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ANALYSIS OF PRECIPITATES AND WATERS ASSOCIATED WITH AN ALKALINE
LEACHATE, GULF STATE STEEL PROPERTY, GADSDEN, ALABAMA: A
RECONNAISSANCE STUDY

by

CRAIG AARON VANTREES

Under the Direction of Dr. W. Crawford Elliott

ABSTRACT

Calcite stalactites ranging in length from several inches to a foot long are found forming on the southeastern slag pile at the former Gadsden Steel Mill of the Gulf States Steel Corporation. Analyses of samples collected per EISOPQAM guidelines include the following: petrographic, conductivity, pH, XRD, XRF, TDS, and major cations and anions. Preliminary field pH and conductivity measurements indicate that waters near the slag pile have a pH ranging from 11-12 and a conductivity ranging from 1115-6300 $\mu\text{S}/\text{cm}$. Titration data indicate that the maximum pH value is 12.5. These calcite stalactites and stream coatings result from the dissolution of the steelmaking slag by rainfall. These alkaline waters precipitate calcite when they are in contact with atmospheric CO_2 . Improper management of slag products can lead to aesthetically impacted environments and ecosystems. Several studies and this study show that steel slag could be used to sequester atmospheric CO_2 .

INDEX WORDS: Alkaline leachate, Alkaline drainage, Gulf states steel, Slag, High pH, Calcite precipitation , Carbon dioxide sequestering

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by

CRAIG AARON VANTREES

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of

Master of Science

in the College of Arts and Sciences

Georgia State University

2010

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Craig Aaron VanTrees
2010

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LEACHATE, GULF STATE STEEL PROPERTY, GADSDEN, ALABAMA: A
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Office of Graduate Studies
College of Arts and Sciences
Georgia State University
May 2010

DEDICATION

I dedicate this thesis to my loving family and friends. For eight years you have supported me, and I thank you for being there as I worked towards this milestone!

ACKNOWLEDGEMENTS

I acknowledge the following faculty members of the Georgia State University Geosciences department for making the completion of this thesis possible: Dr. W. Crawford Elliott for guidance, support, facilities, field assistance and funding; Dr. Jordan Clayton for field assistance, guidance, and critique of research; Dr. Seth Rose, Dr. Timothy LaTour, and Dr. Daniel Deocampo for guidance and use of lab facilities; Dr. Eirik Krogstad and Dr. Hassan Babaie for support and guidance, and Dr. Scott Harris for petrographic assistance.

I would also like to acknowledge Jordan Garrard for providing invaluable field leadership as well as Gulf State Steel property information, insight and access; Cindy Pena for field assistance and pH meter calibration; Cindy Broda for peer review, Steve Anthony Arnstein for field assistance, titration assistance, and peer review; Beth Lavoie for editing and general geology advice; ActLabs and TestAmerica for providing quantitative field data; and Burnham Petrography for providing thin sections.

I would like to acknowledge the following Bank of America leaders (former and current) for enabling me to realize my dreams by supporting my decision to go back to school while continuing to work full-time: Kate Milburn, Mike E. Miller, Elaine White, Robin Hofferbert, Kim Goldsmith, Greg Farr, Kelly DiCuffa, Katy Rowell, Fiona O'Neal, Jill Hartley, Daniel McCurdy, and Matthew Cobb.

TABLE OF CONTENTS

ACKNOWLEDGEMENTS	v
LIST OF FIGURES	ix
CHAPTER 1—INTRODUCTION	1
SECTION 1.1—OCCURRENCES OF ALKALINE DRAINAGE, LEACHATE, AND PRECIPITATES	5
SECTION 1.2—GULF STATES STEEL BACKGROUND AND STUDY AREA.	6
SECTION 1.3—OBJECTIVES.	8
CHAPTER 2—METHODS	19
SECTION 2.1—PETROGRAPHY AND ROCK SAMPLE PREPARATION	19
SECTION 2.2—X-RAY DIFFRACTION	20
SECTION 2.3—X-RAY FLUORESCENCE	20
SECTION 2.4—FIELD WATER & SEDIMENT COLLECTION	21
SECTION 2.5—PH MEASUREMENTS	23
SECTION 2.6—CONDUCTIVITY	25
CHAPTER 3—RESULTS	33
SECTION 3.1—PETROGRAPHY	33
SECTION 3.2—X-RAY DIFFRACTION	33
SECTION 3.3—X-RAY FLUORESCENCE	33
SECTION 3.4—PH ANALYSES	34
SECTION 3.5—CONDUCTIVITY ANALYSES	36
SECTION 3.6—TDS ANALYSES	36

SECTION 3.7—TRACE ELEMENT ANALYSES	37
SECTION 3.8—MAJOR ANION AND CATION ANALYSES	38
CHAPTER 4—DISCUSSION	55
CHAPTER 5—CONCLUSION	60
WORKS CITED	61

LIST OF TABLES

Table 2.1 Samples with description and GPS.	30
Table 3.1 Stalactite (E) XRD analysis.	47
Table 3.2 The major oxide weight percents of the stalactite (E), slag (G-1 and G-2), and precipitate below the slag pile (H).	47
Table 3.3 Lost on ignition (LOI) data for the slag samples (G-1 and G-2).	48
Table 3.5 pH titrations conducted at Georgia State University.	51
Table 3.6 Trace element analyses of stalactite (E) and precipitate below the slag pile (H).	52
Table 3.7 Major anions and cations in (mg/L) from TestAmerica.	54
Table 3.8 Major anions and cations (Table 3.7) converted to mEq/L.	54

LIST OF FIGURES

Figure 1.1 Cross section of a blast furnace (OSHA 2008).	9
Figure 1.2 Geology of Alabama (University of Alabama 2008).	10
Figure 1.3 Aerial view of the GSS site, Black Creek, and Lake Gadsden. Drainage is from northwest to southeast.	11
Figure 1.4 Aerial view of the Gulf States Steel property, southeast slag pile, and Black Creek.	12
Figure 1.5 Stalactites on south slag pile at Gulf States Steel.	13
Figure 1.6 Leachate-coated sediment and organic material in drainage ditch at Gulf States Steel property.	14
Figure 1.7 Alkaline drainage observed inside the fence and adjacent to Hickory Road.	15
Figure 1.8 South slag pile and drainage ditch inside the fence and adjacent to Hickory Road.	16
Figure 1.9 Drainage ditch inside the fence leaking into drainage ditch outside the fence.	17
Figure 1.10 Alkaline drainage flowing into wetland area.	18
Figure 2.1 Aerial view of all sample sites (samples A through AG). Black Creek drains from north to south as denoted by a blue arrow. Each sample collection trip denoted by different color schemes.	27
Figure 2.2 Upstream water samples (U, K, and J).	28
Figure 2.3 Alkaline drainage represented by magenta arrows. Black Creek flow direction represented by blue arrows. Map shows all samples (A through AG).	29
Figure 3.1 Petrographic photo of slag in thin section—quartz, vesicles, and amorphous material. Magnification is 10x.	40

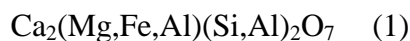
- Figure 3.2 Petrographic photo of slag in thin section—calcite and unknown minerals.
Magnification is 40x. 41
- Figure 3.3 Petrographic photo of slag in thin section—unknown mineral, vesicles, and
amorphous material. Magnification is 10x. 42
- Figure 3.4 XRD reflections of the stalactite (E). D-spacings in Å. Cc refers to calcite. 43
- Figure 3.5 Piper Plot (in mEq/ L). Water samples U, V, W, X, and Y measured by
TestAmerica. 44
- Figure 3.6 Location of upstream samples (J, K, and U) showing pH and approximate TDS.
Black Creek flows from North to South. 45
- Figure 3.7 Alkaline drainage around the southeastern slag pile shown by magenta arrows. pH
and approximate TDS data shown in red text for select samples. 46

CHAPTER 1—INTRODUCTION

Highly alkaline waters occur naturally in thermal springs, ultramafic terrains, evaporative lakes, and in some rift zones (e.g., Roadcap et al., 2005). Alkaline waters also are found in and around steelwork sites, coal combustion residue, solvay process waste, cementitious construction sites, and cementitious and demolition waste (e.g., Mayes et al., 2006). This thesis is a reconnaissance study to define the aerial extent of an alkaline drainage, leachate, and resulting precipitates emanating from one slag pile at the Gulf States Steel site in Gadsden, Alabama.

Slag is a by-product of smelting a mixture of iron ore (hematite) and coke (heated, purified coal), and flux (limestone or dolomite), heated with oxygen to form carbon monoxide in a blast furnace (Schwab et al., 2006). After a charge of iron ore, coke, and flux is smelted with hot air (O_2), the charge melts and produces a layer of liquid iron. This liquid iron sinks below the level of the drain spout for the slag while a layer of slag forms on top of the layer of liquid iron up to the level at the tuyeres (Figure 1.1, OSHA, 2008). After the liquid iron is drained through the drain tap, the slag is then poured off through the same tap. The slag is either kept on site or it is sold as aggregate for roads as described below. The slag cools from liquid state at temperatures nearing the boiling point of iron ($1,482^\circ C$, $2,700^\circ F$) (Ziemkiewicz, 1998) to form a porous, glassy solid. Amorphous glass constitutes 80-95 % of the slag; however, the minerals melilite, oldhamite, merwinite, åkermanite, anorthite, ferdusilicite, gehlenite, khamrabaevite, larnite, monticellite, rankinite, spinel, wollastonite, and wüstite can be detected in thin sections of slag samples (Schwab et al., 2006; Bayless and Schulz, 2003).

According to the National Slag Association (2008) the general formula for slag can be approximated as follows:

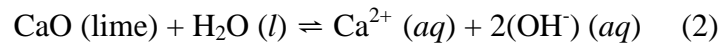


However, slag chemical compositions will vary due to the differences in the chemical make-up of the source rocks and ores that are used in the iron and steel-making processes. Using X-ray fluorescence analysis for major elements, Scott et al. (1986) determined that iron and steelmaking slags will typically contain the following major element oxides: CaO , SiO_2 , Al_2O_3 , and MgO . The purpose of slag is to sequester non-ferrous metals and remove sulfur (as sulfide (in CaS and MgS)) from the liquid iron during smelting. Consequently, slag can also contain gangue metals from iron ore, coke and limestone such as manganese, cadmium, iron (as fayalite or ferrosilite), and a small amount of other metals.

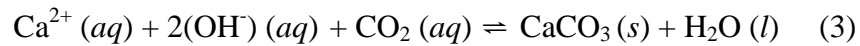
Slags are used as aggregate materials for concrete and asphalt as well as stream bed reinforcement (Koryak et al., 2002). Furthermore, slags are used to neutralize acid mine drainage due to their ability to produce alkaline drainage (Ziemkiewicz, 1998). However, despite their beneficial uses, the reaction of slags with runoff or precipitation can produce hyper-alkaline drainage ($\text{pH} \geq 11$) and precipitates of carbonates. The leachates can have an aesthetically negative impact on the environment, and they can lead to litigation from adjoining properties if not corrected (Feldmann et al, 1980).

Under the neutral to slightly acidic conditions observed during dissolution or weathering, calcium is released from silicate glasses, while calcium is retained in the glass matrix under alkaline conditions (e.g., Grambow, 1992). Therefore, slag will likely react with slightly acidic

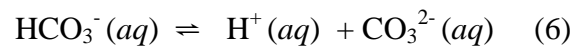
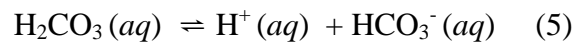
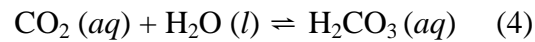
rain water to release calcium and other ions and create alkaline waters. This reaction is approximated below by showing the reaction of calcium oxide (CaO), a predominant constituent in slag, with water to produce a strong alkaline solution and calcium ions forming a hyper-alkaline drainage (pH >11). In the following reactions *aq*, *l*, and *s* represent aqueous, liquid, and solid, respectively (Schwab et al., 2006):



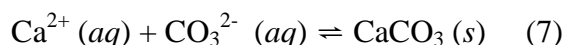
If aqueous calcium ions combine with atmospheric carbon dioxide, then calcite will precipitate as given in the following net reaction (Astrup et al., 2006):



Implicit in this mechanism is the formation of carbonic acid and its dissociation to bicarbonate or carbonate ions by the following reactions (Hurd, 1988):



At alkaline pH conditions, bicarbonate ion disassociates to form carbonate ions which can then bond with calcium ions from the slag to form calcite.



Numerous studies have investigated the formation of calcite from alkaline leachates or drainage. For example, Roadcap et al., (2005) used X-ray diffraction to demonstrate that calcite precipitates in slag-filled aquifers. In laboratory experiments, Schwab et al., (2006) reacted slag with distilled-deionized water in sealed Erlenmeyer flasks producing a leachate with a pH range of 11-12. As the leachate physically moved into neutral waters, Schwab et al, (2006) found that once the alkalinity of the solution decreased through a series of reactions involving carbon dioxide, various calcium carbonate and sulfate minerals precipitated. Calcite, clays, dolomite (Bayless et al., 2003) feldspathoids, gypsum, iron oxides, sodalite, and zeolites appear to be the most common precipitated minerals from the reaction of steel slag with both runoff and groundwater (Schwab et al, 2006). Calcium carbonate-based minerals—specifically calcite and travertine—are the most abundant minerals precipitating from the alkaline drainage of the slag piles (Roadcap et al., 2005; Schwab et al., 2006).

Although the calcium-based coating caused by the drop in alkalinity has negative aesthetic qualities observed by Feldmann et al (1980) and Feldmann (1981) and occasionally costly remediation efforts (e.g., Boyd 1994), slag can have potential positive uses beyond road aggregate and mitigating acid mine drainage. Recent research is looking into slag as a potential carbon dioxide sink (e.g. 3 p. 3) according to Hiujgen et al (2005) and Renforth et al (2009).

Atmospheric carbon dioxide can be sequestered by soils mixed with slag through similar reaction mechanisms as noted above.

SECTION 1.1—OCCURRENCES OF ALKALINE DRAINAGE, LEACHATE, AND PRECIPITATES

Slag-produced carbonate precipitates observed in the field are similar in appearance to travertine, flowstones, or tufa that have formed by the movement of alkaline water, leachate, through sediment, stream beds (Koryak et al., 2002), or road fill used for highway construction (Feldman, 1981 and Boyer, 1994). The color of the precipitates has been described as a milky white or yellow (Moreno, 1999). Schwab et al. (2006) note precipitates can appear green when sulfur is present in an anoxic environment. It is important to note that slag – unlike lime – does not react readily with atmospheric CO₂. Therefore slag, according to Ziemkiewicz (1998), will not be neutralized by air, thus enabling the slag to produce hyper-alkaline drainage for an extended period of time, even when it is exposed to dissolved carbon dioxide found in rain and surface waters. This high yield of alkaline drainage over a long period of time poses a potential threat to organisms that live in close proximity to the drainage from slag piles. For example, studies have shown a correlation between the highly alkaline waters produced by reaction with slag to toxicity to local organisms. Moreno (1999) found that blast furnace slag mixed with fresh water increased the pH of the fresh water above 11.0, resulting in conditions toxic to the freshwater organism *Ceriodaphnia dubia* and larvae of the marine scallop *Mimachlamys asperima*.

Slag-produced alkaline drainage can also affect structures constructed with slag aggregate material, as well as underlying groundwater flow patterns. For example, Bayless and Schulz (2003) noted that quartz becomes pitted above pH 9.9. Increased porosity of geologic formations could lead to structural weaknesses at sites where slag has been used as building aggregate such as highway-bridge approaches. Bayless and Schulz (2003) used the scanning electron microscope and the electron microprobe to show that authigenic minerals – calcite, dolomite, gypsum, clays, and iron oxides—formed rapidly and directly beneath slag piles. They conclude that these minerals can form a hardpan which may divert or impede infiltration of shallow groundwater flow to deeper aquifers. Roadcap et al., (2005) observed calcite precipitate and highly alkaline drainage in the field. They recorded a range of total dissolved solids from 1,900 to 5,000 mg/kg (ppm) for their field samples. Schwab et al. (2003) observed leachate from corroding northeastern Indiana highways built with blast furnace slag. Near Consett, Co. Durham England, Mayes et al., (2006) measured an electrical conductivity between 1,164 and 3,747 $\mu\text{S}/\text{cm}$, and recorded a pH range of 11.48 – 12.48 at waters directly down gradient from slag piles. Their XRD data show the precipitates to be mostly calcite with little impurities.

SECTION 1.2—GULF STATES STEEL BACKGROUND AND STUDY AREA.

Gulf States Steel (GSS) is situated on a 767 acre site located in Gadsden (Etowah County), Alabama. Steel manufacturing began on the site in 1902. Since that time the plant has changed hands through many owners until GSS took charge of operations in 1986. In 1993 the *Gadsden Times* (Kanwar 1993) reported the occurrence of fish kills in nearby Lake Gadsden. This led to a 1993 EPA (Environmental Protection Agency) ecological assessment of the

discharged waste water entering Black Creek and ultimately Lake Gadsden downstream. A fish health assessment targeting the largemouth bass indicated severe impairment of the species. As a result on September 27, 1994 GSS entered into a Consent Decree with the EPA. In December 1994 *The Gadsden Times* reported that GSS agreed to pay a settlement of \$500,000, without admitting guilt for any negative environmental impact to the state of Alabama (Kanwar 1994).

GSS committed several Resource Conservation and Recovery Act (RCRA) violations from 1982 through 1998. In 1999, GSS filed a voluntary petition for bankruptcy and terminated most of its operations in 2000. On January 9, 2004 the Alabama Department of Environmental Management (ADEM) deferred all RCRA activities at GSS. The EPA then took ownership of the GSS site remediation under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) or Superfund program. Today the majority of the former GSS property is owned by the Gadsden Industrial Park.

The slag deposits at GSS occur predominantly in two large piles in the southeastern area of the property. These piles are approximately 80 feet high and have an approximate volume of four-million cubic yards. Figures 1.3 and 1.4 show aerial views of the slag piles at the GSS site, and data for this reconnaissance study was collected from the southern slag pile and surrounding area. Stalactites forming on the east side of the southern slag pile, as seen in Figure 1.5, were observed for this study. A white leachate coats sediment in the drainage ditches surrounding the slag piles, as seen in Figure 1.6. This leachate extends radially from the slag pile only on the meter scale. The relative short distance of leachate observed is most likely due to its adsorption by surface sediments—a natural leachate retardation method also observed by Indraratna et al., (1999). The drainage ditches surrounding the east side of the slag pile flow to a wetland. The

wetland and ditches are coated with a milky, white sediment (Figures 1.7, 1.8, 1.9, and 1.10). Some loss of vegetation is observed in the wetland.

The EPA (2006) classifies the surficial soils at the GSS site as Urban Land, meaning the soils have been so altered by on-site activities that they cannot be assigned to a recognized soil order. The top soil layer is fill material consisting of 5 feet of slag with intermixed coal, coke, and metal fragments. Underneath the fill layer lies a 10 to 15 foot thick clay layer followed by bedrock. According to the EPA (2006) the bedrock of Etowah County is composed of Paleozoic rocks ranging in age from Cambrian to Pennsylvanian Periods. The GSS property is underlain by the Conasauga Group (University of Alabama and Figure 1.2). The Conasauga Group has a thickness here of about 850 feet, with limestone and dolostone upper members, and a shale lower member.

SECTION 1.3—OBJECTIVES.

The focus of this thesis was to conduct a reconnaissance study to characterize the residual precipitates and the alkaline leachate associated with the GSS slag piles. Mineral and chemical analysis were conducted on water, leachate, and sediment samples. In addition, the scope of work included a rudimentary analysis of waters in the immediate area of the GSS site. From these characterizations, a mechanism of leachate production and precipitate formation will be proposed. The presence of alkaline leachates and associated precipitates occur less commonly compared to acidic drainage found elsewhere in the Piedmont Province (for example, Boyd, 1998). This study describes the occurrence of these leachates and associated precipitates and should add to a growing body of research on slag pile alkaline drainages and precipitates.

Cross Section of a Typical Furnace

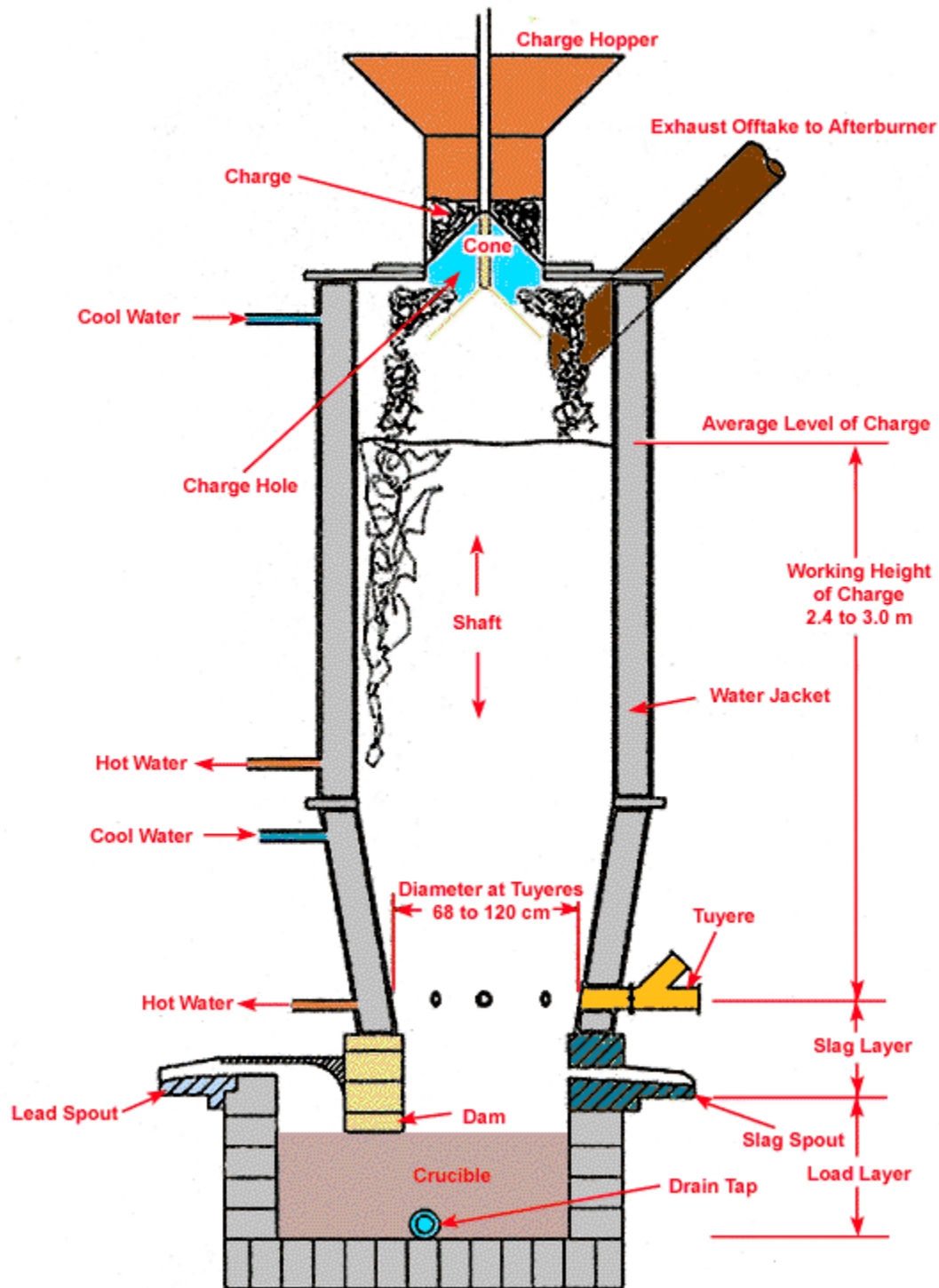


Figure 1.1 Cross section of a blast furnace (OSHA 2008).

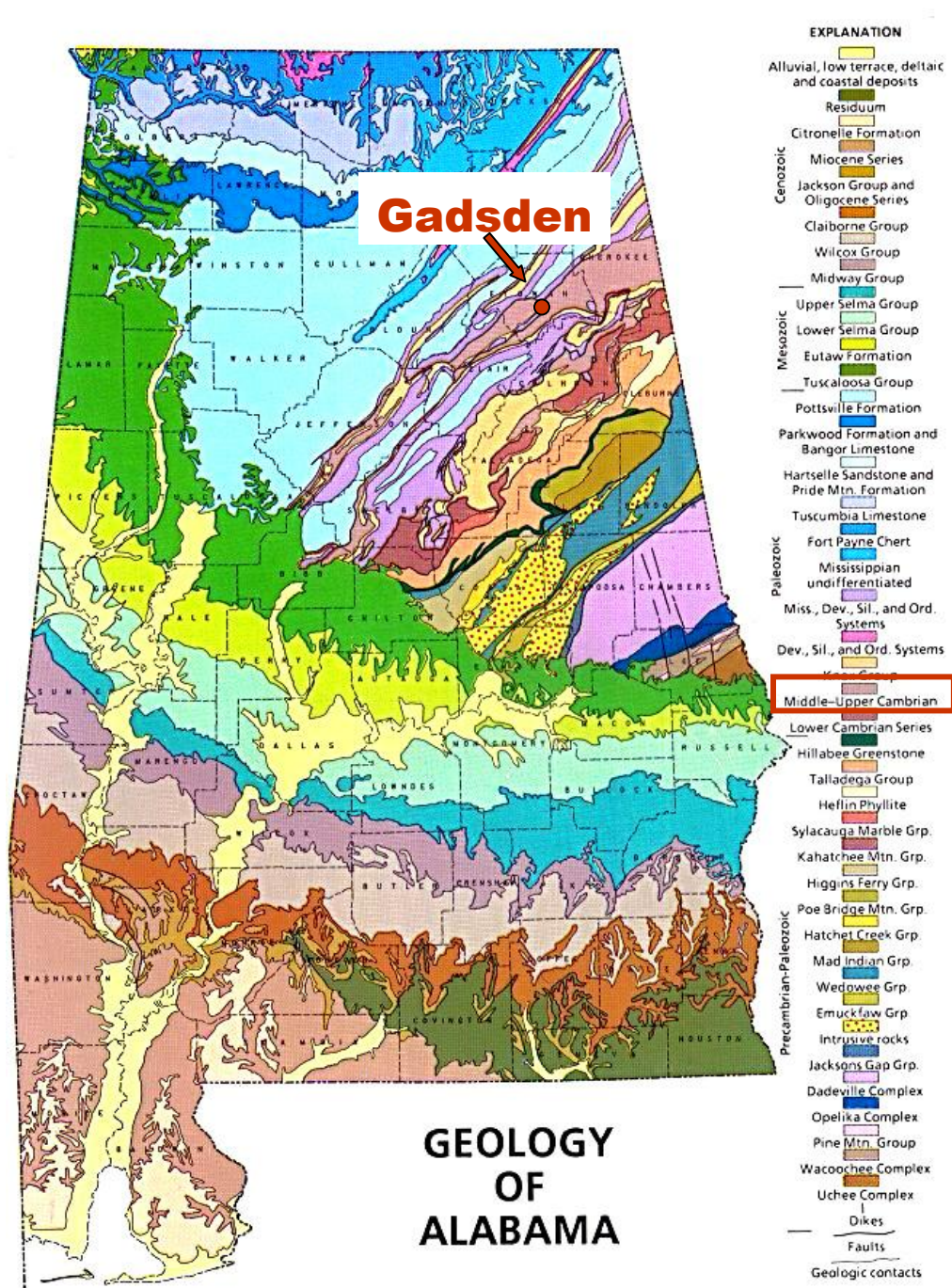


Figure 1.2 Geology of Alabama (University of Alabama 2008).

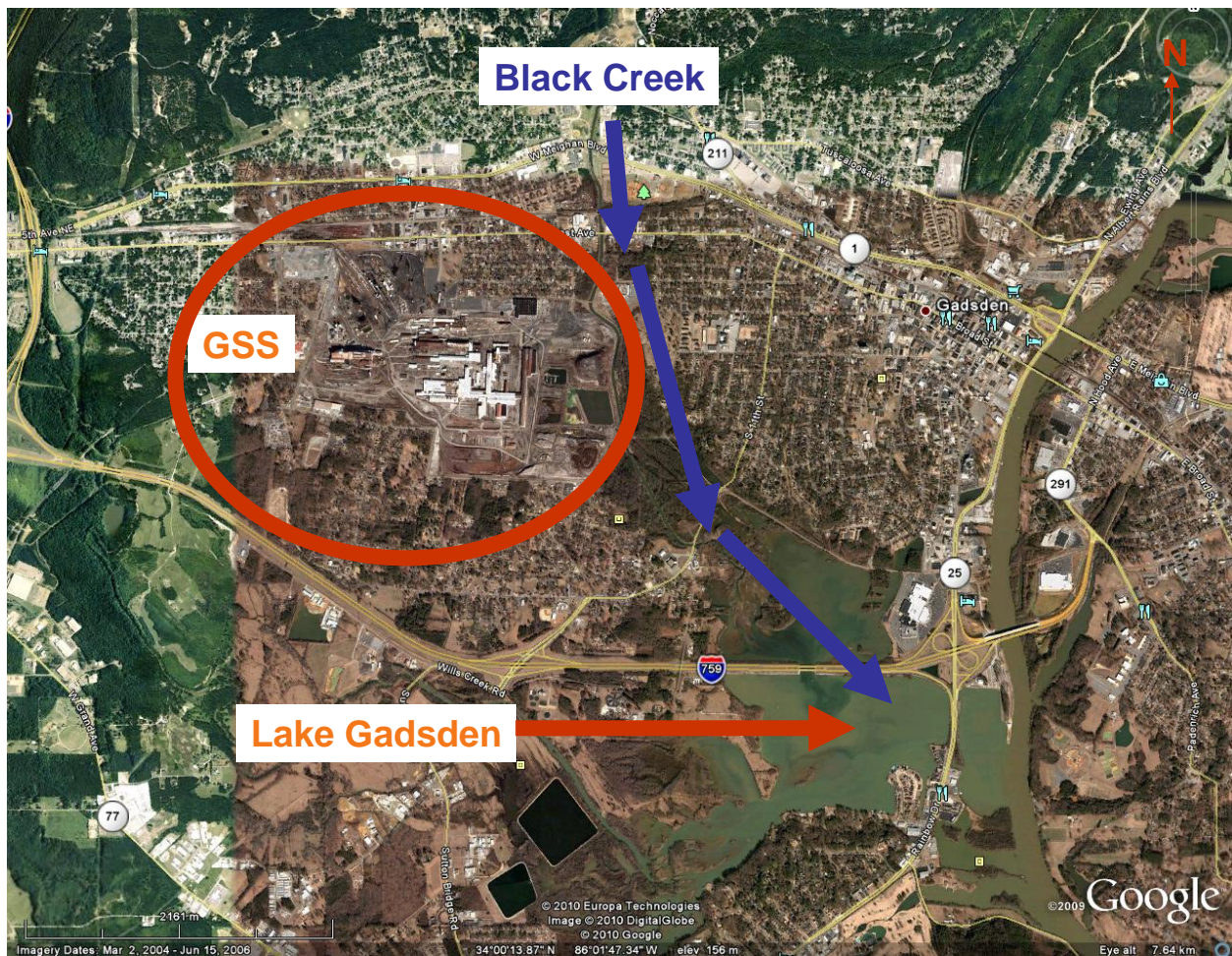


Figure 1.3 Aerial view of the GSS site, Black Creek, and Lake Gadsden. Drainage is from northwest to southeast.



Figure 1.4 Aerial view of the Gulf States Steel property, southeast slag pile, and Black Creek.



Figure 1.5 Stalactites on south slag pile at Gulf States Steel.



Figure 1.6 Leachate-coated sediment and organic material in drainage ditch at Gulf States Steel property.



Figure 1.7 Alkaline drainage observed inside the fence and adjacent to Hickory Road.



Figure 1.8 South slag pile and drainage ditch inside the fence and adjacent to Hickory Road.



Figure 1.9 Drainage ditch inside the fence leaking into drainage ditch outside the fence.



Figure 1.10 Alkaline drainage flowing into wetland area.

CHAPTER 2—METHODS

Methods employed were as follows: Environmental Protection Agency (EPA) approved field collection of water and sediment samples, *in situ* pH measurements, *in situ* conductivity, laboratory pH, laboratory conductivity, X-ray diffraction of applicable sediments, X-ray fluorescence of slag, and thin section analysis of slag. Refer to Figures 2.1, 2.2, and 2.3; and Table 2.1 for sample details.

SECTION 2.1—PETROGRAPHY AND ROCK SAMPLE PREPARATION

The Rock Preparation Laboratory in the Georgia State Department of Geosciences was used to prepare the stalactite (E¹) for X-ray diffraction (XRD) and X-ray fluorescence (XRF). Additionally, the slag (G) and the drainage ditch sediment (H) samples were prepared for XRF. Two slag samples were set aside for petrographic analysis. Burnham Petrographics LLC prepared two 30 micro-meter thin sections of these slag samples for petrographic analyses. Initially a metal mortar and pestle were used to reduce the size of the stalactites (E) and drainage ditch sediment (H). A Braun Chipmunk Rock Crusher was used on the slag (G) to reduce the size of the crystalline slag so that it could be ground to a fine, clay-sized powder in the shatter box. In addition, the stalactites (E) and drainage ditch sediment (H) samples were crushed and powdered for further reduction in size in the shatter box.

After initial preparation, each sample was ground using the shatter box for approximately 45 seconds. A clean metal spatula was used to transfer the powdered rock sample from the metal

¹ Samples are represented by a letter, or letters, in parentheses. Refer to Table 2.1 for sample details.

canister to an unused, labeled, plastic snap-cap vial. Due to the small size of the metal canister, several cycles were conducted to obtain 50 grams of each sample powdered. The shatter box, metal spatula, and the metal canister were decontaminated with soap and water and allowed to air dry between each sample.

SECTION 2.2—X-RAY DIFFRACTION

The stalactite sample (E) was analyzed with X-ray diffraction for mineralogical characterization. Random back-filled mounts were prepared using methods described in Moore and Reynolds (1997). The mounts were scanned from 2 to 60 degrees two-theta (2θ) using copper radiation filtered with a graphite monochromator. The step size was 0.02 degrees two theta and the count time for each step was one second. The MDI Databox® was used for control of scanning and acquisition of intensity data.

SECTION 2.3—X-RAY FLUORESCENCE

X-ray fluorescence (XRF) of the powdered slag sample (G) was conducted at Georgia State University. XRF analyses of the stalactites (E) and precipitate below slag pile (H) was conducted at ActLabs in Canada. For the XRF analyses at Georgia State University, all samples and crucibles were weighed using an electric Metter PM200 balance. 0.5000 grams of powdered slag (G) and 4.5000 grams of lithium metaborate flux were mixed and added to a platinum crucible, and then heated at 1100 °C for 10 minutes in an oven. After being heated in the oven, the crucible was then further heated over a Meker burner while being gently shaken in horizontal,

circular motion to remove the air bubbles from the melt. The melt was poured onto a template to form a glass disk. The glass disk was labeled with the sample number for XRF analysis. The platinum crucible was placed into acid to remove any remaining residue. After cooling and labeling, the resulting glass disks were analyzed with a Rigaku 3270 X-Ray Fluorescence Spectrometer. A standard glass disk was also placed in rotary to ensure proper calibration of the equipment. XRF analyses were run for all samples, and data results were printed out from the computer.

Lost on ignition (LOI) was calculated by subtracting the weight of the sample after heating and cooling from the weight of the sample before heating. LOI percentage was calculated by dividing the LOI weight by the initial weight of the sample before heating.

SECTION 2.4—FIELD WATER & SEDIMENT COLLECTION

The drainage ditch water outside the fence (A), drainage ditch sediment outside the fence (B), wetland water (C), encrustation on wetland sediments (D), stalactites (E), drainage ditch sediment inside the fence (F), slag (G), precipitate below slag pile by parking lot (H), and mouth of drainage into Black Creek (I) samples were collected on December 1st, 2007 by Craig VanTrees, Jordan Garrard, and Dr. W.C. Elliott. The stalactite (E) and precipitate on sediment (H) samples were sent to ActLabs for pH, conductivity, and trace element analysis. The slag sample (G) was sent to Burnham Petrography for thin section preparation.

Upstream Black Creek water (J), upstream Black Creek water (K), southeast ditch water adjacent to slag pile (L), southeast ditch water adjacent to slag pile (M), wetland water (N), drainage ditch water adjacent to slag pile (O), drainage ditch water adjacent to slag pile (P),

Black Creek water at Hickory Road Bridge (Q), stagnant wetland (R), drainage pipe water into Black Creek (S), and south of the drainage pipe water into Black Creek (T) samples were collected on January 24th, 2009 by Craig VanTrees, Cindi Pena, and Dr. W.C. Elliott.

Approximate coordinates for sample collecting are latitude 34°00'32.2"N, longitude 86°01'8.63"W at an elevation of approximately 684 feet above mean sea level. Exact latitude and longitude values presented in Table 2.1 were recorded onsite via a hand-held GPS unit issued by Georgia State University.

All field samples were collected, transported, and stored according to the Environmental Protection Agency's Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM.) EISOPQAM methods for collecting water require using a non-reactive plastic bottle. New 250 mL plastic Nalgene bottles washed in nitric acid were utilized for this collection effort.

Upstream Black Creek water (U), drainage ditch below stalactites (V), tributary into Black Creek from wetland (W), center of Black Creek downstream from the slag (X), and center of Black Creek farthest downstream from the slag (Y) samples were collected, transported, and stored adhering to EISOPQAM methods by Craig VanTrees, Dr. Jordan Clayton, and Dr. W. Crawford Elliott on August 15th, 2009. These samples were sent to TestAmerica for pH, TDS, major cation, and major anion analysis on the week of August 17th, 2009.

The drainage ditch waters below the stalactites (Z), slag pile seepage at Jackson and Hickory inside the fence (AA), slag pile seepage mixing with drainage ditch water at Jackson and Hickory inside the fence (AB), telephone pole between Jackson and Main on Hickory inside the fence (AC), outside fence between Jackson and Main on Hickory (AD), outside fence at the bend by Hickory and Harrison (AE), moving water directly north from the drainpipe outside the fence

(AF), and at the bend of Hickory and Harrison inside the fence (AG) samples were collected by Craig VanTrees, and Steve Arnstein, and Jordan Garrard on October 20th, 2009.

The samples were collected with the bottles facing upstream to prevent disturbed sediment from contaminating the water sample. Water samples are stored and transported in a cooler on ice and then placed into refrigeration. Samples were analyzed within 30 days of collection.

EISOPQAM methods for collecting sediment collection require use a non-reactive, stainless steel spoon for collecting the sample and a clean air-tight non-reactive glass container. Mason jars and a stainless steel spatula were used in this sampling effort. For both water and sediment sampling, disposable, sterilized latex gloves were worn. Distilled water and dish soap were used two times to decontaminate the following items in the field between each sample collection: rubber gloves, metal bowl, plastic scoopers, and two metal scoopers.

SECTION 2.5—PH MEASUREMENTS

Approximate pH measurements were conducted using litmus paper and a pH meter *in situ* prior to quantitative measurements taken in a lab setting. Field pH was measured using pH indicator strips ranging from pH 1-14.

Quantitative pH measurements of drainage ditch water outside the fence (A), wetland water (C), and mouth of drainage into Black Creek (I) were conducted by pH meter at Georgia State University. Quantitative pH measurements of samples drainage ditch waters below the stalactites (Z), slag pile seepage at Jackson and Hickory inside the fence (AA), slag pile seepage mixing with drainage ditch water at Jackson and Hickory inside the fence (AB), telephone pole

between Jackson and Main on Hickory inside the fence (AC), outside fence between Jackson and Main on Hickory (AD), outside fence at the bend by Hickory and Harrison (AE), moving water directly north from the drainpipe outside the fence (AF), and at the bend of Hickory and Harrison inside the fence (AG) were conducted by titration at Georgia State University. ActLabs provided quantitative pH measurements for the stalactite slurry paste (E) and leachate-coated sediment slurry paste (H). TestAmerica provided quantitative pH measurements and TDS data for samples upstream Black Creek water (U), drainage ditch below stalactites (V), tributary into Black Creek from wetland (W), center of Black Creek downstream from the slag (X), and center of Black Creek farthest downstream from the slag (Y).

Samples A, C, and I through T (refer to Table 2.1 for descriptions) were transported to Georgia State University to conduct quantitative pH measurements using a pH meter. Calibration of the pH meter at Georgia State University was performed 3 times using buffers with pH values of 4 and 11 to calibrate the electrode. Values of 4 and 11 were manually input into the device as the reading never landed precisely on pH 4 and 11 during the calibration effort. Note that some of the samples were higher than 11. Sixty mL of sample was placed into a cleaned glass beaker and held in contact with the pH electrode for approximately 3 minutes prior to the reading being recorded. Note that the pH meter value continued to increase, and the meter was not able to land on a value. pH was recorded after three minutes if the value would not lock on one number. The pH meter electrode was rinsed with distilled water between each calibration and sample pH reading. After completion of the pH analyses, each sample was transferred to a clean plastic graduated cylinder to be used for the conductivity test.

The pH was measured by titration for samples Z through AG (refer to Table 2.1 for descriptions). The titration used a 0.5 % phenolphthalein solution made at Georgia State

University. A graduated cylinder was filled with approximately 30 mL of sample, and then 2 drops of phenolphthalein were added. The sample with the phenolphthalein was placed under a burette containing a 0.1 molar solution HCl. The initial volume of HCl solution in the burette was measured and recorded. The sample was then titrated with the HCl solution until the sample turned fuchsia then faded to a faint pink in color. The final HCl volume is recorded. The following formula was used to calculate the pH for each titration trial:

$$\text{pH} = 14 + \log [(1 \text{ L} / 1000\text{mL sample in titrant}) * (\text{volume HCl used in mL}) * (0.1 \text{ M HCl}) * (1000\text{mL} / 1\text{L}) * (1 / (\text{volume sample in mL}))] \quad (8)$$

$$\text{pH} = 14 + \log [\text{OH}^-] \quad (9)$$

SECTION 2.6—CONDUCTIVITY

Conductivity was measured *in situ* for water samples when possible by placing the conductivity meter directly in the water so that all the coils were covered. After the conductivity value stabilized, the conductivity was recorded in the field note book. If the water level was too shallow for proper conductivity measurements, then a large plastic beaker was rinsed several times before slowly collecting the water in the upstream direction. The conductivity meter was then placed into the plastic beaker to obtain a measurement. Conductivity measurements were taken again at Georgia State University in the Hydrogeology Laboratory. The conductivity electrode attached to the conductivity meter was placed in the graduated cylinder so that the top (or fifth) rubber spiral would be right at the water line. The conductivity value was recorded

after the electrode rested in the cylinder for approximately 2 minutes. The conductivity electrode and graduated cylinder were both rinsed with distilled water between, and after, each sample reading. Total Dissolved Solids (TDS) in parts per million (ppm) were approximated by multiplying the conductivity by 0.67 and changing the units from $\mu\text{S}/\text{cm}$ to mg/kg . This method outlined by Hem (1970) advises using a value between the range of 0.55 and 0.75, and 0.67 falls within this range.

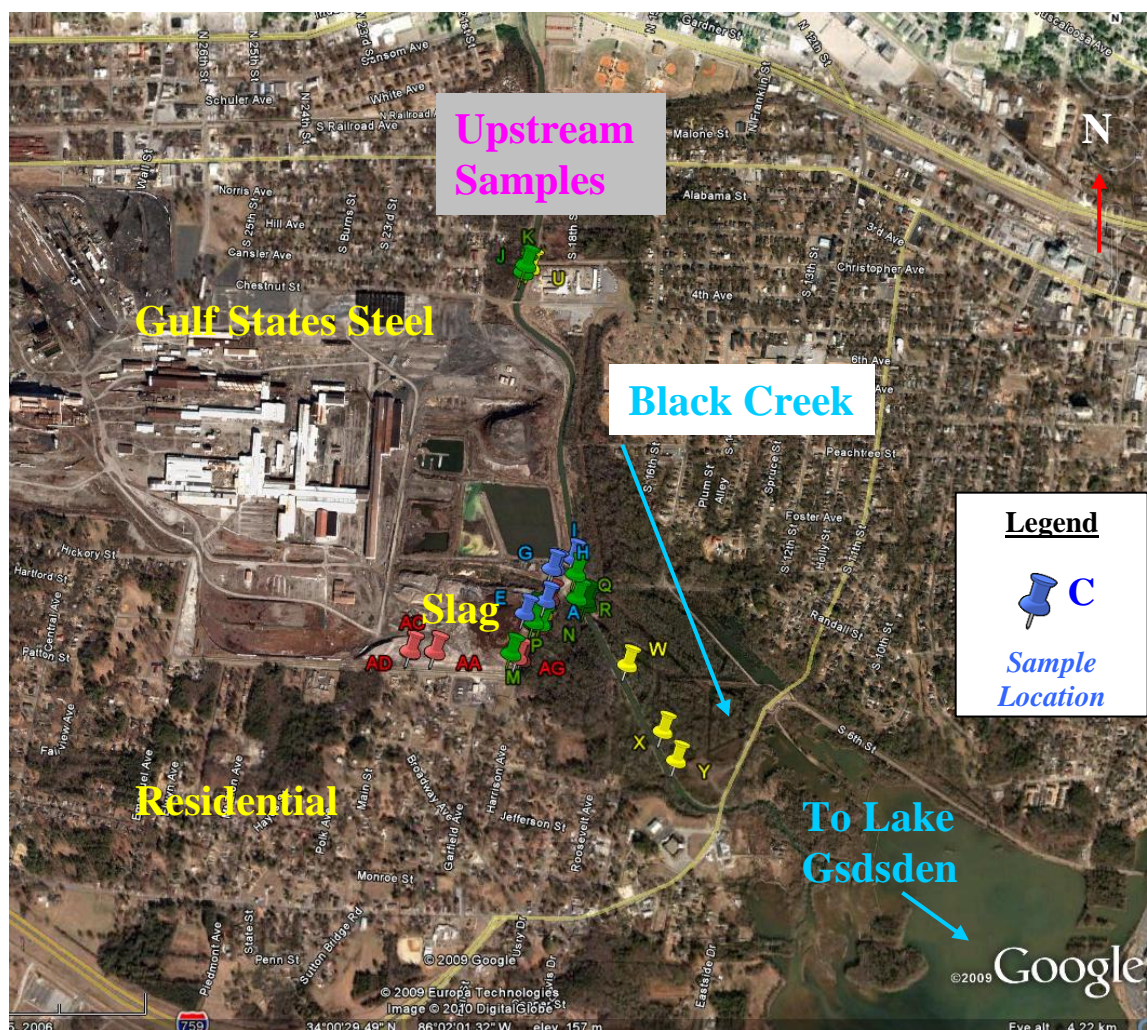


Figure 2.1 Aerial view of all sample sites (samples A through AG²). Black Creek drains from north to south as denoted by a blue arrow. Each sample collection trip denoted by different color schemes.

² Samples are represented by a letter, or letters, in parentheticals. Refer to Table 2.1 for sample details.



Figure 2.2 Upstream water samples (U, K, and J).

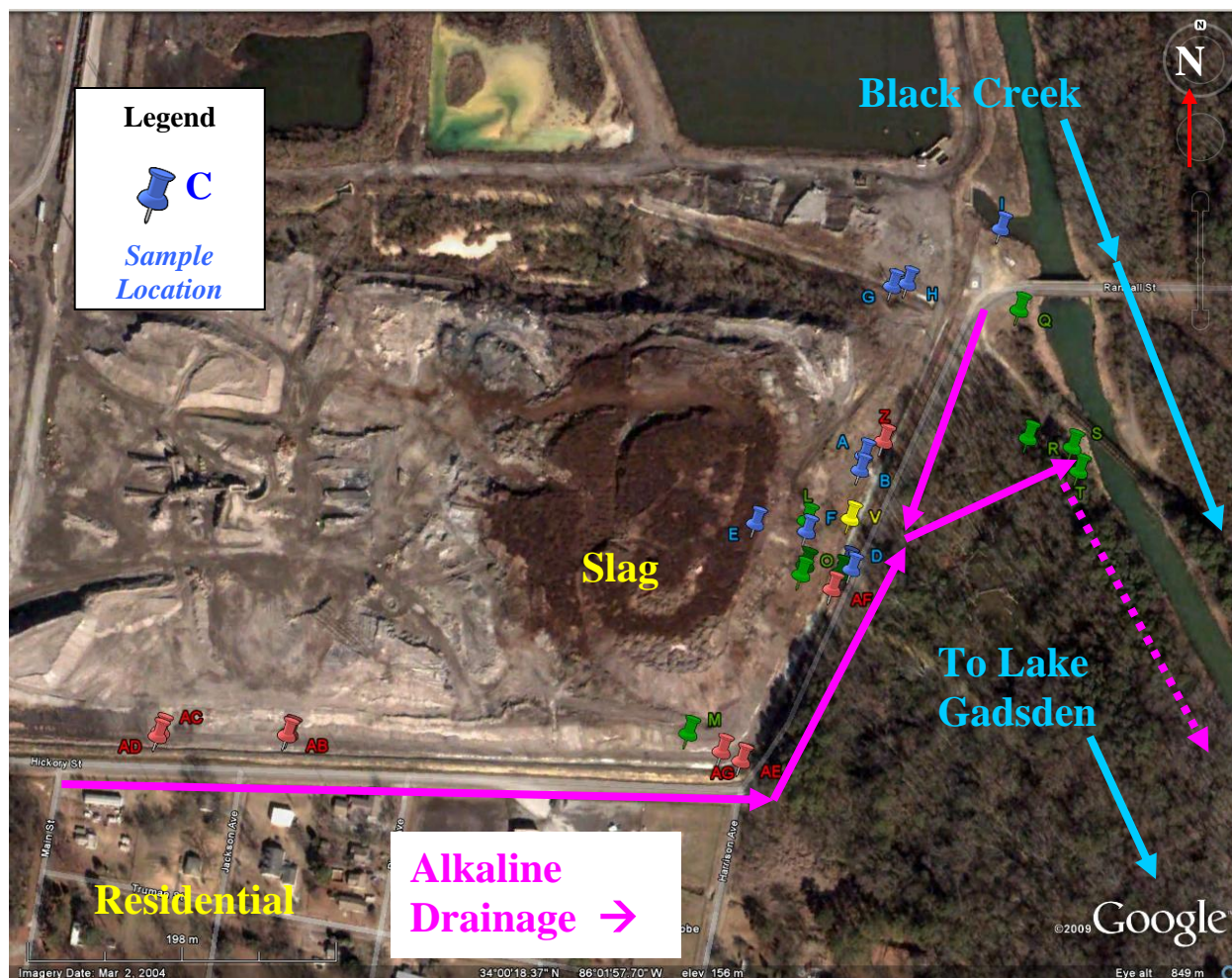


Figure 2.3 Alkaline drainage represented by magenta arrows. Black Creek flow direction represented by blue arrows. Map shows all samples (A through AG).

Table 2.1 Samples with description and GPS.

Sample	Sample Description	Analyses Performed*	Latitude	Longitude
A	Drainage ditch water directly below the slag pile, outside the fence.	Conductivity pH	34°00'18.9"N	86°01'51.8"W
B	Drainage ditch sediment, outside the fence.	X-Ray Diffraction	34°00'18.5"N	86°01'52.2"W
C	Stagnant wetland water across the road from the slag pile.	Conductivity pH	34°00'15.4"N	86°01'51.7"W
D	Stagnant wetland encrustation.	X-Ray Diffraction	34°00'15.4"N	86°01'51.7"W
E	Stalactites, inside the fence.	Conductivity FUS-ICP FUS-MS INAA MULT INAA / TD-ICP pH TD-ICP X-Ray Diffraction X-Ray Florescence	34°00'32.2"N	86°01'8.63"W
F	Drainage ditch sediments, inside the fence.	X-Ray Diffraction	34°00'20"N	86°01'50"W
G	Slag sample, inside fence.	Petrographic Analysis X-Ray Diffraction X-Ray Florescence	34°00'20"N	86°01'50"W
H	White precipitate, directly below slag pile by second parking lot, inside fence.	Conductivity FUS-ICP FUS-MS INAA MULT INAA / TD-ICP pH	34°00'23.3"N	86°01'49.4"W

Sample	Sample Description	Analyses Performed*	Latitude	Longitude
		TD-ICP		
I	Mouth of drainage into river.	Conductivity pH	34°00'23.4"N	86°01'48.1"W
J	Upstream water sample.	Conductivity pH	34°00'9.24"N	86°01'9.14"W
K	Upstream water sample.	Conductivity pH	34°00'9.31"N	86°01'9.01"W
L	SE Adjacent to the Slag Pile	Conductivity pH	34°00'32.0"N	86°01'8.57"W
M	Corner adjacent to slag pile	Conductivity pH	34°00'21.7"N	86°01'9.22"W
N	Wetland stagnant water.	Conductivity pH	34°00'26.6"N	86°01'8.69"W
O	Trough adjacent to slag pile.	Conductivity pH	34°00'27.3"N	86°01'8.78"W
P	Trough adjacent to slag pile.	Conductivity pH	34°00'28.6"N	86°01'8.71"W
Q	Black Creek at Hickory Road Bridge.	Conductivity pH	34°00'37.5"N	86°01'7.91"W
R	Stagnant Wetland.	Conductivity pH	34°00'32.7"N	86°01'7.88"W
S	Drainage Pipe into Black Creek.	Conductivity pH	34°00'32.5"N	86°01'7.69"W
T	South of Drainage Pipe.	Conductivity pH	34°00'32.2"N	86°01'7.67"W
U	Upstream water.	Conductivity Major Anion and Cations pH TDS	34°00'56.2"N	86°01'54.1"W
V	Ditch below stalactites.	Conductivity Major Anion and Cations pH TDS	34°00'18.6"N	86°01'51.6"W
W	Tributary into Black Creek from the Marsh area.	Conductivity Major Anion and Cations pH TDS	34°00'12.5"N	86°01'40.4"W

Sample	Sample Description	Analyses Performed*	Latitude	Longitude
X	Center of Black Creek, downstream from South Slag.	Major Anion and Cations pH TDS	34°00'05.6"N	86°01'35.8"W
Y	Center of Black Creek, farthest downstream.	Major Anion and Cations pH TDS	34°00'02.3"N	86°01'33.9"W
Z	Drainage ditch below the stalactite inside the fence.	Conductivity pH	34°00'19.2"N	86°01'51.3"W
AA	Seepage at Jackson & Hickory inside the fence.	Conductivity pH	34°00'13.1"N	86°02'06.3"W
AB	In ditch by seepage at Jackson & Hickory inside the fence.	Conductivity pH	34°00'13.1"N	86°02'06.3"W
AC	Telephone pole between Jackson and Main on Hickory inside the fence.	Conductivity pH	34°00'13.0"N	86°02'09.5"W
AD	Outside fence between Jackson and Main on Hickory.	Conductivity pH	34°00'13.1"N	86°02'09.7"W
AE	Outside fence at the bend by Hickory and Harrison.	Conductivity pH	34°00'12.5"N	86°01'54.9"W
AF	Moving water directly north from the drainpipe outside the fence.	Conductivity pH	34°00'16.1"N	86°01'52.6"W
AG	At the bend of Hickory and Harrison inside the fence.	Conductivity pH	34°00'12.7"N	86°01'55.4"W

**Key for analyses methods is as follows: FUS-ICP - Fusion-Inductively Coupled Plasma , FUS-MS - Fusion-Inductively Coupled Plasma-Mass Spectrometry, INAA - Instrumental Neutron Activation Analysis, and TD-ICP - Total Digestion- Inductively Coupled Plasma-Optical Emission Spectrometry*

CHAPTER 3—RESULTS

SECTION 3.1—PETROGRAPHY

The thin section of the slag (G) appears to contain abundant quartz as seen in Figure 3.1. An optically negative, off-centered uniaxial interference figure was observed to confirm calcite grains in Figure 3.2. The other large mineral grain (Figure 3.3) was not fully identifiable, and no interference figure could be obtained due to both grain orientation and high birefringence. However, it is likely that the mineral in Figure 3.3 corresponds to muscovite based on shape and high birefringence (Deer et al, 1992). The remaining mineral grains were too small to be identified using the petrographic microscope. Amorphous material and vesicles (or holes) appeared to be present in the majority of both thin sections.

SECTION 3.2—X-RAY DIFFRACTION

The diffraction data for the stalactite (E) are shown in Table 3.1 and Figure 3.4. The following d-spacings observed for the stalactite (E) correspond to calcite: 3.85, 3.04, 2.50, 2.39, 2.10, 2.03, 1.91, 1.88, and 1.61Å.

SECTION 3.3—X-RAY FLUORESCENCE

The concentration of major elements for the stalactite (E), two powdered slag samples (G-1 and G-2), precipitate adjacent to the slag pile (H) are listed in Table 3.2. Table 3.3 lists the

LOI (lost on ignition) data for the slag samples (G-1 and G-2). The stalactite (E) and precipitate adjacent to the slag pile (H) were sent to ActLabs for XRF analyses. XRF analyses for the slag samples (G-1 and G-2) were conducted at Georgia State University.

The analyses of slag samples G-1 and G-2 (Tables 3.2 and 3.3) indicate that calcium (39.7% and 39.5%), iron (22.5% and 22.4%), and silica (19.9% and 20.4%), magnesium (3.0% and 3.0%), manganese (2.5% and 2.5%), phosphorous (1.8% and 1.8%), and aluminum (1.6% and 1.6%) are its predominant oxides. These predominant oxides correspond to the general slag formula. All other measured oxides—titanium, sodium, and potassium— are < 1.0%. LOI ranged from 3.8% to 4.1%.

Both the stalactite (E) and precipitate on the sediment below the slag pile (H) have high calcium oxides (54.9% and 54.8%). LOI is also high (39.4% and 43.6%). The high CaO and LOI are consistent with calcite. All other measured oxides—silica, titanium, aluminum, iron, manganese, magnesium, sodium, potassium, and phosphorous are < 0.5%. According to ActLabs, titanium is below the detection limit for the stalactite (E) and potassium is below the detection limit for the precipitate on the sediment (H).

SECTION 3.4—PH ANALYSES

The results for the preliminary water analyses collected during the winter and spring (A, C, E (slurry paste), H (slurry paste), I, J, K, L, M, N,O, P, Q, R, S, and T) are listed in Tables 3.4, 3.5, 3.7, and 3.8. The pH ranges from 6.55 to 12.70. pH data in the range of 6 to 8 in value correspond to the waters surrounding Black Creek (K, J, R, T, S, I, and Q). pH data in the 6

range correspond to the upstream waters and wetland (J, K, and R). pH data in the 7-8 range correspond to Black Creek waters near and down-gradient from the GSS property (I, Q, S, and T). pH data in the range of 11 to 12 correspond to the drainage and waters near the slag pile (A, H, N, O, P, C, M, L, and E). Refer to Table 3.4, Figure 3.6, and Figure 3.7 for preliminary data.

The upstream water (U), ditch below stalactites (V), marsh tributary (W), downstream Black Creek (X), farthest downstream Black Creek (Y) samples collected in the summer were sent to TestAmerica for quantitative analysis of pH and these data are displayed in Tables 3.4, 3.7, and 3.8 and in Figure 3.5. The pH data of the waters in Black Creek range from 8.09 to 8.66 (U, W, X, and Y). The pH at the drainage ditch near the slag pile (V) is greater than 12, and TestAmerica could not provide a more accurate pH reading as this was beyond their detection limits.

Eight samples were collected in the Fall of 2009 for quantitative analysis (Z, AA, AB, AC, AD, AE, AF, and AG), and those pH data are shown in Tables 3.4 and 3.5 and in Figure 3.7. The highest pH 12.52 is observed for direct slag pile seepage (AA) and lowest pH observed is from the drainpipe area at 11.96 (AF). Seepage water that has mixed with the ditch water (AB) has an observed pH of 12.39. The water directly below the largest stalactite concentrations (Z) has a similar pH at 12.38. The pH remains in the 12 range in the ditches both inside and outside the fence until it approaches and mixes with the water by the drainpipe moving the waters towards Black Creek.

SECTION 3.5—CONDUCTIVITY ANALYSES

Conductivity ranges from 47.8 to 6300.0 $\mu\text{S}/\text{cm}$ (microsiemens per centimeter). Data ranging from 47.8 to 327.5 $\mu\text{S}/\text{cm}$ (J, K, Q, T, S, U, H, W, R, and I) correspond to the waters surrounding Black Creek, with the exception of the leachate and sediment below the slag (H) and a wetland sample (R). Data ranging from 430.0 to 6300.0 $\mu\text{S}/\text{cm}$ (V, O, N, A, P, C, AF, L, M, AE, AC, AD, E, Z, AG, AB, AA) correspond to the waters near the slag pile. The conductivity of 430.0 $\mu\text{S}/\text{cm}$ for the ditch waters below the stalactites (V) appears to be low for the pH recorded. Refer to Table 3.4 for sample description and data. The seepage coming directly out of the slag pile (AA) has the highest observed value of 6300.0 $\mu\text{S}/\text{cm}$.

SECTION 3.6—TDS ANALYSES

Approximate TDS ranges from 32 to 4221 mg/ L. These values were approximated by calculating the conductivity of the waters by 0.67 to convert $\mu\text{S}/\text{cm}$ to mg/ L. The upstream water (U), drainage ditch water below stalactites (V), wetland tributary (W), downstream Black Creek (X), and farthest downstream Black Creek (Y) samples were sent to TestAmerica for quantitative analysis of TDS, and these data are displayed in Table 3.4. Data ranging from 47.8 to 327.5 mg/ L (J, K, Q, T, S, U, H, W, R, and I) correspond to the waters surrounding Black Creek, with the exception of the leachate and sediment below the slag (H) and a wetland sample (R). TDS ranging from 288.1 to 4221 mg/ L (V, O, N, A, P, C, AF, L, M, AE, AC, AD, E, Z, AG, AB, AA) correspond to the waters near the slag pile. The seepage coming directly out of the slag pile (AA) has the highest approximated value of 4221 mg/ L.

SECTION 3.7—TRACE ELEMENT ANALYSES

Trace elements were analyzed at ActLabs, Canada, for the stalactite (E) and precipitate near the slag pile (H) as shown in Table 3.7. The following 16 trace elements were determined both in the stalactite (E) and precipitate below the slag pile (H): Ba, Br, Co, Cr, Cu, Nb, Ni, Pb, S, Sc, Sr, V, Zn, Zr, Nd, and Eu. The following 7 trace elements were determined in the precipitate below the slag pile (H) in addition to what is determined in the stalactite: Eu, Sb, U, La, Ce, Pr, Sm, and Yb.

The stalactite (E) has measured values above the detection limits for the following trace elements: Ba (214 ppm), Sr (179 ppm), Pb (96 ppm), Ni (77 ppm), Zn (30 ppm), V (18 ppm), and Cr (13 ppm), Cu (3 ppm), and Zr (2 ppm). The precipitate below the slag pile (H) has measured values above the detection limits for the following trace elements: Ba (229 ppm), Sr (468 ppm), Pb (8 ppm), Ni (3 ppm), Zn (51 ppm), V (26 ppm), and Cr (7.7 ppm), and Cu (13 ppm).

The stalactite (E) has significantly higher levels of lead, nickel, and chromium compared to the precipitate below the slag pile (H,) but it has lower levels of all other trace elements. Neodymium and europium were the only 2 rare earth elements (REE) with an analysis above minimum detection limits in the stalactite (0.009 ppm and 0.006 ppm), and the precipitate below the slag pile (H) has more Nd and Eu comparatively than the stalactite (E).

The precipitate below the slag pile (H) appears to be enriched in Sr compared to the average Earth's crust (Krauskopf 1967), and this is the only trace element observed that is enriched compared to the average of Earth's crust. Compared to average of Earth's crust, the

stalactite (E) and slag leachate (H) are observed to be depleted in the following trace elements:
As, Ba, Be, Bi, Cd, Co, Cr, Cs, Cu, Ga, Ge, Hf, Mo, Nb, Rb, Sc, Sn, Th, U, V, W, Y, Zr, La, Ce,
Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Yb, and Lu.

SECTION 3.8—MAJOR ANION AND CATION ANALYSES

The upstream water (U), drainage ditch water below stalactites (V), wetland tributary (W), downstream Black Creek (X), and farthest downstream Black Creek (Y) samples were sent to TestAmerica for quantitative analysis of major cations and major anions, and these data are displayed in Tables 3.7 and 3.8 and Figure 3.5. EPA methods conducted were as follows:
6010B—calcium, magnesium, potassium, and sodium analyses using inductively coupled plasma (ICP), 325.2—chloride (colorimetric, automated ferricyanide AAII), 340.2—fluoride (potentiometric, ion selective electrode), 375.4—sulfate (turbidimetric), and 310.1—alkalinity (titrimetric, pH 4.5). The major anions and cations in Table 3.7 were converted to milliequivalents per liter (Table 3.8) by the following formula:

$$\text{mEq/L} = (\text{Cation or Anion (mg/L)}) \times (\text{Absolute Value of the Charge of Cation or Anion}) / (\text{Atomic Weight of Cation or Anion (g/mol)}) \quad (10)$$

These converted values are plotted on a Piper diagram (Figure 3.5). Carbonate and bicarbonate concentrations were at or near minimum detection limits of 5 mg/L. The values are similar for all the samples with the exception of bicarbonate being much lower for sample drainage ditch waters below stalactite (V) at 5 mg/L and higher for the upstream Black Creek

waters (U) at 82 mg/L. The wetland tributary (W), downstream Black Creek (X), and farthest downstream Black Creek (Y) samples display similar cation and anion data values. Sulfate (93 mg/L) is highest in Sample upstream Black Creek waters (U) and lowest in drainage ditch waters below stalactite (V) at 14 mg/L, but both chloride and fluoride are highest for V (45 and 2.3 mg/L) and lowest for U (7.1 and 0.31 mg/L.) For the cations, sample V exhibits a significant spike in all major cations (calcium (1000 mg/L,) magnesium (5.7 mg/L,) potassium (100 mg/L,) and sodium (55 mg/L) compared to the other samples. Potassium (2.4 mg/L) is significantly lower in upstream Black Creek water (U) than all other samples (12 – 100 mg/L.)

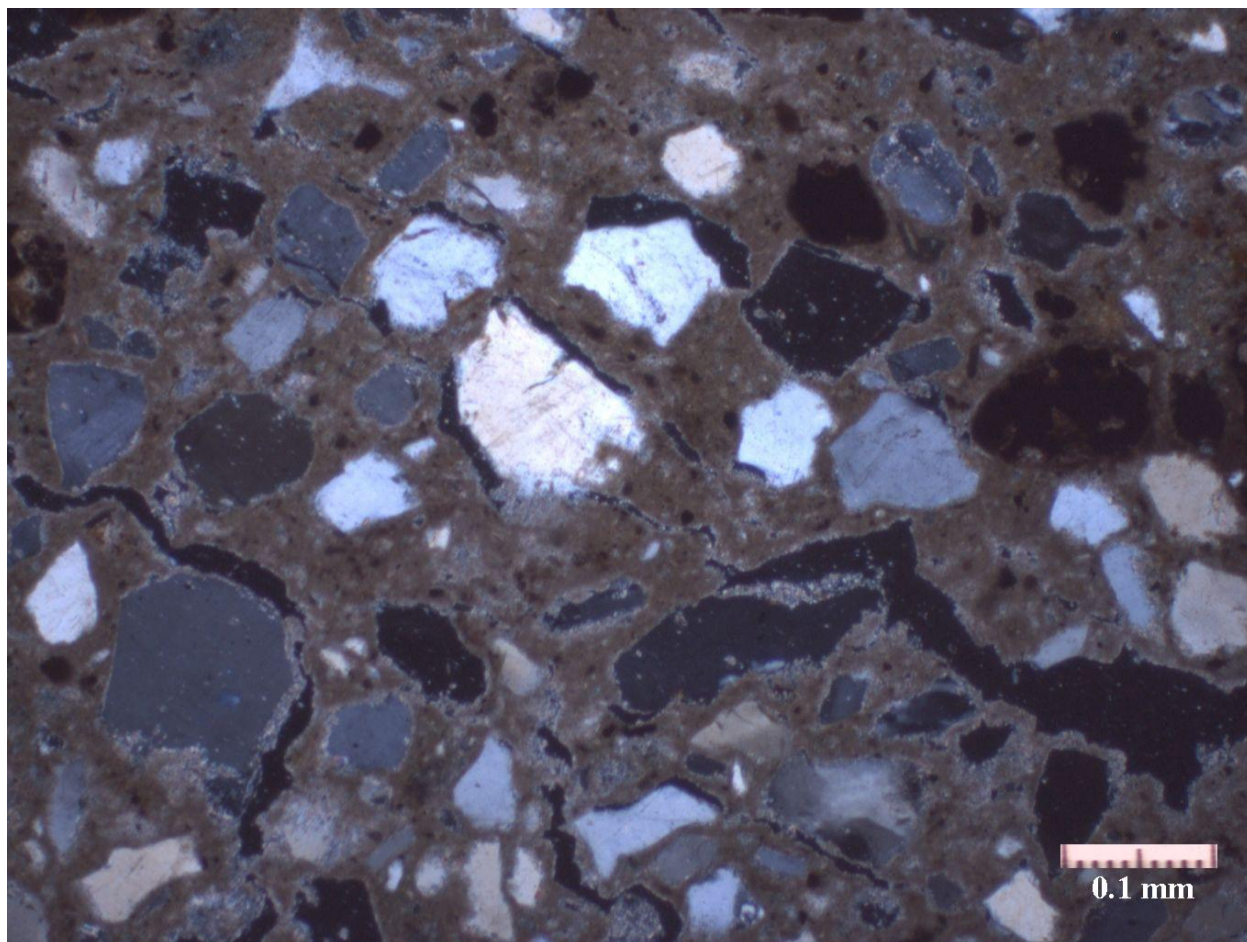


Figure 3.1 Petrographic photo of slag in thin section—quartz, vesicles, and amorphous material. Magnification is 10x.

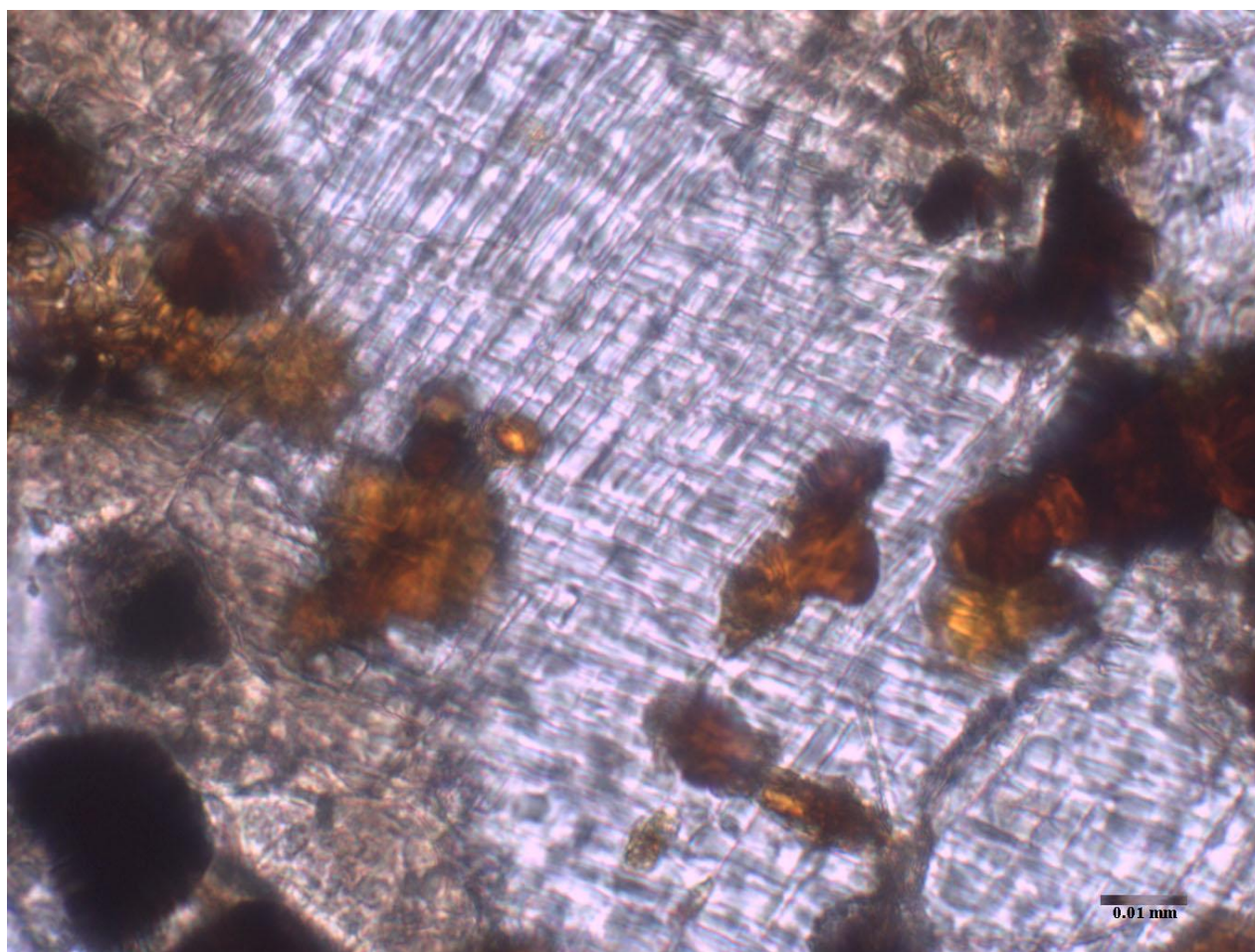


Figure 3.2 Petrographic photo of slag in thin section—calcite and unknown minerals.

Magnification is 40x.

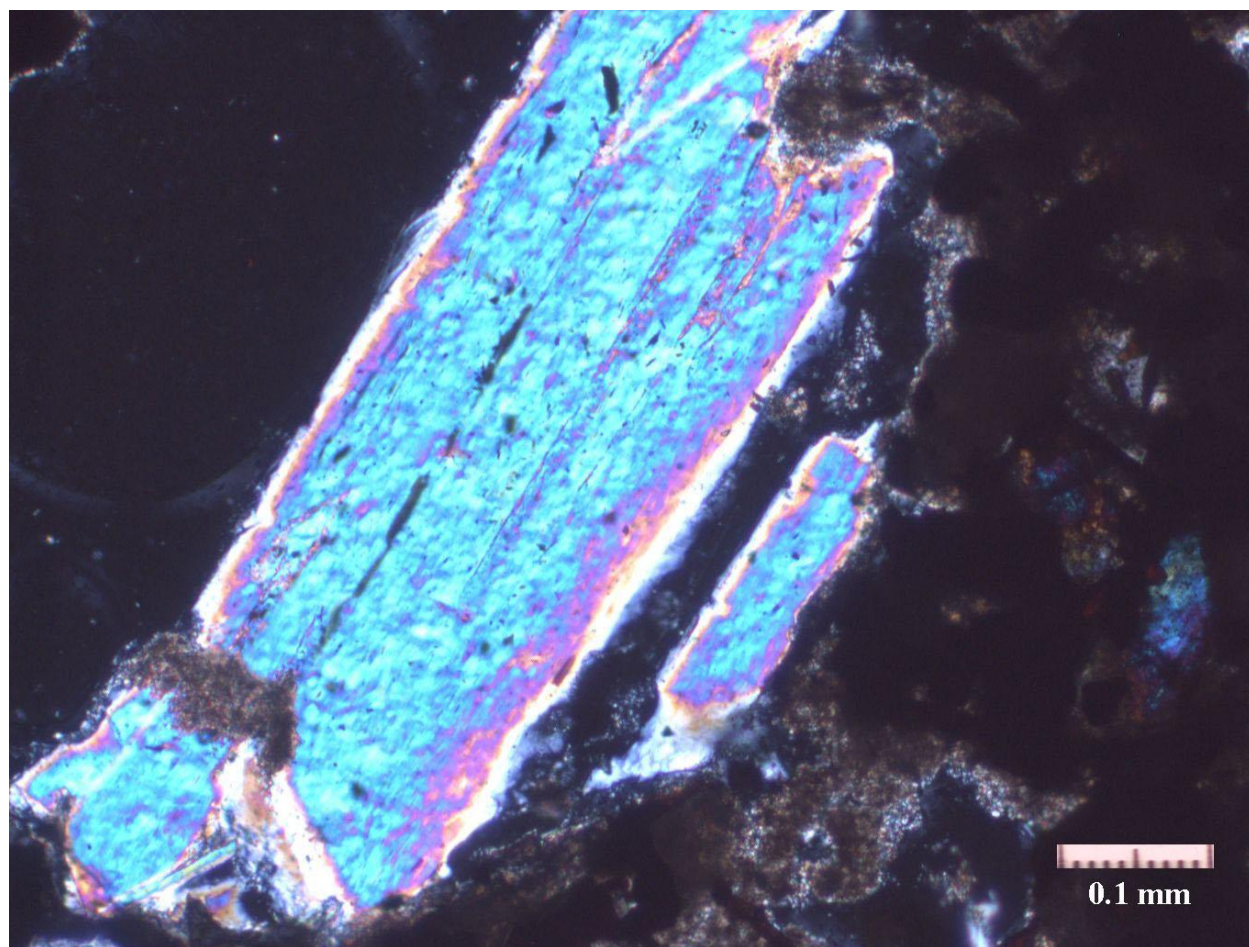


Figure 3.3 Petrographic photo of slag in thin section—unknown mineral, vesicles, and amorphous material. Magnification is 10x.

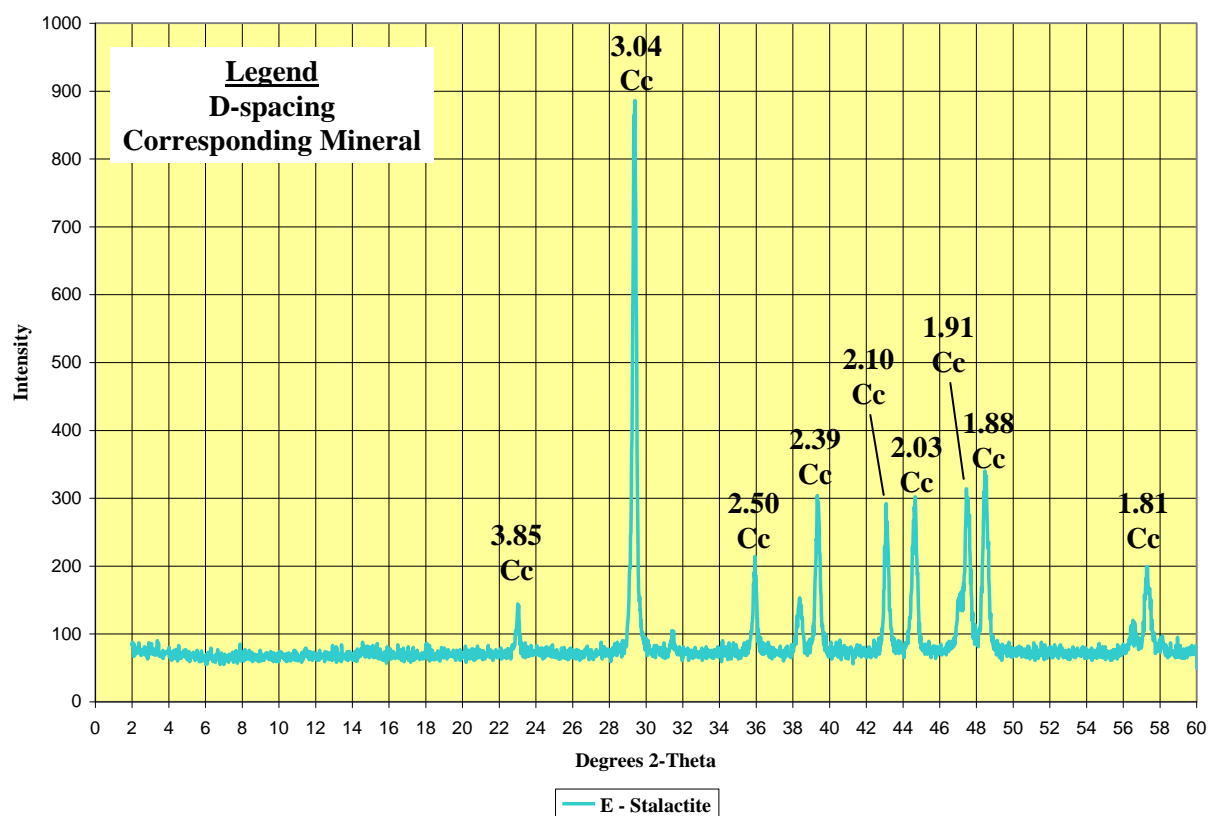


Figure 3.4 XRD reflections of the stalactite (E). D-spacings in Å. Cc refers to calcite.

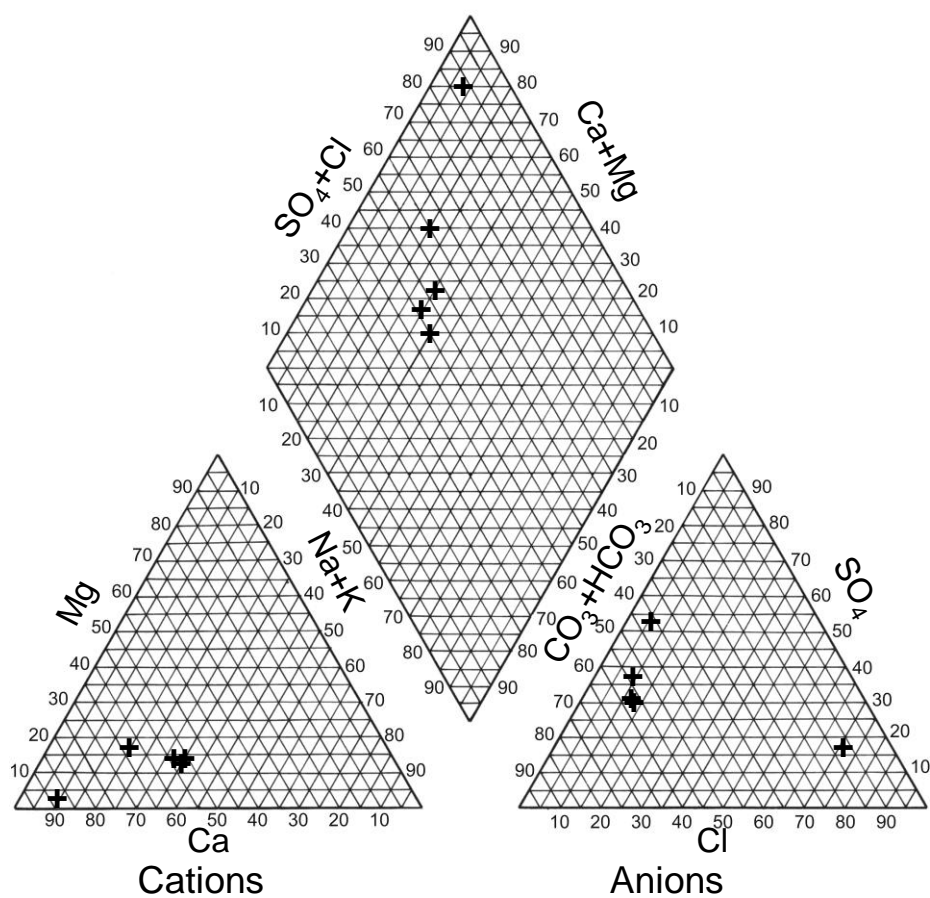


Figure 3.5 Piper Plot (in mEq/ L). Water samples U, V, W, X, and Y measured by TestAmerica.

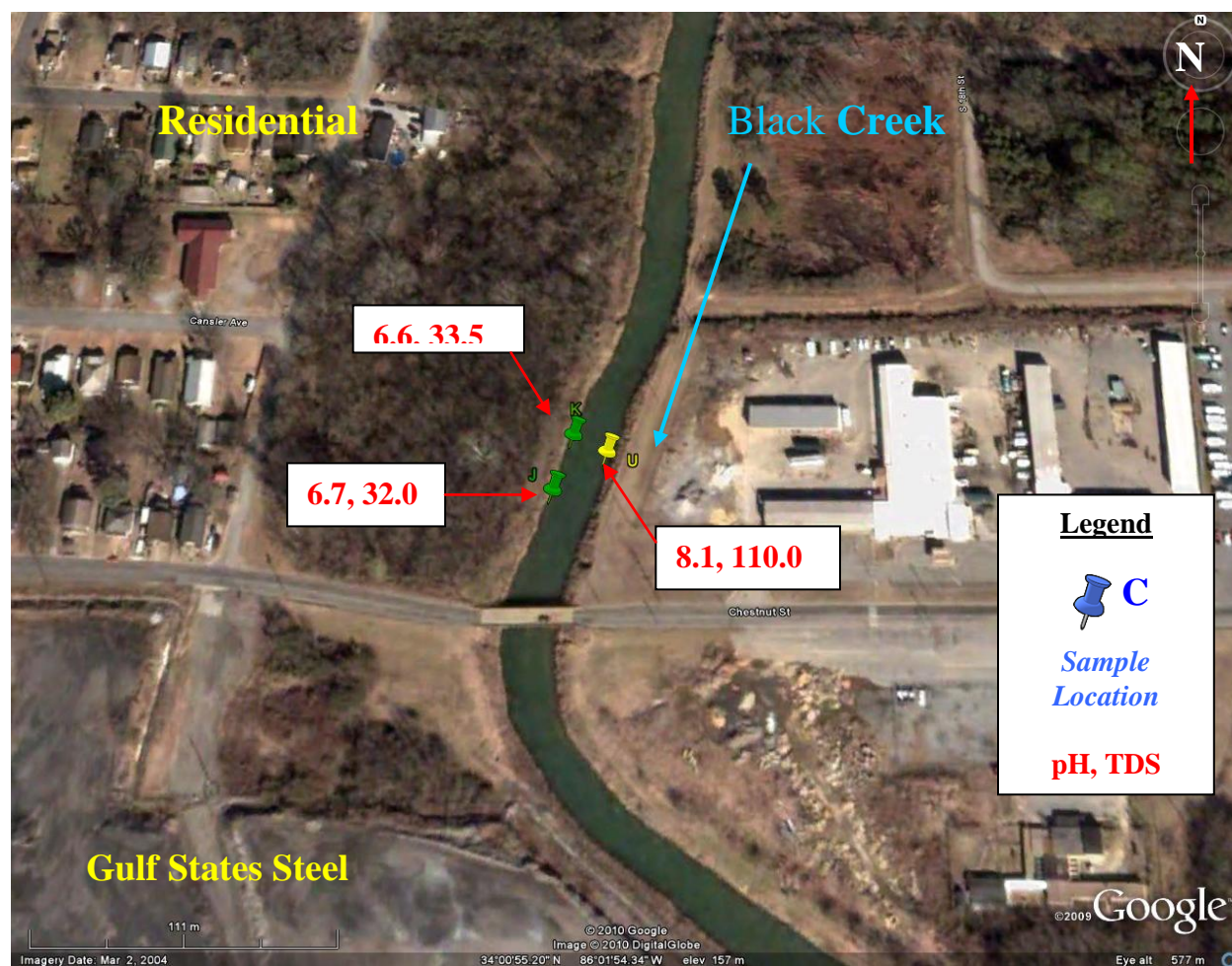


Figure 3.6 Location of upstream samples (J, K, and U) showing pH and approximate TDS.

Black Creek flows from North to South.

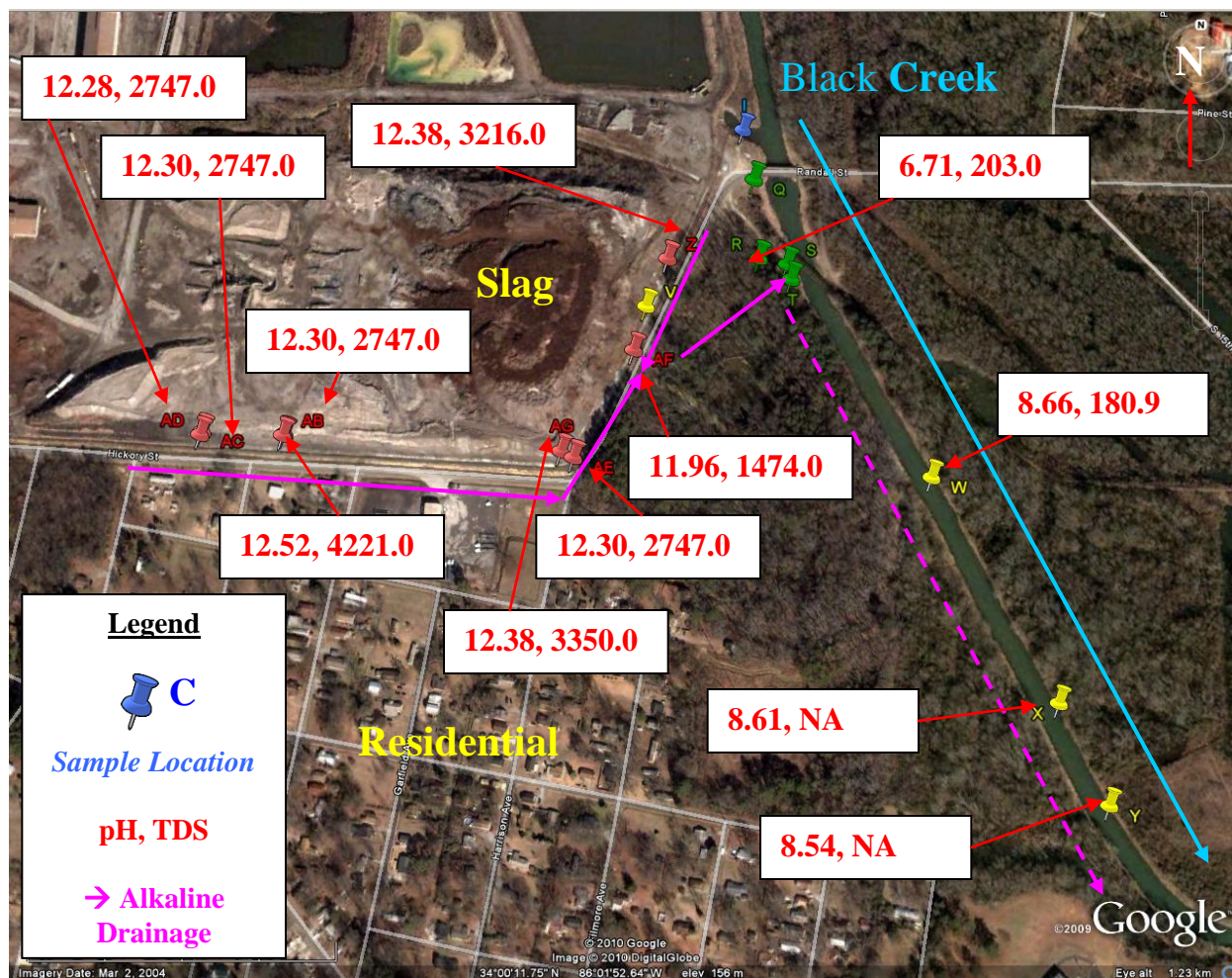


Figure 3.7 Alkaline drainage around the southeastern slag pile shown by magenta arrows. pH and approximate TDS data shown in red text for select samples.

Table 3.1 Stalactite (E) XRD analysis.

Significant peaks Observed	Degrees 2 theta	d-spacing (Å)	Intensity (Counts per Second)	Corresponding Mineral
1	23.06	3.85	142	Calcite
2	29.40	3.04	886	Calcite
3	35.94	2.50	214	Calcite
4	39.40	2.29	304	Calcite
5	43.08	2.10	292	Calcite
6	44.68	2.03	302	Calcite
7	47.46	1.91	314	Calcite
8	48.46	1.88	290	Calcite
9	57.32	1.61	199	Calcite

Table 3.2 The major oxide weight percents of the stalactite (E), slag (G-1 and G-2), and precipitate below the slag pile (H).

Major Oxide Weight Percent (weight %)	Stalactite (E*) (Weight %)	Slag (G-1) (Weight %)	Slag (G-2) (Weight %)	Precipitate below the Slag Pile (H*) (Weight %)
SiO ₂	0.30	19.89	20.4	0.42
TiO ₂	< 0.001	0.42	0.42	0.004
Al ₂ O ₃	0.04	1.55	0.42	0.06
Fe ₂ O ₃	0.06	22.47	22.38	0.22
MnO	0.002	2.49	2.49	0.016
MgO	0.03	3	2.98	0.11
CaO	54.87	39.72	39.52	54.81
Na ₂ O	0.1	0.26	0.26	0.12
K ₂ O	0.02	0.07	0.07	< 0.01
P ₂ O ₅	< 0.01	1.76	1.77	0.002
LOI	39.4	4.326	4.38	43.6
Total Percent	94.82	95.956	96.27	99.362

*Analyses conducted by Actlabs Incorporated, CANADA

Table 3.3 Lost on ignition (LOI) data for the slag samples (G-1 and G-2).

#	Crucible (g)	Weight of Crucible and Powdered Slag (g)	Initial Weight of Sample (g)	Weight of Crucible and Powdered Slag after Heating (g)	LOI (g)	LOI (%)
G-1	5.101	12.036	6.935	11.736	0.3	4.326
G-2	5.137	11.415	6.278	11.140	0.275	4.38

Table 3.4 pH, conductivity, and approximate TDS of all applicable samples.

Sample	Sample Description	pH	<i>In Situ</i> Conductivity (in $\mu\text{S}/\text{cm}$)	<i>Approximate</i> TDS (in ppm)++	T ($^{\circ}\text{C}$)
A	Drainage ditch water directly below the slag pile, outside the fence.	11.1	1640	1098.8	25
B	Drainage ditch sediment, outside the fence	Not Available	Not Available	Not Available	Not Available
C	Stagnant wetland water across the road from the slag pile.	11.9	1937	1297.8	25
D	Stagnant wetland encrustation.	Not Available	Not Available	Not Available	Not Available
E*	Stalactites, inside the fence. Samples made into both powder and slurry by ActLabs.	12.7	4750	3182.5	25
F	Drainage ditch sediments, inside the fence.	Not Available	Not Available	Not Available	Not Available
G	Slag sample, inside fence.	Not Available	Not Available	Not Available	Not Available
H*	White precipitate, directly below slag pile by second parking lot, inside fence.	11.2	264	176.9	25
I	Mouth of drainage into river.	8.5	327.5	219.4	25
J	Upstream water sample.	6.71	47.8	32	25
K	Upstream water sample.	6.55	50	33.5	25
L	SE Adjacent to the Slag Pile	11.98	3490	2338.3	25
M	Corner adjacent to slag pile	11.96	3930	2633.1	25
N	Wetland stagnant water.	11.26	1118	749.1	25
O	Trough adjacent to slag pile.	11.26	1115	747.1	25
P	Trough adjacent to slag pile.	11.61	1449	970.8	25

Sample	Sample Description	pH	<i>In Situ</i> Conductivity (in $\mu\text{S}/\text{cm}$)	<i>Approximate</i> TDS (in ppm)++	T ($^{\circ}\text{C}$)
Q	Black Creek at Hickory Road Bridge.	8.68	95	63.7	25
R	Stagnant Wetland.	6.71	303	203.0	25
S	Drainage Pipe into Black Creek.	8.22	130	87.1	25
T	South of Drainage Pipe.	7.78	124.5	83.4	25
U+	Upstream water.	8.09	220	110	25
V+	Ditch below stalactites.	>12	430	1300	25
W+	Tributary into Black Creek from the Marsh area.	8.66	270	150	25
X+	Center of Black Creek, downstream from South Slag.	8.61	Not Available	140	25
Y+	Center of Black Creek, farthest downstream.	8.54	Not Available	150	25
Z	Drainage ditch below the stalactite inside the fence.	12.38	4800	3216	14.3
AA	Seepage at Jackson & Hickory inside the fence.	12.52	6300	4221	20.7
AB	In ditch by seepage at Jackson & Hickory inside the fence.	12.39	5800	3886	20.1
AC	Telephone pole between Jackson and Main on Hickory inside the fence.	12.3	4100	2747	17
AD	Outside fence between Jackson and Main on Hickory.	12.28	4100	2747	18.6
AE	Outside fence at the bend by Hickory and Harrison.	12.27	4000	2680	19.3
AF	Moving water directly north from the drainpipe outside the fence.	11.96	2200	1474	15
AG	At the bend of Hickory and Harrison inside the fence.	12.38	5000	3350	16.9

*Analyses performed by ActLabs.

+Analyses performed by TestAmerica.

++Test America TDS analyses are quantitative, not approximate.

Table 3.5 pH titrations conducted at Georgia State University.

Sample	Trial #	Initial HCl (mL)	Ending HCl (mL)	Total .1 M HCl Used (mL)	Sample Used (mL)	pH
Z	1	1.3	7.2	5.9	24.3	12.4
Z	2	7.2	12.9	5.7	23.8	12.4
Z	3	13	18.8	5.8	24	12.4
AA	1	10.6	18.7	8.1	24.2	12.5
AA	2	19.2	27.2	8	23.6	12.5
AA	3	35.6	43.5	7.9	23.9	12.5
AB	1	19	25	6	23.9	12.4
AB	2	25	31.1	6.1	24.7	12.4
AB	3	31.1	37.1	6	24.3	12.4
AC	1	2.8	7.5	4.8	23.8	12.3
AC	2	7.5	12.3	4.8	24.2	12.3
AC	3	12.3	17	4.8	24.2	12.3
AD	1	17	21.7	4.7	24.1	12.3
AD	2	21.8	26.3	4.5	23.6	12.3
AD	3	26.5	31.1	4.7	24.2	12.3
AE	1	1.1	5.6	4.5	24.4	12.3
AE	2	5.6	10	4.4	23.6	12.3
AE	3	10	14.4	4.4	23.4	12.3
AF	1	18.1	20.3	2.3	24.5	12
AF	2	20.3	22.6	2.3	24.8	12
AF	3	22.6	24.8	2.2	25	11.9
AG	1	0.7	6.6	5.9	24.4	12.4
AG	2	6.6	12.4	5.8	23.8	12.4
AG	3	12.4	18.3	5.9	24.8	12.4

Table 3.6 Trace element analyses of stalactite (E) and precipitate below the slag pile (H).

Analyte Symbol	Unit Symbol	Detection Limit	Analysis Method*	Stalactite (E)	Leachate and Sediment (H)	Average Abundance in Earth's Crust (ppm)**
Au	ppb	1	INAA	< 1	< 1	<0.05
Ag	ppm	0.5	MULT INAA / TD-ICP	< 0.5	< 0.5	0.07
As	ppm	1	INAA	< 1	< 1	1.8
Ba	ppm	1	FUS-ICP	214	229	425
Be	ppm	1	FUS-ICP	< 1	< 1	2.8
Bi	ppm	0.1	FUS-MS	< 0.1	< 0.1	0.17
Br	ppm	0.5	INAA	0.9	1.3	2.5
Cd	ppm	0.5	TD-ICP	< 0.5	< 0.5	0.2
Co	ppm	0.1	INAA	0.8	1	25
Cr	ppm	0.5	INAA	13	7.7	100
Cs	ppm	0.1	FUS-MS	< 0.1	< 0.1	3
Cu	ppm	1	TD-ICP	3	13	55
Ga	ppm	1	FUS-MS	< 1	< 1	15
Ge	ppm	0.5	FUS-MS	< 0.5	< 0.5	1.5
Hf	ppm	0.1	FUS-MS	< 0.1	< 0.1	3
Hg	ppm	1	INAA	< 1	< 1	0.08
In	ppm	0.1	FUS-MS	< 0.1	< 0.1	0.1
Ir	ppb	1	INAA	< 1	< 1	<0.05
Mo	ppm	2	FUS-MS	< 2	< 2	1.5
Nb	ppm	0.2	FUS-MS	0.5	1.7	20
Ni	ppm	1	TD-ICP	77	3	75
Pb	ppm	5	TD-ICP	96	8	12.5
Rb	ppm	2	FUS-MS	< 2	< 2	90
S	%	0.001	TD-ICP	0.052	0.002	260
Sb	ppm	0.1	INAA	< 0.1	0.2	0.2
Sc	ppm	0.01	INAA	0.04	0.06	22
Se	ppm	0.5	INAA	< 0.5	< 0.5	0.05
Sn	ppm	1	FUS-MS	< 1	< 1	2
Sr	ppm	2	FUS-ICP	179	468	375
Ta	ppm	0.1	FUS-MS	< 0.1	< 0.1	Not Available
Th	ppm	0.05	FUS-MS	< 0.05	< 0.05	9.6
U	ppm	0.05	FUS-MS	< 0.05	0.08	2.7
V	ppm	5	FUS-ICP	18	26	135
W	ppm	1	INAA	< 1	< 1	1.5

Analyte Symbol	Unit Symbol	Detection Limit	Analysis Method*	Stalactite (E)	Leachate and Sediment (H)	Average Abundance in Earth's Crust (ppm)**
Y	ppm	1	FUS-ICP	< 1	< 1	33
Zn	ppm	1	MULT INAA / TD-ICP	30	51	70
Zr	ppm	1	FUS-MS	2	2	165
La	ppm	0.05	FUS-MS	< 0.05	0.09	25
Ce	ppm	0.1	FUS-MS	< 0.1	0.2	67
Pr	ppm	0.02	FUS-MS	< 0.02	0.02	6.5
Nd	ppm	0.05	FUS-MS	0.05	0.09	28
Sm	ppm	0.01	FUS-MS	< 0.01	0.02	7.3
Eu	ppm	0.005	FUS-MS	0.006	0.01	1.2
Gd	ppm	0.02	FUS-MS	< 0.02	< 0.02	7.3
Tb	ppm	0.01	FUS-MS	< 0.01	< 0.01	1.1
Dy	ppm	0.02	FUS-MS	< 0.02	< 0.02	5.2
Ho	ppm	0.01	FUS-MS	< 0.01	< 0.01	1.5
Er	ppm	0.01	FUS-MS	< 0.01	< 0.01	2.8
Tl	ppm	0.05	FUS-MS	< 0.05	< 0.05	0.45
Tm	ppm	0.005	FUS-MS	< 0.005	< 0.005	0.25
Yb	ppm	0.01	FUS-MS	< 0.01	0.01	3
Lu	ppm	0.002	FUS-MS	< 0.002	< 0.002	0.8
Mass	g		INAA	1.262	1.253	Does Not Apply

*Key for analyses methods is as follows: FUS-ICP - Fusion-Inductively Coupled Plasma , FUS-MS - Fusion-Inductively Coupled Plasma-Mass Spectrometry, INAA - Instrumental Neutron Activation Analysis, and TD-ICP - Total Digestion- Inductively Coupled Plasma-Optical Emission Spectrometry.

**Average crustal abundances listed are according to Krauskopf (1967).

Table 3.7 Major anions and cations in (mg/L) from TestAmerica.

Anion or Cation	Sample ID (mg/ L)					Detection Limit (mg/L)
	U	V	W	X	Y	
Ca ²⁺	26	1000	26	26	25	0.5
Mg ²⁺	4.7	5.70	4.4	4.4	4.2	0.5
K ¹⁺	2.4	100	14	13	12	1
Na ¹⁺	8.7	55	10	9.9	9.4	1
SO ₄ ²⁻	93	14	41	39	40	10
Cl ¹⁻	7.1	45	9.9	9.2	9.3	1
F ¹⁻	0.31	2.3	0.64	0.63	0.63	0.2
CO ₃ ²⁻	5	5	5	5	5	5
HCO ₃ ¹⁻	82	5	69	69	69	5

Table 3.8 Major anions and cations (Table 3.7) converted to mEq/L.

Anion or Cation	Sample ID (mEq/ L)				
	U	V	W	X	Y
Ca ²⁺	1.30E+00	4.99E+01	1.30E+00	1.30E+00	1.25E+00
Mg ²⁺	3.87E-01	4.69E-01	3.62E-01	3.62E-01	3.46E-01
K ¹⁺	6.14E-02	2.56E+00	3.58E-01	3.32E-01	3.07E-01
Na ¹⁺	3.78E-01	2.39E+00	4.35E-01	4.31E-01	4.09E-01
SO ₄ ²⁻	1.94E+00	2.91E-01	8.54E-01	8.12E-01	8.33E-01
Cl ¹⁻	2.00E-01	1.27E+00	2.79E-01	2.59E-01	2.62E-01
F ¹⁻	1.63E-02	1.21E-01	3.37E-02	3.32E-02	3.32E-02
CO ₃ ²⁻	1.67E-01	1.67E-01	1.67E-01	1.67E-01	1.67E-01
HCO ₃ ¹⁻	1.34E+00	8.19E-02	1.13E+00	1.13E+00	1.13E+00

CHAPTER 4—DISCUSSION

The major elemental composition of the slag is unlike that of common rocks and minerals. The slag analyzed in this study has high CaO and MgO—totaling 42.72% for G-1 and 42.5% for G-2—and low in SiO₂ and Al₂O₃—totaling 21.44% for G-1 and 20.82% for G-2. Vesicles are also abundant, which makes slag appear to be similar to an extrusive volcanic rock. The high CaO and MgO content of slag removes sulfur during smelting to produce S-free iron (Richardson and Fincham, 1954).



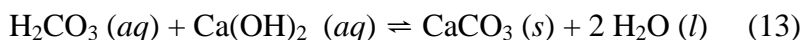
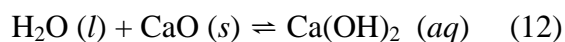
The presence of the stalactites on the vertical surface of the slag, the white coated streams and wetland, and alkaline waters (pH of 8 to 12.5) nearby are distinct features of this GSS site. The stalactites consist of calcite as observed in the X-ray diffraction data (Figure 3.4). This identification is supported from the major element analyses of the stalactites (Table 3.2). The stalactites did not sequester appreciable trace elements while they were formed. The stalactites are forming presently.

The other distinct feature is the presences of high approximate total dissolved solids (1098.8 to 4221 mg/L, Table 3.4) and high pH (12.5) found in trenches adjacent to the slag pile (Samples A, C, L, M, O, P, V, Z, AA, AB, AC, AD, AE, AF, and AG). In terms of commonly measured anions and cations in waters, the high TDS (ranging from 110 to 1300 mg/L) is composed of high carbonate (bicarbonate) and high dissolved Ca (Table 3.4 and Table 3.7).

However, compared to the wetland and Black Creek samples that have been affected by high biological activity in the summer months, the drainage ditch (V) shows elevated Ca, K, Na, and Cl and depleted sulfate and bicarbonate. This drainage attenuates quickly as it migrates through soils as noted by the change in conductivity between samples V and W. The drainage, once thought to have impacted the nearby Black Creek, no longer flows into Black Creek (Figure 2.3) as evidenced by the low conductivity values and near neutral pH (Table 3.4) for waters in flowing through carbonate terrains.

A simple model is proposed for the formation of the alkaline pH, high conductivity waters and the stalactites based on the data gathered in this study and consistent with prior ideas given in the Introduction. The reaction of water and a CaO and MgO slag produces strong bases such as Ca(OH)_2 or Mg(OH)_2 , as noted by Yan et al (2000). The solution forming here is most likely composed of Ca(OH)_2 based on the high content of CaO in slags. This solution originates from the reaction of rainfall with slag.

The presence of these calcite stalactites on the surface of the slag led us to propose that this Ca(OH)_2 base is reacting with runoff containing some carbonic acid. Carbonic acid dissociates to carbonate anions (CO_3^{-2}) and combines with Ca^{+2} to form the calcite stalactites. The sequence is summarized below.



The rate limiting constituent for forming the stalactite and perhaps the white surface coating seen in the adjacent wetlands and drainage ditches is the amount of CO_2 coming from

either rainfall or the atmosphere directly and the availability of free Ca ions from the slag. In terms of Ca availability, the formation of a thin Fe-rind (the brownish rind seen on the slag) after weathering may inhibit further reaction of the slag with rainwater. Huijgens and Comans, (2005), note that the presence of oxides limits slag carbonation reactions. In terms of availability of CO₂, if limestone is present in the slag pile as is possible given other common minerals are being found in slag (see Chapter 3, where calcite, quartz, and possibly muscovite are found in thin section), then it is possible that limestone is present, especially since the bedrock is the Conasauga Formation. The dissolution of limestone is a potential source of CO₂ in the waters percolating through the slag. Otherwise, the source of CO₂ is likely atmospheric. It is possible that the measurement of both $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ from calcite as done by Renforth et al (2009) might differentiate carbonates resulting from limestone versus those formed from high pH solutions.

The flux of the high pH drainage however is related to the amount of slag being dissolved by surface or rainwater and its flow into the area wetlands and perhaps Black Creek. It is fortunate that most of the drainage does not make it to Black Creek; however, some of the drainage does flow directly into the wetlands and then into Black Creek. Most of the drainage inside the fence flows to an onsite GSS reservoir; however during times of heavy rainfall, drainage flows directly into the wetland along with the drainage from the ditches outside of the fence. Apparently, both pH and TDS are strongly attenuated by flowing into the urban soils in this area (see pH and conductivity of samples in Table 3.4) such that the adjacent waters in Black Creek presently are not impacted by the drainage inside the fence. This attenuation is evident by the pH measurements and conductivity measurements in the creek upstream from the slag piles, at the slag piles and downstream from the slag piles (Table 3.4). The pH of Black Creek varies

from 6 to 8 and consistent with it being underlain by the Conasuauga Formation (a limestone in this area). The compositions of these waters are bicarbonate dominant waters (Figure 3.5).

While the presence of high pH drainage, white coated wetlands and streams nearby, are distinct features, such features are observed elsewhere, resulting from a similar dissolution of steelmaking slags. Mayes et al (2006) found pH levels of 12 and greater in leachate draining through a basic slag used as cover in a derelict land near a steelmaking district at Consett, County Durham, England (Renforth et al (2009)). Renforth et al also noted a calcareous hardpan of 400 km² adjacent to a calcareous wetland. The calcareous wetland and hardpan appear to be comparable to similar observed features in the ditches and wetlands adjacent to the slag pile at the GSS site. As mentioned earlier the active drainage of high pH water appears to be attenuated at present by a small soil berm of a few feet in width and connection to the wetlands is active only during rainfall and after periods of heavy rain.

The results of this study potentially point to this reaction process being able to sequester or remove atmospheric CO₂ and store it in the calcite stalactites. These stalactites could then be processed to form cement releasing CO₂ or used as lime in agricultural areas (especially if further study shows that trace elements are not sequestered while these stalactites are formed). This concept of slag carbonation reactions and CO₂ sequestration was not discussed in the initial stages of this work. The removal of CO₂ by weathering basic rocks is possible and has been discussed lately in a session of papers at the National GSA meeting in Portland, OR (e.g. Matter et al., 2009). In this study, the removal of CO₂ apparently results from the formation of calcite stalactites actively forming on these slag piles via the removal of Ca from the slag followed by calcite precipitation. Huijgen et al (2005) and Huijgen and Comens (2006) also find the carbonation of steelmaking slag (i.e. formation of calcite by reaction of slag and water) as a

possible way to remove CO₂. Given the large quantities of slag conceivably generated each year (approximately 21 million tons in the United States, Proctor et al (2000)), the carbonation reaction in slag, soils, and the rock record are promising and useful topics for continued study.

CHAPTER 5—CONCLUSION

- 1) The stalactite precipitating on the slag piles came from the calcium trapped in the glassy matrix of the slag and was released when contacted with acidic rain and CO₂.
- 2) The exposed slag at the GSS site releases highly alkaline drainage that results in carbonate-rich leachate coating surrounding sediments. Placing a berm of soil down gradient from the slag appears to mitigate the high alkalinity until the soil becomes saturated with leachate, forming a hardpan. The operator of the GSS site appears to have placed a berm of soil around the site in the past; however, the soil layer has been both breached and saturated. As a result, visible leachate thoroughly coats the drainage ditches and migrates well beyond the property limits where the public could easily come into contact with it. It is recommended that soil barriers be monitored and maintained on a frequent basis around sites that have exposed slag or slag aggregates. Contaminated soil around sites such as these could be removed and used to mitigate acidic drainage.
- 3) Future research should be conducted to determine the feasibility of utilizing slag as a potential carbon sink for CO₂ sequestering.

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