several pockets of excessively high sulfate values at all depths and must be considered with extreme caution.

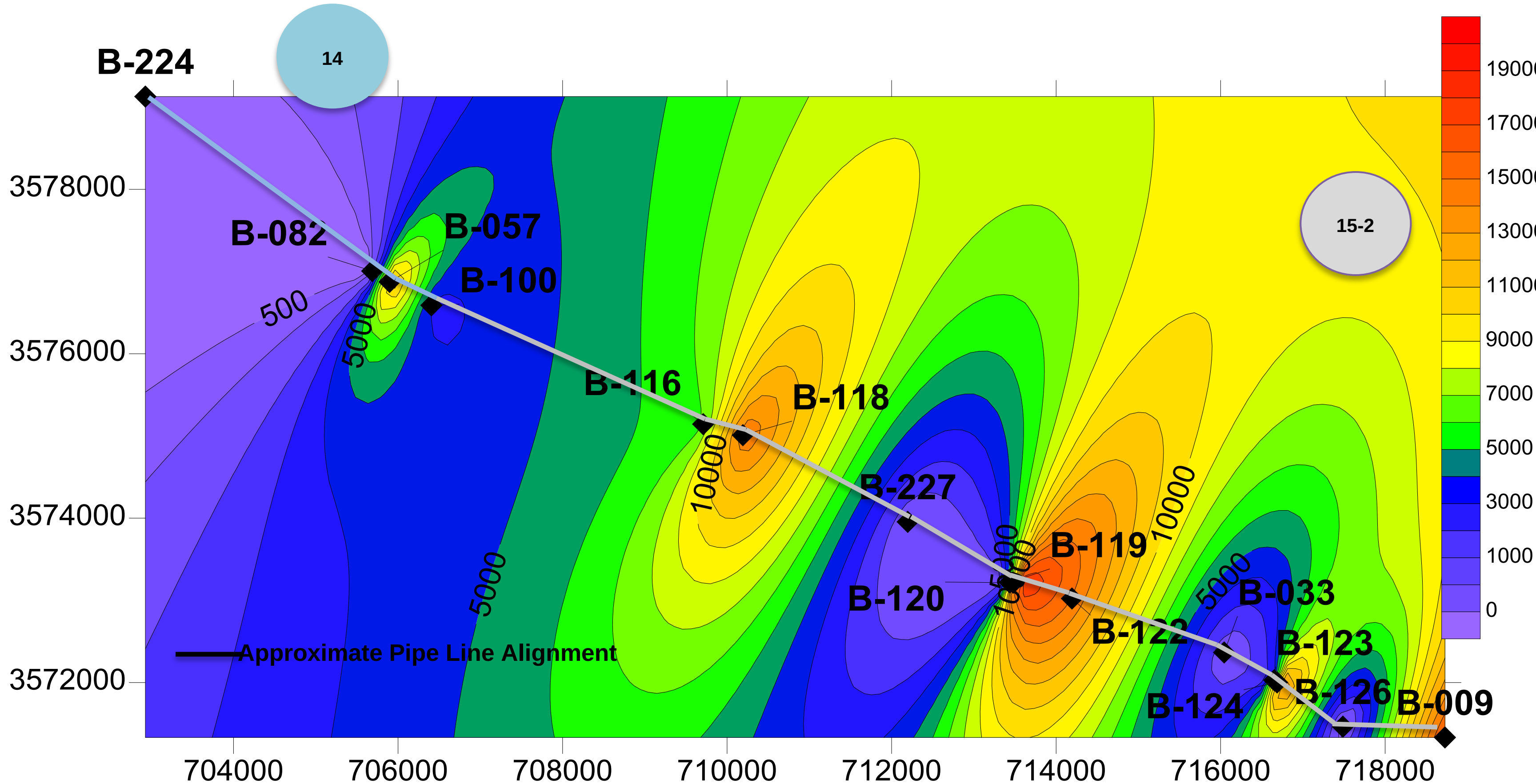


Figure 4.5 Ozan Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers

(Depth – 5ft)

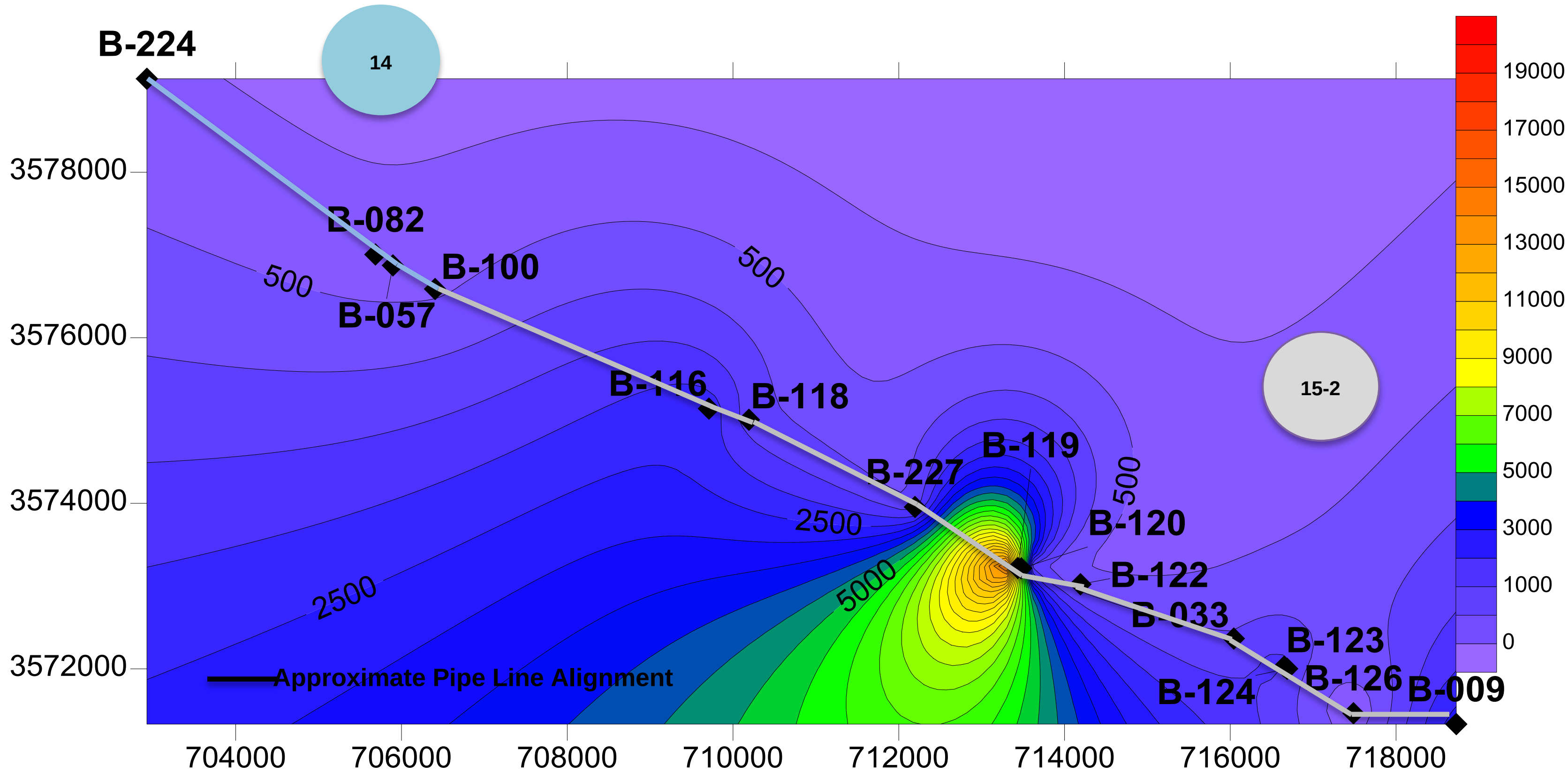


Figure 4.6 Ozan Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers

(Depth – 10ft)

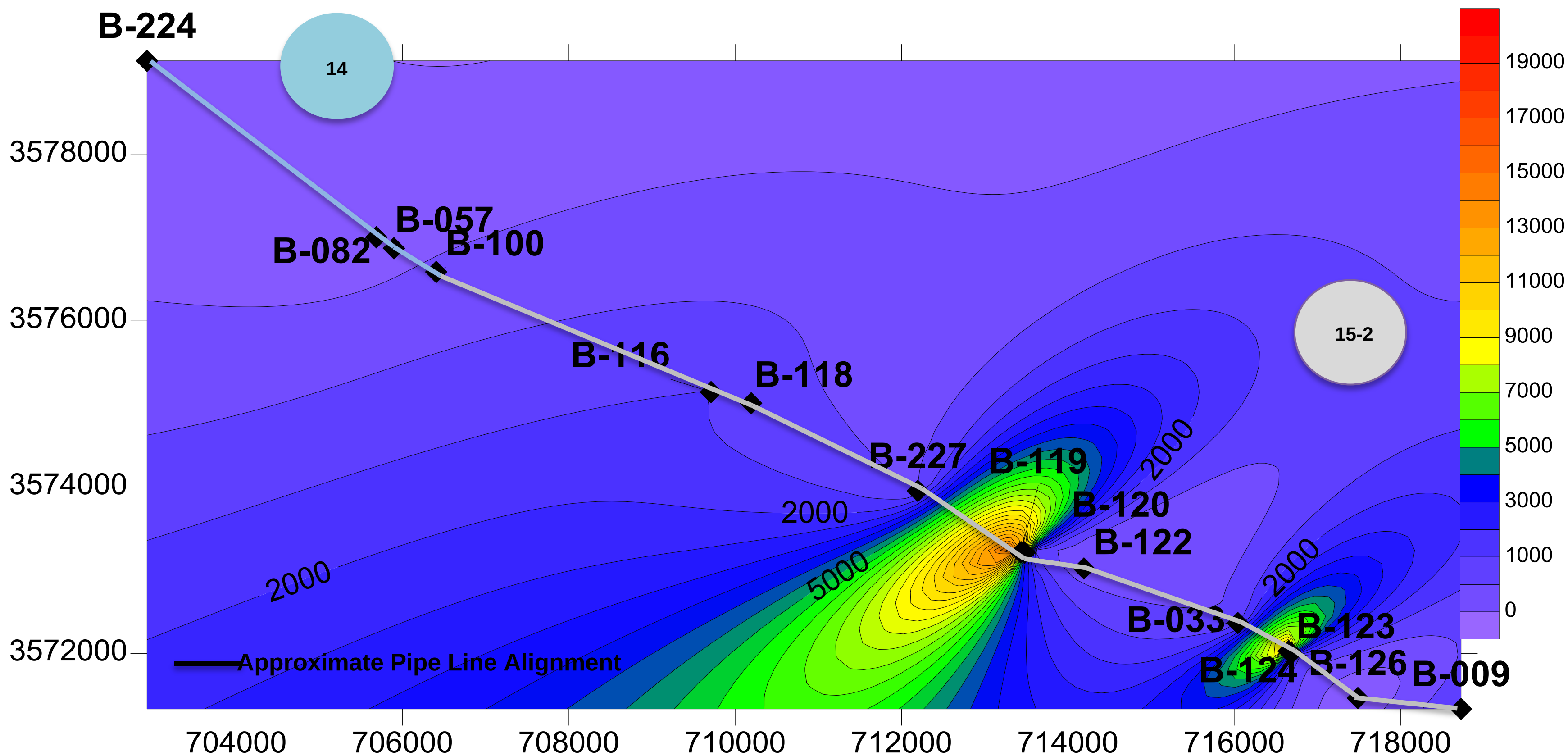


Figure 4.7 Ozan Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers

(Depth – 15ft)

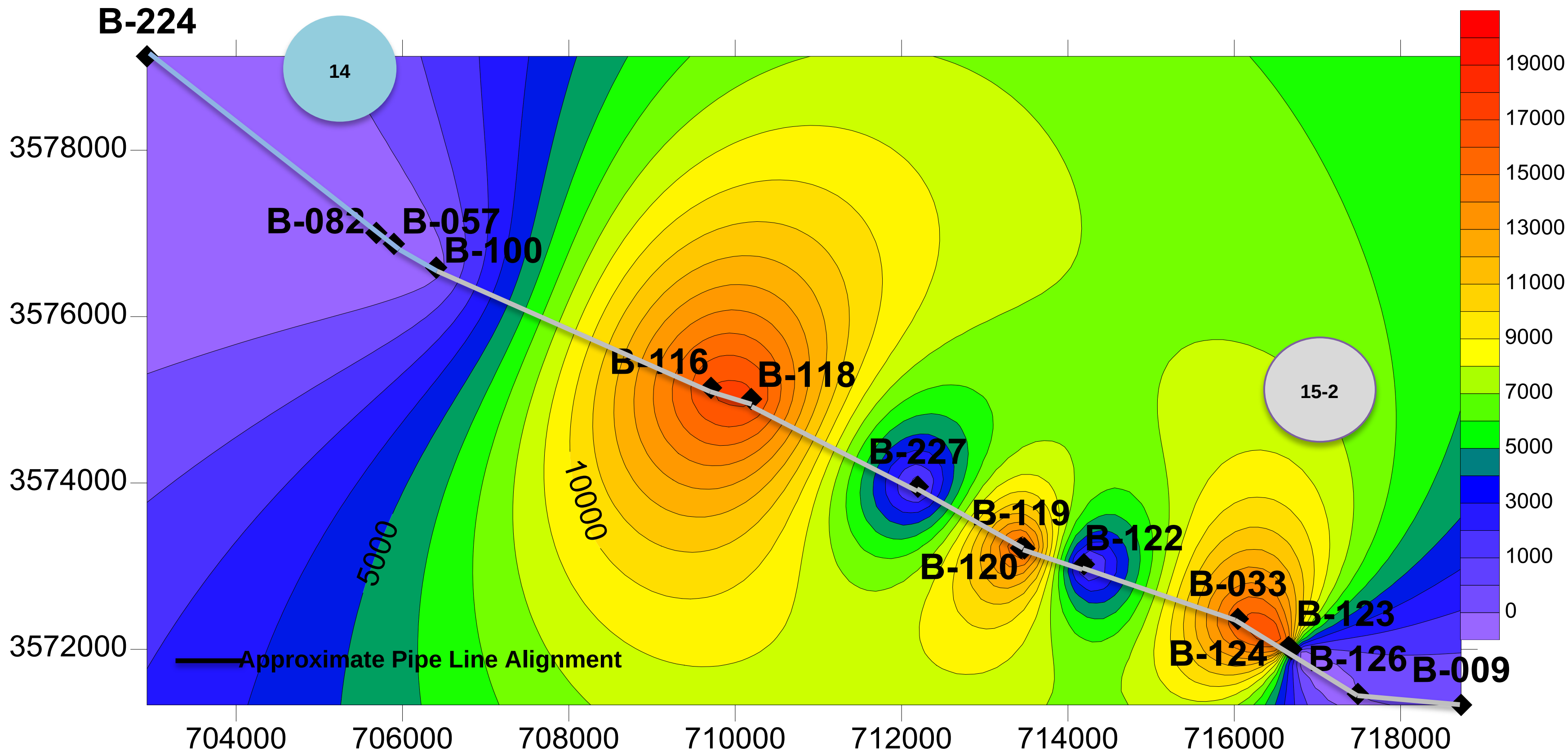


Figure 4.8 Ozan Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers

(Depth – 20ft)

4.4 Wolfe City Sand and Neylandville Formations

The Wolfe City Sand and Neylandville portions of the alignment appear to have very satisfactory sulfate levels. As the Figure 4.9 indicates the entire 5ft. portion of the alignment appears to have sulfate values less than 2000 ppm. Figure 4.10, the 10ft. section, also has sulfate levels predominately below 2000 ppm; however there is a zone of high sulfate in the Wolfe City Sand near B- 34, B-35 and B-36. Figure 4.11, the 15ft. map, also appears to have all sulfate values less than 2000 ppm. Lastly, the 20ft. map, Figure 4.12 shows only one zone of high sulfates at B-89. Specifically, the sulfate value is around 14,100 ppm. Overall, the Neylandville section has consistently low sulfate zones lower than 2000 ppm and the Wolfe City only has two zones of high sulfate at 10ft. depth near B- 34 and B-35, and also at the 20ft. depth near B-89.

4.5 Kemp Clay and Wills Point Formations

It can be seen from the mapping analysis that the Kemp Clay and Wills Point sections show relatively low sulfate values. In fact, the Wills Point borings only showed sulfate values between 5 ppm and 750 ppm throughout all depths and is considered very low risk for chemical stabilization. Looking at Figure 4.13, the 5ft. map shows only one zone of high sulfate located in the Kemp portion of the alignment near B-69 and B-70, and the sulfate level is as high as 11,000 ppm. The rest of the 5ft. sulfate values are at or below 2000 ppm. The 10 ft. map, Figure 4.14, indicates again that the Kemp Clay has a zone of high sulfate near B-97, B-69, B-70, and B-25, with values ranging from 840 ppm to 17,000 ppm. Figure 4.15, the 15ft. map, indicates that the majority of the alignment has levels of sulfate lower than 2000 ppm, but again a hot spot is located at B-70 where the sulfate value increases. Lastly, the 20ft. map, Figure 4.16, shows zones of highly problematic sulfate values near B-70 and B-180. For those particular locations the sulfate values are as high as 18,000 ppm and 15,000 ppm, respectively. Overall, much of this portion of the alignment shows low values of sulfates; however, consistently high values at B-69 and B-70 are observed at all depths, as well as B-180 at 20ft. depth. Therefore, chemical stabilization is cautioned in the Kemp Clay portion of the alignment.

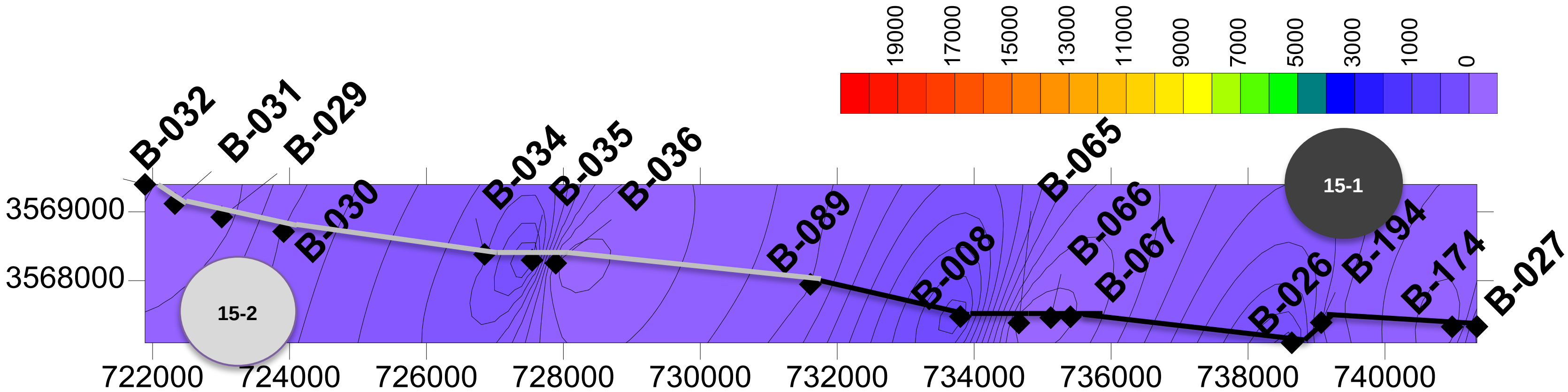


Figure 4.9 Wolfe City Sand and Neylandville Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 5ft)

Approximate Pipe Line Alignment

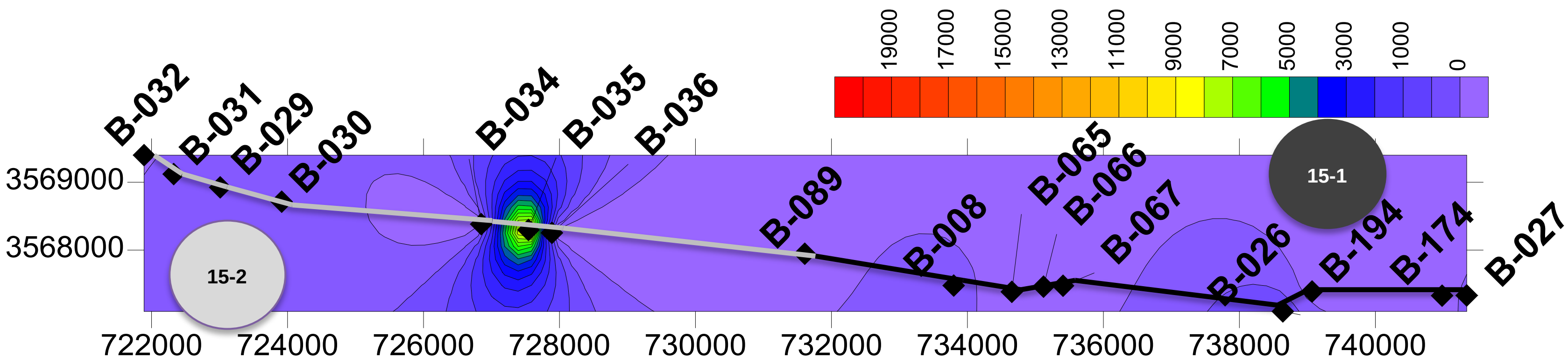


Figure 4.10 Wolfe City Sand and Neylandville Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 10ft)

Approximate Pipe Line Alignment

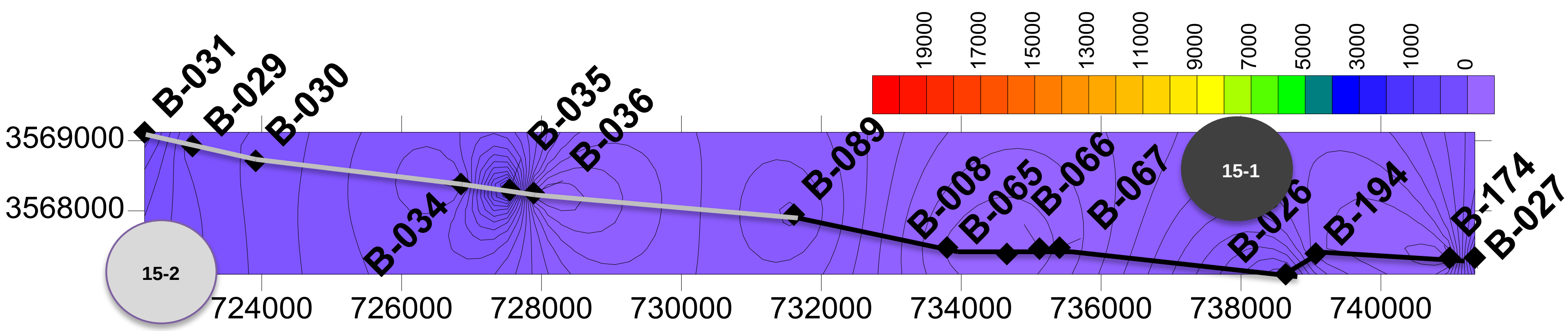


Figure 4.11 Wolfe City Sand and Neylandville Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 15ft)

Approximate Pipe Line Alignment

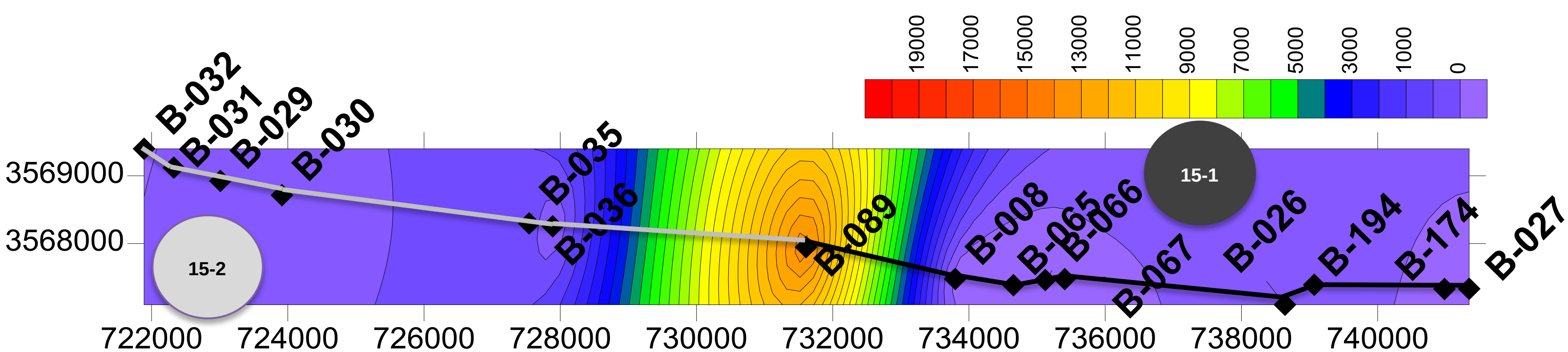


Figure 4.12 Wolfe City Sand and Neylandville Contour Map with Approximate Pipeline Alignment and IPL Parcel Numbers (Depth – 20ft)

Approximate Pipe Line Alignment

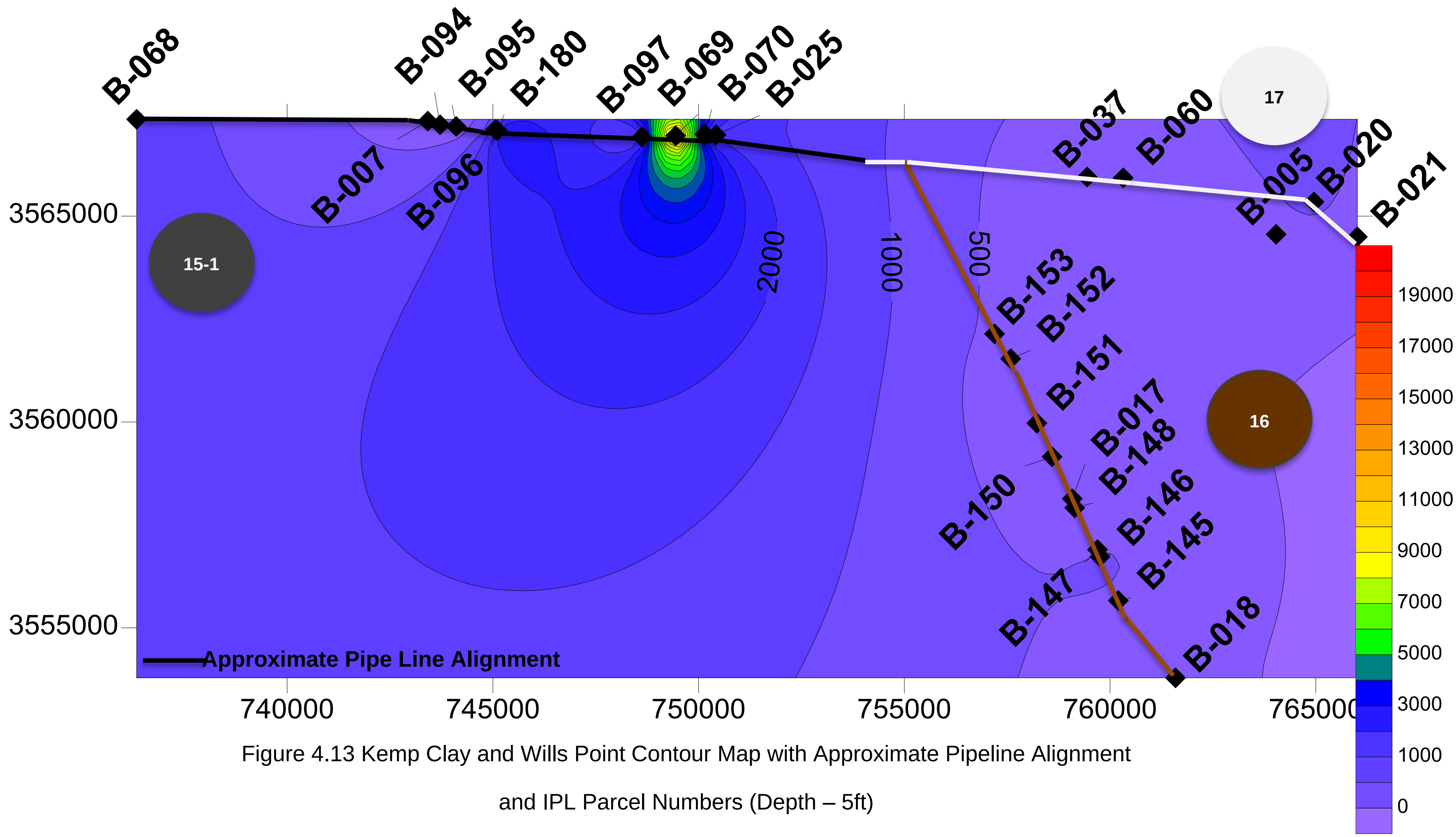


Figure 4.13 Kemp Clay and Wills Point Contour Map with Approximate Pipeline Alignment

and IPL Parcel Numbers (Depth – 5ft)

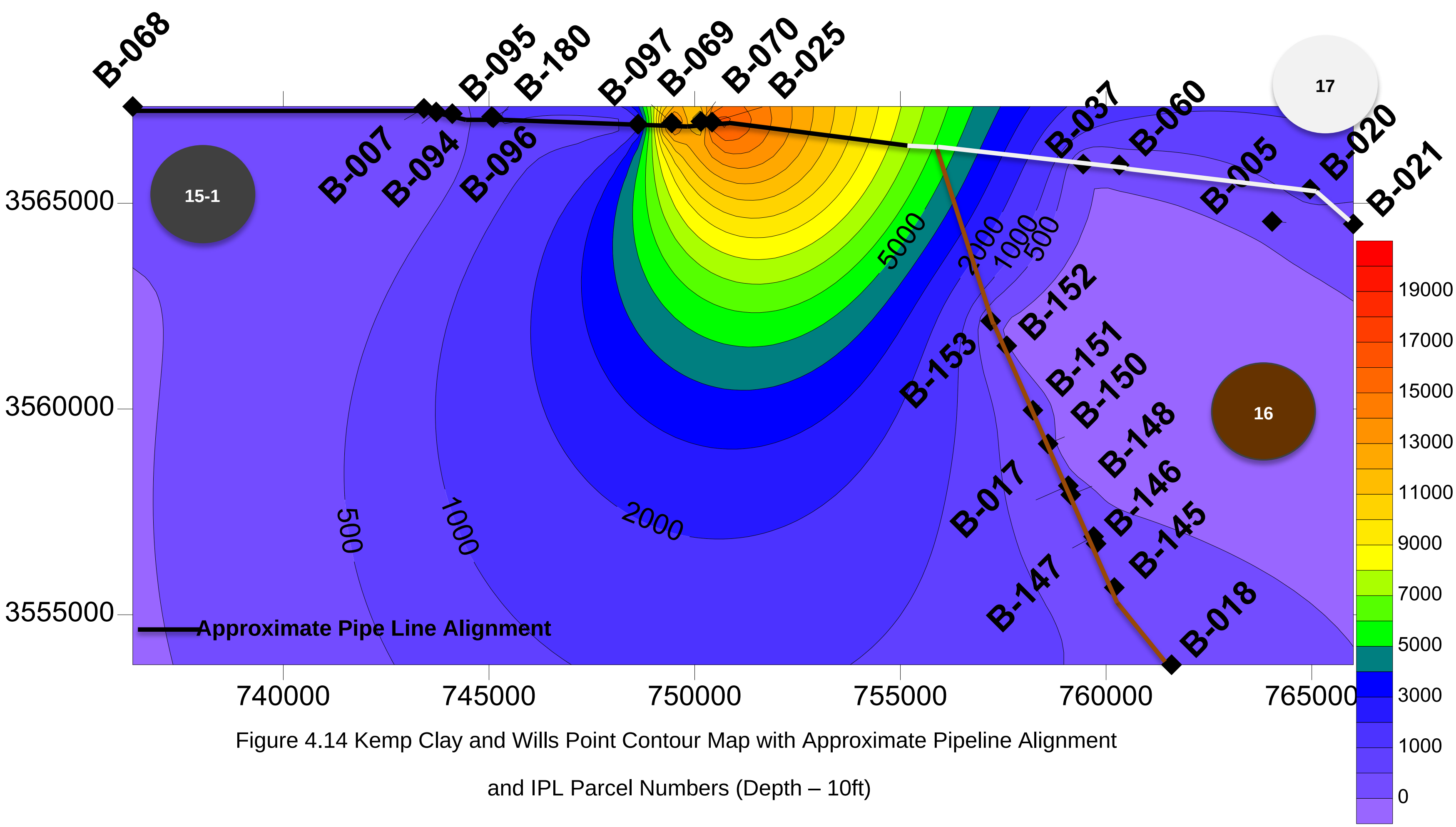


Figure 4.14 Kemp Clay and Wills Point Contour Map with Approximate Pipeline Alignment

and IPL Parcel Numbers (Depth – 10ft)

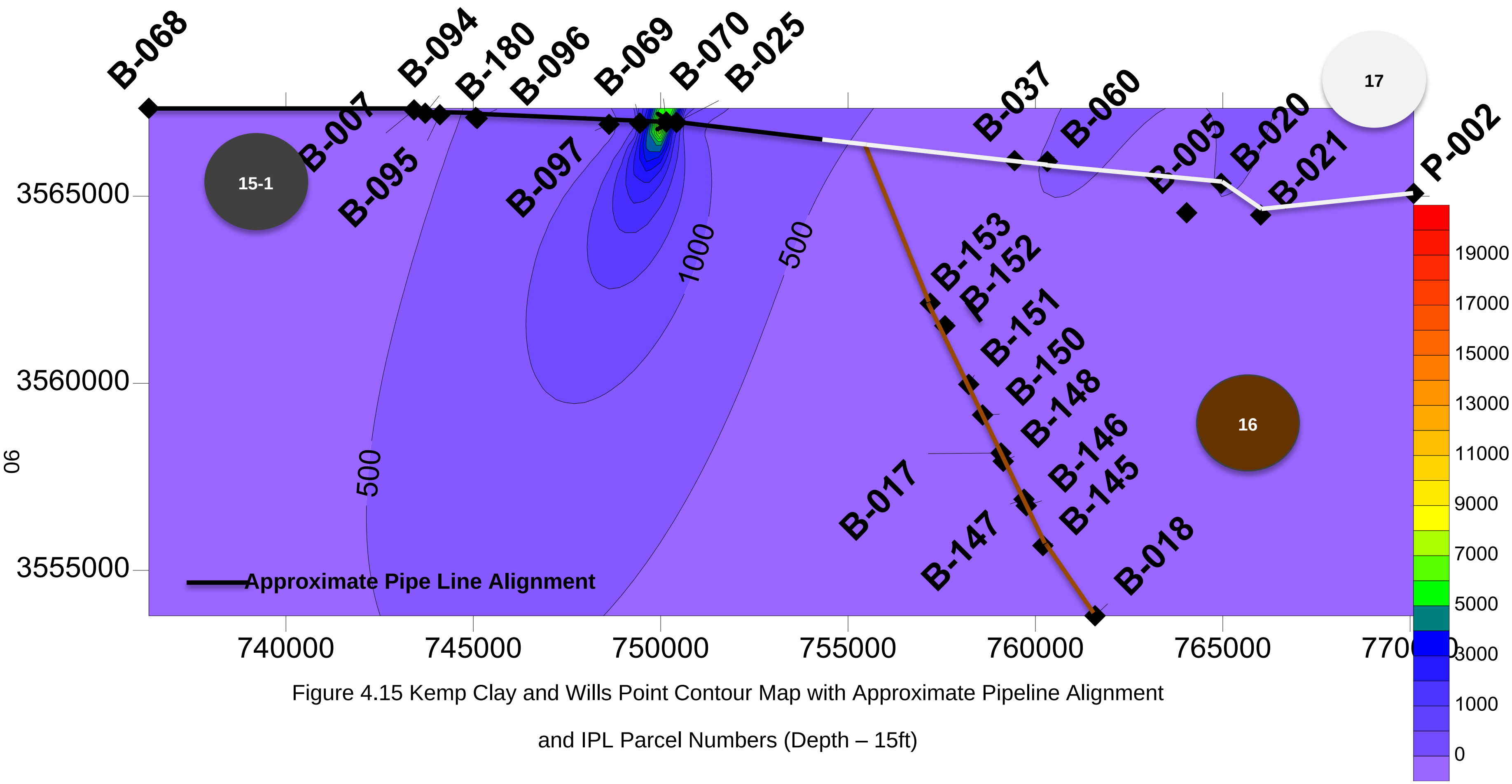


Figure 4.15 Kemp Clay and Wills Point Contour Map with Approximate Pipeline Alignment

and IPL Parcel Numbers (Depth – 15ft)

90

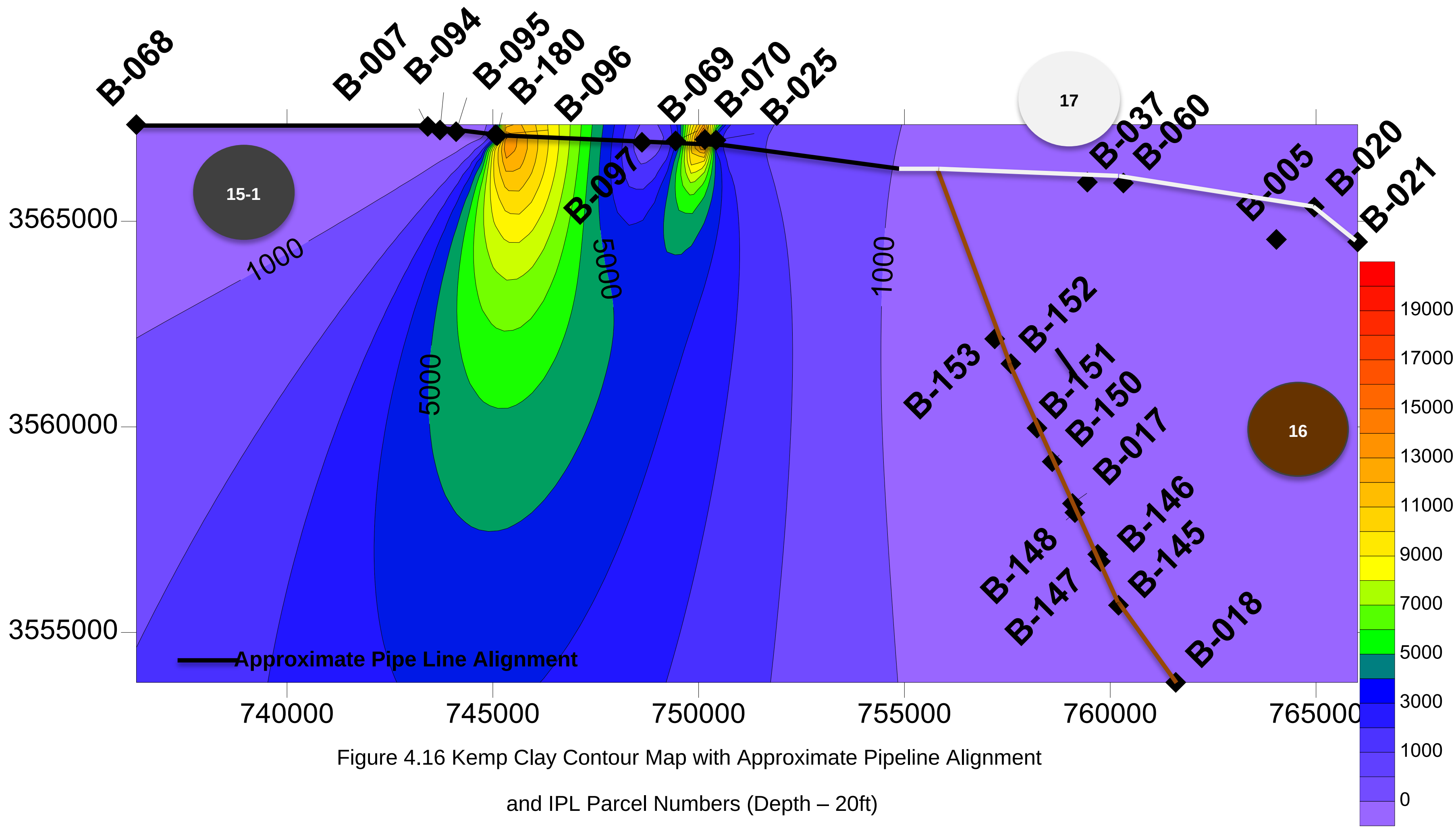


Figure 4.16 Kemp Clay Contour Map with Approximate Pipeline Alignment

and IPL Parcel Numbers (Depth – 20ft)

4.6 All Formations

From Figure 4.17, the 5ft. map, it can be observed that throughout the entire alignment there are trouble zones in the Eagle Ford, Ozan, and Kemp Clay, with highly problematic zones of high sulfates in the Eagle Ford and Ozan. In general, Figure 4.17 parallels greatly with the individual map analysis, indicating consistent results. Figure 4.19 presents the overall map for 10ft. depth; again the parallel with the individual maps generated is present. Large trouble zones in the Eagle Ford and in the Ozan were observed in Figure 4.19. Additionally, the Kemp Clay has one large high sulfate zone in the easternmost portion of the formation. This is the same result as with the individual maps at the corresponding depth interval of 10 feet Figure 4.221, the 15 ft. depth map, indicates the results of collective formation mapping parallel the individual formation maps. High sulfate zones are observed in the Eagle Ford and Ozan, while in the Wolfe City, Kemp Clay and Wills Point contain very low sulfate values. Lastly, the 20ft map, Figure 4.23 indicates the most extreme conditions along the alignment. Several high sulfate zones exist in all formations except for Wills Point and Neylandville. Overall, the same trends are seen in the collective formation map at 20ft. depth as in the individual maps.

The contour map generation indicates that Eagle Ford, Ozan, and portions of Kemp Clay and Wolfe City Sand show high values of sulfate pockets. Conversely, the map generation indicates that Neylandville and Wills Point have very low sulfate values consistently under 2000 ppm. The location and intensities of the sulfate values have a good correlation with all experimental data indicating a good deterministic portion of the geostatistical analysis. As with all geostatistical analyses, the maps generated are a prediction and not to be taken as exact values, only an indication of sulfate values in zones both tested and not tested. Extreme caution should be utilized when interpreting the geostatistical maps generated for this research project. Figures 4.18, 4.20, 4.22, & 4.24 depict the geostatistical maps approximately overlaid onto the geologic formations to give a more clear indication as to the actual zones of sulfate concentration. Further discussion of map validation id discussed in the next section.

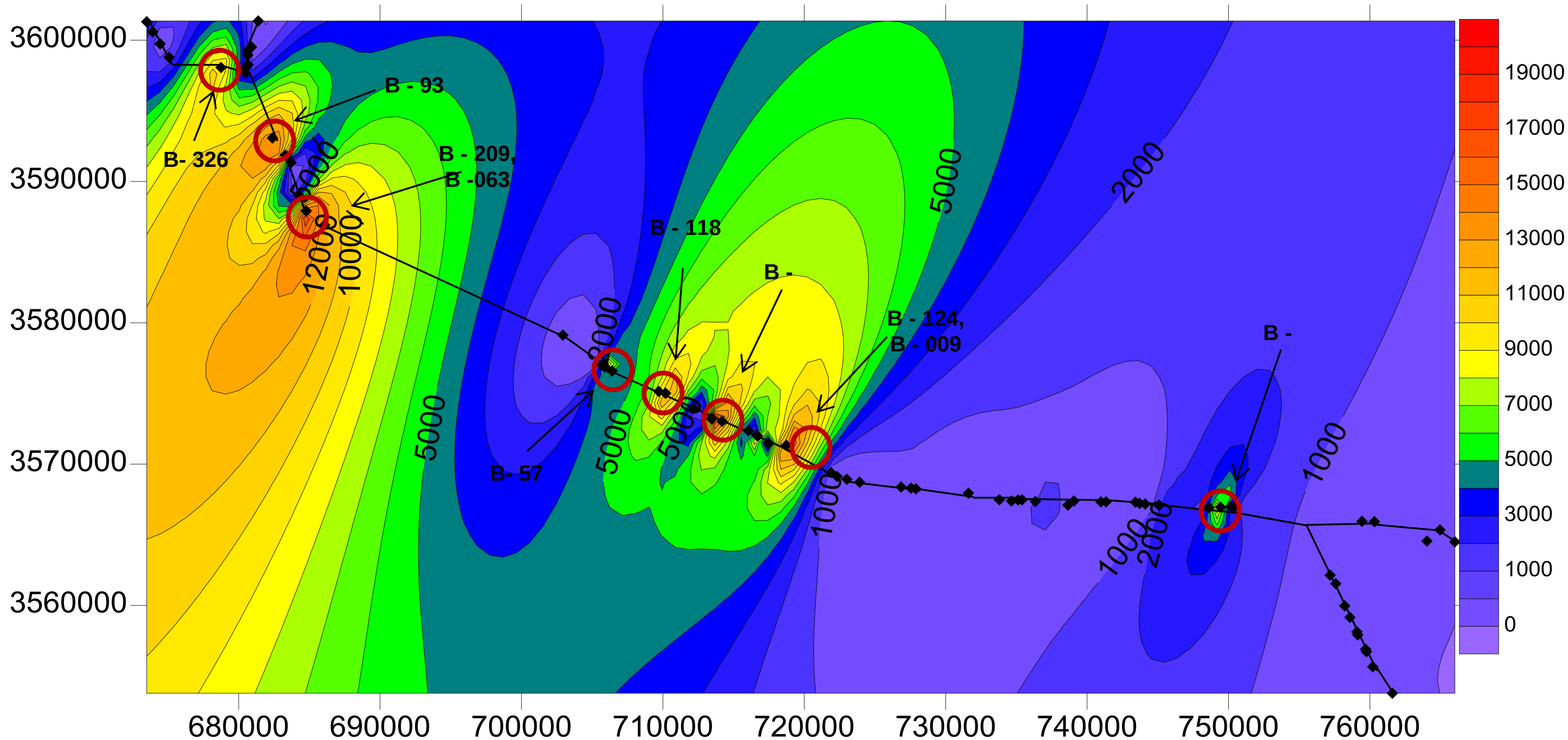


Figure 4.17 All Formations Contour map with Approximate Pipeline Alignment and Highlighting High Sulfate Borings

(Depth – 5ft)

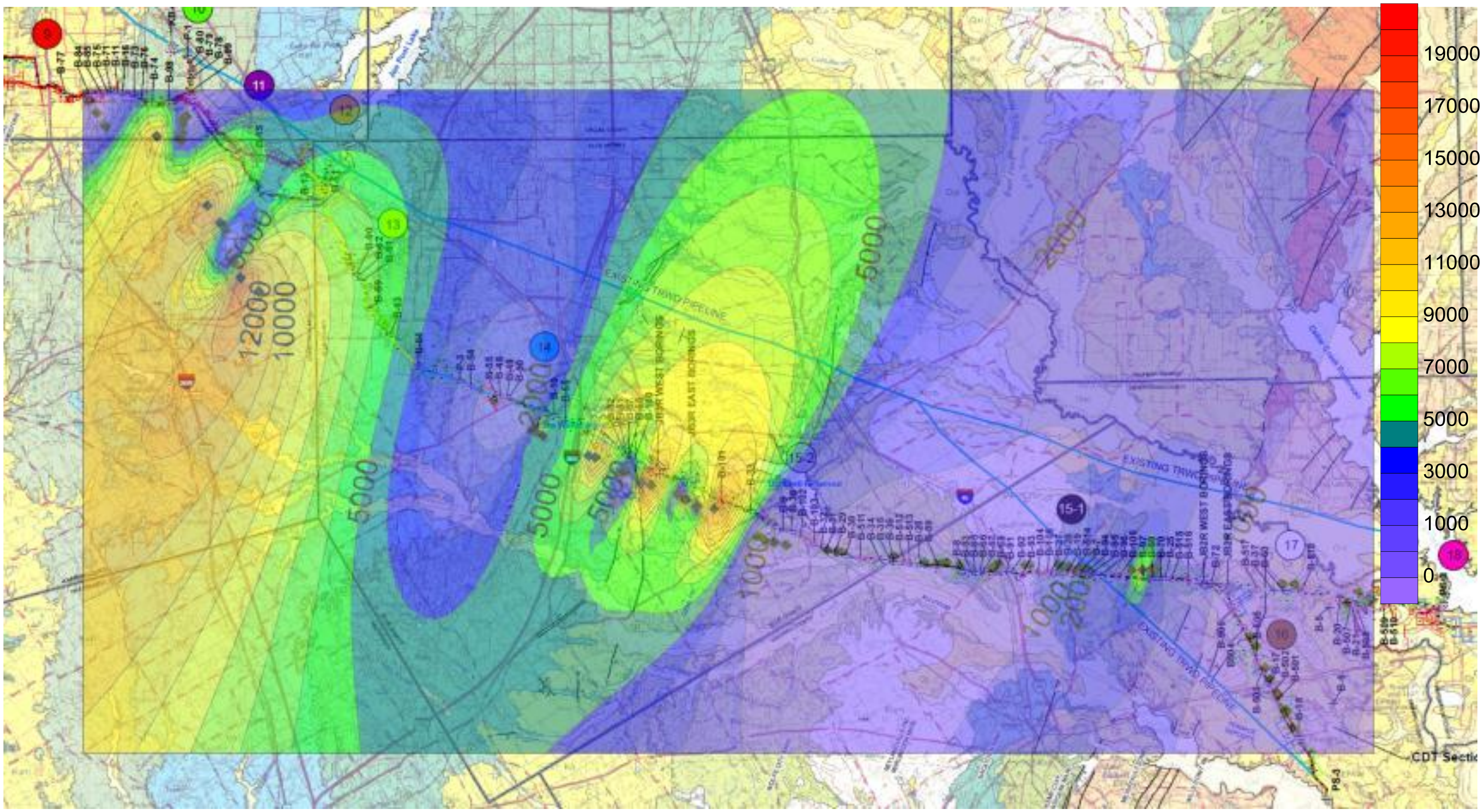


Figure 4.18 All Formations Contour Map Overlaid on Geology Map (Depth – 5ft)

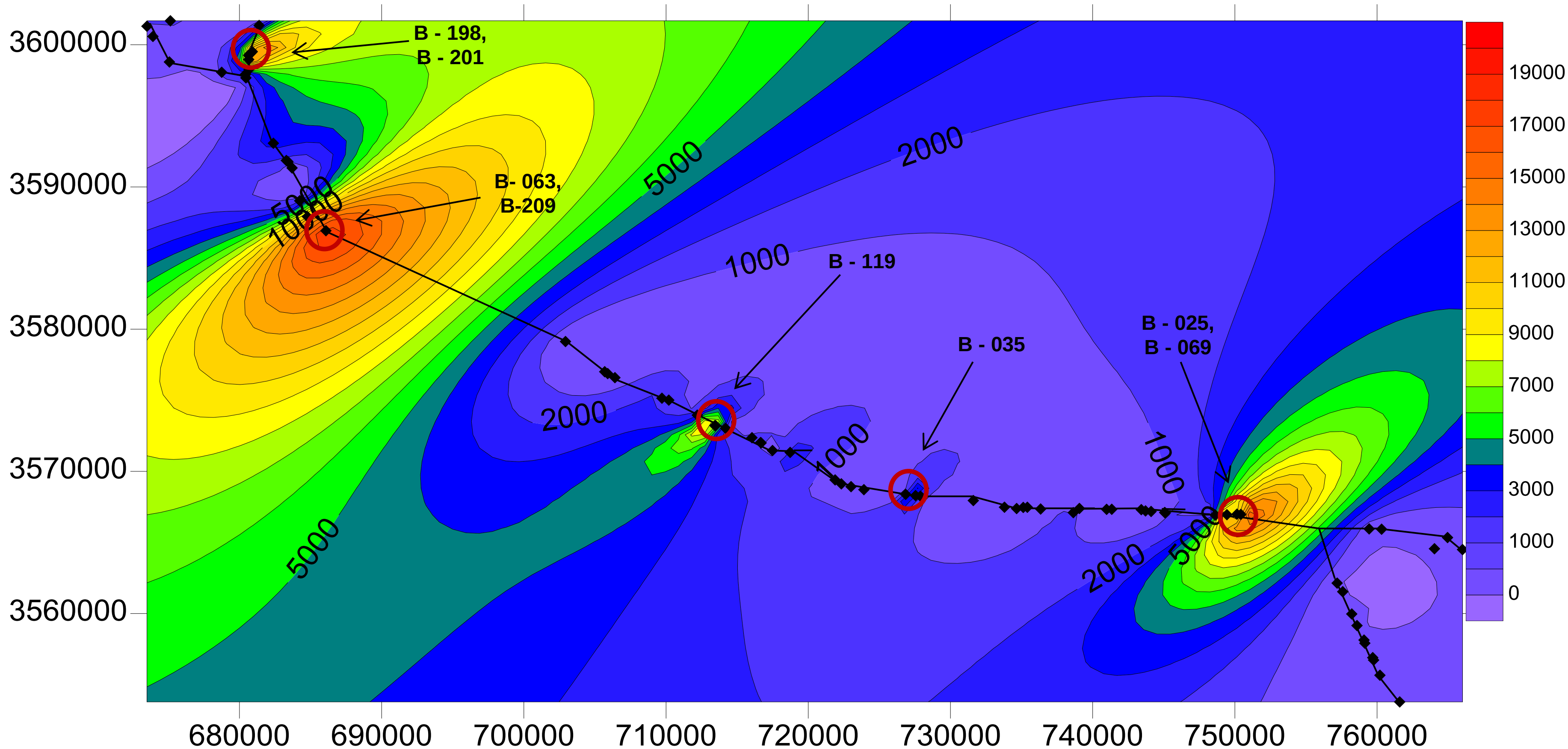


Figure 4.19 All Formations Contour Map with Approximate Pipeline Alignment and Highlighting High Sulfate Borings

(Depth – 10ft)

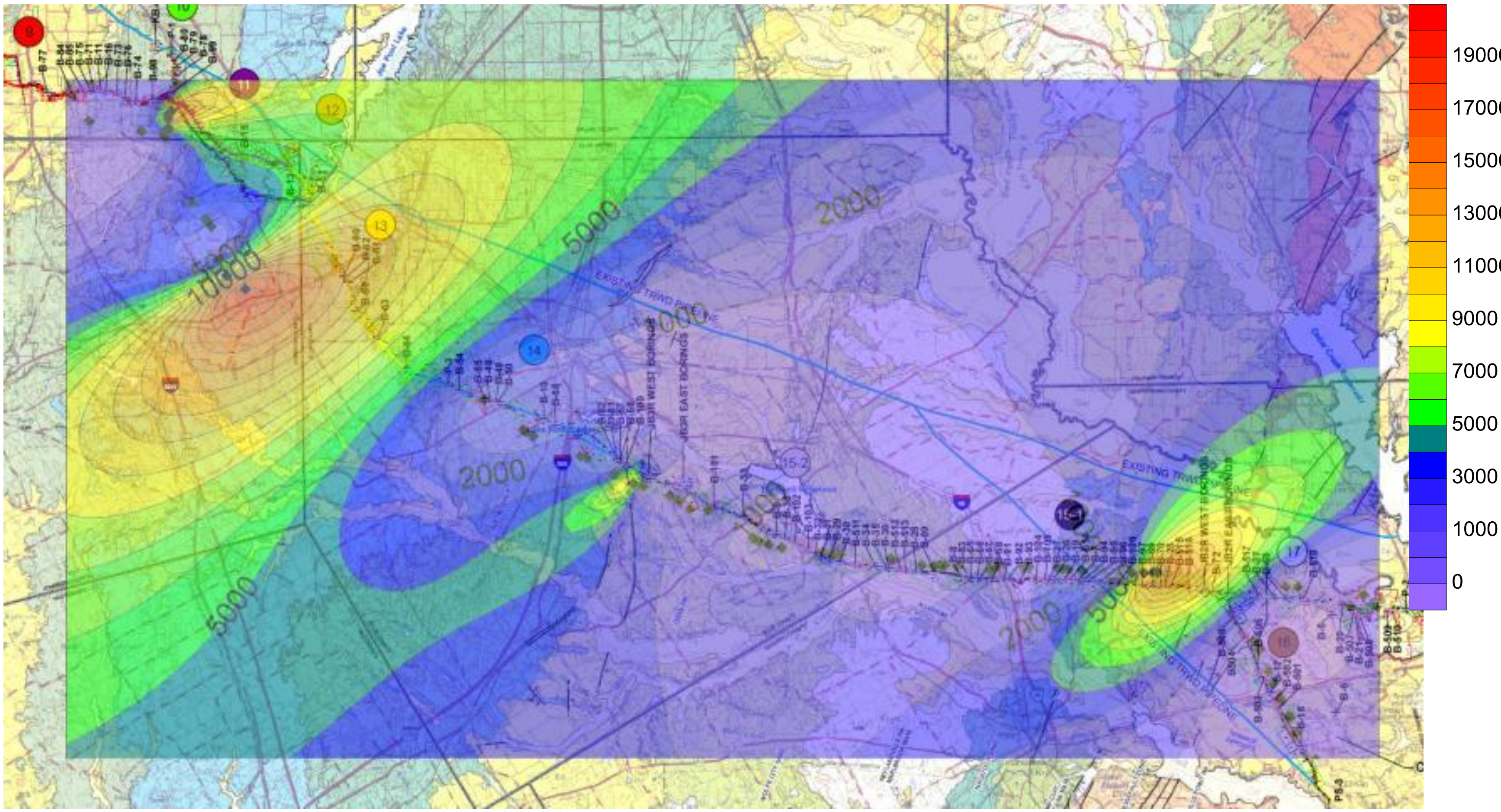


Figure 4.20 All Formations Contour Map Overlaid on Geology Map (Depth – 10ft)

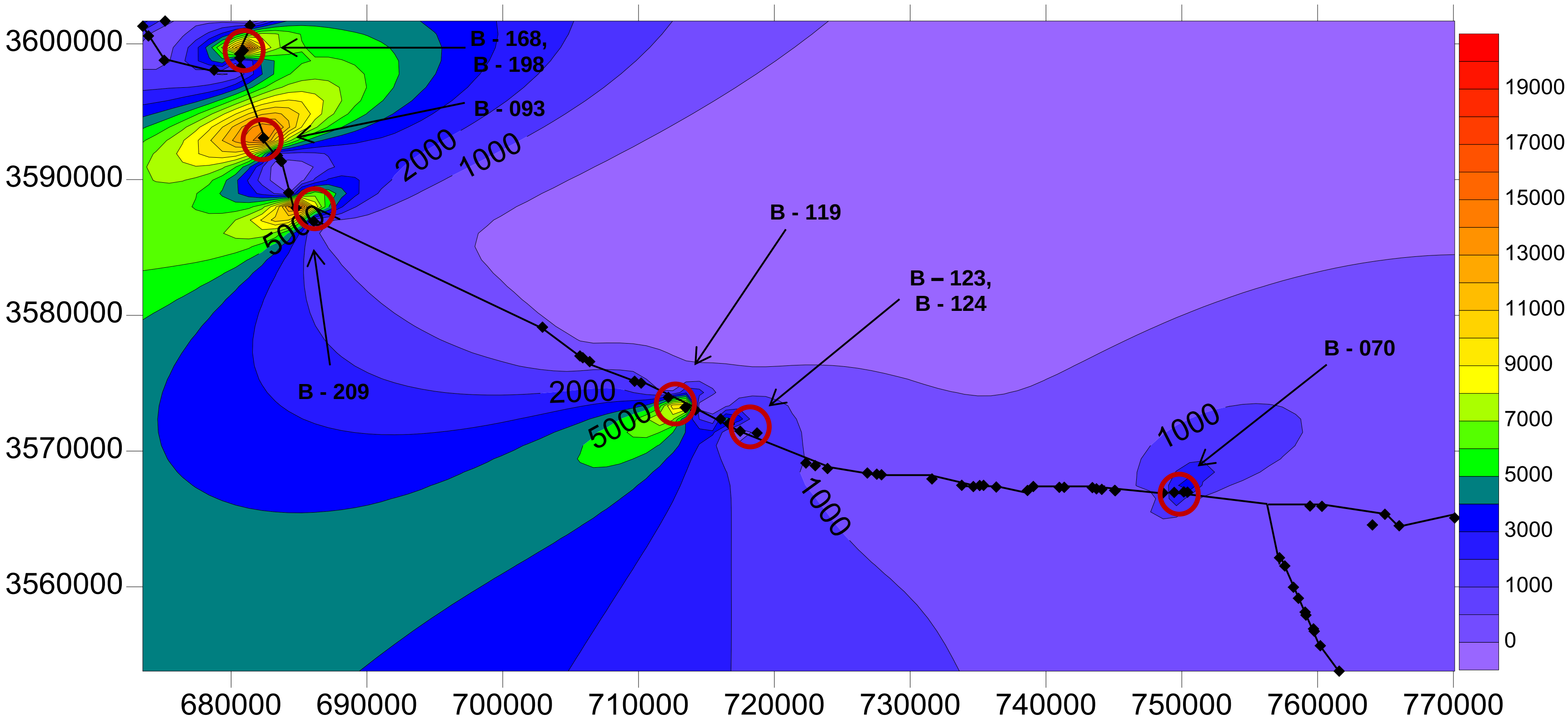


Figure 4.21 All Formations Contour Map with Approximate Pipeline Alignment and Highlighting High Sulfate Borings

(Depth – 15ft)

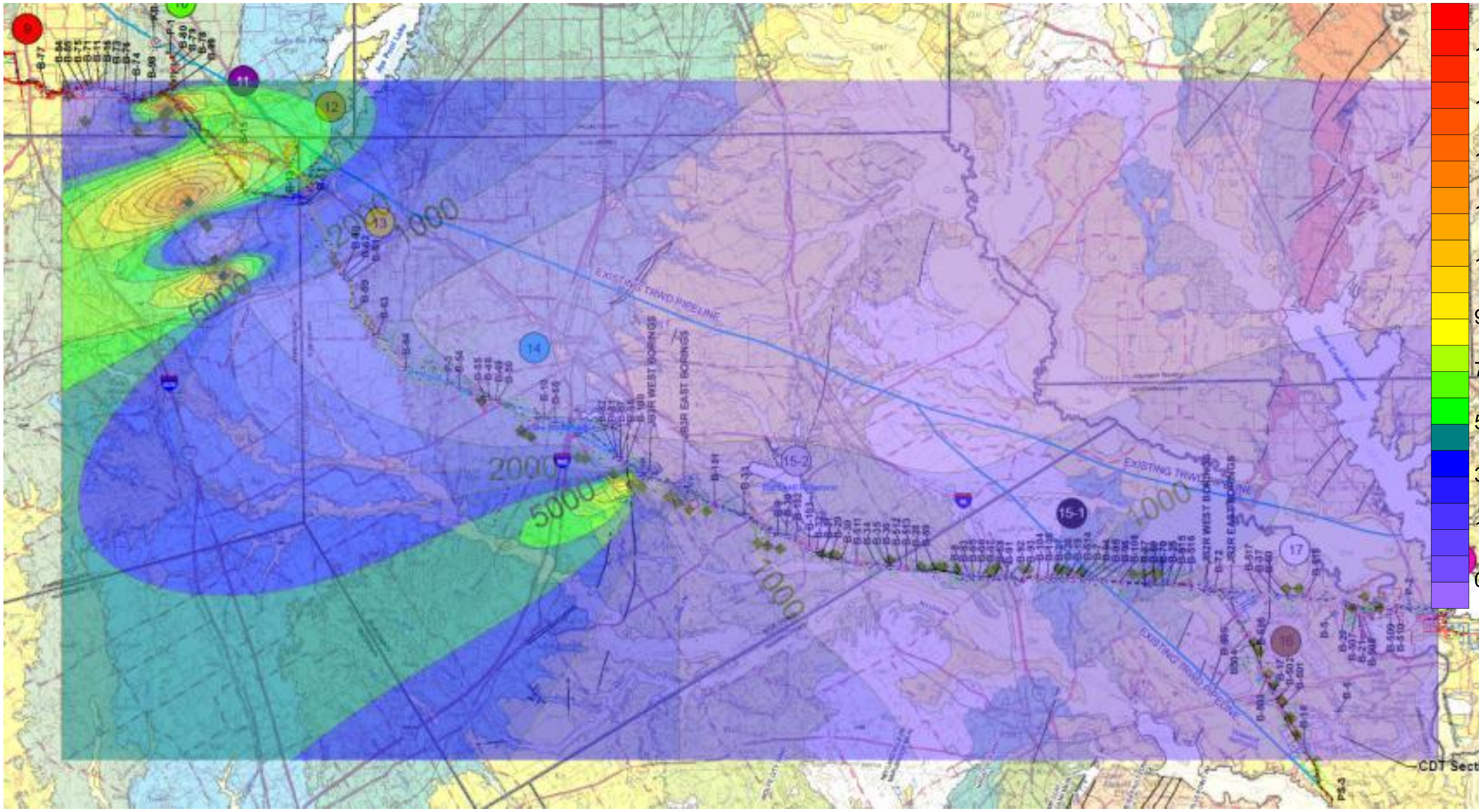


Figure 4.22 All Formations Contour Map Overlaid on Geology Map (Depth – 15ft)

98

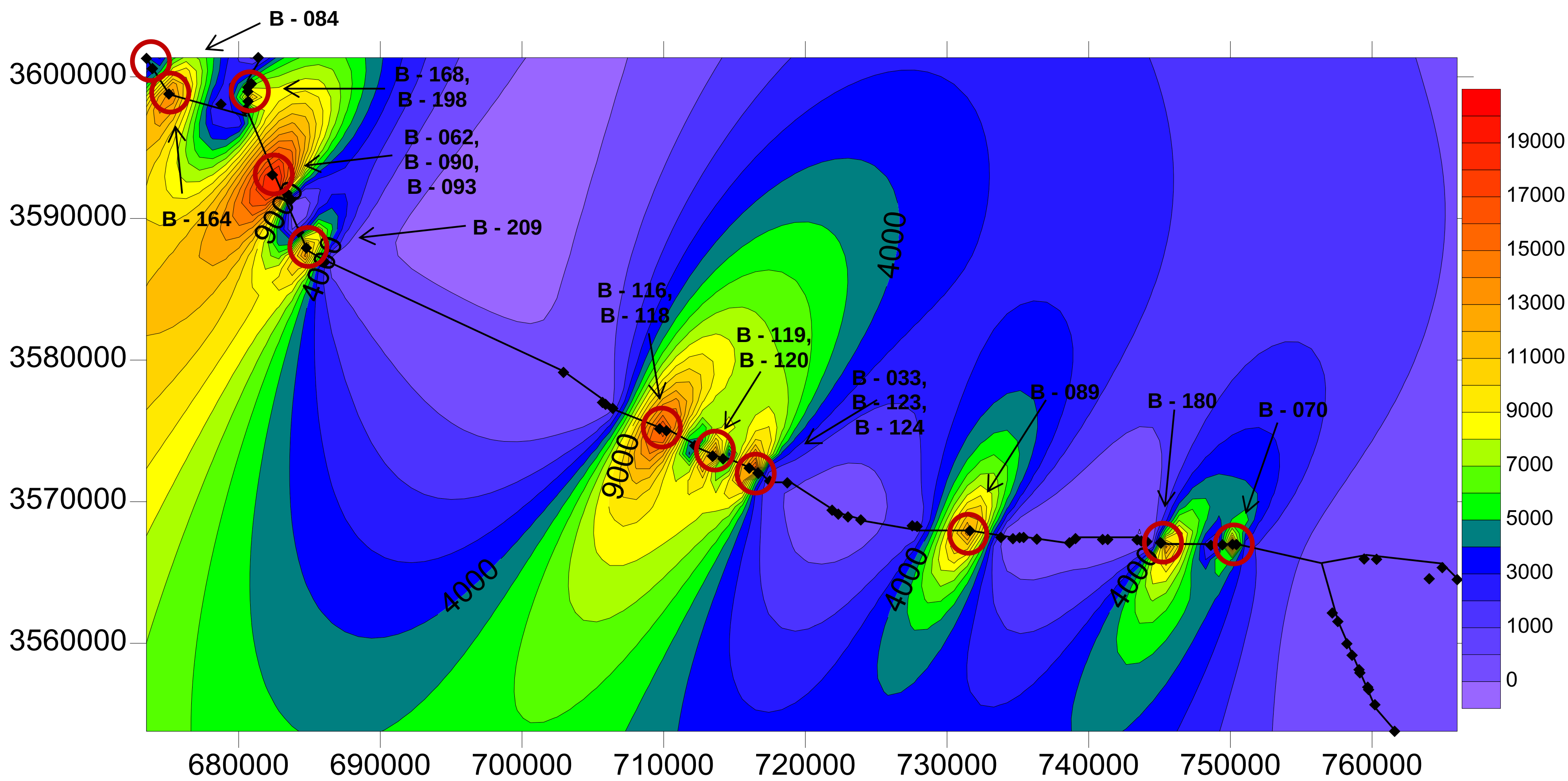


Figure 4.23 All Formations Contour Map with Approximate Pipeline Alignment and Highlighting High Sulfate Borings (Depth – 20ft)

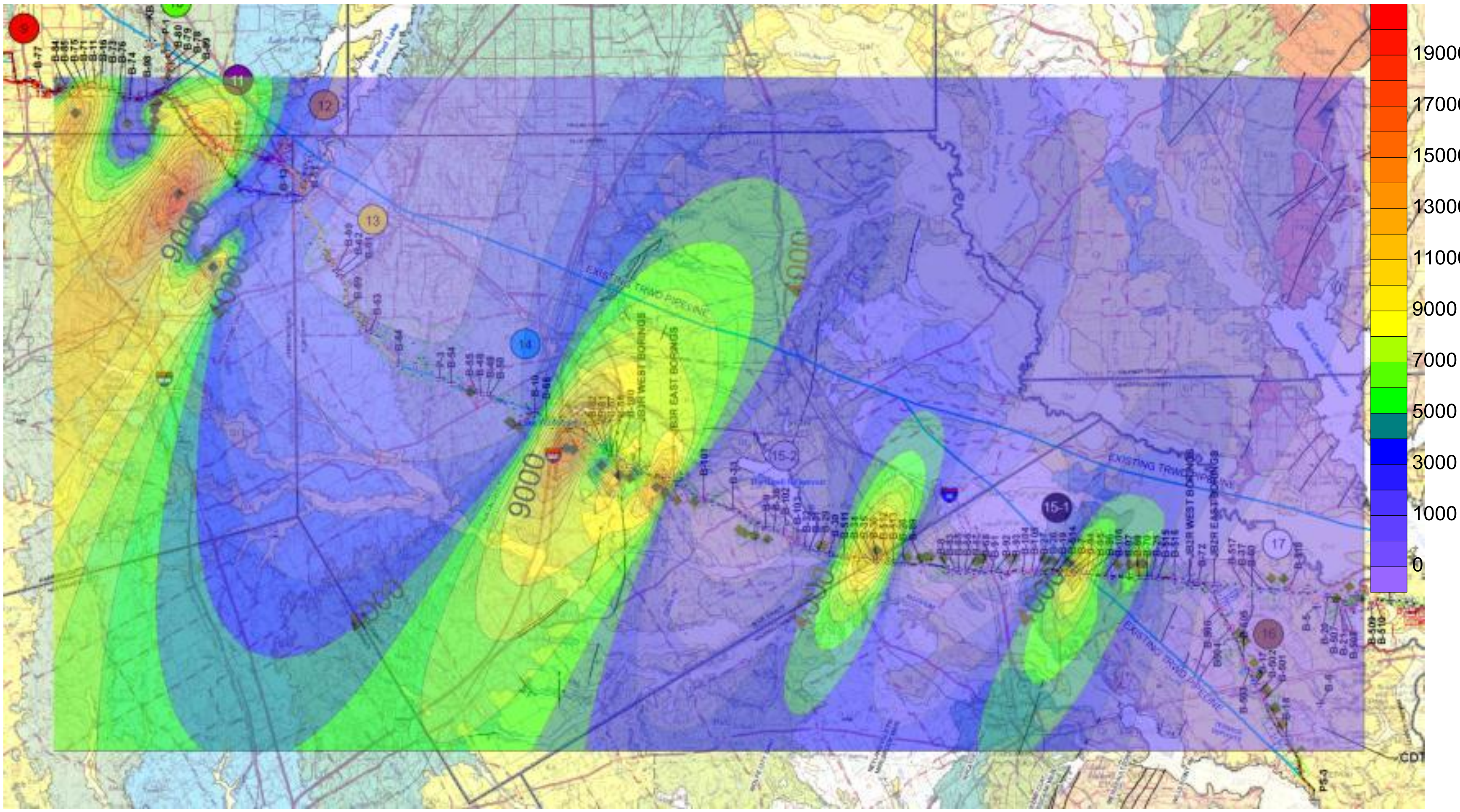


Figure 4.24 All Formations Contour Map Overlaid on Geology Map (Depth – 20ft)

4.7 Geostatistical Validation

Validation of the geostatistical analysis was completed by using a 45° validation line technique comparing actual input data points to predicted data points. The Kriging process is an optimization process, by which the variance between test points and estimations is to be reduced. During this optimization process some of the test point values are adjusted to accommodate the least amount of variance between actual and unknown predicted values. Therefore, an easy approach to determine the validation of Kriging maps is to compare the true input values to the adjusted values created through the optimization and mapping process (Armstrong 1998). If the Kriging process was valid then the true values of the input data versus the adjusted input values will lie close to a 45° validation line, indicating that the true input values are similar to the Kriged output values. No model is perfect, as such, no geostatistical mapping will lie perfectly on the 45° line; however the ultimate goal is to observe little variation from the input points to the optimized and adjusted points. The generated maps were used to identify the borings and their corresponding adjusted values.

The approach to determine the error associated with the Kriging for this project focused on the variance generated by the variogram used in conjunction with the Kriging technique. The maximum variance generated by the variogram was compared for all individual formations. The formations with the lowest and highest variance are compared with the idea in mind that all variance ranges within the highest and lowest will also be valid. Upon completion of the highest and lowest the overall maps at all depths were evaluated using the 45° validation line technique. This was done because a variation from individual maps to the overall maps was observed in some test areas. The maximum cumulative variance associated with all formation's variograms is presented in Table 4.1. For consistency with the geostatistical formation mapping combinations presented in the previous sections, the validation process was carried out in the same manner by analyzing the variance generated by the formations mapped by individually, Eagle Ford and Ozan, as well as the formations mapped in combination, Wolfe City/Neylandville and Kemp Clay/Wills Point. The formations determined to have the lowest and highest maximum cumulative variances were the Wolfe City - Neylandville and Eagle Ford, respectively. Following Table 4.1, all depth intervals for Wills Point and Eagle Ford are presented using the 45° validation line (Figures 4.25 – 4.26). Then, the entire collective formation maps are inspected at all depth intervals using the 45° trend line technique (Figures 4.27). The actual input and adjusted input values are provided next to the figures. Following the validation figures a brief evaluation of the validation results is given. One important note to observe from the validation plots is that points that lie above the validation line indicate that the estimated value was lower than the true value and conversely points that lie below indicate the estimated value is higher than the actual value. In some cases higher estimation are not desired. Such applications would pertain to mineral quality prediction. In this case, estimation higher values would negatively impact the gross profit of the mineral mining operation. In other cases over estimating values may prove helpful or conservative. Such applications would be soluble sulfate prediction and mapping. In this case, the over estimation of sulfate values would be safe and conservative. The validation line technique can indicate exactly how conservative the over estimation may be. If values are over predicted, the analysis becomes too conservative and is counterproductive.

Table 4.1 Variance Comparison from Variograms at Tested Depth Intervals

Formation(s) / Depth	Eagle Ford	Ozan	Wolfe/ Neylandville	Kemp/ Wills
5 ft.	1.00E+08	1.40E+08	6.50E+05	4.50E+07
10 ft.	1.25E+08	5.00E+07	2.50E+07	1.10E+08
15 ft.	1.20E+08	7.50E+07	3.00E+05	1.25E+07
20 ft.	1.80E+08	1.25E+08	1.00E+08	6.50E+07
Cumulative Variance	5.25E+08	3.90E+08	1.26E+08	2.33E+08

4.7.1 Eagle Ford Formation Validation

The Eagle Ford formation exhibited the highest amount of variance as computed by the semivariogram (see Table 4.1). Therefore, the Eagle Ford should provide the largest variation from the 45° validation line. Figure 4.25.a shows very little variation between input and output values indicating good prediction and reliability in mapping. Figure 4.25.b shows some variation between input and output values primarily at very low and very high sulfate levels. One would expect the largest variations in these locations because the optimization process affects values that generate the highest variance. In other words, very low values will be augmented and very high values are reduced. Figure 4.25.c shows very good correlation at low sulfate values; however at high sulfate values the estimated values are often lower than the true values. This is due to the higher frequency of lower sulfate values that cause the higher sulfate values to be reduced. Figure 4.25.d shows the highest variation from input to output values. This is due to the fact the 20ft. depth in the Eagle Ford had the highest variance of any formation at any depth (see Table 4.1). Overall, the most variable formation, Eagle Ford, indicated good results using the 45° validation line technique.

4.7.2 Wolfe City and Neylandville Combination

The Wolfe City and Neylandville formation combination yielded the lowest amount of variance as computed by the semivariogram (see Table 4.1). Therefore, this map combination should provide the smallest variation from the 45° validation line. Figure 4.26.a, the 5 ft. validation plot, shows a good correlation of input and output values. There are some outlying points at the lower and higher end of the sulfate values. As mentioned in the previous Eagle Ford formation discussion, this is due to the optimization procedure carried out in the Kriging process. The lowest and highest values, the ones creating the largest variance, are optimized and increased or decreased to reduce variance between points. The 10 ft. validation plot, Figure 4.26.b also shows a very good correlation of input and output values and only has three points that exhibit a significant amount of error. In the case of the highest value, this error is quite acceptable considering sulfate concentrations above 800 ppm are known to be very problematic. In the case of the lower values, the Kriging output estimated higher values than the true values. For this type of analysis higher values are more conservative and therefore acceptable. Figure 2.26.c, the 15 ft. validation, shows a very good correlation as well with the same trend of over predicted lower values and under predicting higher values. The 20ft. validation map, Figure 2.26.d shows the best correlation with the validation line indicating an almost exact input and output. This is due to the fact that most of the input values were in the same range, from 0 to 200 ppm, and there was only one major variant of 15,000 ppm. This major variant came from the Wolfe City portion and was not directly close spatially to other test points, so its variation was essentially nullified over distance.

4.7.3 All Formations

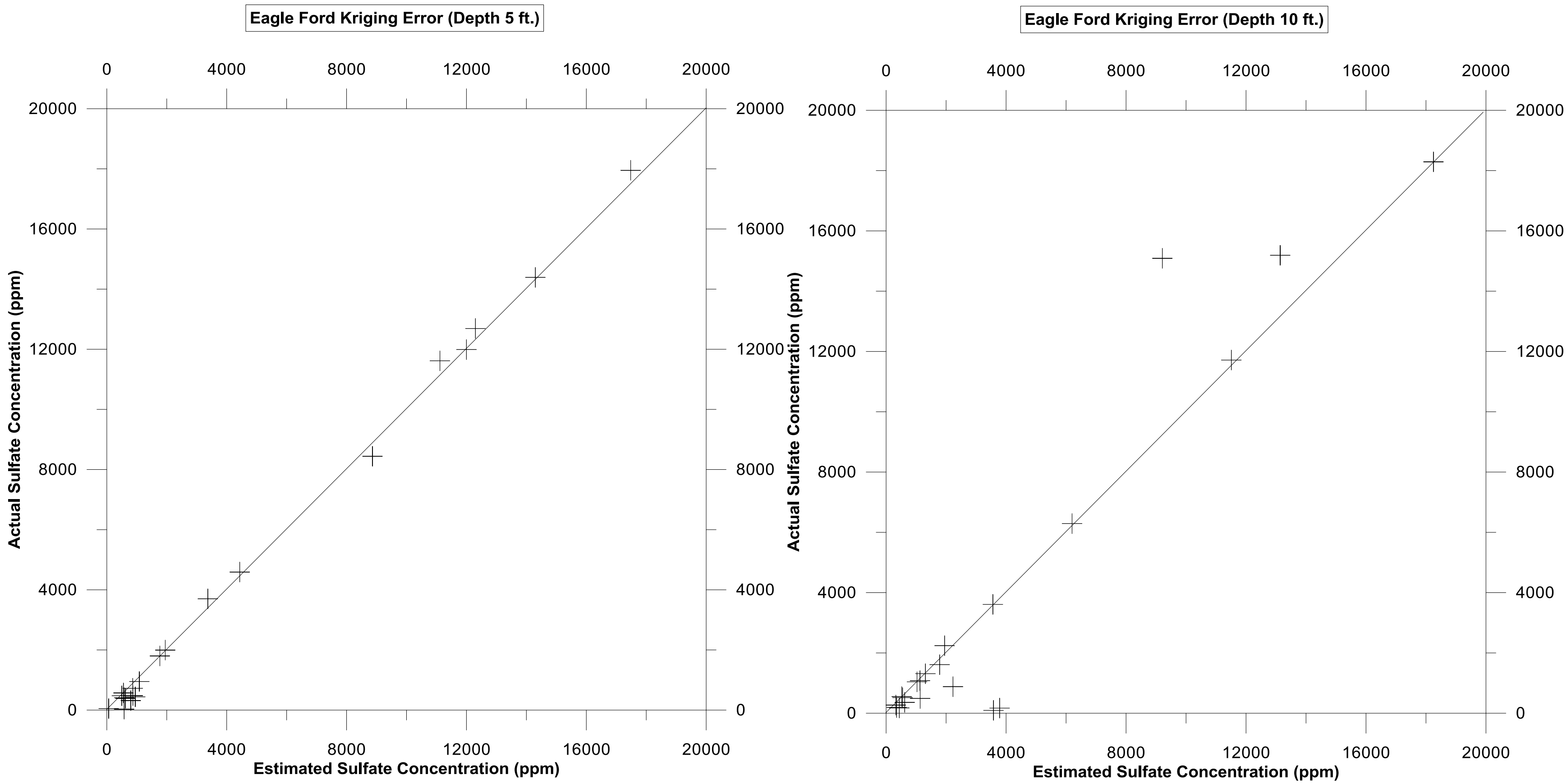
The All formations maps exhibited the highest amount of deviation from the validation line. In most cases, the addition of data points strengthens a geostatistical analysis, however in this case the addition of data points weakened the analysis. This is due to the extreme variation of the added input values. Increasing the data set is helpful if the values do not exhibit large variation. By adding large variation numbers to the data set, one creates a larger overall variance and thus weakens the accuracy of the analysis. This is the case with all formations maps in this research. The overall maps are still quite useful and exhibit good visualization qualities, however the predictability associated with these maps is not as strong as the individual maps. Two reasons exist to explain the large variation in input values. The first deals with the random nature of sulfate values, which is due to the random manifestation and forming of sulfate minerals through natural processes. The second deals with the numerical difference in sulfate values due to the ppm scale. For instance, if one value of sulfate is 1000 ppm and its nearest neighbor, the most influential value in the Kriging process, is 18,000, then the resulting variance of those two numbers as computed by the semivariogram is over 72,000,000 ppm. The exact calculation is provided below.

$$\gamma^{*}(\mathrm{h}) = \frac{\sum[\mathrm{z}(\mathrm{x}) - \mathrm{z}(\mathrm{x}+\mathrm{h})]^2}{2\mathrm{n}}$$

$$\gamma^{*}(\mathrm{h}) = \frac{\sum[1000 - 18000]^2}{2(2)}$$

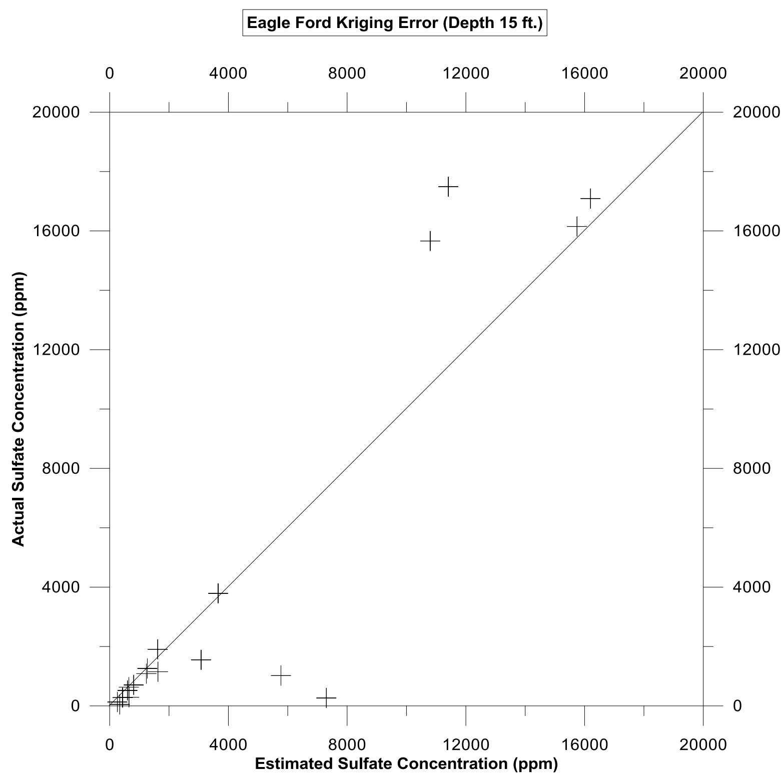
$$\gamma^{*}(\mathrm{h}) = \frac{[-17000]^2}{4} = 7.225\ \mathrm{E}7$$

This is an incredible variance. This number can be reduced by the addition of data points that are closer together in value, thus explaining why the maximum variances presented in Table 4.1 are not much higher than this example calculation. Figures 4.27.a through 4.27.d all exhibit the same type of deviation from the validation line, which is the same as with the Eagle Ford and combined Wolfe City/Neylandville. The deviation from the validation line increases as the values of sulfate are at the very low and very high end. Additionally and further reiterating the increase in optimization error through Kriging, data points in the middle range of analysis also seem to be both over and under predicted, with an equal amount of incorrect predictions on both sides of the validation line.

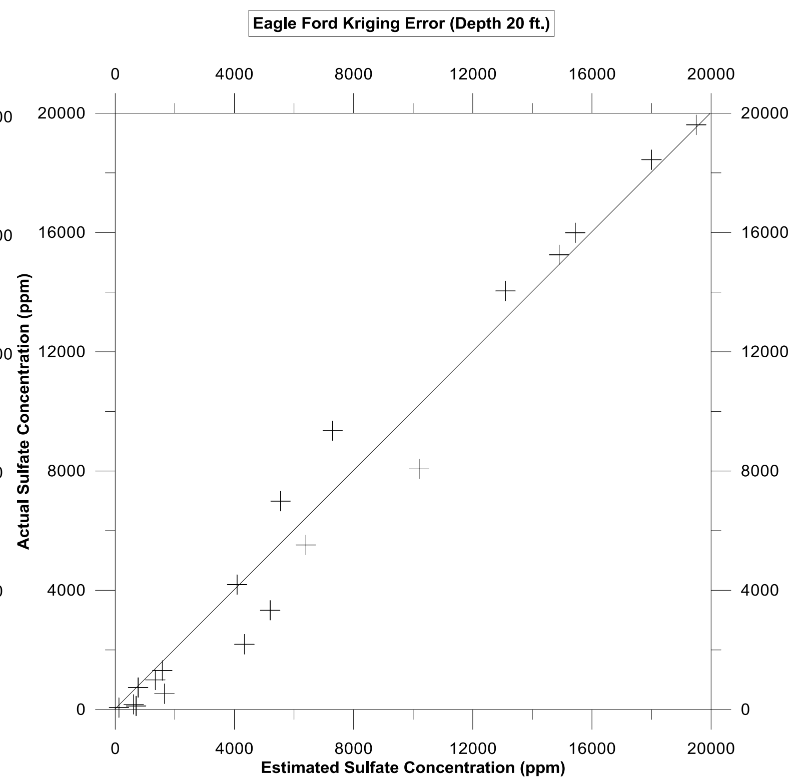


a) b)

Figure 4.25 a&b Eagle Ford Validation Depth 5ft. & 10ft.

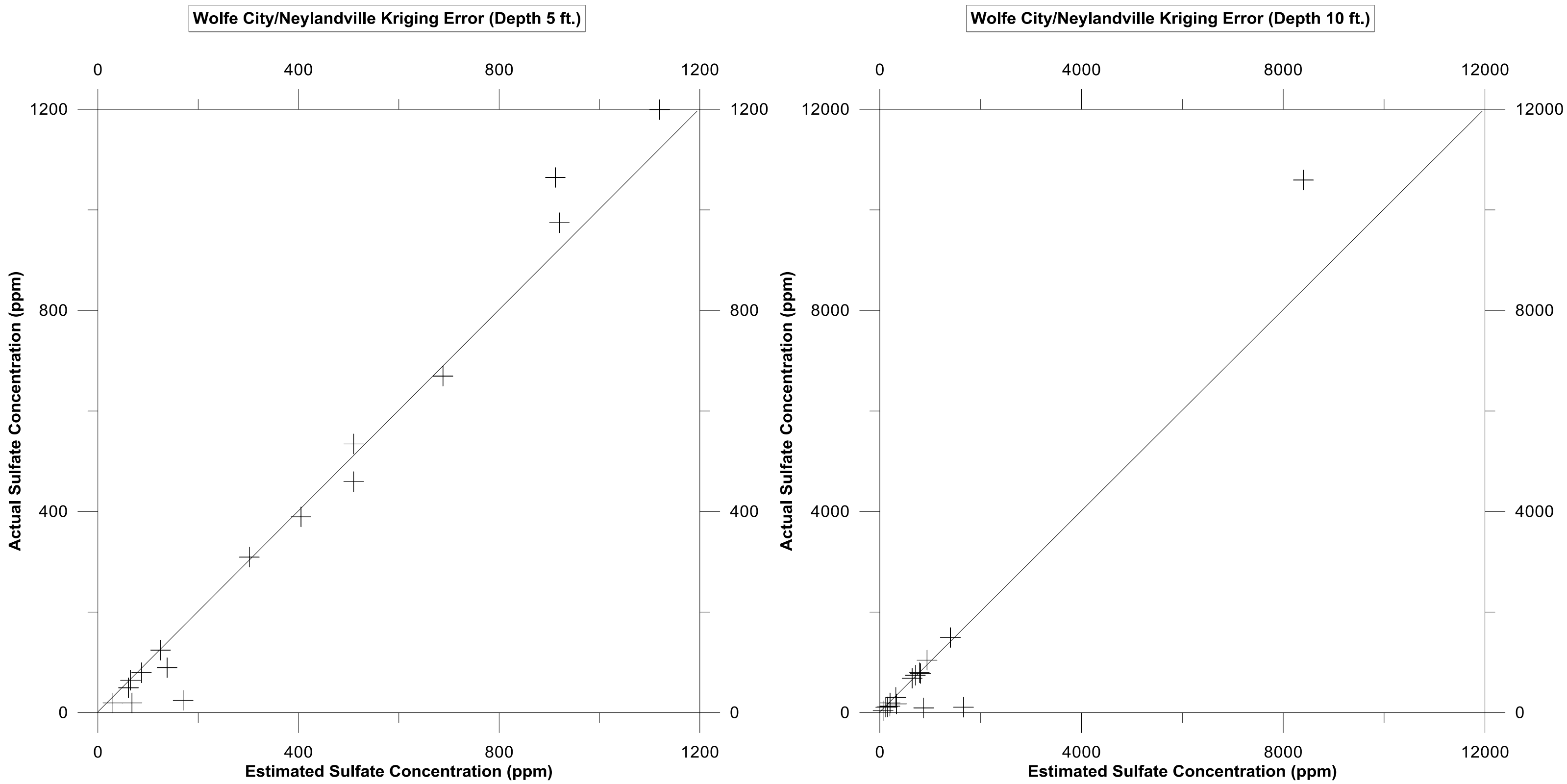


c)



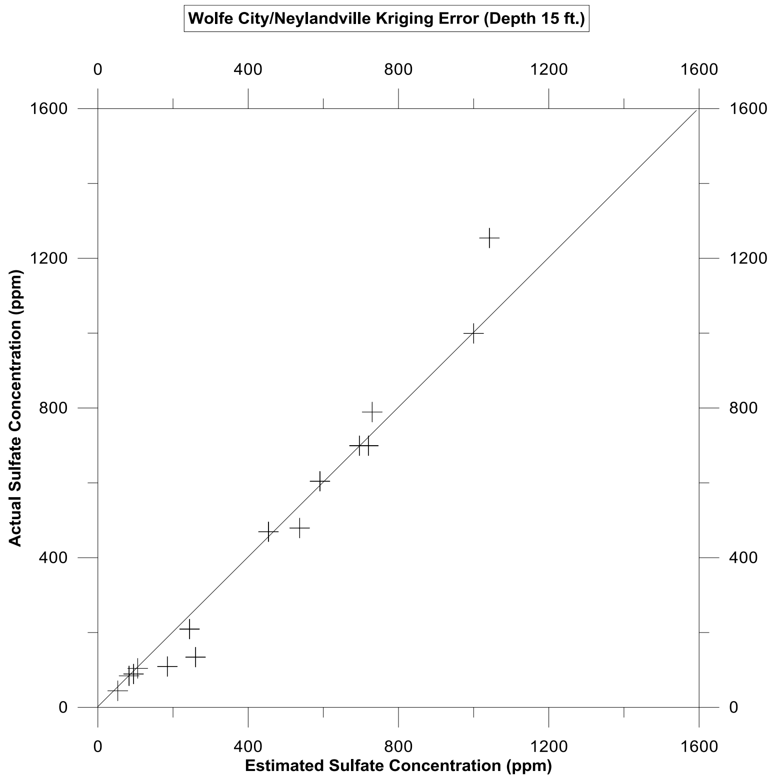
d)

Figure 4.25 c&d Eagle Ford Validation Depth 15ft. & 20ft.

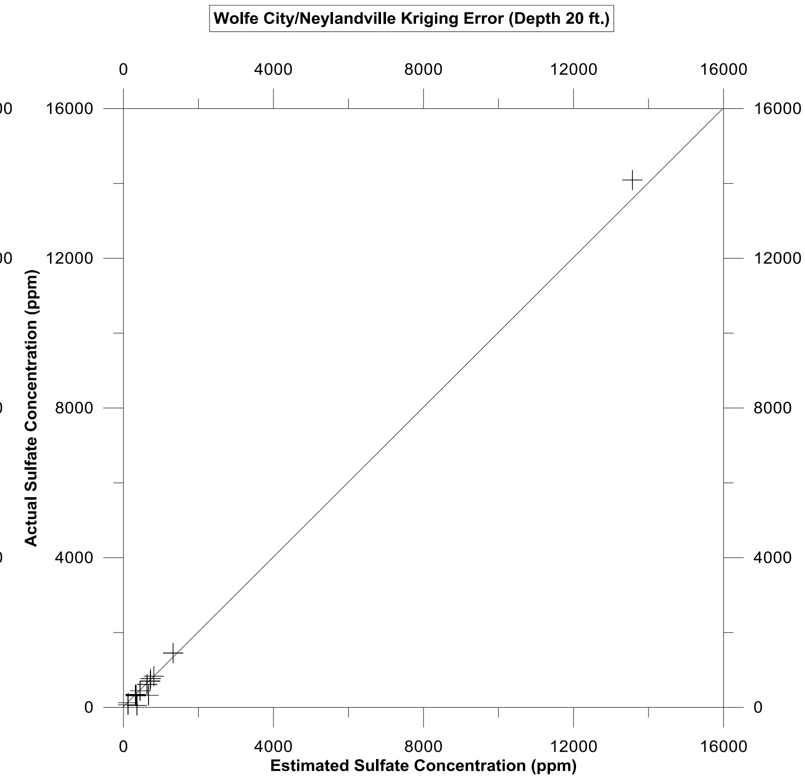


a) b)

Figure 4.26 a&b Wolfe City/Neylandville Validation Depth 5ft. & 10ft.

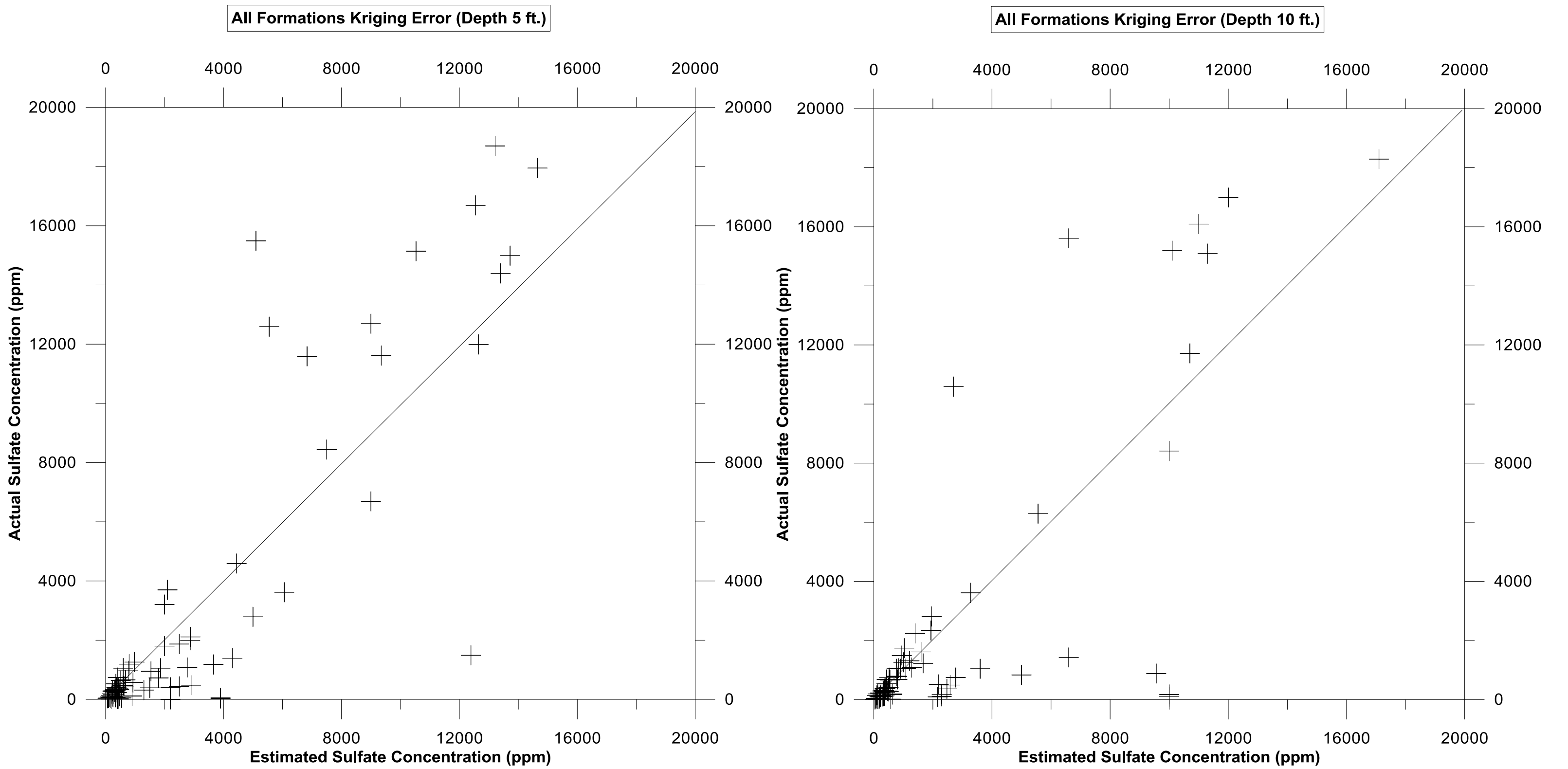


c)



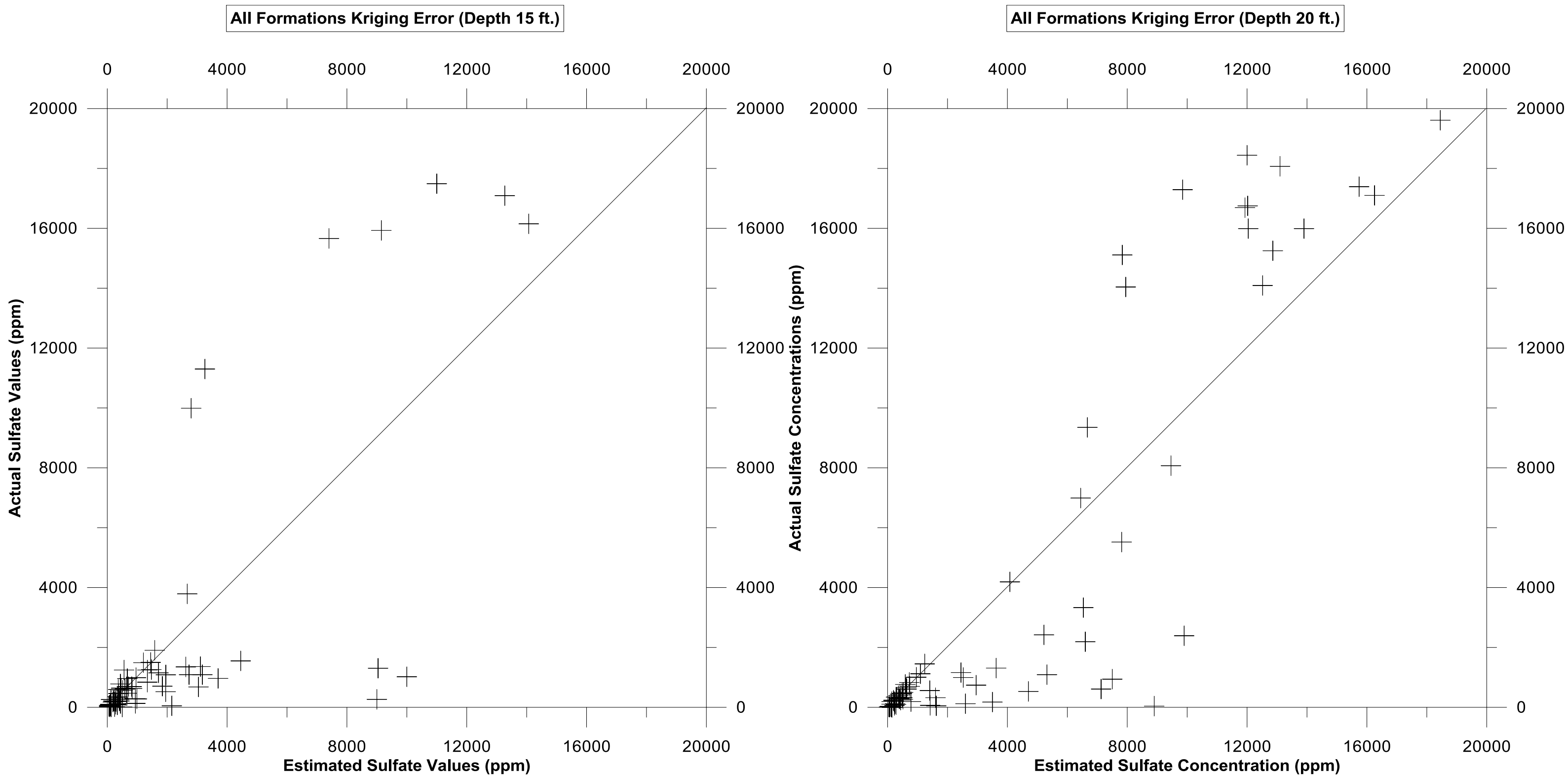
d)

Figure 4.26 c&d Wolfe City/Neylandville Validation Depth 15ft. & 20ft.



a) b)

Figure 4.27 a&b All Formations Validation Depth 5ft. and 10ft.



c)

d)

Figure 4.27 c&d All Formations Validation Depth 15ft. and 20ft.

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions of Testing and Mapping

Based on the data obtained by testing select samples from 6 different soil formations along the IPL pipeline alignment the following conclusions can be made:

1. Over 300 samples were tested using the Modified UTA Method of Soluble Sulfate Determination in Soil. The results of the tests (Tables 3.2 – 3.7) indicate that sulfates in soil are inherently random and often have large variation over spatially related distances as also indicated by the deterministic bar plots (Figures 3.6 – 3.9).
2. The test results also indicate that the Neylandville and Wills Point formations have sulfate values below the typical problematic thresholds and may not have issues if treated with calcium based stabilizers.
3. The test data also clearly indicates that the Eagle Ford and Ozan formations are very problematic at all depths (5 ft., 10 ft., 15 ft., and 20 ft.,) and calcium based stabilization could be problematic leading to the formation of ettringite. However, further testing is recommended to narrow down problematic zones and clear areas that are not an issue along the pipeline.
4. Additionally, test data indicates the Kemp Clay and Wolfe City Sand formations show some zones that appear to be problematic and are considered moderately problematic. Additional testing is recommended in these zones to further assess the problem in these zones.
5. The deterministic data was then used for geostatistical analyses in aims to visualize and predict sulfate values. The analyses were conducted using Linear Geostatistics, specifically Ordinary Point Kriging. The results of the analyses indicate that the geostatistical analysis of sulfate values is quite difficult due to the random nature of sulfate values over spatial regions.

6. Validation was conducted on the various contour maps generated by the geostatistical analysis. The validation indicated that individual formation mapping was more accurate than the overall combination mapping of all formations.

5.2 Recommendations

5.2.1 Eagle Ford Formation

It is recommended that further testing be completed on the Eagle Ford Formation at all depths due to the high intensity and distribution of sulfate concentrations. Specifically, further testing should focus on the zones listed in the previous sections indicated by the contour maps and test results, but are not limited to those zones as sulfate concentrations are inherently random. Further testing will not only aid in the proper stabilization of sulfate soils along the pipeline alignment, but will also provide further validation of the contour mapping.

5.2.2 Ozan Formation

It is recommended that further testing be completed on the Ozan Formation at all depths, especially the 5ft. and 20ft. zones as they appear to be most problematic. Specifically, testing should focus on the zones listed in the previous sections indicated by the contour maps and test results, but are not limited to those zones as sulfate concentrations are inherently random. Further testing will not only aid in the proper stabilization of sulfate soils along the pipeline alignment, but will also provide further validation of the contour mapping.

5.2.3 Kemp Clay Formation

It is recommended that further testing be completed on the Kemp Clay Formation in the vicinity of: B-69 at 5ft. and 10ft. depth, B-70 at all depths, B-25 at 10ft. depth, and B-180 at 20ft. depth. However, sulfate concentrations are inherently random and further testing may be necessary in zones other than previously stated or as indicated by the contour maps and test data.

5.2.4 Wolfe City Formation

It is recommended that further testing be completed on the Wolfe City Formation in the vicinity of: B-35 at a depth of 10ft. and B-89 at 20ft. depth. It appears that other locations and depths have acceptable sulfate values, however sulfate concentrations are inherently random and further testing may be necessary in zones other than previously stated or as indicated by the contour maps and test data.

5.3 Geostatistical Validation

Based on the validation process on the geostatistical maps utilizing Kriging error evaluation, it can be seen that good prediction correlation exists with the mapped data. The approach of examining the formation with the least amount of variability and the formation with largest amount of variability shows that all formations in between should have good correlation from predicted values to true values. As with all geostatistical analyses the prediction accuracy is dependent on data variability, test spacing, and test borehole orientation. Based on the method of Validation, it can be seen that when data points lie below the 45° line, the predicted value is higher than the actual value. Conversely, if the data point lies above the 45 line, the actual value is higher than the predicted value. In most cases, we see that the predicted values are higher than actual values at lower sulfate concentrations and actual values are higher than predicted values at high sulfate concentrations. The implications of such phenomena are addressed in the following section which breaks down the analysis into each validation set

5.3.1 Eagle Ford Validation

The Eagle Ford formation was the most variable formation from a statistical stand point. Therefore, the fact that the validation points were so close to the 45° validation line indicates that the Kriging approach does have validity. There are a few outlying points on the validation maps; however these outliers appear to be in an acceptable range.

5.3.2 Wolfe City/Neylandville

The Wolfe City/Neylandville combination map also had good correlation to the 45° validation line. As with the Eagle Ford formation there were some outlying points, but relatively few. The explanation for the outlying points is simply that this particular data set also includes Wolfe City, which shows much larger variation than the Neylandville alone. Therefore, all outlying points are due to the Wolfe City variation.

5.3.3 All Formations Validation

Upon analyzing the results from the validation on all formations collectively, it can be seen that there is quite a bit of variation from the 45° validation line. In most geostatistical applications the addition of data points is a positive. However, it can be seen that when the addition of data points are quite variable, the accuracy of the analysis is compromised. The addition of variation from formations such as Ozan, Wolfe City, and Neylandville caused the Kriging optimization process to lose accuracy. In this case it appears that the use of all formations collectively is not as accurate or as appropriate and the maps should be studied on an individual formation basis.

5.4 Implications for Further Study

The implications from a study such as this are far reaching. The identification of soluble sulfates in enough abundance to create adverse reactions post soil stabilization is present in many of the test formations. Previous studies conducted by UTA indicate that much of the bedding and haunch material utilized for pipeline construction will be reused and chemically stabilized. If such bedding and haunch material is utilized from certain established problematic formations, such as the Eagle Ford and Ozan, then detrimental sulfate will be observed. If certain bedding and haunch material is stabilized and reused in the Kemp and Wolfe City formations in certain areas, then detrimental sulfate heave will occur. The sulfate heave will ultimately reduce the performance and could fail the pipeline in certain areas. Further sulfate testing should be conducted in situ as the pipeline is being trenched and the bedding and haunch stabilized in order to prevent sulfate heave due to isolated pockets of sulfates. It has also been suggested by UTA that CLSM mixes using soils along the pipeline could save vast amounts of time and money in regards to pipeline construction. Therefore, CLSM mixes utilizing sulfate soil need to be studied rigorously for swelling, shrinkage, Ettringite/Thaumasite formation, and durability effects.

In regards to soluble sulfate reactions, further study must be conducted on the contribution of free alumina to the Ettringite reaction. A link between the alumina silica ratios has been observed in recent studies as being impactful in the reaction; however no studies have been conclusive in determining the extent of free alumina's role in the reaction. Considering the pipe line may be constructed with steel and precast concrete pipe, further study must be carried out to determine if sulfate heave will cause distress issues to these materials.

Lastly, more studies should be conducted in the geotechnical arena using geostatistics as a tool of analysis. Sulfate values show very large variations, making geostatistical analysis more difficult and potentially less accurate. However other aspects of civil engineering properties, such as friction angle, compaction density, permeability, etc. generally exhibit less numerical variation over spatial regions. Therefore analyses using these variables might prove to be easier and more accurate. Additionally, many other geotechnical parameters have correlation with one another. For example density and permeability are related and multivariate geostatistical analyses can be used to predict and map both characteristics at the same time. Overall, there are many advantages of geostatistical tools in geotechnical engineering and the use of such tools in geotechnical investigations is warranted.

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BIOGRAPHICAL INFORMATION

Justin David Thomey was born in Rogers, Arkansas and grew up in both Louisiana and Texas. Thomey graduated with a Bachelor of Science degree in Civil Engineering, emphasizing Geotechnical Engineering, from The University of Texas at Arlington in May of 2011. He worked on two civil engineering projects as an EIT under the direction of Mark A. Thomey, CEO of Structural Engineering, located in Arab, Alabama. The projects included the design and fabrication a steel stage extension for an Arlington, Texas entertainment venue as well as the sizing design for geotechnical stability of two (2) reinforced concrete cantilever retaining walls for a Fuel City in Lufkin, Texas.

In the fall of 2011 the author enrolled in the Master Program at The University of Texas at Arlington College of Engineering as a Master of Science candidate in Geotechnical Civil Engineering and worked under the guidance of Dr. Anand J. Puppala and Bhaskar Chittori. During the course of his study the author worked as a graduate research assistant under Dr. Anand J. Puppala and had a chance to work on various research projects, including: sulfate testing, fully softened shear strength, slope stability using sustainable stabilizers, and sustainability as it pertains to reuse of fill and haunch material for pipelines.