# CHEMICAL CHARACTERIZATION OF RECYCLED CONCRETE AGGREGATES USING A HANDHELD X-RAY FLUORESCENCE DEVICE

by

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A Thesis submitted to the faculty of
The University of North Carolina at Charlotte
for the degree of Master of Science in
Construction and Facilities Engineering

Charlotte

2020

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# ABSTRACT

ARINDAM DEY**.** Chemical Characterization of Recycled Concrete Aggregates Using a Handheld X-Ray Fluorescence Device**.** (Under the direction of DR. TARA CAVALLINE)

Recycled concrete aggregates (RCA) are an important source of material that can be used in construction to mitigate the problems associated with use of virgin aggregates. The volume of waste generated by the construction industry is increasing at a rapid pace with demolished concrete forming a major part of the total waste. Aggregates are the largest component of concrete, and conventional sources of natural aggregates are diminishing due to environmental and cost considerations. Increased use of RCA in lieu of virgin aggregates slows the depletion of non-renewable natural resources and produces a more environmentally friendly infrastructure as our nations strive to progress towards a more sustainable future.

Globally, a large amount of infrastructure construction is planned and underway. One potential way of reducing the consumption of natural aggregates is to replace natural aggregates with RCA extracted from construction and demolition (C&D) waste. However, the use of RCA in concrete applications comes with its own set of challenges due to RCA's non-homogenous composition. Produced by crushing concrete, RCA includes both the virgin aggregate from the original concrete and the adhered mortar. Many of the properties of RCA are influenced by the presence of the mortar adhered to its surface. Because of this, use of RCA has historically been mostly limited to non-structural concrete applications like construction of gutters, pavements and small retaining or barrier walls. RCA is also often used in applications such as in pavement foundations (unbound and bound bases and subbases).

Primary barriers to increased use of RCA include its variability in composition, including the residual mortar content, and the potential to contain contaminants which can negatively affect the properties of concrete (such as chlorides or sulfates) or present environmental concerns (such as heavy metals) (Snyder et al. 2018). Chemical characterization of RCA and determination of residual mortar content prior to use in new concrete or other infrastructure applications provides critical information to users, providing confidence in its usability and potential impacts on the performance of new concrete or base materials.

To date, no method of rapid chemical characterization of RCA has been developed and accepted. Some of the existing methods implemented in the industry for elemental analysis of samples include atomic absorption spectrometry (AAS), Neutron Activation Analysis (NAA) and inductively coupled plasma-optical emission spectrometry (ICP-OES) which are non-destructive testing techniques that each require a high investment cost and are time consuming. To overcome the short comings of the aforementioned methods, this research study proposes the use of a portable handheld XRF (PHXRF) for chemically characterizing recycled concrete aggregates and determining the residual mortar content of the samples. In this study the PHXRF was used for collecting quantitative data from the elemental analysis of RCA samples acquired from four different sources (highway, airfield pavement, and C&D waste) across the state of North Carolina.

The results of this quantitative PHXRF data were compared against XRF “whole rock analysis” results for the validation of PHXRF. First, the actual mortar content of the RCA samples was determined using the thermal shock method and then a stepwise regression was performed on the PHXRF results based on size (No.4, No.12 & No.50) to determine a regression model to help compute the predicted values of the mortar content. The predicted mortar content value was compared against the mortar content determined through laboratory testing to determine the accuracy of the model in predicting the mortar.

The second objective of the research was to determine the accuracy of the PHXRF device and choose the best representative size for PHXRF analysis. This objective was achieved by comparing the PHXRF results of the RCA samples with the whole rock analysis test results and using simple linear regression to observe the $R^2$ values for each element. Based on the $R^2$ value, the most appropriate size of the aggregate used for the test was determined, and the regression equation of this size RCA was used to compute the predicted weight % of major and trace elements.

# ACKNOWLEDGEMENTS

I would like to extend my gratitude and appreciation to Dr. Tara Cavalline for believing in my capabilities and granting me the opportunity to work on this research project. Special thanks to my family for supporting me throughout this journey and providing me with the monetary support to fulfil my dreams. I would like to thank my research team Dr. Jiong Hu and Miras Mamirov from the University of Nebraska-Lincoln for their significant contributions towards this research. I would also like to thank my committee members Dr. Jacelyn Rice-Boayue, Dr. Nicole Barclay, and Dr. Don Chen for their advisory and guidance and my research team Mr. Wesley Maxwell, Akshay Bansal and Allison Summers for assisting me in the laboratory portion of this research. Lastly, special thanks to the American Concrete Institute (ACI) Concrete Research Council (CRC) for donating time and providing the funding to support this research study.

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| | |
|---|---|
| ASR | alkali silica reaction |
| ASTM | American Society for Testing and Materials |
| AA | Atomic Absorption |
| COV | coefficient of variation |
| CLT | Charlotte-Douglass International Airport |
| HXRF | handheld X-Ray fluorescence |
| ICP-MS | Inductively Coupled Plasma-Mass Spectrometry |
| ICP-AES | Inductively Coupled Plasma- Atomic Emission Spectrometry |
| IR | Infrared Spectrometry |
| LOI | Loss on Ignition |
| PHXRF | portable handheld X-ray fluorescence |
| QA/QC | quality assurance/quality control |
| RCA | recycled concrete aggregates |
| $R^2$ | coefficient of determination |
| RMC | residual mortar content |
| RMSD | root mean square deviation |
| SC | stabilizer content |
| SCM | supplementary cementitious materials |
| XRF | X-Ray fluorescence |

# CHAPTER 1: INTRODUCTION

The use of recycled concrete aggregates (RCA) in construction is an important step towards a more sustainable infrastructure. RCA is a product manufactured from existing concrete elements, and due to its varying composition, is often linked to questions regarding its quality. Potential users often cite a lack of knowledge about its composition and appropriateness for use in new applications such as unbound base material or as aggregates in new concrete (Cackler 2018; McNeil and Kang 2013). Specifically, the physical, mechanical and chemical properties and characteristics of RCA can vary widely based upon source concrete quality and production techniques. These properties and characteristics are important to know when determining the suitability of using RCA in new unbound or concrete applications.

Currently, several ongoing research studies are aiming to develop a protocol to rapidly and accurately characterize RCA, and to develop specifications and procedures to promote increased use of RCA. In line with these efforts by many stakeholders interested in promoting RCA use, the scope of this research study is to identify an effective method for chemical characterization of RCA.

The research study proposes the use of handheld XRF (PHXRF) to utilize x-ray fluorescence for chemical characterization of RCA. X-ray generation and detection technology has evolved over the last decade leading to the development of portable HXRF units (Shugar and Mass 2012). XRF is a non-destructive destructive analytical technique used to determine the elemental composition of material. It has been widely used in the construction, geology, medical, metallurgy, environmental and mining industries. The device operates on the principle that when a material sample is subjected to an X-ray beam, electrons from the atom are ejected from their atomic orbital positions causing instability inside the atom (Bruker 2019). In order to correct this instability, electrons from a higher orbit move down to the lower orbit to fill the vacancy. This transmission of electron into a lower orbital causes emission of fluorescent radiation which is unique for each element. The detector present in the XRF instrument detects this release of energy and then categorizes the elements by energies.

The handheld XRF can thus be useful for rapid identification and quantification of elements present in RCA and has the capability of identifying most elements heavier than magnesium. Therefore, a chemical analysis of RCA samples from the PHXRF would provide information about its chemical composition and properties that could be useful to designers, contractors, agencies, and other stakeholders. It would also provide an input on the presence of contaminants detrimental to structural performance (such as chlorides and sulfates), its alkali-silica reaction (ASR) potential, and the quantity of mortar adhered to its surface which will impact the performance of concrete produced with it.

Being non-destructive in nature, the PHXRF analyzer can prove to be beneficial over some of the existing methods like Inductively Coupled Plasma-mass spectrometry (ICP) and Atomic Absorption (AA) spectrometry for elemental analysis of samples as they are destructive in nature, time consuming, and also give rise to inaccuracies caused due to improper dissolution or digestion. The PHXRF analyzer's portability, coupled with its ability to produce results within a mere few seconds by simply pointing and shooting at samples makes it a very useful testing technique. The PHXRF analyzer, due to its rapid and non-destructive nature has the potential to support quick and robust testing of RCA samples for use in quality assurance (QA) or quality control (QC) examination. Thus, the main objective of this research is to assess the effectiveness and feasibility of using PHXRF for characterization of RCA chemical composition and for validating and developing a testing protocol for RCA samples, and suggest a testing protocol for future use of the method.

# CHAPTER 2: LITERATURE REVIEW

## 2.1 Recycled Concrete Aggregate

Humankind is affected by issues of sustainability due to large scale consumption of natural resources and other contributing factors. Sustainable engineering will soon become a part of decision making across various industries (Hiller et al. 2011). The construction industry is one of the largest consumers of natural resources due to rapid industrialization and urbanization. It produces large amount of construction waste in the form of bricks, wood, steel, concrete, and other materials. Of all the construction materials, the concrete industry is the largest consumer of natural resources (Behera et al. 2014). The consumption of natural resources can have a negative impact on the environment, energy, and economies. Since, concrete is approximately 75% aggregates, the use of recycled aggregate instead of virgin aggregates is an economically viable and a sustainable decision, since concrete can be manufactured with aggregates obtained from C&D waste materials by either partial or complete replacement of virgin aggregates (Van Dam et al. 2015).

The aggregates obtained by crushing parent or old concrete from construction wastes such as demolished buildings, highways and other structures are called recycled concrete aggregates (RCA). At present, the applications of RCAs primarily extend to pavement base course, fill material, and soil stabilization, although increasingly, RCA is being used as aggregates in hot-mix asphalt or portland cement concrete (Snyder et al. 2018). The underutilization of RCA in new concrete is primarily due to its material properties that can negatively affect the fresh and hardened properties of concrete (Abbas et al. 2009). The presence of residual mortar in RCAs is typically the primary reason behind its inferior strength. Residual mortar affects the mechanical and durability properties of recycled aggregate concrete, which is why special consideration and adequate QC is required when producing concrete using RCAs (Snyder et al. 2018). The variability of RCA, particularly when produced from crushed concrete obtained from two or more sources, can also be a barrier to increased use in new concrete (ACPA 2008a).

### 2.1.1 Production

The production process of RCAs begins with the reduction of the source concrete into smaller fragments by removing contaminants such as plastic, wood, reinforcing steel, and other components, followed by different levels of screening and sorting (Behera et al. 2014). The first step towards production of good quality RCA for use in bound (such as asphalt or concrete) or unbound applications (such as base or fill material) is identifying its source, its constituents, and the amount of mortar adhered to it. Having a sound knowledge of the constituents of RCA can be helpful in assessing the quality of RCA and determining RCA's fitness for potential use in lower grade applications (such as unbound base materials) and higher-grade applications (such as new concrete).

The concrete structures are reduced to smaller sizes by either impact breakers or resonant breakers to prepare them for crushing. Breaking is also done to ensure debonding of reinforcing steel from existing concrete. Additional contaminants such as dowel bars, wire mesh, steel reinforcements, and other materials are removed by electromagnetic separators. The broken concrete might be subjected to further manual screening in case the contaminants are not completely eliminated (Fick 2017).

After the breaking operation, the fractured concrete is crushed using three different types of crushers (jaw, cone and impact crushers). These crushers typically reduce the size of concrete to a level that can be used as concrete. The concrete is first subjected to a primary crusher (often a jaw or a cone crusher) followed by secondary crushing (typically by cone or impact crushers) which reduces the size of the concrete which is uniformly distributed. Occasionally, to meet gradation requirements, final crushing is performed with an impact crusher which removes a significant percentage of adhered mortar from the original aggregates (Hiller et al. 2011).

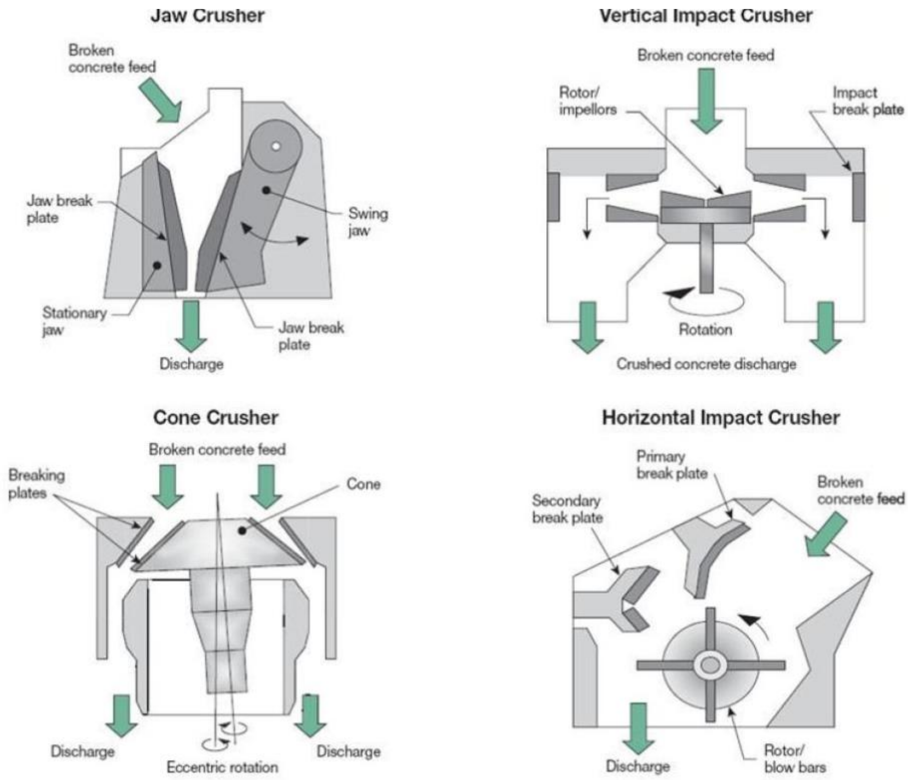



Figure 2.1 Different Types of Crushers (Hiller et al. 2011)

The crushing operation usually leads to the production of large amount of top-sized aggregate and fine material. The dearth of mid-sized materials causes difficulty in achieving gradation specification and can be overcome by additional sieving (Hiller et al. 2011).

2.1.2 Composition and Characteristics:

The quality of the original concrete sourced for use as RCA has a strong influence on the properties of RCA. In particular, the characteristics and composition of RCAs affect the performance of RCA concrete and hence, it becomes necessary to ascertain their composition and characteristics because the history and properties of the source concrete are unknown (Silva et al. 2014). Sometimes, particularly with RCA sourced from non- state highway agency sources or from local crushing and grading facilities, there is a lack of information about the environment and conditions the original materials were exposed to (Snyder et al. 2018). Due to this pronounced difference between the RCA and aggregates obtained from natural resources and its complicated nature, primarily because of the cement mortar attached to the RCA, a thorough understanding of its physical/geometrical properties and chemical composition is vital (Hiller et al. 2011).

2.1.2.1 Physical Properties:

RCAs have several physical similarities and dissimilarities compared to virgin aggregates. Their physical properties include, but are not limited to, mortar content, specific gravity, absorption capacity, and soundness. A brief description of each of these properties along with some supporting information is presented in the following list.

i. Mortar Fraction: The most distinguishing difference between RCA and natural aggregates is the presence of two different materials in RCA. RCA is composed of coarse aggregate and cement mortar attached to its surface originating from the parent concrete (de Juan and Gutiérrez 2009). The mortar clinging to the surface of RCA is generally composed of fine aggregate, hydrated cement particles, and unhydrated cement particles, along with the hardened pore and void system from the original source concrete (Behera et al. 2014). Most of the negative properties and performance associated with RCA have been attributed to the presence of this cement mortar. The attached mortar can have a negative influence on some of the aggregate's important properties like density, absorption, and specific gravity. Of these, the increased absorption of the RCA is often cited as most problematic to use of RCA in new concrete (Snyder et al. 2018). Moreover, the presence of RCA in a concrete mixture creates a light concrete system due to the high porosity and less dense nature of the mortar adhered to the aggregate matrix (Verian et al. 2018).

ii. Particle shape and texture: Due to the crushing operation, RCA generally tends to have a poorer particle size distribution than conventional aggregates (Behera et al. 2014). The presence of adhered cement mortar and the crushing operations result in RCA particles tending to have an irregular, angular shapes with a very rough surface texture. The mortar content can vary from 30 to 60% depending on the aggregate size. Generally, the amount of finer fraction material in RCA is more and RCA tends to have more mortar attached to their surface (Safiuddin et al. 2013). The shape and size of RCAs are largely influenced by the type of crushing devices used. Jaw crushing (typically used as the primary stage crusher) has been known to provide good grain-size distribution of RCA giving them an angular shape, whereas cone crushers (often used in the secondary crushing stage) give a more spherical shape to RCA. The presence of minute pores in the adhered mortar also leads to the development of cracks and fissures inside the aggregates during the crushing operation.

iii. Specific Gravity: The specific gravity of RCA is greatly influenced by the mortar adhered to it. The percentage of adhered mortar tends to increase as the size of the aggregate reduces thereby reducing the specific gravity due to the voids present in the mortar making it less dense. The specific gravity of RCA is lower than that of natural aggregate because of the attached mortar's lower density and greater porosity (Verian et al. 2018). Finer RCA particles in concrete can increase its water demand which would reduce its workability. It can also lead to other issues such as low modulus of elasticity, low fracture resistance and high drying shrinkage in new concrete (Hiller et. al 2011).

iv. Absorption Capacity: The absorption capacity of RCA is higher than natural aggregates due to the adhered mortar on its surface (Hiller et al. 2011). As the presence of adhered mortar increases the porosity of the RCA also increases, causing an increase in the absorption capacity. When RCA is used in new concrete, the higher absorption capacity increases the water demand of the mixture, which (if unaddressed using other methods such as water reducing admixtures) can lead to a higher w/c ratio, a weaker interfacial transition zone (ITZ), poor fracture resistance, and lower strength (Snyder et al. 2018).

v. Soundness: The durability of an aggregate can be partially predicted by performing soundness testing on it (Hiller et al. 2011). Several types of soundness tests exist, but the durability of RCA is often assessed by subjected it to repeated cycles of freezing and thawing in a sodium chloride solution (Verian et al. 2013). The mass loss is calculated after every cycle, which provides an indication of the aggregate's resistance to disintegration by freezing and thawing. The level of deterioration of RCA tends to be higher than virgin aggregates due to higher mass loss when subjected to freezing and thawing cycles (Verian et al.2018).

2.1.2.2 Chemical Composition:

The chemical composition of the RCA will be highly dependent on the chemical composition of the aggregates/paste used in the source concrete. Therefore, most are calcium and silica rich (primarily from aggregate sources), with aluminates, alkalis, and ferrous materials contributed by the cement. Other chemical contaminants may also be present based on the use of the source concrete. These can include organic chemicals from automobiles (hydrocarbons, other automobile fluids) and inorganic chemicals (from deicers, spilled materials, other sources). Other larger contaminants may be present, such as joint filler, patching material, construction debris, or other materials – these may be present as entire particles from the crushing process, or as contaminants on particles primarily consisting of RCA.

### 2.1.3 Challenges to reuse

#### 2.1.3.1 Consistency

The term consistency gives a measure of the fresh concrete's mobility and flowability. Consistency of a fresh concrete mixture can be determined using either a slump-cone test or Vebe's apparatus to get an idea about its ease of flow and workability (Mehta and Monteiro 2006). The presence of RCA in fresh concrete tends to make it a harsher mix and reduces its workability at a faster rate due to the angularity and surface roughness of RCA (Yrjanson 1989). Fresh concrete mixtures made with RCA exhibit shorter initial setting and final setting times compared to mixtures made using virgin aggregates because of continued hydration attributed to the presence of old mortar fraction on the surface of RCA (Garber et al. 2011). Due to higher absorption of RCA, concrete mixtures incorporating RCA also cause a higher slump loss compared to a normal mixture made with virgin aggregates. In order to achieve the same level of workability as natural aggregate concrete, RCA concrete requires more water due to the high porosity of RCA (Padmini et al. 2009).

2.1.3.2 Mortar Content

The presence of residual mortar on the surface of the original aggregates is known to affect the properties of RCA. The mortar content of RCA is partially dependent on the type of the original aggregates. When concrete is crushed into RCA, rounded, less porous aggregates tend to have less residual mortar adhered to them compared to porous or crushed aggregates that do not solely rely on shear resistance for bond (Hiller et al. 2011). The absorption capacity increases while the specific gravity of RCA decreases due to the adhered mortar (Verian et al. 2018). When RCAs are used in a concrete system, two types of interfacial transition zones (i.e. between the aggregate and residual mortar and between new mortar and the aggregate) are created, and are responsible for affecting the properties of recycled aggregate concrete. The ITZ in new concrete produced using RCA tends to be weaker in nature due to the crushing process which causes the formation of continuous cracks and fissures inside the aggregate and the pores present in the adhered mortar (Behera et al. 2014).

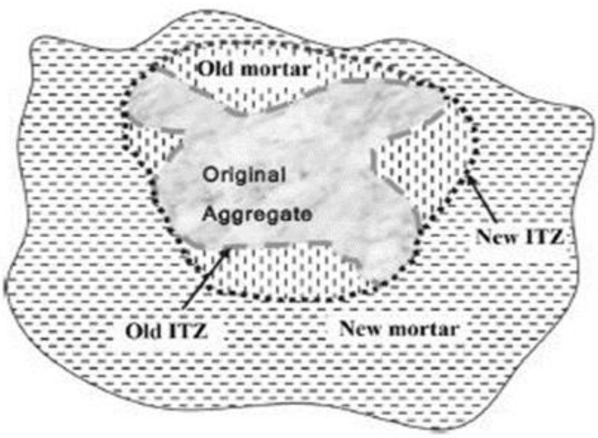



Figure 2.2 Interfacial Transition Zone in RCA (Verian et al. 2018)

2.1.3.3 Potential Contaminants

The presence of deleterious chemicals such as alkalis, sulphates, chlorides and organic impurities etc. greatly influence the chemical properties of RCA concrete and its durability. Sulphates may be present in RCA in the form of water-soluble sulphates sourced mostly from gypsum plaster (Silva et al. 2014). The presence of sulphates in RCA may lead to reactions due to their highly reactive nature. According to de Juan and Gutierrez (2009), the sulphate content in RCA is higher than natural aggregates due to the presence of cement in the adhered mortar. Another deleterious contaminant that affects the durability of concrete is chloride which may be found in RCA due to long-term exposure to de-icing chemicals containing chloride. The chloride causes corrosion of the steel reinforcement which affects the durability of concrete (Anderson et al. 2009).

Another significant concern about using RCA from some sources is its alkali silica reaction (ASR) potential. In the ASR reaction, an amorphous gel is formed when alkalis present in cement react with reactive silica present in the aggregates. This gel is known to produce cracks in concrete as it absorbs water and expands which results in the development tensile forces which eventually lead to cracking and deterioration of the concrete structure (Johnson and Shehata 2016). Lastly, RCA may also contain organic impurities like paper, wood, textile fabrics, joint seals, plastics, rubber and other polymeric materials. The presence of these materials can cause instability in concrete when exposed to freezing/thawing or drying/wetting conditions (Khalaf and DeVenny 2004).

## 2.2 X-Ray Fluorescence

The term ‘fluorescence’ is associated with the emission of light by a substance when subjected to electromagnetic radiation. X-ray fluorescence refers to an element analysis technique where a material excited by high energy X-rays emits secondary X-rays of characteristic energy or wavelength (Brouwer 2006). This non-destructive analytical technique which is used to determine the elemental composition of materials, has a wide range of applications across various industries. XRF has been widely used in the construction, geology, medical, and metallurgy, environmental and mining industries, as will be discussed in subsequent sections of this chapter. PHXRF devices are relatively simple to use and provide accurate analyses of a range of elements.

### 2.2.1 Working Principles of XRF

When a material sample is subjected to an X-ray beam, electrons from the atom are ejected from their atomic orbital positions followed by a release of energy which is characteristic of a specific element. The detector present in the XRF instrument detects this release of energy and then categorizes the energies by element. A detailed explanation of the process is as follows (adapted from Bruker 2019):

1. A high energy X-ray beam is emitted from the front end of an XRF instrument to excite the electrons present in the inner shells of the atoms of a sample.
2. The interaction between the X-rays and the atoms present in a sample causes the electrons to get expelled from the inner orbital shells of the atom. The ejection of electrons from the atom is known as ionization. Ionization is caused when the energy of the incoming x-ray beam is higher than the binding energy of the electrons that holds them in their respective orbits. The orbits where electrons of atoms are fixed is determined by specific energies and the spacing between the orbital shells is unique for different elements.

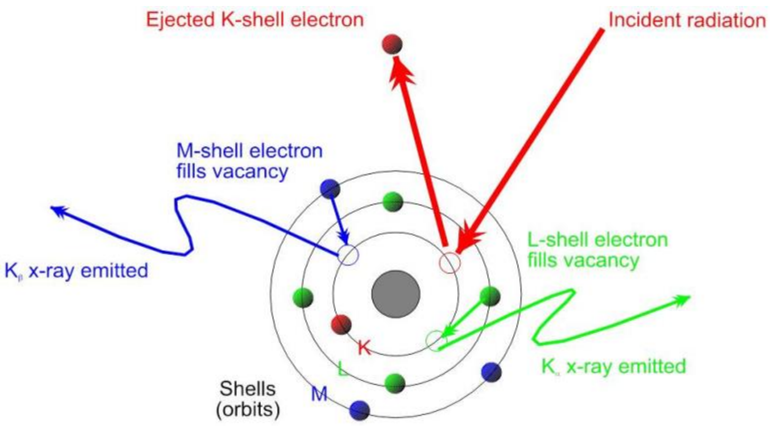



Figure 2.3 XRF Process (Bruker 2019)

3. The atoms become unstable when electrons are ejected out of their orbit, leaving behind vacancies. In order to correct this instability, electrons from a higher orbit move down to the lower orbit to fill the vacancy. If an electron closest to the nucleus of an atom is displaced, then an electron from the next shell occupies the hole left behind by the ejected electron.
4. The binding energies of electrons increase as they move farther away from the nucleus of an atom. As the electron falls into a lower orbital, energy is released. The energy lost by the electron is equal to the energy difference between the two orbitals and is determined by the distance between them. The distance between any two orbitals of an atom is unique to each element.
5. When the electron drops into a lower orbital, a fluorescent radiation characteristic in its energy distribution for a particular element is emitted which is equal to the energy difference between the two orbitals. This fluorescent radiation can then be analysed to determine the elements present in the sample as the energy lost in the fluorescence process is unique for each element.

   The fluorescent radiation can be analyzed using either energy-dispersive analysis or wavelength-dispersive analysis. These analysis techniques, as adapted from Marguí et al. (2013) are described below.

- **Energy-dispersive analysis:** In this analysis, dispersion and detection are a single operation as the fluorescent radiation emitted by the sample strikes a solid-state detector such as PIN diode, Si(Li), Ge(Li), Silicon Drift Detector. On striking the detector, a charge pulse is created which is proportional to the energy of the x-ray. The pulses generated by the detector are converted into voltage pulses which are proportional to the energy of the x-ray by a pulseshaping amplifier. The pulses are then sorted by voltage by a multichannel analyser.

- **Wavelength-dispersive analysis:** In this analysis, a diffraction device is used to separate the characteristic radiation emitted from the sample according to its wavelength. A wavelength-dispersive XRF instrumentation generally consists of an x-ray tube, a collimator and a detector. When the sample is subjected to an x-ray beam, the characteristic fluorescent radiation emitted from the sample is made to pass through a collimator onto the diffraction device (analysing crystal). On passing through the diffraction device, the individual wavelengths of the radiation are diffracted onto the detector satisfying Bragg's law. A gas flow counter/scintillation counter is generally used as a detector system. The detector system converts the diffracted fluorescent radiation into voltage pulses which is proportional to the energy of the original x-ray. The voltage pulses are then displayed as a measure of the characteristic line intensity.

2.2.2 Energy spectrum and nomenclature

During an electron transmission fluorescent radiation is emitted which is unique to each element. When this happens, in an energy spectrum, which is a graphical representation of energy vs intensity, energy peaks are generated. By interpreting the peaks correctly, we can identify the element and compute its concentration (Fisher 2019). To be able to interpret the peaks correctly, it is important to understand the nomenclature associated with peak intensity (Murphy 2006).

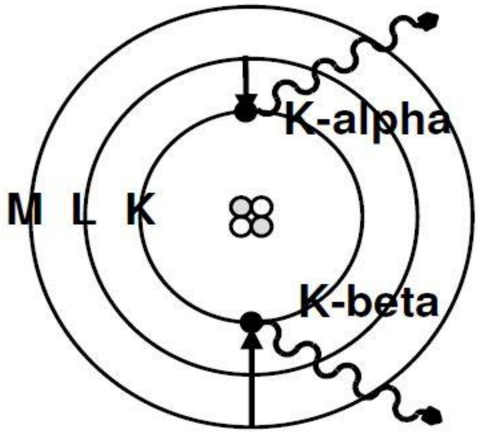



Figure 2.4 Electron Transmission (Murphy 2006)

As shown in Figure 2.4, nomenclature associated with electron transmissions are identified as follows:

1. **K-alpha**: Electron transmission from L shell to K shell

2. **K-beta:** Electron transmission from M shell to K shell

3. **L-alpha**: Electron transmission from M shell to L shell

4. **L-beta**: Electron transmission from N shell to L shell

XRF Energies for various elements (Chemistry LibreTexts 2019):


Figure 2.5 Periodic Table with X-ray energies (Bruker 2020)

Being too low in energy the fluorescence photons from organic elements like H, C, N, O are not efficiently detected by the detector. Therefore, these elements do not give XRF peaks. On the other hand, only K-peaks are given by low atomic number elements like Cl, Ar, K, Ca. The L-peaks are not given by these elements because they low in energy and are not detected by the detector. Lastly, the K peaks from elements with high atomic number like Ba, Hg, Pb, U are too high in energy. Hence, they only give only L-peaks. The elements Rh through I, i.e., middle atomic number elements may give both K and L peaks.

As discussed earlier, electrons on being ejected from the inner shells of an atom are replaced by an electron from an outer shell causing fluorescence. The detector detects the x-rays pertaining to the quantity of K-shell and L-shell which is proportional to the number of atoms of a particular element in the sample (Murphy 2006). Figure 2.6 shows a typical energy vs intensity graph.

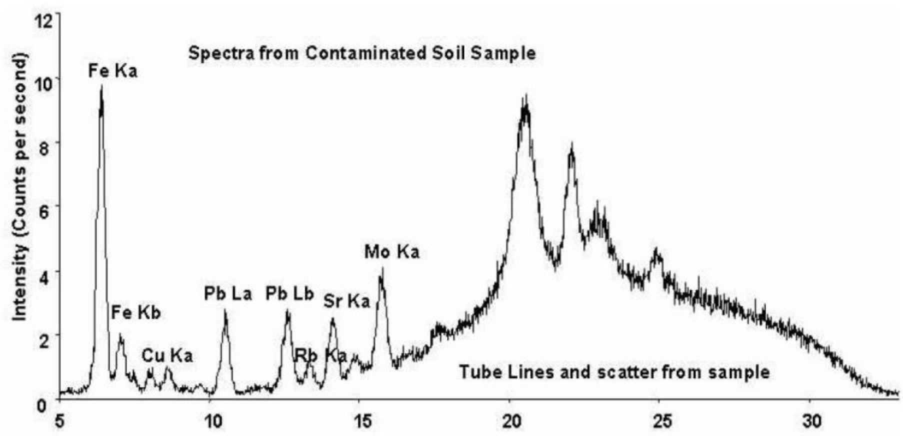


Figure 2.6 Energy Vs Intensity (Murphy 2006)

### 2.2.3 Energy Peaks

The interpretation and production of x-ray energy spectrum comes with its own set of challenges. Matrix absorption effects, Bremsstrahlung radiation, overlapping pulses, interactions in the detector and low-energy background are some of the common challenges one may encounter while interpreting an energy spectrum (Murphy 2006). A brief description of the different types of phenomenon exhibited in an x-ray energy spectrum are described below.

- *Bremsstrahlung x-rays*: The German term Bremsstrahlung translates to "braking." In this phenomenon, x-rays are produced in an x-ray tube of the handheld XRF device, when a negative cathode emits electrons accelerated by a voltage applied across the tube. The accelerated electrons are suddenly impacted by a metal target (cathode) causing it to decelerate. This sudden deceleration generates x-rays with an energy equal to the voltage applied the x-ray tube (Murphy 2006).
- *Characteristic x-rays*: Characteristic x-ray lines are usually superimposed on Bremsstrahlung x-ray radiation. These x-rays are emitted when the electron drops from a higher energy shell to a lower energy shell as discussed earlier. The energy of these x-rays is equivalent to the difference in energies between the inner and outer shells of the atom. Figure 2.7 shows how characteristic x-rays are superimposed on Bremsstrahlung radiation (Murphy 2006).

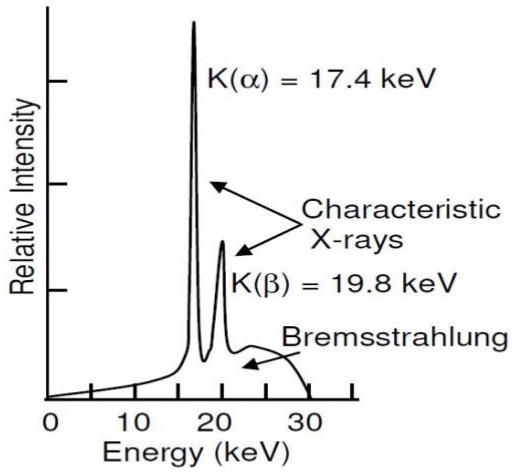



Figure 2.7 Bremsstrahlung and characteristic x-rays in an energy spectrum

- *Sum Peaks:* It might often happen that a detector may encounter two or more x-rays at the same time and may convert them into a single pulse with energy equal to the sum of the two pulses combined. This may give rise to a visible peak in the energy spectrum called as sum peaks. The sum peaks can occur in variety of combinations. It can occur as a sum of Kα+ Kα, Kα+Kß or Kß+ Kß. As seen in the figure, there are two sum peaks in the spectrum. The first one corresponds to Kα+ Kα peaks of Cu which is equal to a value of 16.094 KeV and the second one is a sum of Kα+Kß peak which is equal to 16.951 KeV (Speakman 2015).

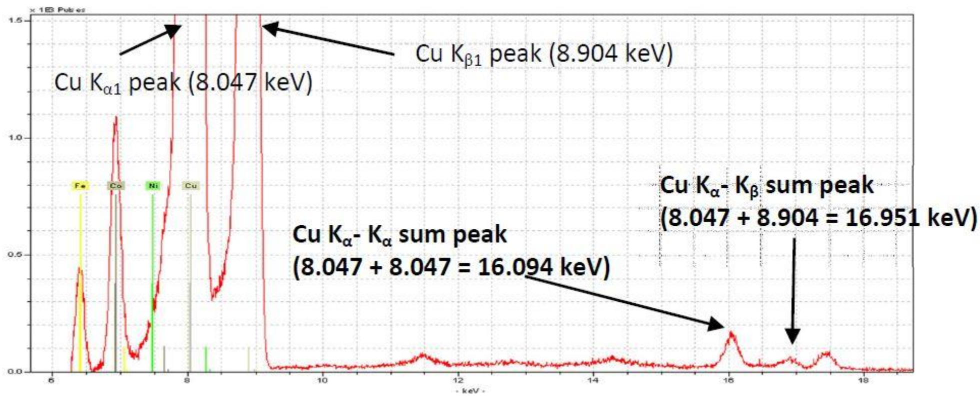



Figure 2.8 Energy Spectrum showing Sum peaks (Speakman 2015)

- *Escape Peaks*: An atom present in the detector which is silicon in the case of Tracer-III SD series, can get excited by an incoming x-ray and cause fluorescence within the detector. The escape peak will appear in the spectrum with energy equal to the difference of the incoming x-ray and the characteristic energy of the atom present in the detector. As shown in Figure 2.9, the escape peak is equal to 6.26 KeV which is equal to the difference of incoming Cu x-ray and the characteristic energy of silicon atom present in the detector. Their intensity is much less compared to the characteristic x-ray peaks (Speakman 2015).

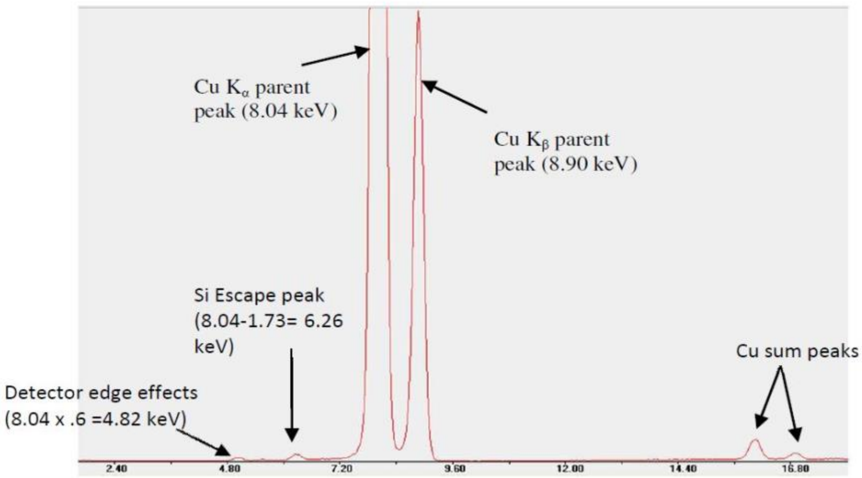



Figure 2.9 Escape peaks in energy spectrum (Speakman 2015)

- *Rayleigh Scattering*: The x-ray tube which emits x-rays may get absorbed by photoelectric effect or they might get scattered when they reach the sample. Rayleigh scattering occurs when the sample reflects the incident x-ray beam towards the detector without causing any loss of energy. The detector identifies this reflected x-ray with an energy peak which corresponds to the energy of the tube element which is rhodium in this case. During this phenomenon, the energy of both the outbound x-ray and inbound x-ray are equal. The scatter peaks may occur as sharp peaks as shown in Figure 2.10 (Kaiser and Wright 2008).

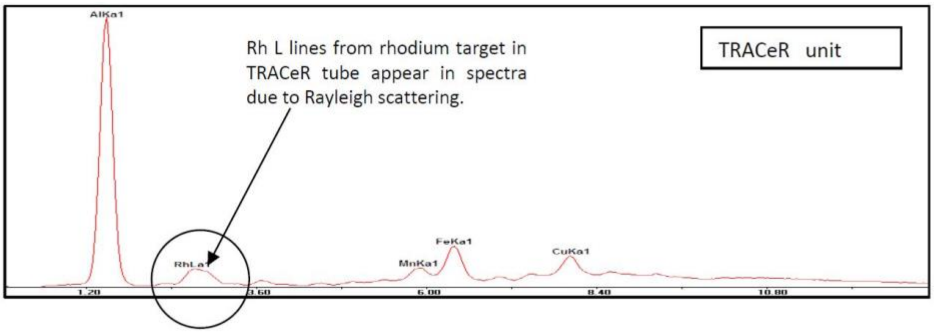



Figure 2.10 Rayleigh Scattering (Kaiser and Wright 2008)

- *Compton Scattering*: Fluorescence may not always occur when an x-ray beam is incident on a sample. Sometimes, the x-ray beam might cause excitation of the electrons in an atom without losing its entire energy. This type of scattering occurs when an x-ray beam has sufficient amount of energy to excite an inner-shell electron but not enough to eject it out of the inner shell to create a vacancy. However, no characteristic energy is released in this process as no vacancy is created. This phenomenon causes the x-ray beam to scatter in all directions and they are generally low in energy compared to Rayleigh scattering as only a small amount of energy is lost in this process. This type of scattering is common in low Z elements and can be seen in Figure 2.11 (Kaiser and Wright 2008).

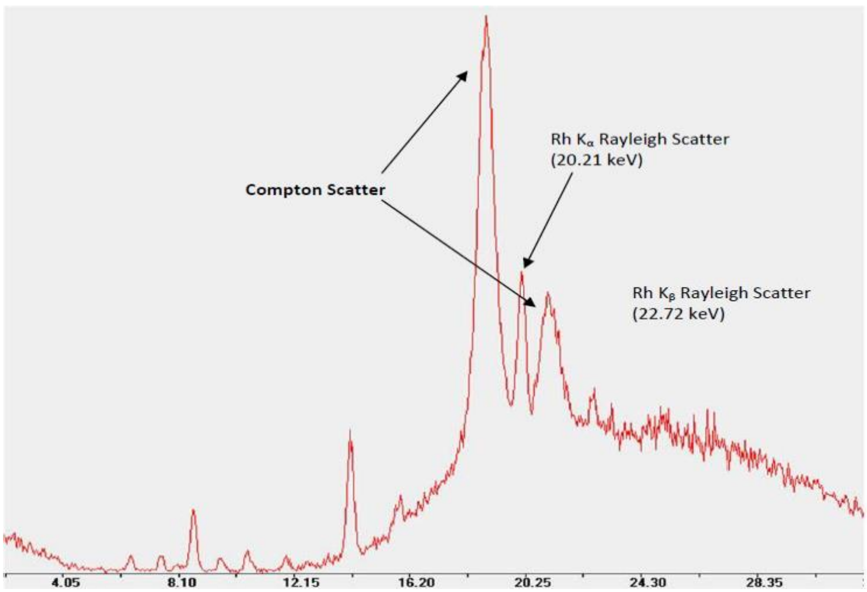



Figure 2.11 Compton scattering (Kaiser and Wright 2008)

## 2.3 Materials Analysis Using XRF

### 2.3.1 General

#### 2.3.1.1 XRF Application in Gold Assaying

The high precious metal value of gold supported the development of an accurate and non-destructive method of analysis to determine the content of gold in an alloy. The ISO identifies the Cupellation method for analysis purpose as it has high accuracy. Despite having high accuracy, it has several drawbacks as it is a destructive form of testing, it is time-consuming and involves the use of strong acids at high temperatures that leads to the production of toxic fumes (Marucco 2004). Another method which has been widely used across the industry for assaying is the Touchstone test. The analysis accuracy of this test has been questioned, as is influenced by several parameters such as quality of stone, skill of the operator, strength of touch acids. Lastly, this test is inappropriate for high carat and white gold alloys.

Over the last few years, techniques such as the Cupellation and Touchstone methods have been replaced by Wavelength/Energy dispersive X-ray Fluorescence. XRF spectrometry can be used for the analysis of precious alloy constituents. This technique’s applications extend primarily to the jewelry manufacturing industry and gold assaying laboratories for anti-fraud control. An experiment was conducted using FISCHERSCOPE X-RAY XAN spectrometer with two different analytical procedures; 1) standard-less analysis for sample of unknown composition and 2) a semi-empirical method based on calibration curves for alloys of known constituents. The experiment concluded that low- energy XRF spectrometry is appropriate for quick-sorting determination of fineness of finished gold articles. This user-friendly, time-saving and non-destructive analysis technique can be used in assaying laboratories for preliminary testing of gold articles and it has the potential to substitute Touchstone method for gold assaying as it offers more accuracy and less sample preparation.

2.3.1.2 Food Chemistry

The presence of heavy metals in food can have significant health impacts on the human body. It is associated with a number of diseases including cardiovascular, neurological and renal diseases (Chailapakul et al. 2008). Heavy metals like lead (Pb), cadmium (Cd) and molybdenum (Mo) are significant environmental pollutants that enter the food chain through various biochemical process. Therefore, in order to safeguard human health, it is necessary to determine the level of Mo and Pb in food and their dietary intake. There are several analytical methods like Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) and Atomic Absorption (AA) which can be used to determine the presence of Mo and Pb in complex matrix foods. The aforementioned analytical methods are often time consuming, complicated and involve dry ashing which can cause losses of Mo and Pb and the use of chemical reagents can cause contamination.

XRF provides an easy solution to this problem as it provides better sensitivity when the sample is in pellet form. Research conducted on food samples like cereals, vegetables, meats, fishes, milk from three different countries (USA, Bangladesh, Australia) using the energy-dispersive x-ray spectrometer was successful in determining the Mo and Pb content in food samples as the measurement accuracy varied over a range of 2-11% with respect to the certified values which were derived from reference materials (bovine liver, rice flour, horse kidney and orchard leaves) (Ali et al. 2014). These reference materials were selected to minimize the matrix effects as much as possible. The measurements of Mo and Pb in certain food sample groups were further validated by using a proton induced x-ray emission.

2.3.1.3 Archaeology

Researchers in the field of archaeology have benefitted significantly from XRF technology as it gives them the opportunity to study various materials with greater flexibility. PHXRF spectrometers in particular have simplified the in-situ elemental analysis of excavated objects, monuments and materials used in finishes and construction of standing buildings. Being non-destructive in nature, they preserve the material culture by traveling to the object under analysis rather than bringing delicate, fragile and unstable objects to the spectrometer (Shugar and Mass 2012).

ED-XRF can be used for preliminary analysis of archaeological ceramics. Compositional analysis under this instrumentation can help establish ceramic provenances and enables the validation of solid hypotheses with respect to its historical significance. The detection of key elements like Al, Si, K, Ti, Fe, Zr, Ca, and Sr can serve as a preliminary approach for analysis of archaeological ceramics (Calparsoro et al. 2019). Furthermore, ED-XRF has been used to differentiate between shells and bones which enabled the determination of prehistoric personal ornaments. An Archaeological study was conducted in Spain using ED-XRF spectrometry on prehistoric ornaments to distinguish between bones and shell materials. The study revealed the presence of three main elements, calcium (Ca), strontium (Sr), and platinum (P), in the samples. The statistical analysis of these three elements led to the differentiation between bones and shells and the determination of nature of prehistoric ornaments (Sánchez De La Torre et al. 2018).

## 2.4 XRF use in Construction Applications

### 2.4.1 XRF Testing for Presence of Corrosive Drywall

Certain types of drywalls produced from 2001 to 2008 were known to produce gases like hydrogen sulphide and sulphur dioxide which led to the corrosion of copper wiring and air conditioning coils in buildings. A film of black corrosion coating was reported to be deposited on the copper components of the building. Studies confirmed that the range of strontium contents in corrosive drywall differs from that of noncorrosive drywall (Unified Engineering 2009; EPA 2009). Although, the presence of strontium is not linked to the production of sulfur compounds, strontium could serve as an indicator for the presence of corrosive drywall.

The primary identification method recognized for identifying corrosive drywall by Consumer Product Safety was a general visual inspection of all the copper components for the presence of a black deposit. This method was to be validated by analyzing the drywall core for the presence of strontium. A methodology for categorization of corrosive and noncorrosive drywalls was performed utilizing XRF and laboratory exposure testing. Samples of drywall were obtained from home-improvement stores and from a high-rise residential building in the south-eastern United States (Steiner 2011). The strontium levels of the gypsum core of the drywall samples obtained from home-improvement stores were tested in the laboratory by handheld XRF and atomic absorption (AA). The XRF data was collected after removal of facing paper, paint, and topping compound on its surface. After collection of XRF data, the samples were subjected to the highly sensitive AA test and were validated by AA test measurements on certified reference gypsums. Data obtained from both the methods were correlated which led to the conclusion that the XRF data was consistent. Furthermore, field testing of strontium content was carried out at the building by taking measurements at each eight feet or less in horizontal direction and at 3 and 5 feet from the floor level and one column of measurements was taken at 3 and 5ft for walls less than eight feet in length (Steiner 2011).

To categorize the drywalls as corrosive and non-corrosive based on the strontium content, 75 samples were further selected for laboratory exposure testing. Under this testing method, the drywall core sample along with a copper coupon were placed in a closed chamber under controlled conditions and inspected at regular intervals. Corrosive drywall caused the tarnishing of copper coupon within a couple of days whereas noncorrosive drywall produced minimal tarnishing. The degree of tarnish was then measured according to CEILAB color coordinate system using a colorimeter. The color change ($\triangle E$) was defined as measurement of color before and after color change. A known control sample (corrosive specimen and noncorrosive specimen) was established followed by measurement of color change after 5 and 10 days. A wide range of $\triangle E$ values were observed primarily due to variations in samples caused by different amount of topping compound on the surface and other unknown factors. Correlation was established between the laboratory exposure test results and XRF by plotting $\triangle E$ against XRF measurements for 5 and 10 days of exposure respectively (Steiner 2011).

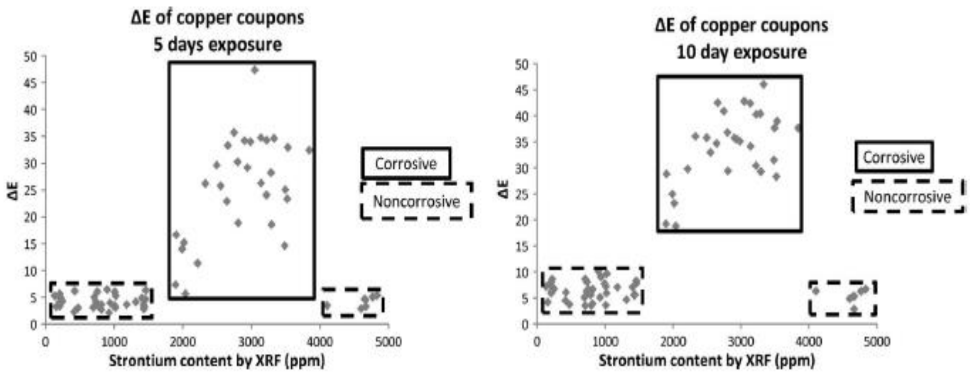



Figure 2.12 Strontium content by XRF Vs color change (Steiner 2011)

The above figure shows how the data has been categorized into 3 categories: (1) non-corrosive drywall with low strontium content (2) non-corrosive drywall with a high strontium content (3) corrosive wall with a high strontium content. The cut off values between corrosive and non-corrosive drywall must be further validated by visual inspection of copper components in the building and laboratory testing. Although strontium does not cause the release of corrosive gases, the measurement of strontium content of corrosive wall using XRF indicates a range of values that distinguishes it from non-corrosive wall. Thus, PHXRF can be a useful tool for quick analysis of drywalls without testing every drywall sheet in the laboratory. One key finding that came into light from this examination was that it was possible to have strontium content of non-corrosive drywall higher than that of corrosive wall (Steiner 2011).

2.4.2 Geotechnical Applications

Many transportation projects use stabilizers to improve the mechanical properties of soil, including increasing shear strength, lowering compressibility, and preventing volume change. Hence, from a quality assurance and quality control (QA/QC) standpoint it becomes necessary to determine the stabilizer content present in the soil to ensure that the field stabilization amount is close to the laboratory design amount. Previous research studies have concluded that XRF can be used for the purpose of determining stabilizer content in the subgrade which aids the process of geotechnical inspections by detecting irregularities in the behavior of roadways (Ferraro 2016).

A research study was conducted to validate the use of PHXRF on stabilized subgrade for the purpose of geotechnical investigations and quality control (Cerato et al. 2017).This research study was conducted on 5 road-widening projects in the state of Oklahoma, where the sites were stabilized with quicklime or high calcium fly ash (Class C). Two portable handheld XRF (PHXRF) devices were used in this research (Bruker S1 Titan and the Thermo Scientific XL3t-950 GOLDD+). The stabilizer content readings of these devices were compared to Laboratory readings using the "Whole

Rock Analysis” to identify sample preparation and analysis techniques producing accurate results. Statistical techniques like ANOVA and regression analysis were used to determine the accuracy of the sample preparation and analysis techniques. In order to study the effects of sample preparation and analysis techniques on the measurements, the impact of several variables like scan duration, scan technique, particle size and the type of sampling were evaluated by the researchers (Cerato et al. 2017).

A 60 second scan duration was determined to be appropriate, since longer scanning durations did not yield significant benefits in terms of precision. Two types of scanning techniques were evaluated to assess the homogeneity of the sample. A standard scanning technique was chosen over the quartering technique as it required a smaller number of scans per sample which saved time and had little to no effect on the accuracy. Particle size played a prominent role in the accuracy of PHXRF Stabilizer content measurements. Two types of soils (super gel-X Bentonite and kaolinite clay) were mixed with stabilizing agents like lime, Class C fly ash, or cement kiln dust (CKD) to create 14 mixtures. The mixtures were further milled to create a matrix of 56 total samples. The mixtures were milled and made to pass through sieve sizes of No. 4, No. 40, No. 100 and No. 200. A matrix of 70 samples of varying stabilizer contents, sample types and particle sizes were created out of which 56 were compacted pellets and 14 were loose powder samples.

The effect of particle size on RMSD and $COV_{RMSD}$ of both the samples revealed that milling samples to smaller particle sizes improve the accuracy of readings. To save time and cost, the field samples were milled to pass a No. 40 sieve because the accuracy of PHXRF begin to level off after they are reduced passed the No. 40 sieve. In terms of sampling type, a powdered sample was considered to be more advantageous for field implementation as it requires less amount of time for preparation compared to pressed pellets (Cerato et al. 2017).

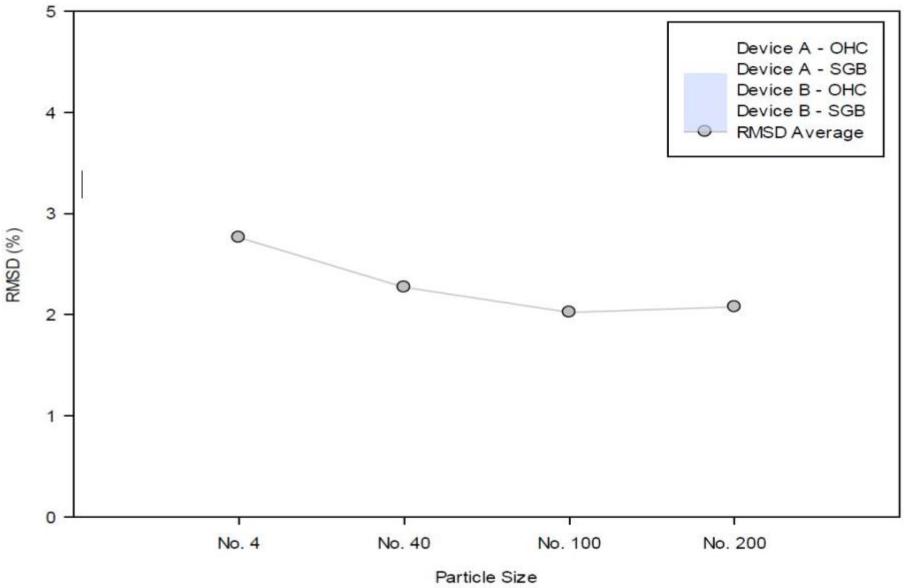



Figure 2.13 Particle size vs RMSD % curve (Cerato et al. 2017)

The field testing was performed by taking measurements in a grid pattern (in situ and ex situ). In situ refers to no sample preparation and ex situ refers to samples placed in cups after processing over a No. 40 sieve. Taking measurements in a grid pattern allowed the researchers to assess the spatial homogeneity of the sampled area. Contour plots of the 5 different sites indicated depth heterogeneity of SCs. As shown in Figure 2.14, the stabilizer content for site 1 (in situ) stayed above the design value of 15%, site 2 falls to a value of 11% from 16% for 8 inches of depth.

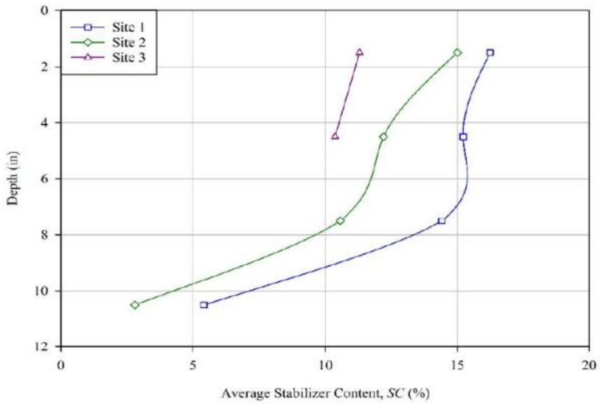



Figure 2.14 Average stabilizer content % vs depth (inches)

The ex-situ PHXRF SC measurements produced consistent measurements, which was attributed to the ex-situ samples being more homogenized than the in situ samples. To assess the accuracy of both in situ and ex situ measurements, they were compared against laboratory XRF measurements. In case of the ex-situ measurements, the combined trend line fit the data points well with an $r^2$ value of 0.925. These values were corrected mathematically using the equation of the combined trend line to achieve accurate measurements. On the contrary, in situ measurements produced vague results and did not fit the trend line as expected due to heterogeneous conditions like presence of clay, pebbles and other debris. The regression analysis indicated poor precision of XRF measurements (in situ). The figures down below show the regression analysis of both in situ and ex situ measurements.

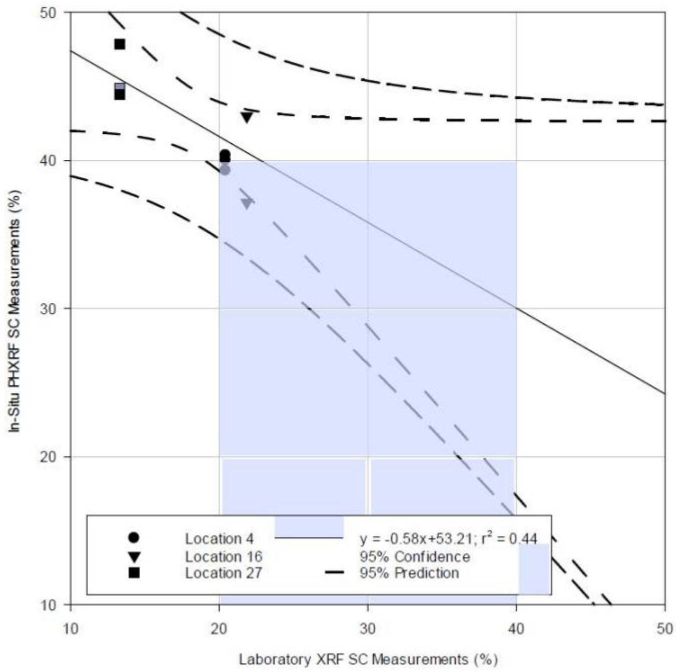



Figure 2.15 Linear regression of in-situ measurements with laboratory XRF measurements.

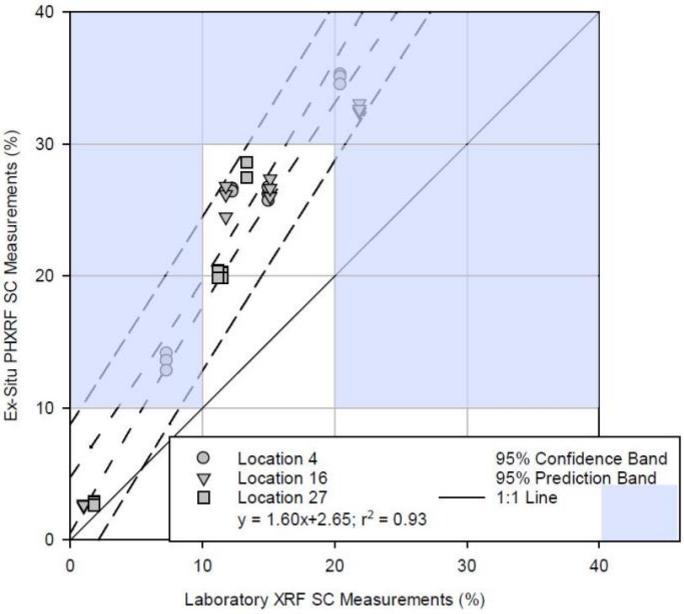



Figure 2.16 Linear regression of ex-situ measurements with laboratory XRF measurements.

Thus, this study demonstrated that with certain level of sample preparation, XRF can be a useful tool which will enhance QA and QC capabilities in future roadway inspections. More importantly for the present work on RCA characterization, this study demonstrated the capability of XRF to successfully characterize granular, silicate and calcarious materials (Cerato et al. 2017).

2.4.3 Assessment of Concrete Mixture Proportions Using XRF

The durability performance of concrete pavements will depend greatly on the proportioning and uniformity of concrete mixtures (Wang and Hu 2005). The current methodology for examining the proportion of any batch requires tracking of batch tickets at the batching plant. This methodology is sometimes not reliable due to calibration errors and addition of water after dispatch. Therefore, use of a handheld XRF device to examine and estimate the proportion of concrete mixtures before construction begins was investigated by Taylor et al. (2012).

One of the major challenges faced by the XRF technique is sample preparation as this analysis often requires a flat and homogenous surface. Also, the device poses problems in detection of elements lighter than magnesium. Thus, an examination was conducted to investigate the feasibility of portable XRF device in determining the proportions of fresh concrete (Taylor et al. 2012).

A handheld Niton XL3t900 GOLDD+ analyzer was used for this research study. The analyses were performed on powder, paste and mortar samples. Powder samples were tested in open topped container sealed with 6-micron polypropylene. The mortar and paste samples were prepared according to ASTM C305 and ASTM C109. The individual chemical composition of cementitious materials (slag cement, silica fume, Class C fly ash, Class F fly ash, type I portland cement) were tested using the XRF device. Fine aggregates were oven-dried in order to reduce the moisture content and paste mixtures were prepared with a w/b ratio of 0.45 with different combinations of cementitious materials. Lastly, mortar samples were prepared in a ratio of 1:3 by mass (cement to sand). The results were averaged after testing 3 samples from each mixture. The results of the examination are as follows:

Cementitious Materials

The results of cementitious materials were represented as elemental percent by mass and using the atomic weights of the data, they were converted into oxides. Once the data was obtained, they were compared to a typical range of Type I portland cement obtained from the PCA (Kosmatka et al. 2002).

Table 2.1 Typical range of Type I portland cement adapted from Kosmatka et al. (2002)

| Oxide | Mass percent | |
|---|---|---|
| | Typical range (min-max) | Test data |
| $SiO_2$ | 18.7-22.0 | 18.21 |
| $Al_2O_3$ | 4.7-6.3 | 3.67 |
| $Fe_2O_3$ | 1.6-4.4 | 4.63 |
| CaO | 60.6-66.3 | 62.95 |
| MgO | 0.7-4.2 | 3.12 |
| $SO_3$ | 1.8-4.6 | 8.55 |

After comparison of the data, it was observed that $SO_3$ was above the expected range.

The fine aggregates were tested using laboratory and portable XRF. On comparison of both the data sets, a large difference was observed between the percentage of undetected elements in both the devices.

For paste mixtures, as seen in Figure 2.18, the handheld XRF was found to provide an adequate correlation between the calculated SCM contents for both binary and ternary mixtures. When water was included in the calculation of actual SCM content, large prediction errors were observed.

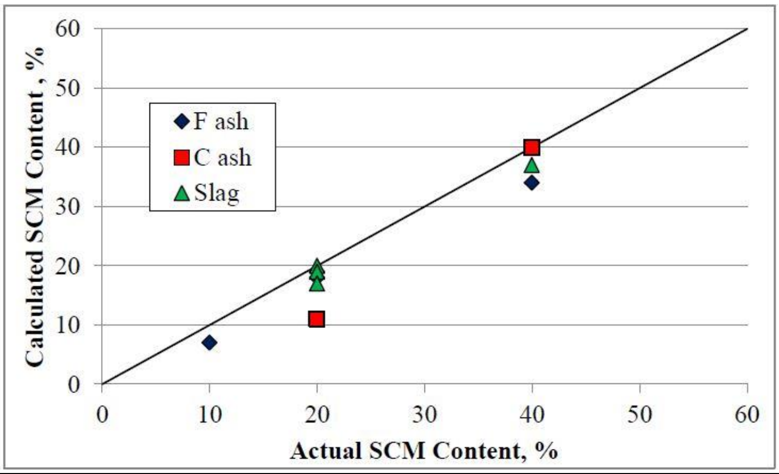



Figure 2.17 Relationship between tested and batched SCM contents

The SCM content of mortar was calculated in a similar fashion as that of paste mixtures considering the data from sand analysis. The sand content was determined to be 30% by mass from the calculations. When a relationship was established between tested and actual SCM including the sand content, errors were observed between tested and actual SCM content. The calculations were repeated assuming a fixed sand content of 15% because the depth of penetration of the beam into the sample analyzing the sand content was not representative of the mixture when the sand content was not fixed. Although, as shown in Figure 2.19, the relationship between actual and calculated SCM content still showed errors which was marginally less than the errors previously observed.

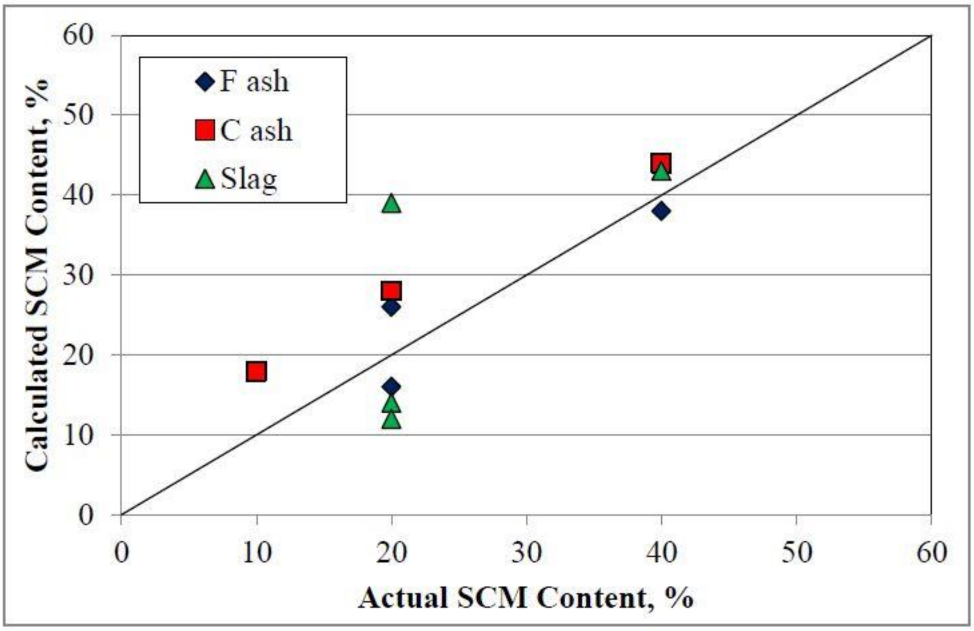



Figure 2.18 Relationship between tested and designed SCM content (Taylor et al. 2012)

This device seems to work reasonably well for reporting SCM dosages of paste mixtures, but the inhomogeneity of the mortar samples affected the analysis of mortar which gave rise to significant errors.

## 2.5 Whole Rock Analysis:

Whole rock geochemistry is a technique used to analyze a rock sample for determining the presence of a wide variety of major and trace elements. This technique usually involves careful preparation of the sample before undergoing tests like ICP-AES, ICP-MS etc. The samples are generally pulverized to the size of few microns and are fused using a fusion flux (Twelker et al. 2017). Using this technique, several major and trace elements are quantified on a weight % basis. The abovementioned techniques used as part of the Whole Rock Analysis procedure have been discussed below.

### 2.5.1 Inductively Coupled Plasma-Mass Spectrometry

ICP-MS is an elemental analysis technique that is used in several industries like the pharmaceutical, medical, geochemical, environmental etc. for detecting elements present in the sample. This analysis technique operates on the phenomena of ionization where the sample is completely ionized by an ionized source into constituent elements which are further transformed into ions. To form the plasma, the energy is coupled to argon gas using an induction coil (Scientific 2020). The simplest sampling technique for this analysis is to prepare a solution in dilute nitric acid. This test set up consists of a sample introduction system which requires the sample to be digested in concentrated nitric, hydrochloric/hydrofluoric acid on hot plates (Godfrey and Glass 2011). An ICP-MS system generally comprises of five basic parts:

i. Sample introduction system
ii. Plasma
iii. Vacuum interface
iv. Ion optics with mass analyzer
v. Ion detection system

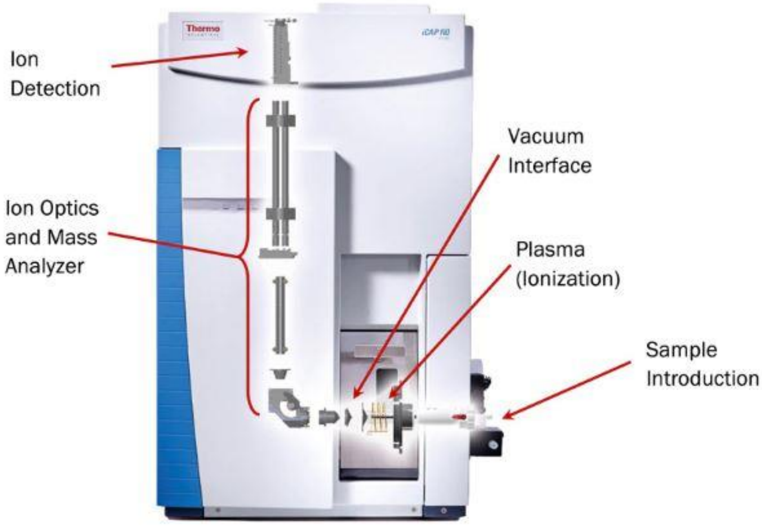


Figure 2.19 ICP-MS system

Depending on the phase of the sample in which it exists, different processing methods are implemented. The processing is fairly straight forward for gases as they can be directly analyzed by the plasma. On the contrary, solids and liquids require conversion into aerosol form using different means. Liquid samples are usually digested in an aqueous matrix which contains nitric and hydrochloric acid in different volumes to stabilize certain elements. Whereas, solid samples are digested in stronger acids and sometimes even hydrogen peroxide is used to break down organic matter efficiently (Scientific 2020). When the sample is prepared, it is introduced into the system in the form of liquid through a spray chamber and nebulizer. The liquid is turned into a fine mist by the nebulizer which causes supersonic expansion of gas and then any large droplets are further removed by the spray chamber before they are processed in plasma. The plasma ionizes the sample and enters the high vacuum region. The ions from the vacuum region then get transferred to the mass analyzer where the elemental masses are separated. The last step in the process is ion detection which occurs after mass separation. There are usually two types of detectors: faraday cups and electron multipliers. Each of these types of detectors detects the ions and then amplifies them to compute their intensities (Scientific 2020).

2.5.2 Inductively Coupled Plasma-Atomic Emission Spectrometry:

This is another widely used technique and is sometimes also referred to as Inductively Coupled Plasma-Optical Emission Spectrometry. In this emission spectrometry, the analytes are atomized by subjecting the sample to high temperature (approximately 7000°C) in an argon plasma. The photomultiplier tube then detects the ionic spectra which is emitted upon the collision of atoms with excited argon species (Cheremisinoff 1996). Relatively, the detectors present in atomic emission spectrometry are highly sensitive and offer a dynamic range.

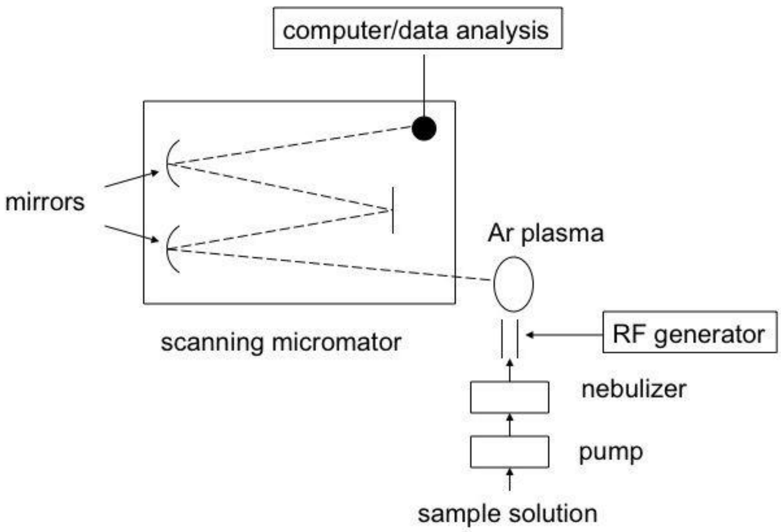



Figure 2.20 ICP-AES set-up (Barron and Raja 2019)

Just like the ICP-MS system, the ICP-AES system comes with a nebulizer and a spray chamber which forms aerosols and selects aerosol droplets of the required diameter. The aerosol analytes rapidly desolvate and get transiently excited when they enter the plasma. Just like the XRF, these excited electrons emit electromagnetic radiation once they go back to their ground state which is characteristic of the change in the energy states (difference between the excited energy level and ground state). Charged coupled device detectors are then used to detect the wavelength of the radiation and the wavelengths per analyte are measured and sometimes, internal standard elements are mixed to subdue the sample matrix interferences in the sample caused during the analysis (Smith and Nordberg 2015).

2.5.3 Infrared Spectroscopy:

Infrared (IR) spectroscopy is another widely used analytical technique in which the oscillation of molecular dipoles caused by molecular vibrations. The vibrations in the bonds are different for every molecule as they depend on factors like orientation of bonds with respect to the molecule, number of bonds and atoms present in the bond. Thus, the spectra generated will be unique for different molecules which helps in identifying the unknown constituents in a sample (Barron and Raja 2019).

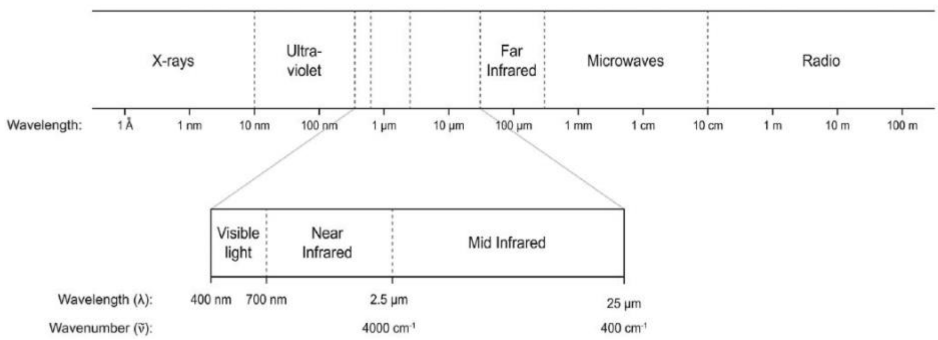


Figure 2.21 Electromagnetic Spectrum

In the electromagnetic spectrum the infrared region ranges from 700 nm to 1mm. Despite having less energy compared to the energy of visible light, infrared radiation causes rotations and vibrations in covalent bonds of molecules with sufficient energy. This property of the infrared radiation helps it to identify molecules. The basic principle behind IR spectroscopy is the vibration and rotation about the bonds of atoms present in molecules. The vibrations occur in two different forms namely; bending and stretching vibrations. Each vibration variation type has its own unique frequency corresponding to the electromagnetic spectrum where the infrared region lies. When samples are subjected to infrared radiation, the radiation causes a change in the dipole moment of the molecule present in the sample which leads to the absorption of photons whose energies are equivalent to the difference in the vibrational energy levels of two molecules. The IR spectroscopy method is then used to measure these energy absorptions. The analysis technique is able to analyze various types of samples like solids, liquids and gases. This testing system consists of a light source which splits into two beams in which one of the split beams passes through the sample while the other beam passes through the air and then the beams are analyzed by the monochromator on recombining. This is recorded by the monochromator in the form of a spectrum which is a function of the wavelength of the beam (Monnier 2018).

2.5.4 Loss on Ignition (LOI):

The LOI is a test which is used to measure the amount of volatiles ($H_2O$, $CO_2$, F, Cl, S etc.) present in a sample in order to determine the accuracy of Whole Rock Analysis by adding up the oxides and verifying whether the total adds up to 100.0 ± -1.0% or not (Dean 1974).

The volatiles normally estimated by LOI include organic matter, inorganic carbon and water content etc. The LOI is a two-step sequential process which first involves oxidizing organic matter to carbon dioxide in ash at a temperature between 500-550°C in a muffle furnace. In the next step, carbon dioxide evolves from carbonate when heated at 900-1000°C. Once the heating procedure is complete, the weight loss of the sample is determined before and after heating to compute the LOI (Bengtsson 1986; Dean 1974).

2.6 Research Needs

As evidenced by this literature review, in recent years, the application of PHXRF technology has been successfully utilized in both construction and non-construction industries. It has been used successfully in the analysis of granular soil materials and cementitious/pozzolanic binders and has also been used to characterize concrete mixture proportions. Therefore, PHXRF shows good promise for use in analysis and characterization of RCA.

The biggest challenge impeding the use of RCA in concrete applications is the absence of an established testing protocol to support use of PHXRF in RCA. Using PHXRF to characterize RCA would provide critical information regarding RCAs chemical composition and physical characteristics (specifically, adhered mortar content) that would promote its use in concrete applications. Specific challenges associated with development of a test method to utilize PHXRF for chemical analysis of RCA are identified and discussed in Section 2.6.1 below.

### 2.6.1 Testing Protocol Development Challenges

Several variables must be given careful consideration in order to obtain accurate PHXRF measurements of the elements present in the samples. The variables are as follows:

1. *Duration of each scan*: Due consideration must be given to this variable as scanning duration can affect the precision and accuracy of PHXRF intensities when scanning for light and heavy elements. The data precision can be improved by effectively using the measurement time (Löwemark et al. 2019; Profe and Ohlendorf 2019). Different combinations of scan durations must be evaluated for the purpose of optimising the measurement time and to exclude the differences arising due to random sampling variability. The effect of duration of each scan with respect to variables like sample type, particle size of sample, thickness of sample and scanning technique must be carefully evaluated to obtain the desired level of accuracy and precision (Cerato et al. 2017).

2. *Scanning Technique*: Scanning technique plays an important role and it should be carefully evaluated in order to assess the homogeneity of the sample. Different types of

scanning technique can be implemented for example; a quartering technique, where a sample is divided into four quadrants and then each quadrant is scanned ‘n’ times. Another way of scanning would be to perform ‘n’ scans in the same place in a sample. Cerato et al. (2017) noted that scanning technique influenced the precision of PHXRF measurements more than the accuracy. Hence, the scanning technique must be selected from an efficiency standpoint. If two different scanning techniques do not show significant differences in the results, then the technique which requires less time and is more convenient to perform should be adopted.

3. *Sample Thickness*: Applying use of the PHXRF to RCA will require consideration of sample thickness, since the PHXRF intensities are influenced by the thickness of sample. An experiment was conducted to measure the harmful elements like Pb, Se, Cd and Fe in soils samples with PHXRF method was investigated by studying the influence of thickness of soil layer and grain size of soils (Imanishi et al. 2010). It was observed that x-rays with high energy saturated at a higher soil thickness compared to low-energy x-rays. PHXRF intensity was shown as a function of thickness of the soil sample to evaluate the optimum thickness of soil sample. Figure 2.20 shows the relationship between thickness of soils and XRF intensities. Therefore, the minimum thickness must be carefully evaluated because the minimum thickness for each element is correlated to its characteristic x-ray energy (Padilla et al. 2019).

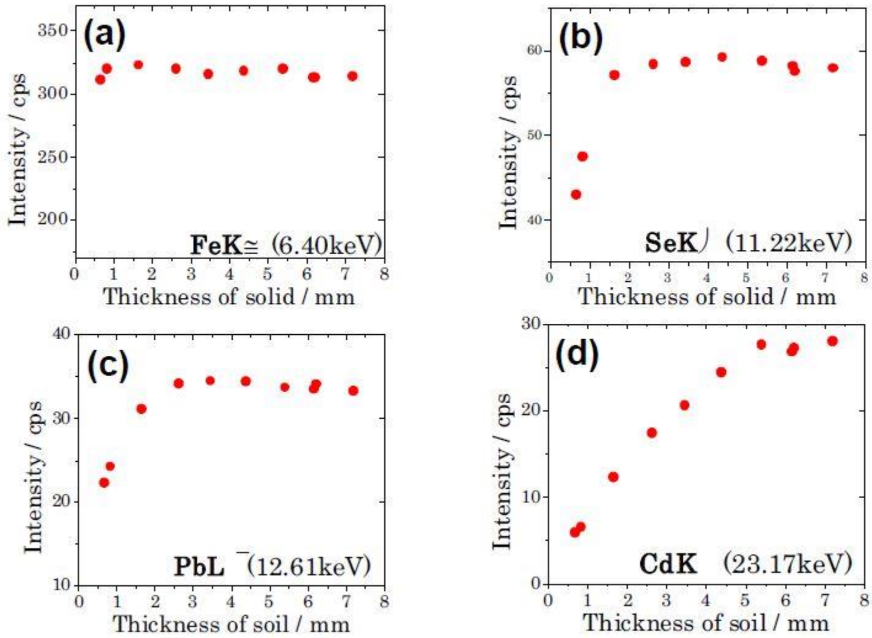



Figure 2.22 Thickness of soil vs XRF intensity (Imanishi et al. 2010)

4. *Particle Size*: Particle size tends to have a significant impact on the accuracy of PHXRF measurements. As discussed previously, Cerato et al. (2017) noticed significant drops in RMSD and $COV_{RMSD}$ when the particles were reduced to smaller sizes. When the particle size was compared against the RMSD values, it was observed that beyond the No. 40 sieve the RMSD values levelled off indicating no noteworthy reduction in the accuracy of XRF measurements. Similarly, Imanishi et al. (2010) also observed that the grain size of the soil sample also influenced XRF intensity. As grain size decreased the XRF intensities increased for low-energy x-rays (Ba, Fe) and high-energy x-rays (Pb, Cd). This is shown in Figure 2.24.

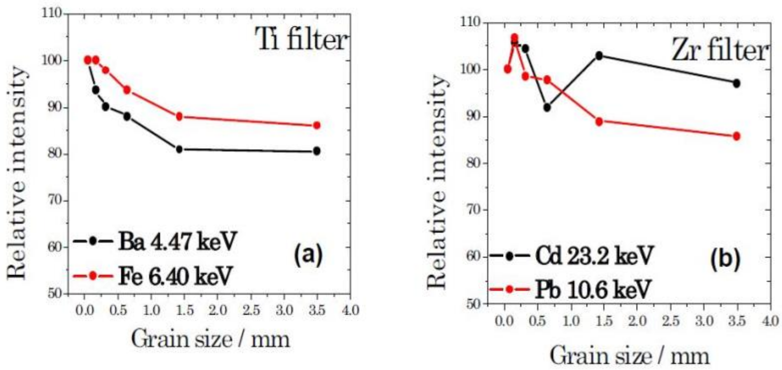



Figure 2.23 Grain size Vs XRF intensity (Imanishi et al. 2010)

5. *Sample Type*: Sample preparation is also known to influence PHXRF measurements. Different sample preparation types such as loose powder, pressed pellets, loose aggregates or unprepared samples can be used for XRF analysis. Inaccurate results may be obtained if the sample is non-homogenous and therefore, some amount of sample preparation should be done to obtain accurate results. The sample type should be selected from a practicality standpoint because samples like pressed pellet may require significant amount of time, cost and effort for its preparation which may not be feasible in field applications (Cerato et al. 2017).

Thus, research is needed to develop this testing protocol to support use of PHXRF in construction applications. Ultimately, this method should provide confidence to stakeholders considering use of RCA, since the characteristics of RCA could be rapidly determined and assessed. Benefits of successful development of this protocol would include the ability for stakeholders to perform rapid QA or QC analysis of RCA, identify RCA inappropriate for use, and determine appropriate uses for RCA materials deemed suitable for use. If successful and subsequently utilized in the field, the long-term benefit of this research could be the promotion of sustainability through greater use of RCA in construction.

# CHAPTER 3: METHODOLOGY

## 3.1 Introduction

Using RCA acquired from four different sources, several experiments were performed to develop a test protocol for estimating the mortar content of RCA, to quantify the elements present within an RCA sample, and identify potential contaminants. Further analysis was performed to determine the accuracy and precision of the PHXRF. As detailed in Chapter 2, in the recent past XRF has been used for heavy and light element analysis across various fields like mining, scrap processing, geotechnical industry, food safety & agriculture, art & archaeology, environment testing etc. A limited amount of research has been conducted with respect to the chemical characterization of RCA using the PHXRF. Therefore, the aim of this section is to illustrate and develop a testing protocol for chemical characterization of RCA in detail suitable for use as a rapid QA/QC test to assist in evaluating the quality of RCA and its suitability for targeted uses. The protocol developed as part of this work will include information regarding the experimental set-up, sample preparation, scanning duration, scanning technique, and data analysis and manipulation required to obtain reasonably accurate results in a time-efficient manner for both laboratory and field applications.

## 3.2 Materials Description and Characterization:

This section provides information about the RCA samples acquired from several sources. Four different RCA types obtained from 1) sites where concrete pavements were demolished and 2) a commercial crushing and grading facility (processing both structural and pavement concrete as well as other C&D waste) to provide a range of types of RCA to explore characteristics and variability. Properties associated with the aggregates acquired from different sources were determined through laboratory testing, and results are presented in this chapter. Physical property and characterization tests included 1) “Standard Test Method for Sieve Analysis of fine and Coarse Aggregates” (ASTM C136/C136M-14), 2) “Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate” (ASTM C 127), and (3) “Standard Test Method for Bulk Density (“ Unit Weight”) and Voids in Aggregate” (ASTM C 29/C 29 M).

One sample (NC_CT1) was collected from building demolition rubble at D.H.Griffin which is a crushing and grading facility in Charlotte, NC (Figure 3.1). A second sample (NC_HW1), shown in Figure 3.2 was obtained from a highway pavement reconstruction project near Durham, NC (contractor: S.T. Wooten), the third sample (NC_CT2) shown in Figure 3.3, is an RCA produced from source concrete containing coastal limestone acquired from Atlantic Coast Industrial, LLC located in Wilmington, NC. Lastly, the fourth sample (NC_AP1), shown in Figure 3.4 was obtained from an airfield pavement reconstruction project at Charlotte-Douglas International Airport (CLT). The aggregates were recycled at the batching plant located in the airport itself and were crushed according to NCDOT’s ABC gradation.

While the first three samples appeared to be fairly “clean” and free of contaminants, the NC_AP1 sample from the airport pavement appeared to contain a significant amount of earthen material and potentially other contaminants. The first three samples have a maximum and nominal maximum aggregate size of 1½” and 1” respectively whereas the airport sample has a maximum and nominal maximum aggregate size of 2” and 1½” respectively. Each of the samples was judged to have an angular shape with a rough texture, except for the coastal aggregate which apeared to have a more porous. This is consistent with the inclusion of North Carolina’s marine limestone as the coarse aggregate in the source concrete used to produce the coastal RCA. The characterization tests and results are provided in the sections below.


Figure 3.1 NC_CT1 RCA Sample (D.H. Griffin)

Figure 3.2 NC_HW1 RCA Sample (S.T. Wooten)


Figure 3.3 NC_CT2 RCA Sample (Coastal)

Figure 3.4 NC_AP1 RCA Sample (CLT Airport)

3.3 Characterization Tests

Approximately 20 5-gallon buckets of each aggregate were retrieved from a representative location on the stockpile and was returned to UNC Charlotte's laboratories. For the purpose of testing material from the three non-airport samples, three buckets which were judged to be representative of the whole 20-bucket sample, were chosen at random and the average value of material tested from those 3 buckets is utilized as the final value for the first three aggregate samples. For the airport sample, the samples were more carefully selected as materials contained in each bucket did not appear uniform. To maintain uniformity, 3 buckets were selected from 3 different groups of aggregates judged to be most uniform.

3.3.1 Sieve Analysis (ASTM C136/C136M-14)

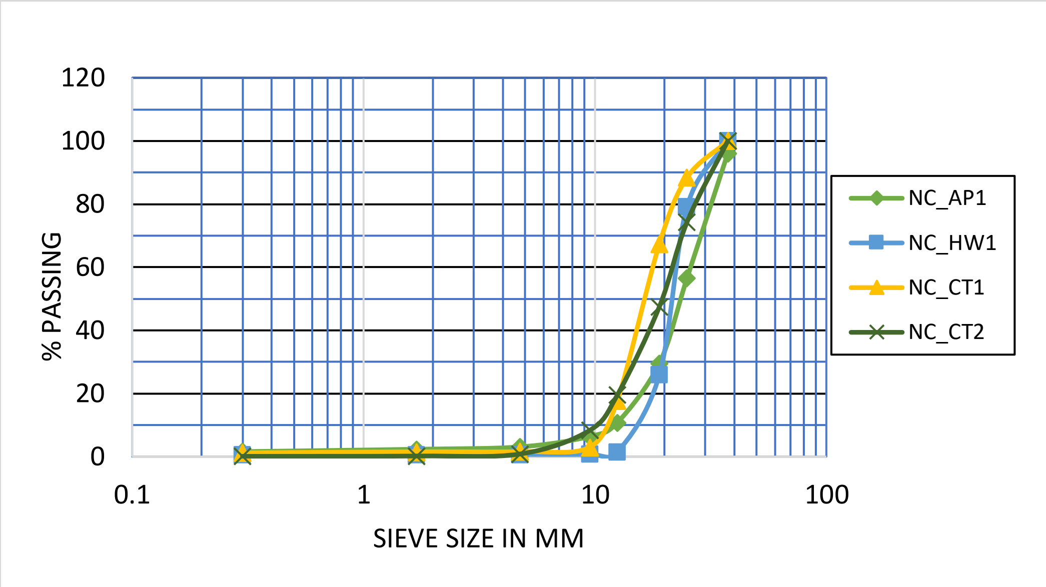



Figure 3.5 Sieve analysis gradation curves

Table 3.1 NC_CT1 Sieve analysis results

| **D.H Griffin RCA Sample** | | | | |
|---|---|---|---|---|
| Sieve Size | Weight Retained (Kg) | Individual % Retained | Cumulative % Retained | %passing |
| 1 1/2" | 0 | 0 | 0 | 100 |
| 1" | 1.16 | 11.6 | 11.6 | 88.4 |
| 3/4" | 2.095 | 20.95 | 32.55 | 67.45 |
| 1/2" | 4.97 | 49.7 | 82.25 | 17.75 |
| 3/8" | 1.475 | 14.75 | 97 | 3 |
| #4 | 0.14 | 1.4 | 98.4 | 1.6 |
| #12 | 0.005 | 0.05 | 98.45 | 1.55 |
| #50 | 0.035 | 0.35 | 98.8 | 1.2 |
| Pan | 0.125 | 1.25 | 100 | 0 |
| **Total** | 10.01 | | | |

Table 3.2 NC_HW1 Sieve analysis results

| **S.T Wooten** | | | | |
|---|---|---|---|---|
| Sieve Size | Weight Retained (Kg) | Individual % Retained | Cumulative % retained | % passing |
| 1 1/2" | 0 | 0 | 0 | 100 |
| 1" | 2.085 | 20.85 | 20.85 | 79.15 |
| 3/4" | 5.315 | 53.15 | 74 | 26 |
| 1/2" | 2.45 | 24.5 | 98.5 | 1.5 |
| 3/8" | 0.075 | 0.75 | 99.25 | 0.75 |
| #4 | 0.005 | 0.05 | 99.3 | 0.7 |
| #12 | 0 | 0 | 99.3 | 0.7 |
| #50 | 0.01 | 0.1 | 99.4 | 0.6 |
| pan | 0.055 | 0.55 | 99.95 | 0 |
| **Total** | 9.995 | | | |

Table 3.3 NC_AP1 Sieve Analysis Results

| Airport | | | | |
|---|---|---|---|---|
| Sieve Size | Weight Retained (kg) | Individual % Retained | Cumulative % retained | % passing |
| 1 1/2" | 0.39 | 3.9 | 3.9 | 96.1 |
| 1" | 3.955 | 39.55 | 43.45 | 56.55 |
| 3/4" | 2.695 | 26.95 | 70.4 | 29.6 |
| 1/2" | 1.88 | 18.8 | 89.2 | 10.8 |
| 3/8" | 0.44 | 4.4 | 93.6 | 6.4 |
| #4 | 0.33 | 3.3 | 96.9 | 3.1 |
| #12 | 0.075 | 0.75 | 97.65 | 2.35 |
| #50 | 0.075 | 0.75 | 98.4 | 1.6 |
| Pan | 0.145 | 1.45 | 99.85 | |
| **Total** | 9.985 | | | |

Table 3.4 NC_CT2 Sieve Analysis Results

| Coastal | | | | |
|---|---|---|---|---|
| Sieve Size | Weight Retained | individual % Ret | Cumulative % ret | % passing |
| 1 1/2" | 0 | 0 | 0 | 100 |
| 1" | 2.57 | 25.7 | 25.7 | 74.3 |
| 3/4" | 2.675 | 26.75 | 52.45 | 47.55 |
| 1/2" | 2.8 | 28 | 80.45 | 19.55 |
| 3/8" | 1.12 | 11.2 | 91.65 | 8.35 |
| #4 | 0.75 | 7.5 | 99.15 | 0.85 |
| #12 | 0.065 | 0.65 | 99.8 | 0.2 |
| #50 | 0.01 | 0.1 | 99.9 | 0.1 |
| pan | 0.03 | 0.3 | 100.2 | 0 |
| **Total** | 10.02 | | | |

3.3.2 Density, Relative Density (Specific Gravity), and Absorption (ASTM C 127)

Table 3.5 RCA Samples Specific Gravity and Absorption Test

| RCA Sample | Bulk Specific Gravity, average (range) | Bulk Specific Gravity, average (range) | Apparent Specific Gravity, average (range) | Absorption %, average (range) |
|---|---|---|---|---|
| NC_CT1 | 2.293 (2.27-2.32) | 2.434 (2.40-2.46) | 2.671 (2.61-2.71) | 6.110 (5.70-6.39) |
| NC_HW1 | 2.210 (2.15-2.30) | 2.354 (2.30-2.37) | 2.581 (2.53-2.61) | 6.493 (6.07-6.70) |
| NC_CT2 | 2.172 (2.16-2.19) | 2.320 (2.31-2.33) | 2.544 (2.53-2.55) | 6.712 (6.36-6.93) |
| NC_AP1 | 2.407 (2.39-2.42) | 2.546 (2.53-2.55) | 2.790 (2.77-2.80) | 5.700 (5.22-5.95) |

3.3.3 Bulk Density and Voids in aggregates (ASTM C 29/C 29 M)

Table 3.6 RCA Samples Bulk Density and Voids in aggregates

| **RCA Sample** | **Bulk Unit Weight (kg/m$^3$), average (range)** | **% Voids, average (range)** |
|---|---|---|
| NC_CT1 | 1346 (1321-1364) | 41.16 (40.24-42.93) |
| NC_HW1 | 1281 (1270-1300) | 41.80 (39.45-43.18) |
| NC_CT2 | 1235 (1223-1248) | 42.98 (42.40-43.55) |
| NC_AP1 | 1506 (1470-1540) | 37.29 (35.93-38.35) |

3.3.4 Thermal shock method

The Thermal Shock Method works on the principle of dehydration of cement paste causing a reduction in strength and increase in pore pressure when subjected to high temperatures. The cement paste undergoes thermal shock when it is exposed to a sudden temperature drop making it extremely brittle. Using this principle, the residual mortar was separated from the natural aggregates by crushing (Mamirov et al. 2020). The detailed procedure of Thermal Shock Method is as follows (Mamirov et al. 2020):


Figure 3.6 Thermal shock method test apparatus (from Mamirov et al. 2020)

1) Prior to testing, any contaminants such as wood, plastic, brick and asphalt etc. present in the sample are removed. Once the sample is free of contaminants, it is sieved and 500g of the sieved sample retained on #4 and above is considered for testing.
2) To provide additional vapor pressure, the sample is soaked in water at a temperature of 23±2 °C.
3) After soaking, the RCA sample is heated for 2 hours by placing it in a furnace and after heating, to cause thermal shock, the sample is submerged in 1350g of water for a duration of 30 minutes at a temperature of 23±2 °C.
4) The RCA sample is then oven dried for 24±4 hours at 110±5˚C. After oven drying, the process of removal of residual mortar begins as the thermal shock makes the residual mortar brittle.
5) The removal process is conducted using a jar mill which consists of a 13/16 inch x 13/16 inch cylindrical alumina grinding media. The RCA sample is ground with 575g of the grinding media at a speed of 70 rpm and the progress is inspected at regular intervals. When the removal process is completed, the RCA sample is separated into coarse aggregate and residual mortar by sieving through the No. 4 sieve. The RMC content is determined using the following formula:

$$RMC\ (\%) = \frac{\text{Mass of material passed \#4 sieve}}{\text{Total mass}} \times 100\%$$

This procedure was repeated for all four of the RCA samples acquired for this work (Airport, D.H Griffin, S.T Wooten and Coastal). The following mortar % were computed from this procedure:

Table 3.7 RCA mean RMC%

| RCA Sample | Mean RMC% |
|---|---|
| Airport | 33.67 |
| DH Griffin | 41.93 |
| ST Wooten | 43.93 |
| Coastal | 55.76 |

# CHAPTER 4: DEVELOPMENT OF PROTOCOL FOR USE OF HANDHELD XRF FOR CHEMICAL CHARACTERIZATION OF RCA

## 4.1 Approach and Data Analysis

For this research study the capability of the PXHRF to determine the mortar content and chemical composition of each RCA sample was compared to reference values. The reference values for the composition of each RCA sample, including mortar (alone), coarse aggregate (alone) and fine aggregate (alone) was determined using the results of laboratory testing described subsequently in this chapter. The weights of selected elements present in the samples were determined by a commercial laboratory via conventional "whole rock analysis." By comparing PHXRF results to values determined by outside laboratories using more extensive, time consuming techniques, the accuracy and repeatability of the results obtained from the PHXRF could be evaluated, and selected sample preparation and analysis techniques refined into a recommended testing protocol. This approach is shown in Figure 4.1. An additional "reference sample" was considered to be the test results from the PHXRF testing of the mortar (alone) and aggregate (alone) removed from each RCA sample. Although this approach does not provide an indication of the accuracy of the device, it should provide insight into the repeatability of results of testing fractionated sizes of RCA.

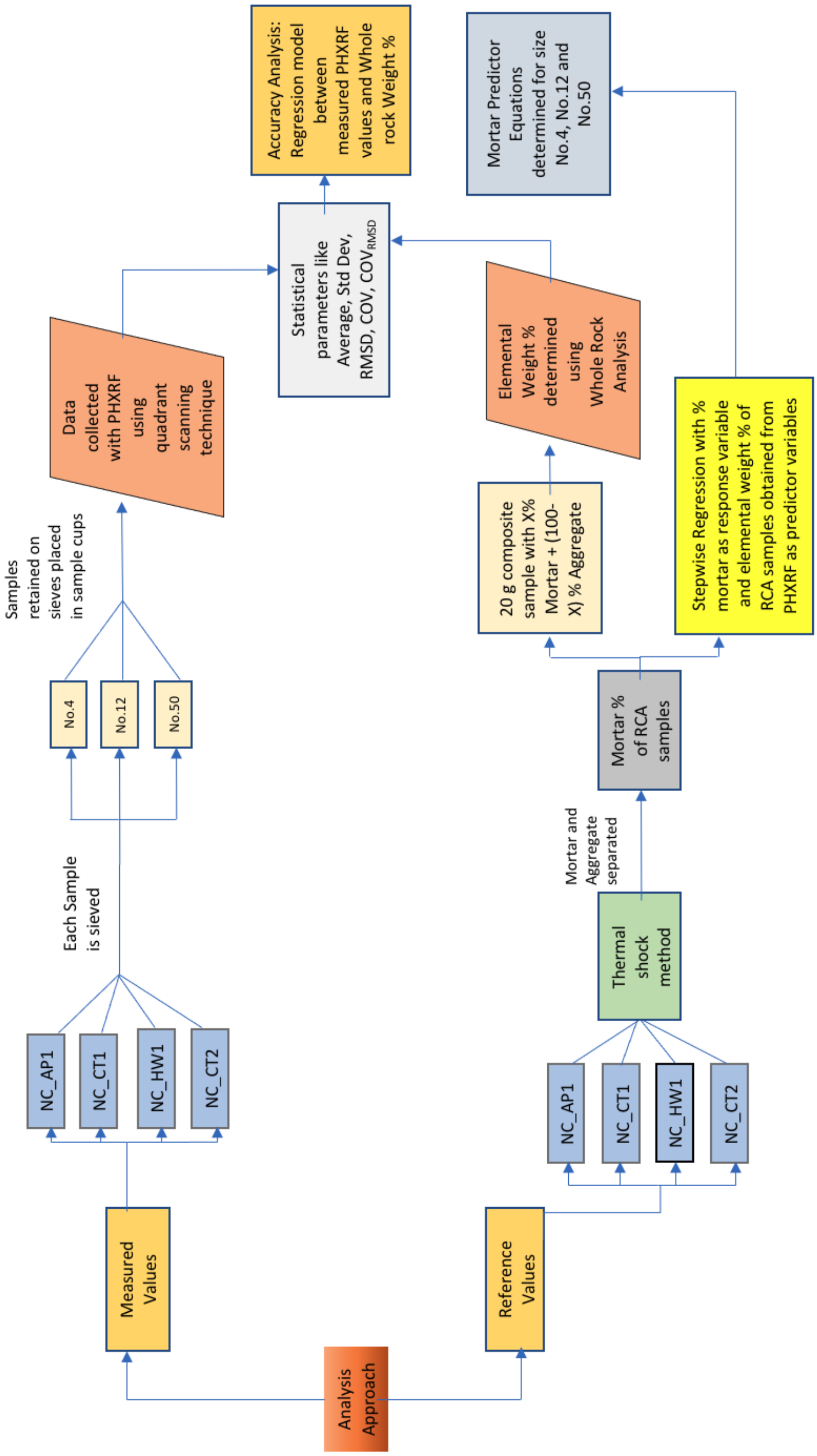



Figure 4.1Approach utilized to evaluate accuracy and precision of handheld XRF for chemical characterization of RCA

As shown in the figure, the approach of the analysis was to obtain values from PHXRF for the RCA samples and then compare the results against reference values. To obtain measured values, the samples were sieved through sieve sizes 1½”, 1”, ¾”, ½”, 3/8”, No. 4, No. 12 and No. 50. The samples retained on sieve sizes No. 4, No. 12 and No. 50 were bagged for analysis. The elemental weight % of the elements present in the samples were determined by PHXRF analysis. For the reference samples, the thermal shock method was first used to separate the mortar and aggregate from RCA samples to determine the percent mortar (by weight) and percent aggregate (by weight). Using the mortar percent, a 20 g composite sample was prepared which comprised of X% of 20g + (100-X) % of 20 g, where X = mortar content and 100-X is the aggregate content. This process was repeated for each of the RCA samples and then was sent for “whole rock analysis” to determine its chemical composition in weight percent. After data collection, models were computed to predict the mortar content using stepwise regression from the PHXRF results and the mortar % computed from Thermal Shock Method. For determining the chemical composition of RCA samples, a simple linear regression was performed to predict the relationship between the PHXRF elemental weight % and the weight % of elements obtained from laboratory testing (Whole Rock Analysis). Based on the $R^2$ values observed from this regression analysis, the best particle size was recommended for the PHXRF analysis. The procedure for the PHXRF analysis will be described in the subsequent sections (Section 4.2)

Statistical analysis tools utilized for this work included root-mean-squared deviations (RMSD), regression analysis with coefficients of determination ($r^2$), 95% confidence and prediction intervals, and between the PHXRF and reference laboratory measurements and calculated quantities (Cerato et al. 2017). The standard deviation indicates how dispersed the data is around its mean. Data clustered around its mean is indicated by a low standard deviation value (closer to 0) and data spread out around the mean is represented by a high value (National Library of Medicine 2019). Another, statistical term $COV_{STDEV}$ is the ratio of standard deviation of a data set to its mean. This parameter determines the measure of the variability of a random variable from its mean (Koopmans et al. 1964). The repeatability or precision of the PHXRF can be determined using this parameter. A low value of $COV_{STDEV}$ (closer to 0) indicates more repeatability. The RMSD is defined as the standard deviation of the residuals. The purpose of RMSD is to indicate how spread out the data points are from the regression lines. Values closer to 0 suggest higher accuracy (Glen 2020). Lastly, $COV_{RMSD}$ is used in a model setting and is represented as the ratio of RMSD to the mean of the dependent variable. This parameter indicates the variability of measurements relative to true deviation. Once these statistical parameters were calculated, a regression analysis was used to compare the accuracy of the measured values vs. reference values.

## 4.2 Test configuration and procedure:

For conducting this research study, a Bruker Tracer III-SD series handheld XRF analyzer was used. This device is based on the energy dispersive X-ray fluorescence technology and uses a rhodium target (x-ray source) and a Silicon Drift Detector system for x-ray detection. The PHXRF analyzer can be operated as either a handheld device or in a bench-top setup. Based on the testing requirements, the bench-top test setup was deemed as the most suitable configuration. In this testing setup, the instrument is mounted on a desktop stand and a sample table is fixed to the nose of the instrument to provide a flat working surface. The sample cup is then placed on the sample table in an inverted fashion such that the open end of the sample cup secured with mylar film rests on the nose of the device. The sample cup is then enclosed in a sample shield to prevent radiation exposure to the user. Once the instrument is physically configured, the instrument is connected to a PC notebook with a USB cable. This connection enables the user to control the instrument and analyse the spectrum generated from the analysis of the sample via the S1PXRF software. The elements detected by the instrument are quantified using the relevant calibration file which comes with the software.

## 4.3 List of influencing factors considered:

List of factors influencing readings of the HXRF (refer to Section 2.5.1 for additional background information on each factor):

i. Scan duration
ii. Scanning technique
iii. Sample type
iv. Particle sizes
v. Sample thickness
vi. Calibration and instrument settings

### 4.3.1 Scan Duration:

A scan duration of 180 seconds and 60 seconds was selected for major (Na to Fe) and trace (Fe to U) elements respectively for the analysis of samples. A 180 second scan duration was utilized to excite light elements (Na to Fe) and the 60 second scan duration was utilized to excite heavier elements (Fe to U). Despite the observation made by Cerato et al. (2017) that “longer scan durations do not yield appreciable benefits in terms of precision or accuracy and are, therefore, unnecessary,” the scan duration was selected based on the ‘Murdock/Ceramic’ calibration file suggested for use by the PHXRF manufacturer (Bruker). For the ‘Mudrock/Ceramic’ calibration file to quantify results accurately, the instrument was used under the same settings under which it was calibrated. A detailed description about the Mudrock/Ceramic calibration and the corresponding instrument settings is discussed in a subsequent section (Section 4.3.6). Hence, the testing was done with a scan duration of 180 and 60 seconds for light and heavy elements, respectively.

4.3.2 Sample Type and Preparation:

Loose aggregate samples were used in this analysis. This sample type was selected due to it relatively easy preparation. Alternative sample preparations suggested in the literature, such as a pressed pellet sample, require substantial amount of time to prepare and may not be feasible to prepare in the field.

The loose aggregate sample retained on sieve No. 4, No. 12 and No. 50 were tested, one size fraction per sample cup. Each sample was placed into a cylindrical sample cup with an internal diameter of 37 mm and an internal height of 15 mm and was secured using a 6 µm thick mylar film over the opening of the sample cup.

Figure 4.2 Sample Preparation

Apart from samples prepared from the fractionated sizes of the four types of RCA, other samples were also prepared for the validation. These additional samples included mortar (alone) and aggregate (alone) obtained from each RCA. The mortar and aggregate samples were separated using the Thermal Shock method as described in Section 3.3.4. Once the mortar and aggregate were separated, they were placed in different sample cups and secured using mylar film.

In addition to these samples, other samples like cement (Type I/II), fly ash (class F) and a mixture of cement and fly ash was also prepared to assist in evaluating the PHXRF measurements of the RCA samples. The cement sample was prepared by making a cement paste with a water to cement (w/c) ratio of 0.45 whereas the cement/fly ash mixture was prepared in a manner similar to the cement paste, but by substituting Class F fly ash for cement at a 20% replacement by weight. A water to cementitious (w/cm) ratio of 0.45 was used to prepare this sample. Both of these samples were prepared in a manner similar to that used by (Taylor et al. 2012).

4.3.3 Scan Technique:

For the analysis, a quartering technique was used similar to the approach developed by Cerato et al. (2017). Each sample cup was divided into 4 quadrants, and each quadrant was scanned 3 times at 3 different locations for a total of 12 scans per sample cup. The average value of the 12 scans was computed for both major and trace elements. Major and trace elements, respectively, are listed in Table 4.1. As shown in Figure 4.3 below, the sample cup was divided into 4 quadrants and was placed on the nose of the instrument in an inverted position.

Table 4.1: Major elements and trace elements quantified by the PHXRF for this study, listed in order of atomic weight

| Major elements | Trace elements |
| --- | --- |
| Na | Co |
| Al | Ni |
| Si | Cu |
| P | Zn |
| S | As |
| K | Rb |
| Ca | Sr |
| Ti | Y |
| V | Zr |
| Cr | Nb |
| Mn | Mo |
| Fe | Sn |
| | Sb |
| | Ba |
| | Th |
| | U |

Figure 4.3 Quadrant scanning technique

4.3.4 Surface Thickness:

Another important influencing factor, surface thickness, was also taken into consideration. As previously described in the literature review (Section 2.5.1), the minimum thickness of each element is correlated to its characteristic x-ray energy (Padilla et al. 2019). Each sample must meet the minimum thickness criteria, which describes the minimum depth of the sample required to absorb the primary x-ray emitted from the PXRF followed by emission of characteristic x-ray from the sample. There are two essential terms associated with this concept; ‘penetration depth,’ which refers to how deep into the sample the primary x-ray penetrates, and the second term, ‘Escape Depth’ refers to the depth from which the secondary radiation can be detected from. It is normally assumed that escape depth is exceeded upon exceeding the penetration depth (Bruker 2020). Therefore, it was important to evaluate surface thickness of a sample because it will be different for every material. Denser samples tend to require less thickness. In one experiment conducted to differentiate Near Eastern obsidian source using PHXRF, the minimum depth required for precise and accurate results for obsidian was determined to be 3mm (Forster and Grave 2012). As described in Section 2.5.1, in the experiment conducted by Imanishi et al. (2010) to quantify the presence of harmful elements in soil using XRF, the minimum soil thickness was determined to be 6mm and for the sake of analysing all elements from light to heavy, a thickness of 10 mm was fixed for the experiment. Since the manufacturer-prepared Mudrock/Ceramic calibration file is being used for this work, the manufacturer has provided a list of minimum thickness for various elements at different characteristic energies. The list is shown in Table 4.2:

Table 4.2 Mudrock/Ceramic analysis depth (from Bruker)

| Element | Photon Emitted energy (keV) | Analysis depth in Ceramic(cm) |
|---|---|---|
| O | 0.53 | 0.000001 |
| Na | 1.04 | 0.0007 |
| Mg | 1.2 | 0.00096 |
| Al | 1.47 | 0.0017 |
| Si | 1.74 | 0.0027 |
| P | 2.01 | 0.0013 |
| Ca | 3.69 | 0.0064 |
| Cr | 5.41 | 0.0192 |
| Fe | 6.4 | 0.03 |
| Cu | 8.01 | 0.058 |
| Zn | 8.64 | 0.077 |
| Pb | 10.55 | 0.113 |
| Zr | 15.78 | 0.384 |

Keeping in mind the above observations related to the minimum depth, the sample cup chosen for this analysis had a depth of 15 mm to mitigate the inaccuracies caused due to infinite thickness phenomenon. A cross-section of the sample cup used for the purpose of experimentation is shown in Figure 4.4.

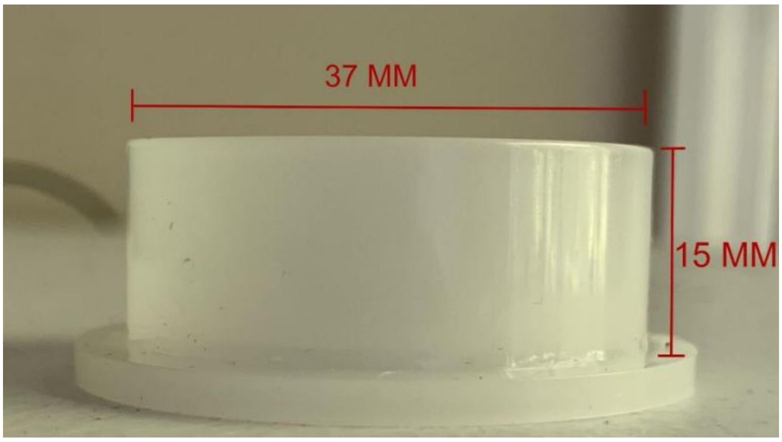
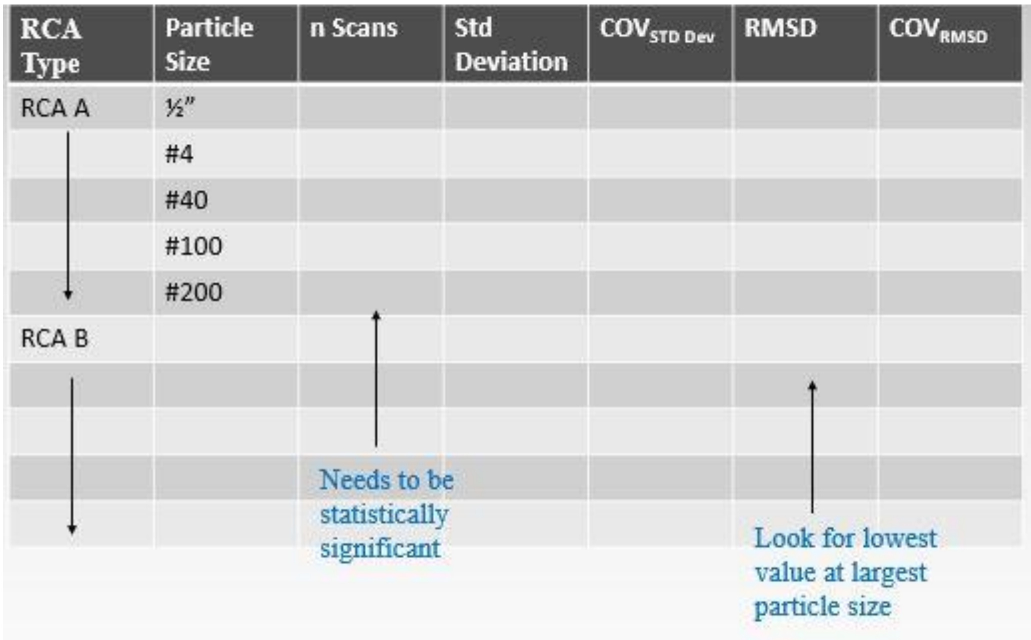


Figure 4.4 Sample Cup Dimension

4.3.5 Particle size:

As described in the literature review, the particle size has been shown to plays the most significant role in the accuracy of the results obtained from the handheld XRF. The RCA samples were sieved through the sieve sizes 1½ inch, 1 inch, ¾ inch, ½ inch, 3/8 inch, No. 4, No. 12, and No. 50. The objective was to achieve highly accurate results by selecting the appropriate particle size for analysis. After sieving, the sample retained on sieve size No. 4, No. 12 and No. 50 were collected and bagged for XRF analysis. The main goal was to identify the lowest value of RMSD at the largest particle as shown in Figure 4.5. Beyond this value, the effects of particle size on RMSD would level off (Cerato et al. 2017). This is also shown in Figure 2.13.



| RCA Type | Particle Size | n Scans | Std Deviation | COV<sub>STD Dev</sub> | RMSD | COV<sub>RMSD</sub> |
|---|---|---|---|---|---|---|
| RCA A | ½" | | | | | |
| | #4 | | | | | |
| | #40 | | | | | |
| | #100 | | | | | |
| | #200 | | | | | |
| RCA B | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |



Figure 4.5 Effects of Particle size on XRF STDEV, $COV_{stdev}$, RMSD, $COV_{RMSD}$

4.3.6 Calibration and Instrument Settings:

As per the PHXRF manufacturer's recommendation, the 'Mudrock/Ceramic' calibration file was used for quantifying the elements obtained from PHXRF analysis. The results are reported as weight percentage of the elements that are measured. **Some elements like carbon, oxygen and hydrogen cannot be measured by the device as these elements are not detected by detector of the instrument. Due to this reason, the total weight % will not add up to 100% (Bruker 2013).**

The Mudrock calibration has two parts; the first part is to quantify major (lighter) elements, i.e. Na to Fe and the second part is quantification of trace elements i.e. Fe to U. The instrument requires different settings for the quantification of major and trace elements and are listed in the table below:

Table 4.3 Instrument settings

| Major Element Settings | Trace Element Settings |
|---|---|
| • Voltage: 15 kV<br>• Current: 35 µA<br>• Scan duration: 180 seconds<br>• Filter: None<br>• Vacuum Pump attached | • Voltage: 40 kV<br>• Current: 10 µA<br>• Scan duration: 60 seconds<br>• Filter: Yellow<br>• No Vacuum |

The Major Element settings requires the use of a vacuum pump provided with the device. Use of this vacuum pump aids in obtaining highly accurate measurements of light elements by removing the surrounding air present between detector and the sample which allows maximum number of X-rays to reach the detector. This pump was connected to the PHXRF with a hose which has a slide valve with an open/close mechanism. Once connected, the instrument was ready for measurement when the display of the pump read less than 10 Torr.

Figure 4.6 PHXRF connected to a vacuum pump

Another important setting for trace element analysis included a filter. The primary function of the filter is to optimize the excitation conditions for a group of elements (heavy elements) when used with a certain voltage setting (Bruker 2010). The combination of the filter and the tube voltage made the device more sensitive to certain elements. Hence, a yellow filter, composed of Ti and Al was used for trace element analysis which allowed x-rays from 12 to 40 KeV to reach the sample (Speakman 2015).

Figure 4.7 Yellow filter

4.4 Test Results and Statistical Analysis:

This section provides information about the statistical procedure and parameters used to analyze the data collected by testing the RCA samples. After the physical characterization of the RCA samples, the samples were tested according to the procedure described in Section 4.2 to obtain the PHXRF test results.

4.4.1 Control Sample:

To evaluate the results obtained from the PHXRF testing of RCA samples, control samples were prepared and then sent to an external laboratory for analysis. The control samples were prepared for each of the 4 RCA samples. To prepare the control samples, the Thermal Shock Method was used to separate the aggregate and mortar. Using this method the mean residual mortar content (RMC) was calculated for #67 graded aggregate size which was selected as the representative size for analysis. Once the RMC was determined for each sample, a 20g composite sample (mixture of mortar and aggregate) was prepared according to the laboratory-measured RMC percent shown in

Table 3.6 So the composite mixture of 20 g is comprised of X% of 20g + (100-X)% of 20g, where X = RMC%, and 100-X = Aggregate wt%. The table below shows the RMC percent and weight distribution:

Table 4.4 Control Sample Composition

| RCA Sample | Laboratory-measured Mean RMC% | X% of 20 grams | (100-X)% of 20 grams | Total Weight (grams) |
|---|---|---|---|---|
| NC_AP1 | 33.67 | 6.73 | 13.26 | 20 |
| NC_CT1 | 41.93 | 8.38 | 11.61 | 20 |
| NC_HW1 | 43.93 | 8.78 | 11.21 | 20 |
| NC_CT2 | 55.76 | 11.15 | 8.84 | 20 |

4.4.2 Test Results

The table below provides the average weight % of each element from the 4 RCA samples tested using the PHXRF. As a reminder, these averages represent the mean computed from 12 measurements (3 measurements taken randomly from each of 4 quadrants of the sample). The raw data for all PHXRF measurements has been included in the Appendix A from table A.1 to A.23.

Table 4.5 Average PHXRF concentrations in weight %

| Elements | NC_AP1 | | | NC_CT1 | | | NC_HW1 | | | NC_CT2 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | No.4 | No.12 | No.50 | No.4 | No.12 | No.50 | No.4 | No.12 | No.50 | No.4 | No.12 | No.50 |
| Na | 0.0809 | 0.0000 | 0.0516 | 0.0008 | 0.0112 | 0.0000 | 0.0000 | 0.0329 | 0.0000 | 0.2808 | 0.2490 | 0.1556 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 1.1005 | 1.1205 | 1.4142 | 0.3400 | 0.4525 | 0.4540 | 0.5891 | 0.8335 | 0.9614 | 0.0000 | 0.0042 | 0.0084 |
| Si | 4.2152 | 4.3376 | 5.1253 | 3.8349 | 4.2804 | 6.7317 | 4.5482 | 4.1852 | 4.8632 | 2.3525 | 2.2261 | 3.0400 |
| P | 0.0190 | 0.0040 | 0.0109 | 0.0069 | 0.0147 | 0.0012 | 0.0117 | 0.0014 | 0.0005 | 0.0188 | 0.0091 | 0.0000 |
| S | 0.5025 | 0.4984 | 0.5051 | 0.5628 | 0.5662 | 0.5866 | 0.5608 | 0.5012 | 0.5017 | 0.2613 | 0.2855 | 0.3591 |
| K | 0.2779 | 0.2708 | 0.2967 | 0.2366 | 0.3005 | 0.2588 | 0.5808 | 0.6666 | 0.9526 | 0.1108 | 0.1468 | 0.1474 |
| Ca | 4.1318 | 4.4303 | 4.0024 | 6.3559 | 7.3314 | 5.8792 | 4.4733 | 4.7039 | 5.1282 | 13.3876 | 14.0301 | 13.0233 |
| Ba | 0.3778 | 0.6236 | 0.8227 | 0.2238 | 0.0098 | 0.0966 | 0.0480 | 0.0769 | 0.0027 | 0.0000 | 0.0287 | 0.0103 |
| Ti | 0.2132 | 0.0970 | 0.0812 | 0.0246 | 0.1109 | 0.0701 | 0.0519 | 0.0645 | 0.0883 | 0.0192 | 0.0198 | 0.0325 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0013 | 0.0019 | 0.0055 | 0.0033 | 0.0042 | 0.0063 | 0.0049 | 0.0038 | 0.0054 |
| Cr | 0.0047 | 0.0063 | 0.0066 | 0.0045 | 0.0039 | 0.0060 | 0.0043 | 0.0054 | 0.0052 | 0.0023 | 0.0025 | 0.0034 |
| Mn | 0.0284 | 0.0275 | 0.0282 | 0.0223 | 0.0243 | 0.0281 | 0.0248 | 0.0239 | 0.0240 | 0.0215 | 0.0226 | 0.0224 |
| Fe | 2.0522 | 1.0289 | 0.9035 | 0.3143 | 1.5305 | 0.8296 | 0.9243 | 0.9277 | 1.3025 | 0.2938 | 0.3408 | 0.4266 |
| Co | 0.0345 | 0.0061 | 0.0012 | 0.0027 | 0.0043 | 0.0065 | 0.0057 | 0.0008 | 0.0075 | 0.0022 | 0.0029 | 0.0028 |
| Ni | 0.0017 | 0.0036 | 0.0031 | 0.0026 | 0.0024 | 0.0012 | 0.0012 | 0.0022 | 0.0012 | 0.0021 | 0.0026 | 0.0018 |
| Cu | 0.0019 | 0.0072 | 0.0083 | 0.0042 | 0.0083 | 0.0032 | 0.0006 | 0.0043 | 0.0023 | 0.0038 | 0.0049 | 0.0011 |
| Zn | 0.0027 | 0.0056 | 0.0061 | 0.0053 | 0.0097 | 0.0025 | 0.0027 | 0.0062 | 0.0026 | 0.0031 | 0.0006 | 0.0025 |
| As | 0.0002 | 0.0004 | 0.0004 | 0.0004 | 0.0006 | 0.0002 | 0.0003 | 0.0005 | 0.0005 | 0.0003 | 0.0002 | 0.0002 |
| Pb | 0.0012 | 0.0012 | 0.0009 | 0.0010 | 0.0011 | 0.0011 | 0.0014 | 0.0012 | 0.0015 | 0.0010 | 0.0010 | 0.0011 |
| Th | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0006 | 0.0010 | 0.0008 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0010 | 0.0010 | 0.0015 | 0.0019 | 0.0022 | 0.0007 | 0.0040 | 0.0102 | 0.0060 | 0.0005 | 0.0004 | 0.0005 |
| U | 0.0044 | 0.0004 | 0.0005 | 0.0006 | 0.0007 | 0.0003 | 0.0002 | 0.0005 | 0.0004 | 0.0005 | 0.0004 | 0.0005 |
| Sr | 0.0374 | 0.0818 | 0.0554 | 0.0357 | 0.0354 | 0.0108 | 0.0156 | 0.0392 | 0.0240 | 0.0404 | 0.0358 | 0.0290 |
| Y | 0.0008 | 0.0025 | 0.0026 | 0.0023 | 0.0025 | 0.0018 | 0.0037 | 0.0023 | 0.0010 | 0.0019 | 0.0020 | 0.0017 |
| Zr | 0.0089 | 0.0103 | 0.0115 | 0.0092 | 0.0132 | 0.0072 | 0.0176 | 0.0188 | 0.0112 | 0.0097 | 0.0136 | 0.0093 |
| Nb | 0.0008 | 0.0007 | 0.0007 | 0.0006 | 0.0007 | 0.0005 | 0.0010 | 0.0012 | 0.0010 | 0.0004 | 0.0005 | 0.0005 |
| Mo | 0.0079 | 0.0003 | 0.0005 | 0.0018 | 0.0008 | 0.0082 | 0.0063 | 0.0005 | 0.0069 | 0.0013 | 0.0022 | 0.0031 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0001 | 0.0001 | 0.0002 | 0.0001 | 0.0001 | 0.0005 | 0.0001 | 0.0001 | 0.0004 | 0.0002 | 0.0000 | 0.0003 |

### 4.4.3 Statistical Analysis:

Statistical analysis of the data obtained using the PHXRF began with a one-way analysis of variance that was performed on the data sets to prove a statistically significant differences between the test results obtained for 1) different size fractions of RCA (of the same type), and 2) RCA obtained from different sources (using the same size fraction). The ANOVA test is used on more than two data sets to determine if the population means are equal or not with some statistical certainty. This test helps identify if there is a significant difference between the data sets by using either the F-test or Welch's F-test depending on whether the variances of each data set are equal or not. The ANOVA tests the following hypotheses:

Null Hypothesis: $H_0 = \mu_1 = \mu_2 = \ldots = \mu_n$

Alternate Hypothesis: $H_1$= The mean of at least one population is unequal

The fundamental concept behind the ANOVA test is that the total variation is divided into two parts in the dependent variable: one part is the variation within the samples which is attributed to chance and the second part is the variation between samples which is attributed to specific causes (Molugaram and Rao 2017).

The ANOVA test was performed on data sets to prove a statistically significant difference between the test results for different sizes (No. 4, No. 12 and No. 50) within each RCA sample and, for each fractionated size, across each of the four RCA samples. To conduct this test, Minitab, a general-purpose statistical analysis software, was used. Before conducting the ANOVA test, Levene's test, a test for testing the equality of variances was performed on the data sets, to determine which ANOVA test is used. If the variances are equal, then the standard F-test is used, whereas in case of unequal variances, Welch's F-test is used to test the hypotheses. Based on the equality of variances the post-hoc test also changes. For equal variances, Tukey's test is used and for unequal variances the Games-Howell test is used to show a pairwise comparison that indicates which data sets are statistically different and which are not. These tests were performed at a significance level of 5% ($\alpha$=0.05). Therefore, tests which returned a p-value of less than or equal to 0.05 were considered as statistically significant (highlighted in green). The Summary tables of ANOVA test are shown in Tables 4.6 and 4.7. The null and alternate hypothesis for table 4.6 would be $H_0$: The sample size has no effect on the elemental concentrations and $H_1$: The sample size affects the elemental concentrations of at least one size group. For table 4.7, $H_0$: The sample source has no effect on the elemental concentrations of the samples and $H_1$: The sample source affects the elemental concentrations of at least one sample group. In this table “Yes” means the p-value for the given element is less than 0.05 and is statistically significant, and “No” means p-value for the given element is greater than 0.05 and is statistically insignificant.

Table 4.6 ANOVA test for RCA samples- No.4, No.12, No.50

| Major/Trace Elements | P-value | | | | Statistically significant | | | |
|---|---|---|---|---|---|---|---|---|
| | NC_AP1 | NC_CT1 | NC_HW1 | NC_CT2 | NC_AP1 | NC_CT1 | NC_HW1 | NC_CT2 |
| Al | 0.000 | 0.182 | 0.005 | -- | Yes | No | Yes | -- |
| Si | 0.001 | 0.000 | 0.189 | 0.000 | Yes | Yes | No | Yes |
| P | 0.169 | 0.007 | 0.023 | -- | No | Yes | Yes | -- |
| S | 0.928 | 0.471 | 0.226 | 0.000 | No | No | No | Yes |
| K | 0.236 | 0.171 | 0.000 | 0.090 | No | No | Yes | No |
| Ca | 0.233 | 0.008 | 0.311 | 0.535 | No | Yes | No | No |
| Ba | 0.212 | 0.005 | 0.276 | -- | No | Yes | No | -- |
| Ti | 0.010 | 0.000 | 0.007 | 0.032 | Yes | Yes | Yes | Yes |
| V | -- | 0.058 | 0.075 | 0.064 | -- | No | No | No |
| Cr | 0.007 | 0.012 | 0.020 | 0.001 | Yes | Yes | Yes | Yes |
| Mn | 0.864 | 0.013 | 0.562 | 0.092 | No | Yes | No | No |
| Fe | 0.034 | 0.000 | 0.054 | 0.333 | Yes | Yes | No | No |
| Co | 0.002 | 0.023 | 0.379 | 0.300 | Yes | Yes | No | No |
| Ni | 0.007 | 0.000 | 0.002 | 0.078 | Yes | Yes | Yes | No |
| Cu | 0.000 | 0.000 | 0.000 | 0.073 | Yes | Yes | Yes | No |
| Zn | 0.000 | 0.000 | 0.000 | 0.023 | Yes | Yes | Yes | Yes |
| As | 0.000 | 0.000 | 0.381 | 0.036 | Yes | Yes | No | Yes |
| Pb | 0.020 | 0.788 | 0.248 | 0.830 | Yes | No | No | No |
| Th | 0.074 | 0.013 | 0.000 | 0.001 | No | Yes | Yes | Yes |
| Rb | 0.058 | 0.003 | 0.000 | 0.834 | No | Yes | Yes | No |
| U | 0.018 | 0.082 | 0.630 | 0.940 | Yes | No | No | No |
| Sr | 0.000 | 0.000 | 0.000 | 0.007 | Yes | Yes | Yes | Yes |
| Y | 0.000 | 0.011 | 0.000 | 0.162 | Yes | Yes | Yes | No |
| Zr | 0.058 | 0.000 | 0.001 | 0.042 | No | Yes | Yes | Yes |
| Nb | 0.066 | 0.002 | 0.003 | 0.009 | No | Yes | Yes | Yes |
| Mo | 0.000 | 0.001 | 0.000 | 0.184 | Yes | Yes | Yes | No |
| Sn | 0.000 | 0.000 | 0.001 | 0.016 | Yes | Yes | Yes | Yes |
| Sb | 0.342 | 0.042 | 0.016 | 0.125 | No | Yes | Yes | No |

Table 4.7ANOVA test for NC_AP1/NC_CT1/NC_CT2/NC_HW1 RCA sample-No.4, No.12 & No.50

| Major/Trace Elements | P-value NC_AP1, NC_CT1, NC_HW1, NC_CT2 | | | Statistically significant | | |
|---|---|---|---|---|---|---|
| | No.4 | No.12 | No.50 | No.4 | No.12 | No.50 |
| Al | -- | 0 | 0 | -- | Yes | Yes |
| Si | 0 | 0 | 0 | Yes | Yes | Yes |
| P | 0.381 | 0.025 | 0.008 | No | Yes | Yes |
| S | 0 | 0 | 0 | Yes | Yes | Yes |
| K | 0 | 0 | 0 | Yes | Yes | Yes |
| Ca | 0 | 0 | 0 | Yes | Yes | Yes |
| Ba | -- | 0.027 | 0.002 | -- | Yes | Yes |
| Ti | 0 | 0 | 0 | Yes | Yes | Yes |
| V | -- | -- | -- | -- | -- | -- |
| Cr | 0 | 0 | 0 | Yes | Yes | Yes |
| Mn | 0.004 | 0.001 | 0 | Yes | Yes | Yes |
| Fe | 0 | 0 | 0 | Yes | Yes | Yes |
| Co | 0 | 0 | 0 | Yes | Yes | Yes |
| Ni | 0 | 0 | 0 | Yes | Yes | Yes |
| Cu | 0.055 | 0.001 | 0 | No | Yes | Yes |
| Zn | 0.018 | 0 | 0 | Yes | Yes | Yes |
| As | 0.029 | 0 | 0.25 | Yes | Yes | No |
| Pb | 0.063 | 0.683 | 0.001 | No | No | Yes |
| Th | 0 | 0 | 0 | Yes | Yes | Yes |
| Rb | 0 | 0 | 0 | Yes | Yes | Yes |
| U | 0.01 | 0.394 | 0.815 | Yes | No | No |
| Sr | 0 | 0 | 0 | Yes | Yes | Yes |
| Y | 0 | 0.004 | 0 | Yes | Yes | Yes |
| Zr | 0.021 | 0 | 0.001 | Yes | Yes | Yes |
| Nb | 0 | 0 | 0 | Yes | Yes | Yes |
| Mo | 0 | 0.012 | 0.012 | Yes | Yes | Yes |
| Sn | 0 | 0 | 0.038 | Yes | Yes | Yes |
| Sb | 0.487 | 0.793 | 0.053 | No | No | No |

Based on the ANOVA test results and the interpretation of p-value, it was observed that for particles size within each sample, majority of the elements are statistically significant which implied that the PHXRF results were affected by particle sizes for major and trace elements, although there were few elements that were insignificant and indicated that the results were not affected by particle size, at least for the samples used in this analysis.

Similarly, another set of ANOVA test were run across samples of the same particle size. It was observed that majority of the elements were statistically significant indicating that the results were indeed affected by the source concrete of the samples obtained. However, a few elements including P (No. 4), Cu (No. 4), As (No. 50), Pb (No. 4 and No. 12), U (No. 12 and No. 50) and Sb (No. 4, No. 12, and No. 50) were statistically insignificant which could be attributed to the fact that they were present in very low concentration. The test results (output) obtained from Minitab software are presented in Appendix B.

Once the ANOVA tests were completed, a series of statistical parameters including the mean, standard deviation, RMSD and COV were determined to analyze the accuracy of the PHXRF analysis. As discussed in the literature review section, to understand the effects of particle size on PHXRF results, RMSD values were computed for each element present in the sample. The RMSD was calculated by determining the sample standard deviation of the difference between the laboratory measurements and PHXRF measurements (Cerato et al. 2017). The RMSD values were observed for each particle size to make conclusions about the effects of particle size on the elemental concentration. As mentioned previously, the raw data for the PHXRF measurements has been included in the Appendix A from table A.1 to A.23.

The statistical parameter values discussed above have been presented in the tables below with the RMSD column showing high, medium and low values highlighted in red, yellow and green respectively. As a reminder, a high RMSD (red) indicates low accuracy, while a low RMSD (green) indicates high accuracy. The symbol double hyphen (--) indicates that the RMSD and $COV_{RMSD}$ values could not be computed as the element weight % was 0.

Table 4.8 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.4 NC_AP1 sample:

| No.4 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| Na | 12 | 0.0809 | 0.0930 | 1.1503 | 0.0895 | 1.1551 |
| MgKa1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| AlKa1 | 12 | 1.1005 | 0.5582 | 0.5072 | 5.7109 | 5.4199 |
| Si | 12 | 4.2152 | 1.5705 | 0.3726 | 23.0856 | 5.7203 |
| P Ka1 | 12 | 0.0190 | 0.0308 | 1.6210 | 0.1158 | 6.3739 |
| S Ka1 | 12 | 0.5025 | 0.0599 | 0.1192 | 0.3472 | 0.7218 |
| K Ka1 | 12 | 0.2779 | 0.1402 | 0.5046 | 0.2169 | 0.8155 |
| CaKa1 | 12 | 4.1318 | 1.5787 | 0.3821 | 3.7611 | 0.9508 |
| BaLa1 | 12 | 0.3778 | 0.4352 | 1.152 | 0.5571 | 1.4747 |
| TiKa1 | 12 | 0.2132 | 0.1193 | 0.5597 | 0.5658 | 2.7716 |
| V Ka1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| CrKa1 | 12 | 0.0047 | 0.0015 | 0.3177 | 0.0021 | 0.4630 |
| MnKa1 | 12 | 0.0284 | 0.0054 | 0.1899 | 0.0724 | 2.6587 |
| FeKa1 | 12 | 2.0522 | 1.1233 | 0.5474 | 4.3768 | 2.2276 |
| BaLa1 | 12 | 2.4785 | 1.8903 | 0.7627 | 3.0334 | 1.2783 |
| CoKa1 | 12 | 0.0345 | 0.0206 | 0.5965 | 0.0373 | 1.1297 |
| NiKa1 | 12 | 0.0017 | 0.0022 | 1.2887 | 0.0052 | 3.1411 |
| CuKa1 | 12 | 0.0019 | 0.0020 | 1.0247 | 0.0050 | 2.7261 |
| ZnKa1 | 12 | 0.0027 | 0.0020 | 0.7716 | 0.0050 | 1.9493 |
| AsKa1 | 12 | 0.0002 | 0.0001 | 0.4267 | 0.0001 | 0.5186 |
| PbLa1 | 12 | 0.0012 | 0.0003 | 0.2345 | 0.0007 | 0.5795 |
| ThLa1 | 12 | 0.0003 | 0.0001 | 0.2216 | 0.0002 | 0.4664 |
| RbKa1 | 12 | 0.0010 | 0.0011 | 1.0666 | 0.0011 | 1.0900 |
| U La1 | 12 | 0.0044 | 0.0043 | 0.9846 | 0.0060 | 1.4228 |
| SrKa1 | 12 | 0.0374 | 0.0270 | 0.7227 | 0.0453 | 1.2669 |
| Y Ka1 | 12 | 0.0008 | 0.0009 | 1.0987 | 0.0012 | 1.4401 |
| ZrKa1 | 12 | 0.0089 | 0.0032 | 0.3646 | 0.0041 | 0.4789 |
| NbKa1 | 12 | 0.0008 | 0.0003 | 0.3222 | 0.0003 | 0.3590 |
| MoKa1 | 12 | 0.0079 | 0.0050 | 0.6364 | 0.0092 | 1.2112 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1447 | 0.0001 | 0.5628 |
| SbKa1 | 12 | 0.0001 | 0.0002 | 1.8232 | 0.0002 | 2.0230 |

Table 4.9 Standard deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.12 NC_AP1 RCA sample

| No.12 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| Na | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| MgKa1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| AlKa1 | 12 | 1.1205 | 0.1686 | 0.1504 | 5.8414 | 5.4449 |
| Si | 12 | 4.3376 | 0.4421 | 0.1019 | 22.9181 | 5.5186 |
| P Ka1 | 12 | 0.0040 | 0.0054 | 1.3525 | 0.1270 | 33.0894 |
| S Ka1 | 12 | 0.4984 | 0.0311 | 0.0624 | 0.3397 | 0.7119 |
| K Ka1 | 12 | 0.2708 | 0.0458 | 0.1692 | 0.1828 | 0.7051 |
| CaKa1 | 12 | 4.4303 | 0.6479 | 0.1462 | 3.2061 | 0.7558 |
| BaLa1 | 12 | 0.6236 | 0.6779 | 1.0870 | 0.8697 | 1.4565 |
| TiKa1 | 12 | 0.0970 | 0.1023 | 1.0540 | 0.6775 | 7.2923 |
| V Ka1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| CrKa1 | 12 | 0.0063 | 0.0019 | 0.2967 | 0.0018 | 0.2973 |
| MnKa1 | 12 | 0.0275 | 0.0041 | 0.1487 | 0.0732 | 2.7783 |
| FeKa1 | 12 | 1.0289 | 1.0942 | 1.0634 | 5.3692 | 5.4506 |
| BaLa1 | 12 | 0.5785 | 0.2208 | 0.3816 | 0.5747 | 1.0376 |
| CoKa1 | 12 | 0.0061 | 0.0025 | 0.4114 | 0.0040 | 0.6945 |
| NiKa1 | 12 | 0.0036 | 0.0006 | 0.1771 | 0.0029 | 0.8602 |
| CuKa1 | 12 | 0.0072 | 0.0017 | 0.2350 | 0.0018 | 0.2552 |
| ZnKa1 | 12 | 0.0056 | 0.0008 | 0.1517 | 0.0018 | 0.3441 |
| AsKa1 | 12 | 0.0004 | 0.0000 | 0.0970 | 0.0002 | 0.5949 |
| PbLa1 | 12 | 0.0012 | 0.0003 | 0.2212 | 0.0007 | 0.5819 |
| ThLa1 | 12 | 0.0004 | 0.0000 | 0.1113 | 0.0002 | 0.4575 |
| RbKa1 | 12 | 0.0010 | 0.0004 | 0.4346 | 0.0005 | 0.4954 |
| U La1 | 12 | 0.0004 | 0.0005 | 1.3302 | 0.0006 | 1.5764 |
| SrKa1 | 12 | 0.0818 | 0.0176 | 0.2157 | 0.0184 | 0.2346 |
| Y Ka1 | 12 | 0.0025 | 0.0004 | 0.1655 | 0.0010 | 0.4046 |
| ZrKa1 | 12 | 0.0103 | 0.0026 | 0.2570 | 0.0028 | 0.2846 |
| NbKa1 | 12 | 0.0007 | 0.0001 | 0.1039 | 0.0001 | 0.1094 |
| MoKa1 | 12 | 0.0003 | 0.0004 | 1.4722 | 0.0004 | 1.4722 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.0993 | 0.0001 | 0.4050 |
| SbKa1 | 12 | 0.0001 | 0.0001 | 2.5099 | 0.0001 | 2.6029 |

Table 4.10 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.50 NC_AP1 RCA sample:

| No.50 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0516 | 0.1384 | 2.6855 | 1.5713 | 31.8361 |
| MgKa1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| AlKa1 | 12 | 1.4142 | 0.0725 | 0.0513 | 5.5459 | 4.0961 |
| SiKa1 | 12 | 5.1253 | 0.4513 | 0.0881 | 22.1307 | 4.5100 |
| P Ka1 | 12 | 0.0109 | 0.0099 | 0.9072 | 0.1204 | 11.5011 |
| S Ka1 | 12 | 0.5051 | 0.0318 | 0.0630 | 0.3664 | 0.7576 |
| K Ka1 | 12 | 0.2967 | 0.0213 | 0.0719 | 0.1530 | 0.5386 |
| CaKa1 | 12 | 4.0024 | 0.5089 | 0.1271 | 3.6065 | 0.9412 |
| BaLa1 | 12 | 0.8227 | 0.6702 | 0.8146 | 1.0085 | 1.2802 |
| TiKa1 | 12 | 0.0812 | 0.1017 | 1.2525 | 0.6930 | 8.9133 |
| V Ka1 | 12 | 0.0000 | 0.0000 | 0.0000 | -- | -- |
| CrKa1 | 12 | 0.0066 | 0.0009 | 0.1391 | 0.0010 | 0.1531 |
| MnKa1 | 12 | 0.0282 | 0.0032 | 0.1126 | 0.0725 | 2.6817 |
| FeKa1 | 12 | 0.9035 | 1.1675 | 1.2923 | 5.5061 | 6.3655 |
| BaLa1 | 12 | 0.1371 | 0.0879 | 0.6409 | 0.1255 | 0.9554 |
| CoKa1 | 12 | 0.0012 | 0.0004 | 0.3535 | 0.0017 | 1.4301 |
| NiKa1 | 12 | 0.0031 | 0.0007 | 0.2230 | 0.0035 | 1.1354 |
| CuKa1 | 12 | 0.0083 | 0.0011 | 0.1292 | 0.0020 | 0.2557 |
| ZnKa1 | 12 | 0.0061 | 0.0012 | 0.1891 | 0.0015 | 0.2633 |
| AsKa1 | 12 | 0.0004 | 0.0001 | 0.1181 | 0.0003 | 0.6459 |
| PbLa1 | 12 | 0.0009 | 0.0003 | 0.3391 | 0.0008 | 0.8794 |
| ThLa1 | 12 | 0.0004 | 0.0000 | 0.0415 | 0.0002 | 0.4909 |
| RbKa1 | 12 | 0.0015 | 0.0004 | 0.2612 | 0.0004 | 0.2949 |
| U La1 | 12 | 0.0005 | 0.0004 | 0.8069 | 0.0006 | 1.2026 |
| SrKa1 | 12 | 0.0554 | 0.0138 | 0.2496 | 0.0233 | 0.4390 |
| Y Ka1 | 12 | 0.0026 | 0.0003 | 0.1033 | 0.0010 | 0.4102 |
| ZrKa1 | 12 | 0.0115 | 0.0017 | 0.1485 | 0.0016 | 0.1485 |
| NbKa1 | 12 | 0.0007 | 0.0001 | 0.0798 | 0.0001 | 0.0922 |
| MoKa1 | 12 | 0.0005 | 0.0009 | 1.8570 | 0.0008 | 1.8570 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.0358 | 0.0001 | 0.4686 |
| SbKa1 | 12 | 0.0002 | 0.0003 | 1.4425 | 0.0003 | 1.7207 |

The series of graphs presented below is intended to show the relationship between the particle size and the RMSD values. As per the literature review (Section 2.6.1) significant drops were observed in the RMSD and $COV_{RMSD}$ values with decreasing particle size. But contrary to the observations made in the literature review except for elements K, Cr, Ba, Co, Zn and Zr, the RMSD and $COV_{RMSD}$ values for all the other elements did not decrease with decrease in particle size indicating that the decrease in particle size did not bring about any significant benefits in terms of accuracy. Rather, in some cases the change in RMSD values was either insignificant or the values did not follow the decreasing pattern from No.4 to No.50.

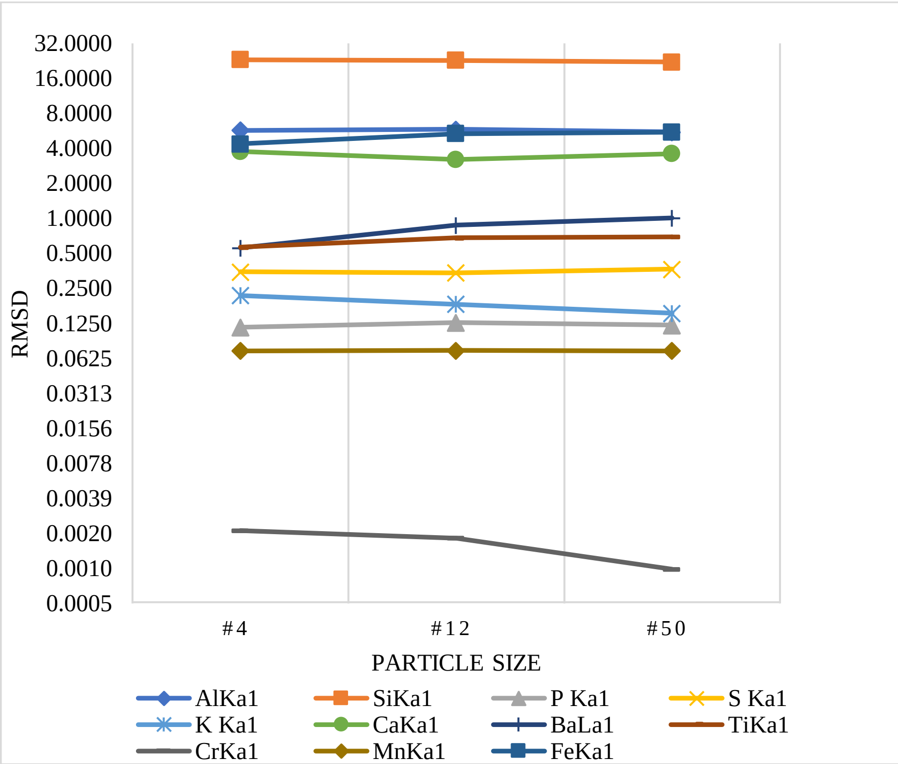


Figure 4.8 Sieve Size Vs RMSD (NC_AP1 RCA Sample-Major Elements)

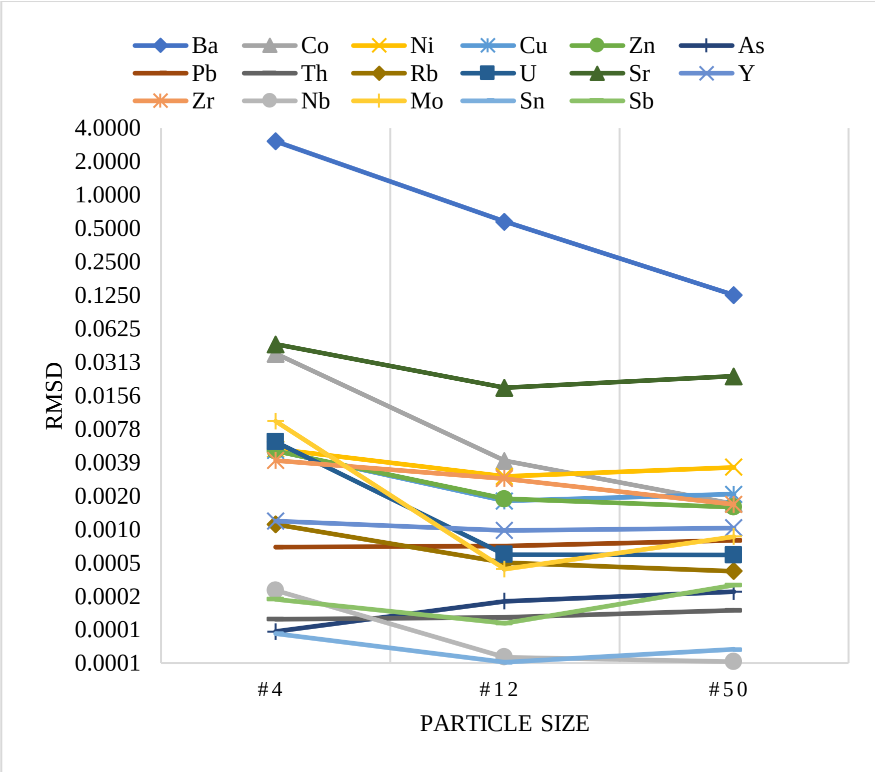


Figure 4.9 Sieve Size Vs RMSD (NC_AP1 RCA Sample-Trace Elements)

Table 4.11 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.4 NC_CT1 sample

| No.4 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0008 | 0.0019 | 2.4164 | 1.3716 | 1806.342 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.3400 | 0.1917 | 0.5639 | 5.1145 | 15.7101 |
| SiKa1 | 12 | 3.8349 | 0.5891 | 0.1536 | 25.9939 | 7.0797 |
| P Ka1 | 12 | 0.0069 | 0.0106 | 1.5287 | 0.1375 | 20.7170 |
| S Ka1 | 12 | 0.5628 | 0.0636 | 0.1130 | 0.7004 | 1.2999 |
| K Ka1 | 12 | 0.2366 | 0.0622 | 0.2629 | 1.1374 | 5.0210 |
| CaKa1 | 12 | 6.3559 | 1.0407 | 0.1637 | 1.8355 | 0.3016 |
| BaLa1 | 12 | 0.2238 | 0.2087 | 0.9325 | 0.2744 | 1.2804 |
| TiKa1 | 12 | 0.0246 | 0.0316 | 1.2868 | 0.2888 | 11.7497 |
| V Ka1 | 12 | 0.0013 | 0.0020 | 1.5757 | 0.0094 | 7.8213 |
| CrKa1 | 12 | 0.0045 | 0.0018 | 0.4036 | 0.0018 | 0.4080 |
| MnKa1 | 12 | 0.0223 | 0.0071 | 0.3196 | 0.0327 | 1.5330 |
| FeKa1 | 12 | 0.3143 | 0.4018 | 1.2784 | 2.8869 | 9.5949 |
| BaLa1 | 12 | 0.2702 | 0.1722 | 0.6375 | 0.2886 | 1.1158 |
| CoKa1 | 12 | 0.0027 | 0.0011 | 0.3996 | 0.0017 | 0.6501 |
| NiKa1 | 12 | 0.0026 | 0.0005 | 0.1818 | 0.0007 | 0.2696 |
| CuKa1 | 12 | 0.0042 | 0.0020 | 0.4772 | 0.0019 | 0.4778 |
| ZnKa1 | 12 | 0.0053 | 0.0030 | 0.5701 | 0.0034 | 0.6846 |
| AsKa1 | 12 | 0.0004 | 0.0002 | 0.4571 | 0.0002 | 0.4648 |
| PbLa1 | 12 | 0.0010 | 0.0004 | 0.3786 | 0.0005 | 0.5098 |
| ThLa1 | 12 | 0.0004 | 0.0001 | 0.2969 | 0.0003 | 0.8345 |
| RbKa1 | 12 | 0.0019 | 0.0014 | 0.7086 | 0.0024 | 1.3348 |
| U La1 | 12 | 0.0006 | 0.0006 | 1.0247 | 0.0007 | 1.1976 |
| SrKa1 | 12 | 0.0357 | 0.0189 | 0.5281 | 0.0199 | 0.5820 |
| Y Ka1 | 12 | 0.0023 | 0.0004 | 0.1760 | 0.0010 | 0.4554 |
| ZrKa1 | 12 | 0.0092 | 0.0016 | 0.1788 | 0.0098 | 1.1159 |
| NbKa1 | 12 | 0.0006 | 0.0001 | 0.1847 | 0.0001 | 0.1862 |
| MoKa1 | 12 | 0.0018 | 0.0015 | 0.8407 | 0.0020 | 1.1373 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0001 | 0.0000 | 0.2428 | 0.0001 | 0.5047 |
| SbKa1 | 12 | 0.0001 | 0.0001 | 1.8858 | 0.0001 | 1.9135 |

Table 4.12 Standard deviation, $COV_{STD\ DEV}$ , RMSD & $COV_{RMSD}$ values for No.12 NC_CT1 RCA sample

| **No.12** | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0112 | 0.0266 | 2.3775 | 1.3615 | 127.2895 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.4525 | 0.2228 | 0.4923 | 5.0033 | 11.5485 |
| SiKa1 | 12 | 4.2804 | 0.6406 | 0.1497 | 25.5496 | 6.2344 |
| P Ka1 | 12 | 0.0147 | 0.0138 | 0.9373 | 0.0482 | 3.4315 |
| S Ka1 | 12 | 0.5662 | 0.0479 | 0.0846 | 0.3989 | 0.7358 |
| K Ka1 | 12 | 0.3005 | 0.1103 | 0.3672 | 0.7283 | 2.5317 |
| CaKa1 | 12 | 7.3314 | 1.2733 | 0.1737 | 1.3441 | 0.1915 |
| BaLa1 | 12 | 0.0098 | 0.0275 | 2.8041 | 0.0370 | 3.9444 |
| TiKa1 | 12 | 0.1109 | 0.0304 | 0.2744 | 0.2030 | 1.9125 |
| V Ka1 | 12 | 0.0019 | 0.0023 | 1.2137 | 0.0089 | 5.0174 |
| CrKa1 | 12 | 0.0039 | 0.0009 | 0.2322 | 0.0012 | 0.3238 |
| MnKa1 | 12 | 0.0243 | 0.0023 | 0.0949 | 0.0300 | 1.2896 |
| FeKa1 | 12 | 1.5305 | 0.4231 | 0.2765 | 1.6940 | 1.1560 |
| BaLa1 | 12 | 0.2992 | 0.2874 | 0.9605 | 0.3826 | 1.3357 |
| CoKa1 | 12 | 0.0043 | 0.0039 | 0.9142 | 0.0047 | 1.1549 |
| NiKa1 | 12 | 0.0024 | 0.0007 | 0.3046 | 0.0007 | 0.3291 |
| CuKa1 | 12 | 0.0083 | 0.0038 | 0.4618 | 0.0056 | 0.7048 |
| ZnKa1 | 12 | 0.0097 | 0.0040 | 0.4150 | 0.0046 | 0.4943 |
| AsKa1 | 12 | 0.0006 | 0.0002 | 0.3635 | 0.0003 | 0.4863 |
| PbLa1 | 12 | 0.0011 | 0.0004 | 0.3429 | 0.0005 | 0.4208 |
| ThLa1 | 12 | 0.0004 | 0.0001 | 0.1489 | 0.0003 | 0.8077 |
| RbKa1 | 12 | 0.0022 | 0.0009 | 0.3928 | 0.0020 | 0.9286 |
| U La1 | 12 | 0.0007 | 0.0005 | 0.7236 | 0.0007 | 1.0073 |
| SrKa1 | 12 | 0.0354 | 0.0119 | 0.3353 | 0.0143 | 0.4215 |
| Y Ka1 | 12 | 0.0025 | 0.0003 | 0.1259 | 0.0012 | 0.5029 |
| ZrKa1 | 12 | 0.0132 | 0.0034 | 0.2548 | 0.0065 | 0.5163 |
| NbKa1 | 12 | 0.0007 | 0.0001 | 0.1252 | 0.0001 | 0.1560 |
| MoKa1 | 12 | 0.0008 | 0.0007 | 0.7992 | 0.0007 | 0.9038 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| SnKa1 | 12 | 0.0001 | 0.0000 | 0.2152 | 0.0001 | 0.6460 |
| SbKa1 | 12 | 0.0001 | 0.0002 | 1.8468 | 0.0002 | 1.9240 |

Table 4.13 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.50 NC_CT1 RCA sample

| **No.50** | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.4540 | 0.0552 | 0.1215 | 4.9975 | 11.4968 |
| SiKa1 | 12 | 6.7317 | 0.4123 | 0.0612 | 23.0944 | 3.5833 |
| P Ka1 | 12 | 0.0012 | 0.0018 | 1.4621 | 0.0599 | 51.3583 |
| S Ka1 | 12 | 0.5866 | 0.0373 | 0.0636 | 0.4181 | 0.7445 |
| K Ka1 | 12 | 0.2588 | 0.0651 | 0.2517 | 0.7649 | 3.0873 |
| CaKa1 | 12 | 5.8792 | 0.9266 | 0.1576 | 2.2046 | 0.3917 |
| BaLa1 | 12 | 0.0966 | 0.1521 | 1.5748 | 0.1578 | 1.7064 |
| TiKa1 | 12 | 0.0701 | 0.0485 | 0.6923 | 0.2460 | 3.6645 |
| V Ka1 | 12 | 0.0055 | 0.0053 | 0.9630 | 0.0071 | 1.3423 |
| CrKa1 | 12 | 0.0060 | 0.0019 | 0.3164 | 0.0022 | 0.3785 |
| MnKa1 | 12 | 0.0281 | 0.0029 | 0.1017 | 0.0262 | 0.9750 |
| FeKa1 | 12 | 0.8296 | 0.5250 | 0.6328 | 2.3991 | 3.0205 |
| BaLa1 | 12 | 0.5322 | 0.4369 | 0.8210 | 0.6511 | 1.2778 |
| CoKa1 | 12 | 0.0065 | 0.0059 | 0.9100 | 0.0076 | 1.2258 |
| NiKa1 | 12 | 0.0012 | 0.0009 | 0.7356 | 0.0012 | 1.0880 |
| CuKa1 | 12 | 0.0032 | 0.0029 | 0.8987 | 0.0029 | 0.9434 |
| ZnKa1 | 12 | 0.0025 | 0.0021 | 0.8182 | 0.0051 | 2.1013 |
| AsKa1 | 12 | 0.0002 | 0.0001 | 0.4261 | 0.0002 | 1.0027 |
| PbLa1 | 12 | 0.0011 | 0.0002 | 0.1877 | 0.0003 | 0.2999 |
| ThLa1 | 12 | 0.0003 | 0.0001 | 0.1614 | 0.0004 | 1.3363 |
| RbKa1 | 12 | 0.0007 | 0.0007 | 1.0290 | 0.0033 | 4.8445 |
| U La1 | 12 | 0.0003 | 0.0003 | 1.2785 | 0.0003 | 1.2823 |
| SrKa1 | 12 | 0.0108 | 0.0074 | 0.6830 | 0.0340 | 3.2950 |
| Y Ka1 | 12 | 0.0018 | 0.0008 | 0.4520 | 0.0009 | 0.5238 |
| ZrKa1 | 12 | 0.0072 | 0.0028 | 0.3922 | 0.0120 | 1.7593 |
| NbKa1 | 12 | 0.0005 | 0.0001 | 0.1873 | 0.0001 | 0.2747 |
| MoKa1 | 12 | 0.0082 | 0.0059 | 0.7168 | 0.0096 | 1.2161 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1813 | 0.0000 | 0.1818 |
| SbKa1 | 12 | 0.0005 | 0.0005 | 1.0262 | 0.0006 | 1.3912 |

Similar to the observations made for NC_AP1, only a handful of elements including Al, Si, V and Mn showed improved accuracy when the particle decreased. The rest of the elements did not show any decrease in RMSD or $COV_{RMSD}$ values with decrease in particle size.

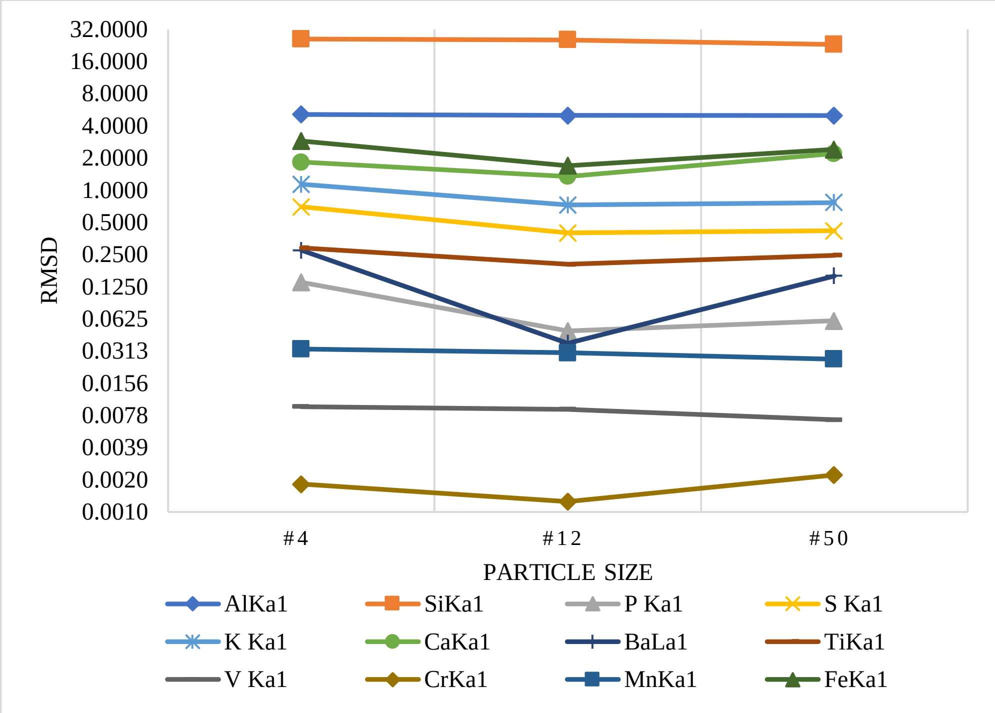


Figure 4.10 Sieve size vs. RMSD (NC_CT1 RCA sample-Major Elements)

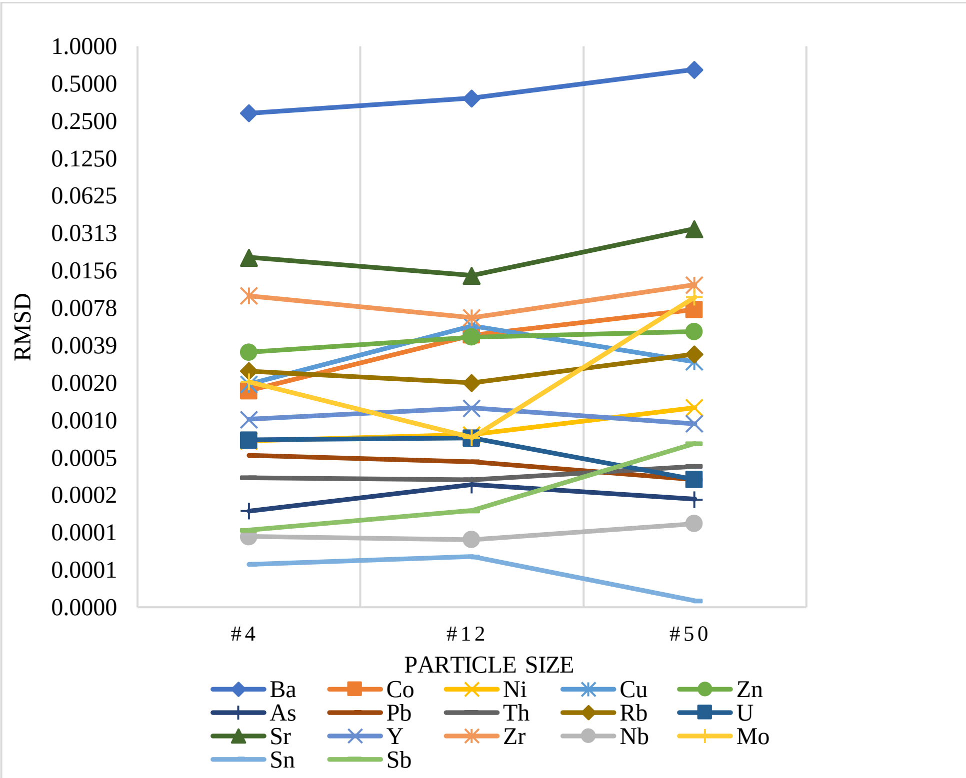


Figure 4.11 Sieve Size Vs RMSD (NC_CT1 RCA Sample-Trace Elements)

Table 4.14 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.4 NC_HW1

| No.4 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.5891 | 0.2491 | 0.4228 | 5.7140 | 10.1315 |
| SiKa1 | 12 | 4.5482 | 1.1262 | 0.2476 | 24.9239 | 5.7236 |
| P Ka1 | 12 | 0.0117 | 0.0122 | 1.0432 | 0.0381 | 3.3960 |
| S Ka1 | 12 | 0.5608 | 0.1114 | 0.1987 | 0.4341 | 0.8085 |
| K Ka1 | 12 | 0.5808 | 0.1433 | 0.2468 | 2.2045 | 3.9647 |
| CaKa1 | 12 | 4.4733 | 1.8633 | 0.4165 | 3.5171 | 0.8212 |
| BaLa1 | 12 | 0.0480 | 0.1005 | 2.0942 | 0.1016 | 2.2112 |
| TiKa1 | 12 | 0.0519 | 0.0373 | 0.7193 | 0.1100 | 2.2152 |
| V Ka1 | 12 | 0.0033 | 0.0021 | 0.6284 | 0.0022 | 0.6923 |
| CrKa1 | 12 | 0.0043 | 0.0009 | 0.2073 | 0.0013 | 0.3032 |
| MnKa1 | 12 | 0.0248 | 0.0033 | 0.1343 | 0.0143 | 0.6020 |
| FeKa1 | 12 | 0.9243 | 0.5198 | 0.5624 | 1.1602 | 1.3111 |
| BaLa1 | 12 | 0.5153 | 0.1966 | 0.3815 | 0.4758 | 0.9644 |
| CoKa1 | 12 | 0.0057 | 0.0020 | 0.3524 | 0.0057 | 1.0399 |
| NiKa1 | 12 | 0.0012 | 0.0006 | 0.4822 | 0.0010 | 0.8936 |
| CuKa1 | 12 | 0.0006 | 0.0008 | 1.3808 | 0.0010 | 1.8903 |
| ZnKa1 | 12 | 0.0027 | 0.0018 | 0.6510 | 0.0040 | 1.5435 |
| AsKa1 | 12 | 0.0003 | 0.0002 | 0.6157 | 0.0002 | 0.6309 |
| PbLa1 | 12 | 0.0014 | 0.0004 | 0.3192 | 0.0007 | 0.5450 |
| ThLa1 | 12 | 0.0006 | 0.0001 | 0.1825 | 0.0010 | 1.7279 |
| RbKa1 | 12 | 0.0040 | 0.0022 | 0.5592 | 0.0074 | 1.9470 |
| U La1 | 12 | 0.0002 | 0.0004 | 2.0780 | 0.0005 | 2.4791 |
| SrKa1 | 12 | 0.0156 | 0.0068 | 0.4343 | 0.0186 | 1.2478 |
| Y Ka1 | 12 | 0.0037 | 0.0016 | 0.4232 | 0.0018 | 0.5162 |
| ZrKa1 | 12 | 0.0176 | 0.0083 | 0.4714 | 0.0090 | 0.5330 |
| NbKa1 | 12 | 0.0010 | 0.0002 | 0.2040 | 0.0002 | 0.2174 |
| MoKa1 | 12 | 0.0063 | 0.0028 | 0.4405 | 0.0064 | 1.0596 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.2662 | 0.0001 | 0.3803 |
| SbKa1 | 12 | 0.0001 | 0.0002 | 1.5108 | 0.0002 | 1.7459 |

Table 4.15 Standard Deviation, $COV_{STD\ DEV}$ , RMSD & $COV_{RMSD}$ Values for No.12 NC_HW1

| **No.12** | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0329 | 0.1140 | 3.4641 | 2.2399 | 71.111 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.8335 | 0.3452 | 0.4142 | 5.4746 | 6.8607 |
| SiKa1 | 12 | 4.1852 | 0.8471 | 0.2024 | 25.2765 | 6.3081 |
| P Ka1 | 12 | 0.0014 | 0.0047 | 3.4641 | 0.0469 | 36.2604 |
| S Ka1 | 12 | 0.5012 | 0.0275 | 0.0549 | 0.3621 | 0.7547 |
| K Ka1 | 12 | 0.6666 | 0.2041 | 0.3062 | 2.1235 | 3.3274 |
| CaKa1 | 12 | 4.7039 | 1.0020 | 0.2130 | 2.9603 | 0.6573 |
| BaLa1 | 12 | 0.0769 | 0.1656 | 2.1528 | 0.1585 | 2.1534 |
| TiKa1 | 12 | 0.0645 | 0.0346 | 0.5359 | 0.0972 | 1.5726 |
| V Ka1 | 12 | 0.0042 | 0.0033 | 0.7927 | 0.0032 | 0.7928 |
| CrKa1 | 12 | 0.0054 | 0.0011 | 0.1966 | 0.0023 | 0.4332 |
| MnKa1 | 12 | 0.0239 | 0.0020 | 0.0841 | 0.0150 | 0.6556 |
| FeKa1 | 12 | 0.9277 | 0.4771 | 0.5142 | 1.1402 | 1.2836 |
| BaLa1 | 12 | 0.1396 | 0.1336 | 0.9575 | 0.1419 | 1.0616 |
| CoKa1 | 12 | 0.0008 | 0.0003 | 0.3664 | 0.0005 | 0.6756 |
| NiKa1 | 12 | 0.0022 | 0.0007 | 0.3241 | 0.0007 | 0.3260 |
| CuKa1 | 12 | 0.0043 | 0.0011 | 0.2607 | 0.0032 | 0.7761 |
| ZnKa1 | 12 | 0.0062 | 0.0018 | 0.2962 | 0.0018 | 0.2974 |
| AsKa1 | 12 | 0.0005 | 0.0001 | 0.2021 | 0.0002 | 0.4589 |
| PbLa1 | 12 | 0.0012 | 0.0006 | 0.4754 | 0.0009 | 0.8537 |
| ThLa1 | 12 | 0.0010 | 0.0001 | 0.0816 | 0.0006 | 0.6321 |
| RbKa1 | 12 | 0.0102 | 0.0016 | 0.1520 | 0.0017 | 0.1772 |
| U La1 | 12 | 0.0005 | 0.0006 | 1.2472 | 0.0006 | 1.2526 |
| SrKa1 | 12 | 0.0392 | 0.0071 | 0.1819 | 0.0092 | 0.2458 |
| Y Ka1 | 12 | 0.0023 | 0.0008 | 0.3285 | 0.0008 | 0.3603 |
| ZrKa1 | 12 | 0.0188 | 0.0019 | 0.0993 | 0.0035 | 0.1924 |
| NbKa1 | 12 | 0.0012 | 0.0001 | 0.0792 | 0.0002 | 0.1643 |
| MoKa1 | 12 | 0.0005 | 0.0005 | 1.0360 | 0.0005 | 1.0380 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.0783 | 0.0000 | 0.3132 |
| SbKa1 | 12 | 0.0001 | 0.0002 | 2.1244 | 0.0002 | 2.2780 |

Table 4.16 Standard Deviation, $COV_{STD\ DEV}$ , RMSD & $COV_{RMSD}$ Values for No.50 NC_HW1

| **No.50** | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.9614 | 0.1504 | 0.1565 | 5.3386 | 5.7999 |
| SiKa1 | 12 | 4.8632 | 0.6149 | 0.1265 | 24.5926 | 5.2818 |
| P Ka1 | 12 | 0.0005 | 0.0018 | 3.4070 | 0.0475 | 91.7218 |
| S Ka1 | 12 | 0.5017 | 0.0218 | 0.0434 | 0.3623 | 0.7542 |
| K Ka1 | 12 | 0.9526 | 0.2054 | 0.2156 | 1.8389 | 2.0162 |
| CaKa1 | 12 | 5.1282 | 0.5817 | 0.1134 | 2.4406 | 0.4971 |
| BaLa1 | 12 | 0.0027 | 0.0051 | 1.8556 | 0.0780 | 29.8666 |
| TiKa1 | 12 | 0.0883 | 0.0132 | 0.1491 | 0.0687 | 0.8129 |
| V Ka1 | 12 | 0.0063 | 0.0038 | 0.6118 | 0.0042 | 0.7013 |
| CrKa1 | 12 | 0.0052 | 0.0008 | 0.1620 | 0.0019 | 0.3889 |
| MnKa1 | 12 | 0.0240 | 0.0009 | 0.0383 | 0.0148 | 0.6446 |
| FeKa1 | 12 | 1.3025 | 0.1909 | 0.1466 | 0.6944 | 0.5568 |
| BaLa1 | 12 | 0.5555 | 0.4205 | 0.7570 | 0.6243 | 1.1739 |
| CoKa1 | 12 | 0.0075 | 0.0060 | 0.8086 | 0.0092 | 1.2815 |
| NiKa1 | 12 | 0.0012 | 0.0008 | 0.6290 | 0.0011 | 0.9683 |
| CuKa1 | 12 | 0.0023 | 0.0029 | 1.2502 | 0.0030 | 1.3356 |
| ZnKa1 | 12 | 0.0026 | 0.0018 | 0.6891 | 0.0041 | 1.6531 |
| AsKa1 | 12 | 0.0005 | 0.0008 | 1.5650 | 0.0007 | 1.6134 |
| PbLa1 | 12 | 0.0015 | 0.0003 | 0.2331 | 0.0006 | 0.4132 |
| ThLa1 | 12 | 0.0008 | 0.0003 | 0.3258 | 0.0008 | 1.0389 |
| RbKa1 | 12 | 0.0060 | 0.0054 | 0.9016 | 0.0073 | 1.2696 |
| U La1 | 12 | 0.0004 | 0.0012 | 2.8189 | 0.0011 | 2.8197 |
| SrKa1 | 12 | 0.0240 | 0.0159 | 0.6630 | 0.0177 | 0.7690 |
| Y Ka1 | 12 | 0.0010 | 0.0011 | 1.0829 | 0.0020 | 1.9905 |
| ZrKa1 | 12 | 0.0112 | 0.0051 | 0.4529 | 0.0116 | 1.0848 |
| NbKa1 | 12 | 0.0010 | 0.0002 | 0.2449 | 0.0002 | 0.2457 |
| MoKa1 | 12 | 0.0069 | 0.0063 | 0.9085 | 0.0088 | 1.3294 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1648 | 0.0000 | 0.1650 |
| SbKa1 | 12 | 0.0004 | 0.0004 | 0.9470 | 0.0006 | 1.3714 |

With the exception of the elements Al, S, K, Ca, Ti, Fe, Ni and Th, no significant benefits were observed for the rest of the elements in terms of improvement in accuracy with reduction in particle size.

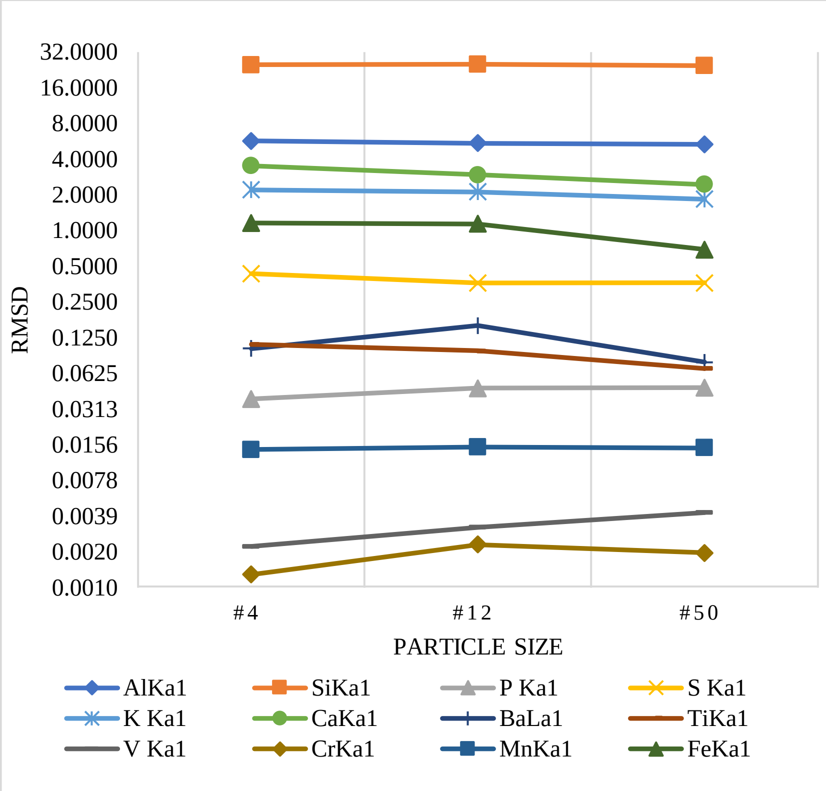



Figure 4.12 Sieve Size Vs RMSD (NC_HW1 RCA Sample-Major Elements)

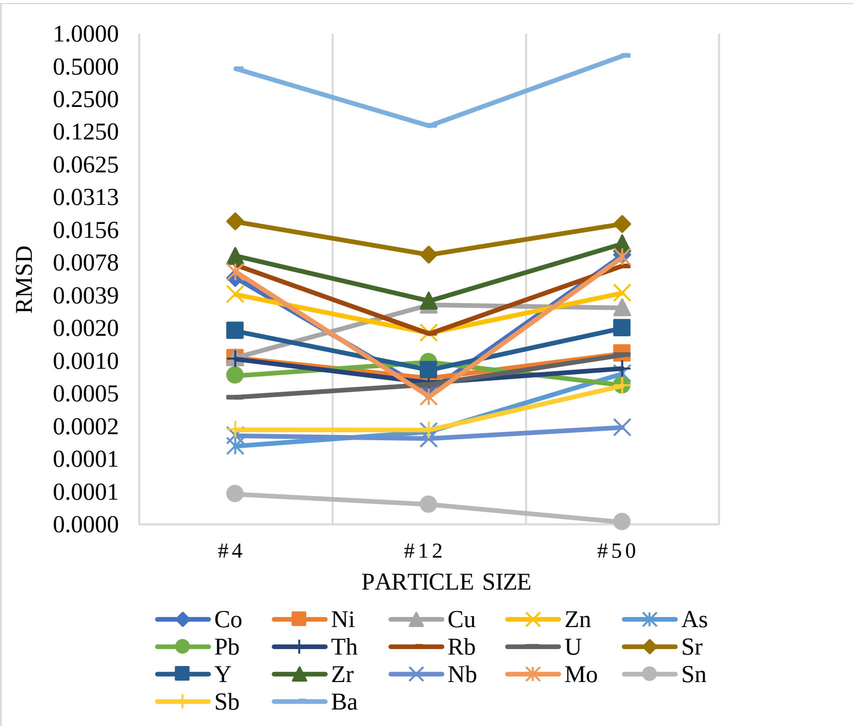



Figure 4.13 Sieve Size Vs RMSD (NC_HW1 RCA Sample-Trace Elements)

Table 4.17 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ Values for No.4 NC_CT2

| No.4 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.2808 | 0.2053 | 0.7309 | 0.2746 | 1.0213 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| SiKa1 | 12 | 2.3525 | 0.6596 | 0.2804 | 7.7233 | 3.4290 |
| P Ka1 | 12 | 0.0188 | 0.0191 | 1.0134 | 0.1265 | 7.0253 |
| S Ka1 | 12 | 0.2613 | 0.0465 | 0.1780 | 0.0679 | 0.2715 |
| K Ka1 | 12 | 0.1108 | 0.0497 | 0.4487 | 0.0668 | 0.6302 |
| CaKa1 | 12 | 13.3876 | 2.2308 | 0.1666 | 17.1918 | 1.3413 |
| BaLa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| TiKa1 | 12 | 0.0192 | 0.0113 | 0.5858 | 0.0364 | 1.9762 |
| V Ka1 | 12 | 0.0049 | 0.0010 | 0.2074 | 0.0030 | 0.6313 |
| CrKa1 | 12 | 0.0023 | 0.0007 | 0.2875 | 0.0066 | 2.9424 |
| MnKa1 | 12 | 0.0215 | 0.0006 | 0.0284 | 0.0095 | 0.4645 |
| FeKa1 | 12 | 0.2938 | 0.2358 | 0.8024 | 0.3930 | 1.3968 |
| BaLa1 | 12 | 0.2577 | 0.2373 | 0.9207 | 0.3393 | 1.3749 |
| CoKa1 | 12 | 0.0022 | 0.0011 | 0.4712 | 0.0021 | 1.0008 |
| NiKa1 | 12 | 0.0021 | 0.0004 | 0.1780 | 0.0007 | 0.3318 |
| CuKa1 | 12 | 0.0038 | 0.0064 | 1.6916 | 0.0069 | 1.9111 |
| ZnKa1 | 12 | 0.0031 | 0.0019 | 0.6285 | 0.0037 | 1.2832 |
| AsKa1 | 12 | 0.0003 | 0.0001 | 0.3965 | 0.0001 | 0.4295 |
| PbLa1 | 12 | 0.0010 | 0.0003 | 0.2795 | 0.0006 | 0.6143 |
| ThLa1 | 12 | 0.0003 | 0.0000 | 0.0985 | 0.0001 | 0.4792 |
| RbKa1 | 12 | 0.0005 | 0.0004 | 0.7541 | 0.0004 | 0.7807 |
| U La1 | 12 | 0.0005 | 0.0004 | 0.9209 | 0.0005 | 1.0197 |
| SrKa1 | 12 | 0.0404 | 0.0089 | 0.2194 | 0.0096 | 0.2476 |
| Y Ka1 | 12 | 0.0019 | 0.0002 | 0.1212 | 0.0014 | 0.7855 |
| ZrKa1 | 12 | 0.0097 | 0.0022 | 0.2222 | 0.0022 | 0.2403 |
| NbKa1 | 12 | 0.0004 | 0.0001 | 0.2270 | 0.0003 | 0.6057 |
| MoKa1 | 12 | 0.0013 | 0.0011 | 0.8051 | 0.0016 | 1.2517 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1489 | | 0.0000 |
| SbKa1 | 12 | 0.0002 | 0.0003 | 1.2734 | 0.0003 | 1.5587 |

Table 4.18 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ values for No.12 NC_CT2

| **No.12** | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.2490 | 0.1717 | 0.6894 | 0.2294 | 0.9621 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.0042 | 0.0146 | 3.4641 | 0.5517 | 136.7464 |
| SiKa1 | 12 | 2.2261 | 0.2591 | 0.1164 | 7.8278 | 3.6727 |
| P Ka1 | 12 | 0.0091 | 0.0156 | 1.7084 | 0.1357 | 15.5643 |
| S Ka1 | 12 | 0.2855 | 0.0969 | 0.3394 | 0.1196 | 0.4376 |
| K Ka1 | 12 | 0.1468 | 0.0352 | 0.2395 | 0.0354 | 0.2517 |
| CaKa1 | 12 | 14.0301 | 2.8714 | 0.2047 | 16.6448 | 1.2391 |
| BaLa1 | 12 | 0.0287 | 0.0828 | 2.8895 | 0.0817 | 2.9774 |
| TiKa1 | 12 | 0.0198 | 0.0132 | 0.6648 | 0.0364 | 1.9208 |
| V Ka1 | 12 | 0.0038 | 0.0015 | 0.3983 | 0.0022 | 0.6082 |
| CrKa1 | 12 | 0.0025 | 0.0006 | 0.2536 | 0.0064 | 2.7024 |
| MnKa1 | 12 | 0.0226 | 0.0022 | 0.0976 | 0.0086 | 0.3968 |
| FeKa1 | 12 | 0.3408 | 0.2304 | 0.6759 | 0.3523 | 1.0797 |
| BaLa1 | 12 | 0.3253 | 0.2937 | 0.9029 | 0.4256 | 1.3667 |
| CoKa1 | 12 | 0.0029 | 0.0009 | 0.3207 | 0.0027 | 0.9759 |
| NiKa1 | 12 | 0.0026 | 0.0009 | 0.3370 | 0.0013 | 0.5389 |
| CuKa1 | 12 | 0.0049 | 0.0024 | 0.4900 | 0.0049 | 1.0466 |
| ZnKa1 | 12 | 0.0006 | 0.0007 | 1.1718 | 0.0057 | 9.6229 |
| AsKa1 | 12 | 0.0002 | 0.0000 | 0.1964 | 0.0002 | 0.9974 |
| PbLa1 | 12 | 0.0010 | 0.0004 | 0.3417 | 0.0007 | 0.6559 |
| ThLa1 | 12 | 0.0003 | 0.0000 | 0.1218 | 0.0001 | 0.5276 |
| RbKa1 | 12 | 0.0004 | 0.0005 | 1.0493 | 0.0005 | 1.1681 |
| U La1 | 12 | 0.0004 | 0.0004 | 0.9619 | 0.0004 | 1.0308 |
| SrKa1 | 12 | 0.0358 | 0.0093 | 0.2589 | 0.0089 | 0.2590 |
| Y Ka1 | 12 | 0.0020 | 0.0003 | 0.1676 | 0.0015 | 0.8108 |
| ZrKa1 | 12 | 0.0136 | 0.0050 | 0.3647 | 0.0056 | 0.4321 |
| NbKa1 | 12 | 0.0005 | 0.0001 | 0.1951 | 0.0003 | 0.6728 |
| MoKa1 | 12 | 0.0022 | 0.0019 | 0.8829 | 0.0028 | 1.3282 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1387 | -- | 0.0000 |
| SbKa1 | 12 | 0.0000 | 0.0001 | 3.0968 | 0.0001 | 3.1229 |

Table 4.19 Standard Deviation, $COV_{STD\ DEV}$, RMSD & $COV_{RMSD}$ values for No.50 NC_CT2

| No.50 | | | | | | |
|---|---|---|---|---|---|---|
| Element | n | Average | Std Dev | $COV_{STD\ DEV}$ | RMSD | $COV_{RMSD}$ |
| NaKa1 | 12 | 0.1556 | 0.0473 | 0.3038 | 0.0805 | 0.5404 |
| MgKa1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| AlKa1 | 12 | 0.0084 | 0.0290 | 3.4641 | 0.5480 | 68.2674 |
| SiKa1 | 12 | 3.0400 | 0.4598 | 0.1513 | 7.0237 | 2.4131 |
| P Ka1 | 12 | 0.0000 | 0.0000 | -- | -- | -- |
| S Ka1 | 12 | 0.3591 | 0.0523 | 0.1456 | 0.1573 | 0.4574 |
| K Ka1 | 12 | 0.1474 | 0.0232 | 0.1572 | 0.0245 | 0.1732 |
| CaKa1 | 12 | 13.0233 | 1.2125 | 0.0931 | 17.4615 | 1.4004 |
| BaLa1 | 12 | 0.0103 | 0.0358 | 3.4641 | 0.0343 | 3.4669 |
| TiKa1 | 12 | 0.0325 | 0.0152 | 0.4664 | 0.0259 | 0.8329 |
| V Ka1 | 12 | 0.0054 | 0.0023 | 0.4194 | 0.0039 | 0.7617 |
| CrKa1 | 12 | 0.0034 | 0.0007 | 0.2123 | 0.0055 | 1.6675 |
| MnKa1 | 12 | 0.0224 | 0.0005 | 0.0220 | 0.0086 | 0.4007 |
| FeKa1 | 12 | 0.4266 | 0.1867 | 0.4375 | 0.2600 | 0.6366 |
| BaLa1 | 12 | 0.1658 | 0.0936 | 0.5648 | 0.1834 | 1.1557 |
| CoKa1 | 12 | 0.0028 | 0.0017 | 0.6246 | 0.0029 | 1.1071 |
| NiKa1 | 12 | 0.0018 | 0.0005 | 0.2933 | 0.0006 | 0.3434 |
| CuKa1 | 12 | 0.0011 | 0.0010 | 0.8631 | 0.0011 | 1.0096 |
| ZnKa1 | 12 | 0.0025 | 0.0031 | 1.2294 | 0.0048 | 1.9956 |
| AsKa1 | 12 | 0.0002 | 0.0001 | 0.5851 | 0.0002 | 0.7347 |
| PbLa1 | 12 | 0.0011 | 0.0003 | 0.2890 | 0.0007 | 0.6505 |
| ThLa1 | 12 | 0.0003 | 0.0000 | 0.0891 | 0.0002 | 0.5695 |
| RbKa1 | 12 | 0.0005 | 0.0006 | 1.1031 | 0.0006 | 1.1245 |
| U La1 | 12 | 0.0005 | 0.0006 | 1.1231 | 0.0006 | 1.2138 |
| SrKa1 | 12 | 0.0290 | 0.0065 | 0.2251 | 0.0094 | 0.3387 |
| Y Ka1 | 12 | 0.0017 | 0.0004 | 0.2281 | 0.0013 | 0.7891 |
| ZrKa1 | 12 | 0.0093 | 0.0021 | 0.2204 | 0.0023 | 0.2619 |
| NbKa1 | 12 | 0.0005 | 0.0001 | 0.1174 | 0.0004 | 0.6735 |
| MoKa1 | 12 | 0.0031 | 0.0032 | 1.0465 | 0.0043 | 1.4523 |
| RhKa1 | 12 | 0.0000 | 0.0000 | -- | 0.0000 | -- |
| SnKa1 | 12 | 0.0002 | 0.0000 | 0.1163 | | 0.0000 |
| SbKa1 | 12 | 0.0003 | 0.0004 | 1.4284 | 0.0005 | 1.7143 |

Similar to the other RCA samples discussed previously, only elements K, Cr, Mn and Fe showed a decreasing trend in RMSD values with decrease in sample size indicating there was improved accuracy in the above-mentioned elements with decrease in particle size.

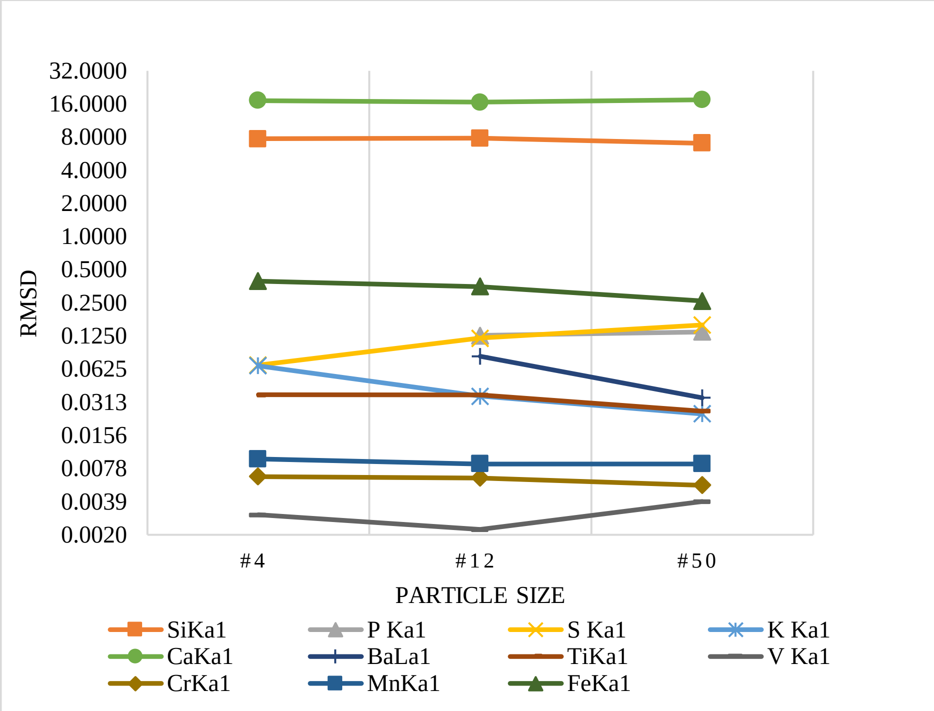



Figure 4.14 Sieve Size Vs RMSD (NC_CT2 RCA Sample-Major Elements)

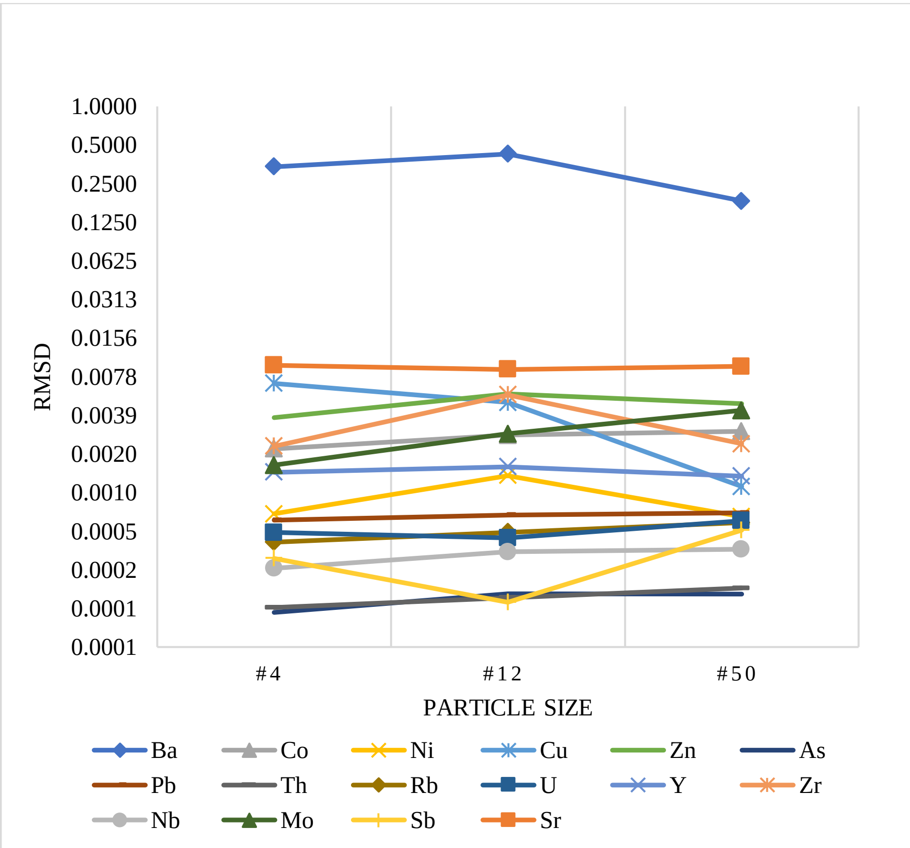



Figure 4.15 Sieve size Vs RMSD (NC_CT2 RCA Sample-Trace Elements)

Based on the RMSD values computed by taking the standard deviation of the difference between the whole rock values and PHXRF results, it was observed that barring a handful of elements, there was no specific trend that the RMSD values followed when going from a larger to a smaller particle size for the sample. The values seemed to vary from element to element across the different samples without any readily discernable pattern.

The RMSD values for Si (NC_AP1, NC_CT1& NC_HW1) and the Ca value (NC_CT2) were high and in the unacceptable range whereas rest of the elements were reasonable with most of the trace elements showing RMSD values under 1 indicating that the trace elements are close to the whole rock values and that the data is concentrated around the line of best fit. Contrary to what was observed in the literature review for a similar analysis of soils, the RMSD values for RCA samples did not decrease with decrease in particle size. This could be attributed to RCA's heterogenous composition due to the presence of adhered mortar and other contaminants whereas the sample under consideration in the literature review was soil which had a more homogenous composition.

Therefore, based on the RMSD values, it can be concluded that the particle size should not have a significant impact on the elemental concentrations of majority of the elements for the samples tested in this research study using PHXRF.

### 4.4.4 Mortar Content prediction

The mortar content was predicted using the stepwise regression method. The stepwise regression method falls under the multiple linear regression category in which the relationship between the response and predictor variables is determined (Minitab 2019). This method uses a combination of the forward selection and backward elimination methods to build a best fitted combination of independent and dependent variable (Chen et al. 2013). During each step, the least significant variables are removed, and the most significant ones are added to the model. To perform this analysis, Minitab software was used to build a regression model equation with the best predictor variables for the mortar content. During each step of the process, variables are added and deleted from the model based on the selected alpha to enter and alpha to remove values. Minitab systematically adds and removes the variables based on the p-values of the variables at each step.

Based on the p-value, Minitab tests the null hypothesis for each regression coefficient to check if it is 0 or not. Thus, predictors with lower p-values make a meaningful contribution to the model. The process stops adding or deleting variables into the model when the p-values of all the predictors present outside of the model are greater than the alpha-to-enter value and when the p-values of the predictors in the model are less than or equal to the alpha-to-remove value (Minitab 2019).

To predict the mortar content, the mortar content computed for the RCA samples from four different sources were chosen as the response variable and the PHXRF results obtained from the separated mortar comprising of both major and trace elements were taken as the predictors. The data was entered into the Minitab software and stepwise regression was performed with an alpha-to-enter value and alpha-to-remove value of 0.15 (PennState Eberly College of Science 2020). A regression model was developed for the RCA particle sizes No. 4, No. 12 and No. 50. The regression equations from Minitab are as follows (refer to Appendix C for supporting data and additional information):

Table 4.20 Regression equations for No.4, No.12, No.50

| Particle Size | Model to predict mortar content | $R^2$ |
|---|---|---|
| No.4 | RCA mortar content (weight %) = 54.52+ 3787×V – 16911×Pb | 0.999 |
| No.12 | RCA mortar content (weight %) =189.97- 524.0×P – 124820×Pb | 0.998 |
| No.50 | RCA mortar content (weight %) = 78.66 – 6588×Cr | 0.958 |

Table 4.21 Percent difference between predicted and actual mortar content for No.4 size

| RCA Sample | True Mortar Content | Predicted Mortar Content | % Difference |
|---|---|---|---|
| NC_AP1 | 33.70 | 33.92 | -0.01 |
| NC_CT1 | 41.90 | 41.64 | 0.01 |
| NC_HW1 | 43.90 | 43.79 | 0.00 |
| NC_CT2 | 55.80 | 55.94 | 0.00 |

Table 4.22 % difference between predicted and actual mortar content for No.12 size

| RCA Sample | Actual Mortar Content | Predicted Mortar Content | % Difference |
|---|---|---|---|
| NC_AP1 | 33.70 | 33.36 | 1.02 |
| NC_CT1 | 41.90 | 42.13 | -0.55 |
| NC_HW1 | 43.90 | 44.29 | -0.88 |
| NC_CT2 | 55.80 | 55.51 | 0.52 |

Table 4.23 % difference between predicted and actual mortar content for No.50 size

| a simple | True Mortar Content | Predicted Mortar Content | % Difference |
|---|---|---|---|
| NC_AP1 | 33.7 | 35.45 | -5.18 |
| NC_CT1 | 41.9 | 39.27 | 6.27 |
| NC_HW1 | 43.9 | 44.59 | -1.57 |
| NC_CT2 | 55.8 | 55.98 | -0.32 |

The actual mortar content was compared to the predicted mortar content determined using regression equations and it was observed that the equations were fairly strong predictors of the mortar content. Although the predicted values for the three different sizes were fairly close to the actual mortar content, based on the difference of the mortar content between the actual and predicted values it can be concluded that the size No.4 was the best predictor of the mortar content as the predicted values are very close to the actual mortar content for the RCA samples. The reason behind greater differences for No.50 particle size compared to No.4 and No.12 could be attributed to its size. With increase in the nominal size of RCA, the adhered mortar fraction tends to decrease (Zheng et al. 2018). Therefore, No.50 Particle size, which is much finer than No.4 and No.12 size differs in composition as compared to No.4 and No.12 particle size as they have both an aggregate portion as well as the adhered mortar portion.

It is notable that the stepwise regression resulted in the selection of trace elements like vanadium (V), phosphorous (P), lead (Pb) and chromium (Cr) as the strongest predictors of mortar content. This could be attributed to the fact that these elements could be present in the paste only (due to their relatively higher presence in cement and fly ash) or they may be present in very low quantities in the aggregate alone. From the PHXRF data obtained for aggregate only (Appendix A) it was observed that these metals were actually present in low concentrations in the aggregates which made them good predictors. On the other hand, major elements like Ca, Si, Fe and Al which were initially thought to be strong potential predictors of mortar content were not identified as predictor variables, likely due to the fact that they were present in both the aggregate and the cement paste in significant quantities. Test results indicated that the elements Ca, Si, Fe and Al were present in significant quantities in both the aggregate (alone) and cement/fly-ash (alone) samples and their relative weight % were high in both of these samples.

Although these models were found to be strong predictors of mortar content, one significant limitation associated with these models would be that the predictor elements would differ based on the source RCA. Additional work would be needed to explore the variability introduced in these models by other RCA sources.

Another drawback to these models is that several predictor variables are heavy metals, which can be detrimental to human health. One barrier to use of RCA, particularly in unbound uses, is the potential for heavy metals from the RCA to leach into waters flowing around/over the RCA (Snyder et al. 2018). Although this approach appears to provide a readily implementable method to predict the mortar content of RCA, it may call attention to a negative aspect of RCA, which is also a detractor to its use.

4.4.5 Prediction of chemical composition using weight percent of elements

Because the PHXRF only tests material to a certain depth (just inside the surface), it was not anticipated that the weight percent value reported by the PHXRF would be directly comparable to the weight % value determined through laboratory testing (Whole Rock Analysis), where the sample is pulverized, and more extensive testing is performed on the full volume of the sample. Therefore, to evaluate the accuracy of the PHXRF results, a simple linear regression analysis was performed. A particle size based simple regression model for each element was created to estimate the relationship between the whole rock values and the PHXRF values from the four RCA samples. For each particle size, “true element content” (from the Whole Rock Analysis) on x-axis vs measured element content on y-axis was plotted for every individual major and trace element (refer to Appendix D).

The plot generated a trendline with an $R^2$ value for each element pertaining to the particle size No.4, No.12 and No.50. The goal of this analysis was to observe the $R^2$ value and comment on how close the PHXRF values were to the regression line and to determine the predicted value of the elemental concentration from the regression equation of the most suitable size. Refer to Appendix D for individual plots. The color-coded table below shows the $R^2$ value for the particle size No.4, No.12 and No.50 for each element where values highlighted in green represent good values, values highlighted in yellow represent medium values and those highlighted in red represent low values:

Table 4.24 $R^2$ values for Major and Trace Elements

| Element | $R^2$ | | |
|---|---|---|---|
| | No.4 | No.12 | No.50 |
| Si | 0.8846 | 0.9718 | 0.7476 |
| Ca | 0.9713 | 0.9222 | 0.968 |
| Al | 0.5756 | 0.8575 | 0.7832 |
| Fe | 0.7278 | 0.2678 | 0.0753 |
| K | 0.9083 | 0.9754 | 0.9377 |
| Ti | 0.8686 | 0.4613 | 0.2612 |
| S | 0.813 | 0.6338 | 0.4722 |
| Mn | 0.7296 | 0.9777 | 0.6424 |
| V | 0.8797 | 0.9476 | 0.8271 |
| Cr | 0.5417 | 0.2254 | 0.2471 |
| Ba | 0.4996 | 0.6139 | 0.0522 |
| Co | 0.7897 | 0.8194 | 0.3271 |
| Ni | 0.0017 | 0.9461 | 0.849 |
| Cu | 0.0002 | 0.6203 | 0.879 |
| Zn | 0.1048 | 0.7822 | 0.221 |
| As | 0.8279 | 0.1985 | 0.4416 |
| Pb | 0.3635 | 0.0244 | 0.7677 |
| Th | 0.9754 | 0.9299 | 0.8021 |
| Rb | 0.9902 | 0.9781 | 0.8741 |
| U | 0.7588 | 0.0433 | 0.0531 |
| Sr | 0.1808 | 0.9067 | 0.6351 |
| Y | 0.3753 | 0.244 | 0.2045 |
| Zr | 0.5204 | 0.5735 | 0.0197 |
| Nb | 0.9293 | 0.8138 | 0.7004 |
| Mo | 0.3915 | 0.0289 | 0.1974 |
| Sb | 0.0249 | 0.2063 | 0.6832 |
| Sn | 0.3227 | 0.5313 | 0.1338 |

Based on the $R^2$ values, it was observed that the particle sizes No. 4 (14 elements out of 27) and No. 12 (13 elements out of 27) show a strong relationship between the Whole Rock Analysis and PHXRF results for major and trace elements. Elements including Si, Ca, K, V, Th, Rb showed exceptional $R^2$ values implying that they were more predictable compared to the other elements. This improved predictability could be attributed to the fact that the relative quantities of these elements present in the samples were significant.

### 4.4.6 Recommendation of Particle Size for Samples

Based on the findings of statistical analysis used to 1) predict the laboratory measured mortar content based on PHXRF measurements and 2) predict the chemical composition of RCA using weight percent of selected elements, it appears that the No. 4 particle size can be recommended as the representative particle size for PHXRF analysis. The No. 4 particle size clearly exhibited the strongest models used to predict the laboratory measured mortar content. Although results were more variable in the elemental analysis, there was not a significantly different improvement in prediction between the No. 4 and No. 12 particle size, and the No. 50 size had the least strong correlations of the three particle sizes tested. Regression analysis indicated that the PHXRF results show reasonable accuracy for most of the major and trace elements detected and quantified by the PHXRF using the No 4 size.

Therefore, in order to simplify a recommended testing protocol and to save time, the No. 4 particle size is suggested for future use of the PHXRF to analyze RCA, since it shows reasonable accuracy for the elements under consideration. Of note, the No. 4 particle size provides an advantage over the No. 12 and No. 50 particle size in that it would likely be present in both the coarse RCA and fine RCA produced from a given source.

# CHAPTER 5: RECOMMENDED TEST METHOD

## 5.1 Scope**:**

This test method determines chemical characteristics of recycled concrete aggregates (RCA) using the portable handheld X-ray fluorescence device (PHXRF). The test method includes a series of procedures for sampling, determination of physical properties and chemical characterization of the aggregates, and estimation of the mortar content and potential contaminant contents using the PHXRF measurements.

## 5.2 Sampling**:**

Care should be taken during sampling to ensure that representative RCA material is selected from the stockpile. The sampling procedure should ensure the sample selected for testing is representative of the total material to be represented by the testing. To obtain samples, the ASTM D75, "Standard Practice for Sampling Aggregates" standard can be followed for guidance. This standard describes the procedure to obtain samples from different sources such as (1) stockpiles, (2) conveyor belts, (3) bins or belt discharges and (4) roadway.

## 5.3 Physical Characterization tests**:**

The RCA samples obtained should undergo physical characterization tests including sieve analysis (ASTM C136/C136M-14), density, relative density (specific gravity), and absorption (ASTM C 127) and bulk density and voids in aggregates (ASTM C 29/C 29 M). These preliminary tests of the RCA assist in mixture design and proportioning so that workable, durable concrete with adequate mechanical properties can be produced (PCA 2019).

## 5.4 Determination of mortar content using the Thermal Shock Method**:**

5.4.1 *Apparatus*- The apparatus required for this test includes:

- standard sieves of sizes 1 inch, ¾ inch, ½ inch, 3/8 inch, and No. 4

- a furnace (with a minimum volume of 0.115 cubic feet and capability of heating to 1200 °F
- a jar mill with cylindrical alumina grinding media of sizes 13/16” x 13/16”.

5.4.2 A representative sample of coarse RCA weighing 500g, retained on the No. 4 sieve and above shall be prepared after removal of any contaminants like brick, metal, wood and asphalt from the sample.

5.4.3 The residual mortar content of the bulk sample of RCA should be determined using the Thermal Shock Method as described in Section 3.3.4 to separate the mortar and aggregate and calculate the RMC % for No. 67 gradation.

5.4.4 The RMC % can be calculated using the equation as follows:

$$RMC\ (\%) = \frac{\text{Mass of material passed \#4 sieve}}{\text{Total mass}} \times 100\%$$

5.4.5 The grinding time and heating temperature should be carefully evaluated for the sample under consideration (Mamirov 2020). The objective behind the selection of both the parameters should be based on selecting the optimal temperature and grinding time which results in a complete removal of residual mortar.

5.5 PHXRF sample preparation and testing procedure:

5.5.1 *Apparatus*- The apparatus for this test procedure includes:

- a PHXRF
- vacuum pump
- desktop stand
- sample cups
- mylar film
- desktop computer

5.5.2 A representative sample weighing 10 kgs shall be sieved through sieve sizes 1 inch, ¾ inch, ½ inch, 3/8 inch, No. 4, No. 12, and No. 50. The portion of the sample retained on sieve sizes No. 4, No. 12, and No. 50 should be set aside for PHXRF analysis.

5.5.3 The selected samples shall be placed in sample cups which have a depth of at least 10 mm to mitigate the inaccuracies caused due to the surface thickness phenomenon.

5.5.4 Secure the sample cups with a mylar film and divide the sample cup into 4 quadrants. Place the sample cup upside down on the sample table such that it rests on the nose of the PHXRF device. Following the PHXRF manufacturer's instructions, select an appropriate calibration file (such as 'Mudrock' for the Bruker Tracer device) which is capable of detecting and quantifying elements present in concrete aggregates.

If an appropriate PHXRF manufacturer's calibration file is unavailable, develop your own calibration file by preparing a fresh reference sample or augment an existing calibration file by adding additional reference samples relevant to the existing file. The Artax software can be used for augmenting or building a new calibration file. Once the device is set-up and the calibration file is ready, take 3 measurements for each sample quadrant using the quadrant scanning technique (described in Section 4.3.3). Calculate the average of 12 readings for each elemental concentration.

5.5.5 The scanning duration for the major & trace element analysis should be as per the calibration file provided by the manufacturer. For Bruker's Mudrock calibration, the recommended scan duration is 180 and 60 seconds for major and trace elements respectively. Use a vacuum for major element analysis if recommended by the manufacturer.

5.6 Whole Rock Analysis:

5.6.1 The purpose of this section is to provide instructions for developing a reference sample for the validation and assessment of the accuracy of the PHXRF results. The control sample must be prepared for each individual RCA sample under consideration. To prepare the reference sample, the mortar and aggregate should be separated and the RMC by percent weight should be determined using the Thermal Shock Method.

5.6.2 Once separated, a composite sample should be developed according to the computed RMC percent weight value. The weight distribution of the reference sample should be: X% of total weight of the sample + (100-X)% of total weight of the sample.

5.6.3 Use the whole rock analysis technique to obtain measurements of major and trace elements present in the sample. Also, test the separated mortar and aggregate from the Thermal Shock Method separately using the PHXRF.

5.7 Statistical Analysis:

5.7.1 After obtaining the measurements from PHXRF and whole rock analysis, run the ANOVA test to test statistical significance between the particle sizes of the same sample and across samples of the same size. Compute statistical parameters including standard deviation, COV, RMSD & $COV_{RMSD}$.

5.5.8 Plot the RMSD vs size graph to observe a trend between both the parameters. The goal should be to identify a decreasing trend of RMSD value with decrease in particle size. The particle size with the lowest RMSD indicates high accuracy.

5.5.9 To do an accuracy comparison, use regression analysis and create a size-based regression model for each element of all the samples tested with whole rock analysis values and PHXRF values. Observe the $R^2$ values for each size. An $R^2$ value closer to 1 would suggest stronger correlation between the whole rock analysis and PHXRF results thereby showing higher accuracy. Based on the $r^2$ values for all the major and trace elements, select the best particle size and use the regression equation for that particle size to compute the predicted values for all elements.

5.8 Mortar Content:

5.6.1 To predict the mortar content of the samples, use stepwise regression analysis.

5.6.2 Take the mortar content computed from the Thermal Shock Method as the response variables and the major and trace elements as the predictor variables. Use statistical software to perform a stepwise regression and obtain the model equation for each size.

5.6.3 Input the values of variables in the model equation to get the predicted values of mortar for each size. Compare the % difference between the true values of mortar and the predicted ones to determine the best predictor of mortar content based on size.

# CHAPTER 6: CONCLUSION AND RECOMMENDATIONS FOR FUTURE WORK

## 6.1 Conclusion:

This research study presents results from the PHXRF analysis of samples acquired from four different sources in North Carolina: from an airport pavement in Charlotte (NC_AP1), from a highway pavement near Durham (NC_HW1), from an unknown building source crushed at a stationary demolition facility in Charlotte (NC_CT1), and from an unknown building source crushed at a stationary facility in Wilmington NC_CT2). The goal of the research was to develop a testing protocol to determine the mortar content of the RCA samples and the presence of any contaminants using the portable handheld XRF.

The purpose of this study was to determine if the PHXRF could be used to quantify elements present in the RCA samples accurately and to develop a model that would predict the mortar content of the samples. In this research study, the collected samples were first subjected to physical characterization tests including sieve analysis (ASTM C136/C136M-14), density, relative density (specific gravity), and absorption (ASTM C 127) and bulk density and voids in aggregates (ASTM C 29/C 29 M) and the Thermal Shock Method.

The Thermal Shock Method was used to separate the aggregate and mortar and the average RMCs in weight percent were computed for the RCA samples for No. 67 graded aggregates. The RMC contents in weight percent for the samples included in this study were 33.70%, 41.90%, 43.90% and 55.80% for NC_AP1, NC_CT1, NC_HW1 & NC_CT2, respectively.

The Bruker Tracer III-SD series handheld XRF was used to test the RCA samples retained on Sieves No. 4, No. 12 and No. 50 and the detected elements were quantified using Bruker's 'Mudrock' calibration file. The ANOVA test was performed on the weight % of the quantified elements between the particle sizes of the same sample and across samples of the same size and it was observed that between the particle size, most of the elements were statistically significant indicating the elemental weight percent changed with size. Similarly, the ANOVA results for samples of the same size across the different samples were also statistically significant for majority of the elements indicating that the concentrations varied across samples.

To test the accuracy of the results, a reference sample was developed for Whole Rock Analysis. Using the quantified data from the reference sample, a comparison was made with the results obtained from PHXRF analysis. Statistical parameters including standard deviation, COV, RMSD & $COV_{RMSD}$ were computed. The RMSD values for the particle sizes No. 4, No. 12, and No. 50 for a majority of the elements did not show a decreasing trend as the particle size decreased, contrary to what was described in the literature review for stabilized soil samples (the closest material to RCA found in the literature). Instead, for this study, the RMSD values for the detected elements did not show a specific trend with change in particle size.

Knowing the mortar content of an RCA sample is important to evaluating its use in bound and unbound applications. The ability to predict the mortar content of RCA should allow it to be more readily utilized by practitioners. In this study, the mortar content of the RCA samples was determined in the laboratory using the Thermal Shock Method (Mamirov et al. 2020), and models were developed to enable the PHXRF elemental analysis in weight percent to predict the RMC.

To accomplish this, the stepwise regression method was used to predict the RMC (in weight percent) based on the elemental weight percent data collected from PHXRF analysis. The RMC% from the thermal shock method was entered in the software as response variables and the quantified elements from the PHXRF analysis of RCA samples was taken as the predictors. Minitab software returned predictor equations for each round of analysis. The three equations generated by the software for the sizes No. 4, No. 12, and No. 50 included as predictor variables the trace elements lead, phosphorus, vanadium and chromium. Once the equations were obtained, the values of these predictor variables were substituted in the equation to obtain the predicted values of mortar content. On comparison of the three equations, it was observed that the regression equation for the No. 4 particle size provided predictions closest to the laboratory-measured value of mortar content for all four of the samples. Based on this observation, it was be concluded that 1) it is possible to predict the mortar content of RCA using the PHXRF and 2) the particle size No. 4 provided the best predictions of the mortar content for the RCA samples tested in this research study.

The PHXRF cannot be expected directly determine the chemical composition of the RCA due to the fact that 1) for each element, the device only measures the material composition to a certain depth, and 2) the device is incapable of detecting elements lighter than Na (so elements such as H, O, and C likely present in the RCA will not be detected and the total weight percent will not add to 100%). To evaluate the ability of the PHXRF to predict the chemical composition of the RCA, a regression model was developed for each element weight percent determined by the PHXRF for each particle size (No. 4, No. 12 and No. 50) of the four samples, correlating the measurement to the corresponding Whole Rock Analysis weight percent. The $R^2$ value obtained from this regression analysis was observed for each particle size for major and trace elements and it was concluded that particle size No.4 and No.12 showed reasonable $R^2$ values for major and trace elements. However, since the models used to predict mortar content suggested the No. 4 size to be the most appropriate, the No. 4

size is recommended for this test as well, primarily to save time and perform characterization testing on a single particle size.

Of note, use of the No. 4 particle size should allow testing of both fine and coarse RCA samples, since this particle size is often present in both gradations of aggregate typically used in building construction. Also, several heavy metals were identified as the strongest predictor variables for mortar content. It is noted that although these elements allowed for strong models to be developed, they are also negatively linked to human health effects, and their use may bring unwanted attention to the RCA as a material containing these substances.

6.2 Limitations and future work:

- One of the most notable limitations of this research would be the limited number of samples obtained for analysis. Due to time and resource constraint, only a limited number of samples could be collected for this research study. Having more samples to test (i.e. samples from other states where the aggregates must have met QA requirements according to different agency specifications) would help in evaluating how the PHXRF fares against diverse samples. Future research studies should focus on incorporating samples from a variety of concrete sources to reduce bias.
- Sample sets from across the U.S would help capture a broader range in composition. The properties of RCA would also vary due to the w/cm ratio of the source concrete and the presence of organic impurities like alkali aggregate reaction, high alumina cement, silt, chloride, sulfate etc (Behera et al. 2014). A wide range of properties and characteristics of the RCA are also influenced by the recycling method and type of crushing equipment utilized in the recycling process. Samples from across the U.S would vary based on crushing methods

and equipment used for production and could help strengthen the analysis and findings.

- The 'Mudrock'calibration file used in the study is a factory installed calibration file that comes with the device and this calibration is capable of quantifying from parts per million to percent weight levels as the set includes matrices ranging from limestone to near pure silicates. This calibration set is based on elemental diversity as it contains 26 well defined reference samples obtained from multiple drill cores. Based on the manufacturer's recommendation, the Mudrock calibration file was used for quantification purpose (Bruker 2014). Further studies should be conducted to recalibrate existing files by adding additional reference samples to the calibration. This would improve this analysis by capturing a broader range of elements from high to low concentrations especially for critical elements present in recycled concrete aggregates.
- In this study, the whole rock analysis method was used for the evaluation of accuracy of the PHXRF device. Due to monetary constraints, only one sample representative of the whole sample was used for developing the reference sample which likely limited the findings. Developing a reference sample for each size would likely give more accurate results.

Based on the observations made in this research study, it can be said that the PHXRF shows strong potential for use for the chemical characterization of RCA, as well as for estimating the residual mortar content. Fostering the use of this technology could be highly useful in promoting the use of RCA in concrete construction. As a non-destructive, timesaving, and easy to use technology, PHXRF has the potential to aid in the QA and QC requirements for qualifying an RCA source for use in concrete. The recommended test method developed as part of the objective of this research could serve as the basis for a technical procedure for contractors and other users interested in this type of characterization of RCA. Broadening the work presented in this thesis with additional RCA sources and validating the models using additional RCA sources should allow users to develop more efficient models for use with their own RCA sources and PHXRF equipment.

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# APPENDIX A: RAW PHXRF DATA FOR RCA SAMPLES

Table A.1 Raw PHXRF data for No.4 NC_AP1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.2117 | 0.0914 | 0.0923 | 0.0000 | 0.0000 | 0.0000 | 0.2207 | 0.1930 | 0.1615 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6188 | 0.3938 | 0.4127 | 1.1389 | 1.7550 | 1.5508 | 0.7110 | 1.0438 | 0.5040 | 1.7727 | 1.7468 | 1.5581 |
| Si | 2.9490 | 2.3260 | 2.4901 | 3.8304 | 5.2984 | 4.8896 | 3.3112 | 3.8419 | 2.7210 | 6.6322 | 6.7445 | 5.5482 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0111 | 0.0078 | 0.0000 | 0.0037 | 0.0020 | 0.0000 | 0.0852 | 0.0714 | 0.0464 |
| S | 0.5808 | 0.5885 | 0.5237 | 0.4754 | 0.5041 | 0.5192 | 0.5215 | 0.5346 | 0.5260 | 0.4081 | 0.3991 | 0.4485 |
| K | 0.1474 | 0.0689 | 0.1112 | 0.3005 | 0.4546 | 0.4226 | 0.2498 | 0.2511 | 0.1319 | 0.3632 | 0.3597 | 0.4734 |
| Ca | 2.7456 | 2.1152 | 3.0364 | 4.1334 | 5.3415 | 4.9817 | 2.6388 | 3.4225 | 2.5991 | 6.1006 | 6.5869 | 5.8799 |
| Ba | 1.3096 | 1.2558 | 0.2572 | 0.3473 | 0.3635 | 0.2371 | 0.1068 | 0.0882 | 0.0000 | 0.2252 | 0.1755 | 0.1670 |
| Ti | 0.0000 | 0.0000 | 0.1609 | 0.1850 | 0.3398 | 0.3172 | 0.2680 | 0.2860 | 0.1341 | 0.3285 | 0.2939 | 0.2453 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Cr | 0.0051 | 0.0064 | 0.0021 | 0.0050 | 0.0054 | 0.0072 | 0.0026 | 0.0034 | 0.0041 | 0.0043 | 0.0050 | 0.0054 |
| Mn | 0.0236 | 0.0279 | 0.0207 | 0.0283 | 0.0338 | 0.0389 | 0.0237 | 0.0294 | 0.0214 | 0.0320 | 0.0303 | 0.0314 |
| Fe | 0.0000 | 0.0000 | 1.2043 | 1.6143 | 3.0653 | 2.7738 | 2.6849 | 3.2957 | 2.0549 | 2.6367 | 2.6592 | 2.6374 |
| Ba | 1.8602 | 1.2426 | 1.0114 | 1.2420 | 1.1659 | 0.9023 | 1.9158 | 2.5324 | 7.1323 | 2.7280 | 2.8191 | 5.1900 |
| Co | 0.0217 | 0.0152 | 0.0131 | 0.0207 | 0.0239 | 0.0121 | 0.0342 | 0.0502 | 0.0805 | 0.0474 | 0.0475 | 0.0474 |
| Ni | 0.0010 | 0.0015 | 0.0023 | 0.0050 | 0.0009 | 0.0070 | 0.0022 | 0.0006 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Cu | 0.0034 | 0.0023 | 0.0003 | 0.0034 | 0.0005 | 0.0041 | 0.0039 | 0.0050 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Zn | 0.0048 | 0.0064 | 0.0023 | 0.0028 | 0.0014 | 0.0015 | 0.0044 | 0.0048 | 0.0022 | 0.0000 | 0.0001 | 0.0010 |
| As | 0.0004 | 0.0004 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0003 | 0.0002 | 0.0001 | 0.0001 | 0.0002 |
| Pb | 0.0011 | 0.0014 | 0.0013 | 0.0013 | 0.0007 | 0.0017 | 0.0016 | 0.0012 | 0.0009 | 0.0013 | 0.0013 | 0.0009 |
| Th | 0.0004 | 0.0005 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0004 | 0.0003 | 0.0002 |
| Rb | 0.0022 | 0.0030 | 0.0018 | 0.0011 | 0.0000 | 0.0015 | 0.0023 | 0.0005 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| U | 0.0014 | 0.0009 | 0.0009 | 0.0009 | 0.0154 | 0.0023 | 0.0021 | 0.0055 | 0.0068 | 0.0028 | 0.0049 | 0.0088 |
| Sr | 0.0670 | 0.0765 | 0.0358 | 0.0527 | 0.0243 | 0.0704 | 0.0542 | 0.0415 | 0.0057 | 0.0067 | 0.0061 | 0.0075 |
| Y | 0.0013 | 0.0014 | 0.0016 | 0.0022 | 0.0000 | 0.0013 | 0.0022 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Zr | 0.0115 | 0.0166 | 0.0091 | 0.0086 | 0.0079 | 0.0061 | 0.0113 | 0.0107 | 0.0056 | 0.0063 | 0.0063 | 0.0063 |
| Nb | 0.0007 | 0.0007 | 0.0005 | 0.0006 | 0.0015 | 0.0007 | 0.0007 | 0.0008 | 0.0008 | 0.0009 | 0.0009 | 0.0009 |
| Mo | 0.0020 | 0.0014 | 0.0055 | 0.0038 | 0.0114 | 0.0026 | 0.0051 | 0.0087 | 0.0136 | 0.0139 | 0.0137 | 0.0132 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0002 | 0.0005 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0001 | 0.0000 | 0.0006 |

Table A.2 Raw PHXRF data for No.12 NC_AP1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #12 | | | Quadrant 2 #12 | | | Quadrant 3 #12 | | | Quadrant 4 #12 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.8815 | 1.1742 | 1.0076 | 1.2316 | 0.9412 | 1.0634 | 1.0118 | 1.5087 | 1.2840 | 1.0742 | 1.1479 | 1.1199 |
| Si | 3.5923 | 4.1603 | 3.8374 | 4.5661 | 4.9957 | 4.0611 | 4.0876 | 4.8028 | 4.9091 | 4.5957 | 4.3821 | 4.0606 |
| P | 0.0129 | 0.0000 | 0.0000 | 0.0020 | 0.0000 | 0.0001 | 0.0117 | 0.0000 | 0.0126 | 0.0000 | 0.0020 | 0.0069 |
| S | 0.4362 | 0.5217 | 0.5187 | 0.5158 | 0.5321 | 0.4800 | 0.4911 | 0.5299 | 0.4903 | 0.5299 | 0.4666 | 0.4682 |
| K | 0.2632 | 0.2616 | 0.2553 | 0.2402 | 0.1559 | 0.2890 | 0.2650 | 0.2975 | 0.2943 | 0.3478 | 0.3018 | 0.2782 |
| Ca | 4.7947 | 3.7568 | 4.6021 | 4.8981 | 2.9174 | 5.1029 | 4.0475 | 4.1286 | 5.0526 | 4.5119 | 5.0557 | 4.2954 |
| Ba | 0.1145 | 1.6630 | 0.0000 | 1.3312 | 0.9949 | 0.2559 | 0.2152 | 1.9374 | 0.3009 | 0.0000 | 0.3496 | 0.3211 |
| Ti | 0.1992 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.1952 | 0.1540 | 0.0000 | 0.2102 | 0.0000 | 0.2051 | 0.2007 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Cr | 0.0049 | 0.0065 | 0.0051 | 0.0064 | 0.0116 | 0.0053 | 0.0057 | 0.0071 | 0.0046 | 0.0063 | 0.0052 | 0.0065 |
| Mn | 0.0249 | 0.0279 | 0.0268 | 0.0279 | 0.0383 | 0.0230 | 0.0233 | 0.0297 | 0.0241 | 0.0266 | 0.0281 | 0.0299 |
| Fe | 2.4632 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 2.1437 | 1.7484 | 0.0000 | 2.3489 | 0.0000 | 1.8498 | 1.7925 |
| Ba | 0.9308 | 0.9564 | 0.7093 | 0.7154 | 0.3752 | 0.6511 | 0.4351 | 0.5775 | 0.5032 | 0.2995 | 0.4801 | 0.3083 |
| Co | 0.0100 | 0.0097 | 0.0058 | 0.0049 | 0.0029 | 0.0043 | 0.0055 | 0.0050 | 0.0095 | 0.0063 | 0.0026 | 0.0062 |
| Ni | 0.0034 | 0.0034 | 0.0033 | 0.0047 | 0.0039 | 0.0042 | 0.0034 | 0.0023 | 0.0037 | 0.0038 | 0.0029 | 0.0037 |
| Cu | 0.0078 | 0.0091 | 0.0058 | 0.0065 | 0.0064 | 0.0062 | 0.0079 | 0.0110 | 0.0077 | 0.0063 | 0.0044 | 0.0075 |
| Zn | 0.0061 | 0.0057 | 0.0046 | 0.0047 | 0.0048 | 0.0064 | 0.0062 | 0.0057 | 0.0063 | 0.0056 | 0.0039 | 0.0065 |
| As | 0.0004 | 0.0004 | 0.0003 | 0.0003 | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0004 |
| Pb | 0.0014 | 0.0012 | 0.0007 | 0.0016 | 0.0015 | 0.0014 | 0.0013 | 0.0013 | 0.0012 | 0.0008 | 0.0014 | 0.0009 |
| Th | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0004 | 0.0003 | 0.0004 | 0.0004 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0013 | 0.0016 | 0.0015 | 0.0012 | 0.0002 | 0.0008 | 0.0006 | 0.0013 | 0.0006 | 0.0015 | 0.0010 | 0.0008 |
| U | 0.0000 | 0.0004 | 0.0000 | 0.0000 | 0.0000 | 0.0002 | 0.0006 | 0.0014 | 0.0000 | 0.0013 | 0.0000 | 0.0006 |
| Sr | 0.0622 | 0.0900 | 0.0818 | 0.0838 | 0.0645 | 0.1005 | 0.0921 | 0.0977 | 0.0868 | 0.0859 | 0.0404 | 0.0963 |
| Y | 0.0030 | 0.0024 | 0.0023 | 0.0027 | 0.0025 | 0.0024 | 0.0018 | 0.0022 | 0.0023 | 0.0023 | 0.0034 | 0.0021 |
| Zr | 0.0140 | 0.0134 | 0.0097 | 0.0101 | 0.0083 | 0.0115 | 0.0083 | 0.0118 | 0.0090 | 0.0074 | 0.0140 | 0.0061 |
| Nb | 0.0006 | 0.0007 | 0.0006 | 0.0007 | 0.0006 | 0.0007 | 0.0006 | 0.0008 | 0.0007 | 0.0005 | 0.0007 | 0.0006 |
| Mo | 0.0010 | 0.0000 | 0.0007 | 0.0001 | 0.0011 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0008 | 0.0000 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0001 | 0.0000 | 0.0001 | 0.0000 | 0.0005 | 0.0000 |

Table A.3 Raw PHXRF data for No.50 NC_AP1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #50 | | | Quadrant 2 #50 | | | Quadrant 3 #50 | | | Quadrant 4 #50 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.4696 | 0.1490 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 1.3353 | 1.3271 | 1.3614 | 1.3881 | 1.4788 | 1.4027 | 1.4229 | 1.3275 | 1.5096 | 1.5513 | 1.4378 | 1.4274 |
| Si | 4.9590 | 4.5240 | 4.3848 | 5.4945 | 5.6603 | 5.5990 | 5.0161 | 4.9600 | 5.4240 | 4.9470 | 4.7902 | 5.7443 |
| P | 0.0000 | 0.0000 | 0.0010 | 0.0201 | 0.0037 | 0.0260 | 0.0111 | 0.0126 | 0.0059 | 0.0274 | 0.0179 | 0.0054 |
| S | 0.5356 | 0.5233 | 0.5267 | 0.5113 | 0.5299 | 0.4608 | 0.4810 | 0.4776 | 0.5311 | 0.4623 | 0.4732 | 0.5488 |
| K | 0.3061 | 0.2672 | 0.2705 | 0.2795 | 0.2875 | 0.3318 | 0.3075 | 0.3110 | 0.2893 | 0.3302 | 0.2982 | 0.2810 |
| Ca | 3.6016 | 3.9080 | 3.4910 | 3.9850 | 3.7441 | 5.1945 | 4.5605 | 4.4247 | 3.8578 | 3.8314 | 4.0492 | 3.3805 |
| Ba | 1.5158 | 1.3651 | 1.3166 | 1.5294 | 1.4793 | 0.2121 | 0.1810 | 0.1764 | 0.0000 | 0.2837 | 0.2711 | 1.5424 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.2191 | 0.2134 | 0.2072 | 0.0000 | 0.1531 | 0.1817 | 0.0000 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Cr | 0.0072 | 0.0075 | 0.0069 | 0.0083 | 0.0064 | 0.0057 | 0.0051 | 0.0054 | 0.0071 | 0.0065 | 0.0064 | 0.0063 |
| Mn | 0.0274 | 0.0282 | 0.0296 | 0.0289 | 0.0305 | 0.0247 | 0.0256 | 0.0266 | 0.0287 | 0.0243 | 0.0283 | 0.0363 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 2.5577 | 2.6150 | 2.5547 | 0.0000 | 1.4282 | 1.6859 | 0.0000 |
| Ba | 0.0426 | 0.1715 | 0.0204 | 0.1138 | 0.1749 | 0.3310 | 0.1058 | 0.1766 | 0.1169 | 0.2405 | 0.0592 | 0.0926 |
| Co | 0.0016 | 0.0009 | 0.0022 | 0.0011 | 0.0006 | 0.0013 | 0.0010 | 0.0011 | 0.0017 | 0.0009 | 0.0010 | 0.0012 |
| Ni | 0.0037 | 0.0022 | 0.0029 | 0.0029 | 0.0041 | 0.0030 | 0.0033 | 0.0026 | 0.0037 | 0.0040 | 0.0032 | 0.0019 |
| Cu | 0.0077 | 0.0088 | 0.0076 | 0.0084 | 0.0079 | 0.0086 | 0.0083 | 0.0091 | 0.0094 | 0.0094 | 0.0085 | 0.0054 |
| Zn | 0.0072 | 0.0064 | 0.0059 | 0.0054 | 0.0058 | 0.0062 | 0.0053 | 0.0069 | 0.0070 | 0.0068 | 0.0074 | 0.0032 |
| As | 0.0005 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0003 |
| Pb | 0.0008 | 0.0008 | 0.0010 | 0.0012 | 0.0009 | 0.0000 | 0.0010 | 0.0011 | 0.0011 | 0.0011 | 0.0009 | 0.0010 |
| Th | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 |
| Rb | 0.0015 | 0.0014 | 0.0014 | 0.0012 | 0.0012 | 0.0021 | 0.0016 | 0.0015 | 0.0007 | 0.0020 | 0.0017 | 0.0011 |
| U | 0.0003 | 0.0005 | 0.0010 | 0.0007 | 0.0003 | 0.0014 | 0.0007 | 0.0004 | 0.0000 | 0.0004 | 0.0004 | 0.0000 |
| Sr | 0.0559 | 0.0537 | 0.0541 | 0.0636 | 0.0685 | 0.0618 | 0.0489 | 0.0587 | 0.0511 | 0.0692 | 0.0630 | 0.0167 |
| Y | 0.0026 | 0.0022 | 0.0025 | 0.0024 | 0.0026 | 0.0023 | 0.0023 | 0.0028 | 0.0032 | 0.0026 | 0.0027 | 0.0025 |
| Zr | 0.0114 | 0.0122 | 0.0115 | 0.0121 | 0.0118 | 0.0113 | 0.0131 | 0.0108 | 0.0147 | 0.0120 | 0.0096 | 0.0078 |
| Nb | 0.0007 | 0.0006 | 0.0007 | 0.0008 | 0.0008 | 0.0007 | 0.0007 | 0.0008 | 0.0008 | 0.0007 | 0.0006 | 0.0006 |
| Mo | 0.0004 | 0.0000 | 0.0005 | 0.0001 | 0.0000 | 0.0005 | 0.0001 | 0.0003 | 0.0000 | 0.0000 | 0.0005 | 0.0032 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0004 | 0.0007 | 0.0001 | 0.0001 | 0.0007 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0002 |

Table A.4 Raw PHXRF data for No.4 NC_CT1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0036 | 0.0000 | 0.0000 | 0.0000 | 0.0059 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.3432 | 0.0390 | 0.2639 | 0.6519 | 0.2953 | 0.2766 | 0.3783 | 0.5582 | 0.6176 | 0.1342 | 0.1524 | 0.3699 |
| Si | 3.9782 | 3.3312 | 3.8273 | 4.0786 | 4.4876 | 2.8092 | 3.7967 | 4.6889 | 4.4936 | 3.1197 | 3.3196 | 4.0878 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0144 | 0.0269 | 0.0222 | 0.0000 | 0.0000 | 0.0197 |
| S | 0.5483 | 0.5150 | 0.6164 | 0.7094 | 0.6171 | 0.5210 | 0.5608 | 0.5632 | 0.5594 | 0.4894 | 0.5758 | 0.4772 |
| K | 0.2708 | 0.2108 | 0.2051 | 0.2572 | 0.1510 | 0.2023 | 0.2469 | 0.2523 | 0.3872 | 0.2858 | 0.1826 | 0.1871 |
| Ca | 8.6432 | 6.9433 | 5.1864 | 6.6564 | 5.9308 | 4.7277 | 6.3160 | 7.0164 | 6.0007 | 5.4266 | 7.1388 | 6.2843 |
| Ba | 0.0296 | 0.0000 | 0.3090 | 0.5113 | 0.3917 | 0.1184 | 0.4044 | 0.3876 | 0.4902 | 0.0169 | 0.0268 | 0.0000 |
| Ti | 0.0827 | 0.0468 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0620 | 0.0555 | 0.0479 |
| V | 0.0017 | 0.0028 | 0.0000 | 0.0000 | 0.0000 | 0.0065 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0014 | 0.0026 |
| Cr | 0.0044 | 0.0073 | 0.0045 | 0.0053 | 0.0065 | 0.0042 | 0.0000 | 0.0048 | 0.0057 | 0.0036 | 0.0033 | 0.0046 |
| Mn | 0.0246 | 0.0231 | 0.0238 | 0.0256 | 0.0274 | 0.0245 | 0.0000 | 0.0242 | 0.0238 | 0.0230 | 0.0234 | 0.0237 |
| Fe | 0.9757 | 0.7091 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.8376 | 0.7352 | 0.5135 |
| Ba | 0.7168 | 0.3881 | 0.2424 | 0.3254 | 0.2242 | 0.3392 | 0.1228 | 0.3327 | 0.1692 | 0.1451 | 0.0702 | 0.1662 |
| Co | 0.0043 | 0.0038 | 0.0021 | 0.0038 | 0.0043 | 0.0022 | 0.0020 | 0.0031 | 0.0021 | 0.0012 | 0.0016 | 0.0022 |
| Ni | 0.0015 | 0.0030 | 0.0021 | 0.0030 | 0.0031 | 0.0025 | 0.0024 | 0.0028 | 0.0026 | 0.0032 | 0.0025 | 0.0023 |
| Cu | 0.0010 | 0.0060 | 0.0019 | 0.0041 | 0.0056 | 0.0045 | 0.0074 | 0.0059 | 0.0032 | 0.0039 | 0.0052 | 0.0014 |
| Zn | 0.0009 | 0.0130 | 0.0025 | 0.0057 | 0.0068 | 0.0034 | 0.0050 | 0.0061 | 0.0044 | 0.0062 | 0.0057 | 0.0035 |
| As | 0.0001 | 0.0008 | 0.0003 | 0.0004 | 0.0005 | 0.0003 | 0.0007 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 |
| Pb | 0.0015 | 0.0014 | 0.0008 | 0.0009 | 0.0004 | 0.0012 | 0.0017 | 0.0006 | 0.0007 | 0.0008 | 0.0013 | 0.0012 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0005 | 0.0005 | 0.0006 | 0.0006 | 0.0005 | 0.0004 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0007 | 0.0011 | 0.0009 | 0.0029 | 0.0046 | 0.0021 | 0.0023 | 0.0040 | 0.0021 | 0.0010 | 0.0008 | 0.0006 |
| U | 0.0002 | 0.0000 | 0.0000 | 0.0000 | 0.0003 | 0.0012 | 0.0000 | 0.0015 | 0.0015 | 0.0004 | 0.0009 | 0.0012 |
| Sr | 0.0254 | 0.0220 | 0.0302 | 0.0645 | 0.0706 | 0.0393 | 0.0307 | 0.0613 | 0.0234 | 0.0209 | 0.0222 | 0.0183 |
| Y | 0.0019 | 0.0027 | 0.0025 | 0.0027 | 0.0022 | 0.0019 | 0.0028 | 0.0017 | 0.0024 | 0.0026 | 0.0021 | 0.0017 |
| Zr | 0.0080 | 0.0120 | 0.0085 | 0.0086 | 0.0085 | 0.0090 | 0.0113 | 0.0100 | 0.0115 | 0.0083 | 0.0077 | 0.0068 |
| Nb | 0.0004 | 0.0005 | 0.0005 | 0.0007 | 0.0007 | 0.0007 | 0.0008 | 0.0007 | 0.0008 | 0.0006 | 0.0005 | 0.0006 |
| Mo | 0.0054 | 0.0024 | 0.0023 | 0.0006 | 0.0006 | 0.0009 | 0.0005 | 0.0000 | 0.0012 | 0.0029 | 0.0018 | 0.0034 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| Sb | 0.0003 | 0.0000 | 0.0000 | 0.0003 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0002 | 0.0000 |

# Table A.5 Raw PHXRF data for No.12 NC_CT1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #12 | | | Quadrant 2 #12 | | | Quadrant 3 #12 | | | Quadrant 4 #12 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0787 | 0.0000 | 0.0554 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.4848 | 0.2815 | 0.2962 | 0.1863 | 0.2053 | 0.4582 | 0.8015 | 0.3285 | 0.5171 | 0.5035 | 0.9165 | 0.4509 |
| Si | 3.3894 | 3.8131 | 3.7818 | 3.5171 | 4.2074 | 4.3048 | 4.8332 | 4.5096 | 4.3839 | 4.8365 | 5.6793 | 4.1087 |
| P | 0.0101 | 0.0022 | 0.0054 | 0.0022 | 0.0000 | 0.0236 | 0.0281 | 0.0040 | 0.0132 | 0.0399 | 0.0122 | 0.0353 |
| S | 0.5223 | 0.5286 | 0.4981 | 0.5391 | 0.6083 | 0.5375 | 0.6571 | 0.6009 | 0.6074 | 0.5798 | 0.5928 | 0.5227 |
| K | 0.3426 | 0.2410 | 0.3547 | 0.1406 | 0.1446 | 0.3062 | 0.3589 | 0.1845 | 0.3242 | 0.3050 | 0.5311 | 0.3723 |
| Ca | 5.7478 | 6.6033 | 7.5313 | 5.7569 | 6.8046 | 8.4040 | 8.7185 | 7.5588 | 9.5803 | 7.8067 | 5.5341 | 7.9308 |
| Ba | 0.0952 | 0.0194 | 0.0000 | 0.0000 | 0.0000 | 0.0030 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.1312 | 0.1394 | 0.1240 | 0.0747 | 0.0629 | 0.1608 | 0.1385 | 0.0800 | 0.1268 | 0.0966 | 0.1067 | 0.0887 |
| V | 0.0000 | 0.0079 | 0.0006 | 0.0009 | 0.0039 | 0.0000 | 0.0000 | 0.0013 | 0.0013 | 0.0023 | 0.0027 | 0.0013 |
| Cr | 0.0035 | 0.0026 | 0.0035 | 0.0032 | 0.0057 | 0.0032 | 0.0041 | 0.0039 | 0.0044 | 0.0047 | 0.0052 | 0.0032 |
| Mn | 0.0273 | 0.0235 | 0.0230 | 0.0224 | 0.0288 | 0.0247 | 0.0228 | 0.0222 | 0.0225 | 0.0242 | 0.0274 | 0.0226 |
| Fe | 1.7538 | 1.3409 | 1.8245 | 1.0612 | 1.0707 | 2.0270 | 2.3293 | 1.1425 | 1.8425 | 1.2259 | 1.5880 | 1.1600 |
| Ba | 0.1801 | 0.8646 | 0.8259 | 0.3531 | 0.3920 | 0.1396 | 0.2501 | 0.0574 | 0.3748 | 0.0237 | 0.0031 | 0.1259 |
| Co | 0.0031 | 0.0108 | 0.0127 | 0.0063 | 0.0032 | 0.0041 | 0.0021 | 0.0017 | 0.0046 | 0.0009 | 0.0013 | 0.0004 |
| Ni | 0.0021 | 0.0012 | 0.0017 | 0.0019 | 0.0027 | 0.0024 | 0.0024 | 0.0034 | 0.0028 | 0.0026 | 0.0015 | 0.0036 |
| Cu | 0.0070 | 0.0042 | 0.0051 | 0.0051 | 0.0069 | 0.0057 | 0.0131 | 0.0148 | 0.0065 | 0.0084 | 0.0079 | 0.0151 |
| Zn | 0.0093 | 0.0083 | 0.0073 | 0.0060 | 0.0114 | 0.0093 | 0.0102 | 0.0166 | 0.0054 | 0.0088 | 0.0054 | 0.0179 |
| As | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0007 | 0.0006 | 0.0008 | 0.0010 | 0.0004 | 0.0006 | 0.0004 | 0.0011 |
| Pb | 0.0014 | 0.0010 | 0.0009 | 0.0004 | 0.0017 | 0.0008 | 0.0013 | 0.0013 | 0.0012 | 0.0015 | 0.0006 | 0.0014 |
| Th | 0.0003 | 0.0004 | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0005 | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0004 |
| Rb | 0.0012 | 0.0019 | 0.0009 | 0.0026 | 0.0013 | 0.0022 | 0.0031 | 0.0036 | 0.0015 | 0.0023 | 0.0030 | 0.0029 |
| U | 0.0003 | 0.0010 | 0.0000 | 0.0016 | 0.0015 | 0.0008 | 0.0010 | 0.0003 | 0.0006 | 0.0000 | 0.0011 | 0.0005 |
| Sr | 0.0577 | 0.0390 | 0.0578 | 0.0271 | 0.0223 | 0.0284 | 0.0298 | 0.0272 | 0.0257 | 0.0363 | 0.0319 | 0.0420 |
| Y | 0.0025 | 0.0020 | 0.0027 | 0.0024 | 0.0023 | 0.0025 | 0.0027 | 0.0031 | 0.0022 | 0.0029 | 0.0023 | 0.0029 |
| Zr | 0.0107 | 0.0077 | 0.0108 | 0.0126 | 0.0147 | 0.0194 | 0.0149 | 0.0164 | 0.0141 | 0.0114 | 0.0094 | 0.0164 |
| Nb | 0.0006 | 0.0006 | 0.0006 | 0.0008 | 0.0008 | 0.0007 | 0.0008 | 0.0009 | 0.0007 | 0.0007 | 0.0007 | 0.0007 |
| Mo | 0.0006 | 0.0013 | 0.0008 | 0.0022 | 0.0012 | 0.0014 | 0.0006 | 0.0000 | 0.0012 | 0.0001 | 0.0005 | 0.0000 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0001 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| Sb | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0003 | 0.0006 | 0.0002 | 0.0000 | 0.0000 | 0.0001 | 0.0000 |

Table A.6 Raw PHXRF data for No.50 NC_CT1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #50 | | | Quadrant 2 #50 | | | Quadrant 3 #50 | | | Quadrant 4 #50 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.4818 | 0.4794 | 0.4451 | 0.4356 | 0.4765 | 0.3783 | 0.5350 | 0.4475 | 0.5310 | 0.3424 | 0.4352 | 0.4606 |
| Si | 6.0842 | 6.7223 | 7.3205 | 6.6436 | 6.9147 | 6.8357 | 7.0088 | 7.0086 | 7.2932 | 6.5228 | 6.1937 | 6.2318 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0005 | 0.0000 | 0.0000 | 0.0034 | 0.0026 | 0.0000 | 0.0000 | 0.0048 | 0.0033 |
| S | 0.6518 | 0.6346 | 0.6489 | 0.5606 | 0.5695 | 0.5646 | 0.5471 | 0.5675 | 0.5533 | 0.5855 | 0.5876 | 0.5676 |
| K | 0.1914 | 0.1684 | 0.1651 | 0.2641 | 0.2693 | 0.2503 | 0.3819 | 0.2815 | 0.3145 | 0.2167 | 0.3213 | 0.2808 |
| Ca | 4.7791 | 4.3799 | 4.3943 | 5.9355 | 6.3006 | 5.9526 | 6.6650 | 6.4048 | 5.6249 | 6.1758 | 7.2644 | 6.6730 |
| Ba | 0.3543 | 0.3880 | 0.2848 | 0.0000 | 0.0000 | 0.0000 | 0.0841 | 0.0000 | 0.0480 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0741 | 0.0720 | 0.0817 | 0.1451 | 0.1008 | 0.1338 | 0.0669 | 0.0873 | 0.0799 |
| V | 0.0000 | 0.0000 | 0.0173 | 0.0037 | 0.0057 | 0.0061 | 0.0000 | 0.0093 | 0.0000 | 0.0079 | 0.0109 | 0.0055 |
| Cr | 0.0078 | 0.0077 | 0.0031 | 0.0046 | 0.0070 | 0.0057 | 0.0052 | 0.0065 | 0.0043 | 0.0097 | 0.0038 | 0.0063 |
| Mn | 0.0302 | 0.0271 | 0.0260 | 0.0273 | 0.0284 | 0.0263 | 0.0316 | 0.0245 | 0.0349 | 0.0275 | 0.0270 | 0.0266 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 1.0397 | 1.0809 | 0.9147 | 1.5176 | 1.0634 | 1.2529 | 0.9336 | 1.1563 | 0.9959 |
| Ba | 1.2016 | 0.7472 | 1.2319 | 0.2470 | 0.1645 | 0.2051 | 0.2980 | 0.0717 | 0.2494 | 0.1407 | 0.9063 | 0.9227 |
| Co | 0.0156 | 0.0093 | 0.0157 | 0.0026 | 0.0025 | 0.0024 | 0.0022 | 0.0015 | 0.0017 | 0.0010 | 0.0139 | 0.0094 |
| Ni | 0.0004 | 0.0002 | 0.0005 | 0.0018 | 0.0018 | 0.0023 | 0.0015 | 0.0014 | 0.0022 | 0.0020 | 0.0001 | 0.0000 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0063 | 0.0052 | 0.0062 | 0.0042 | 0.0059 | 0.0050 | 0.0055 | 0.0000 | 0.0000 |
| Zn | 0.0001 | 0.0010 | 0.0008 | 0.0057 | 0.0035 | 0.0058 | 0.0034 | 0.0029 | 0.0031 | 0.0035 | 0.0000 | 0.0003 |
| As | 0.0001 | 0.0001 | 0.0002 | 0.0004 | 0.0003 | 0.0004 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0001 | 0.0001 |
| Pb | 0.0013 | 0.0011 | 0.0008 | 0.0015 | 0.0010 | 0.0014 | 0.0010 | 0.0008 | 0.0014 | 0.0011 | 0.0012 | 0.0010 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0004 | 0.0004 | 0.0003 | 0.0002 |
| Rb | 0.0000 | 0.0000 | 0.0000 | 0.0010 | 0.0017 | 0.0021 | 0.0010 | 0.0014 | 0.0006 | 0.0009 | 0.0000 | 0.0000 |
| U | 0.0009 | 0.0000 | 0.0005 | 0.0000 | 0.0001 | 0.0007 | 0.0000 | 0.0000 | 0.0000 | 0.0005 | 0.0004 | 0.0000 |
| Sr | 0.0021 | 0.0028 | 0.0025 | 0.0178 | 0.0171 | 0.0174 | 0.0166 | 0.0175 | 0.0158 | 0.0150 | 0.0026 | 0.0024 |
| Y | 0.0008 | 0.0009 | 0.0008 | 0.0023 | 0.0026 | 0.0023 | 0.0026 | 0.0024 | 0.0027 | 0.0023 | 0.0008 | 0.0013 |
| Zr | 0.0048 | 0.0038 | 0.0047 | 0.0092 | 0.0090 | 0.0092 | 0.0093 | 0.0105 | 0.0092 | 0.0093 | 0.0037 | 0.0031 |
| Nb | 0.0005 | 0.0005 | 0.0005 | 0.0006 | 0.0006 | 0.0007 | 0.0006 | 0.0005 | 0.0006 | 0.0007 | 0.0005 | 0.0003 |
| Mo | 0.0161 | 0.0150 | 0.0155 | 0.0036 | 0.0039 | 0.0034 | 0.0038 | 0.0031 | 0.0037 | 0.0029 | 0.0139 | 0.0137 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0009 | 0.0006 | 0.0009 | 0.0009 | 0.0000 | 0.0003 | 0.0000 | 0.0000 | 0.0000 | 0.0002 | 0.0005 | 0.0015 |

Table A.7 Raw PHXRF data for No.4 NC_HW1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.8202 | 0.7575 | 0.9658 | 0.1065 | 0.2443 | 0.4421 | 0.7243 | 0.7013 | 0.4249 | 0.6093 | 0.7425 | 0.5302 |
| Si | 5.6594 | 4.9511 | 6.3012 | 2.3992 | 2.9296 | 3.8391 | 5.3484 | 5.3436 | 3.9659 | 4.6041 | 4.8579 | 4.3788 |
| P | 0.0000 | 0.0022 | 0.0117 | 0.0000 | 0.0000 | 0.0030 | 0.0297 | 0.0367 | 0.0135 | 0.0158 | 0.0207 | 0.0073 |
| S | 0.5398 | 0.5277 | 0.4890 | 0.4739 | 0.4445 | 0.4643 | 0.7488 | 0.7754 | 0.6808 | 0.5367 | 0.5086 | 0.5395 |
| K | 0.6030 | 0.5698 | 0.8534 | 0.4023 | 0.4344 | 0.5171 | 0.7657 | 0.7371 | 0.5740 | 0.5584 | 0.5578 | 0.3962 |
| Ca | 1.7252 | 2.0959 | 3.2817 | 3.0774 | 4.5815 | 3.4956 | 7.3925 | 7.2887 | 6.4515 | 4.7131 | 4.6561 | 4.9200 |
| Ba | 0.2833 | 0.2377 | 0.0417 | 0.0000 | 0.0000 | 0.0000 | 0.0130 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0392 | 0.0172 | 0.0397 | 0.0349 | 0.1129 | 0.1034 | 0.0785 | 0.0679 | 0.0795 | 0.0491 |
| V | 0.0036 | 0.0016 | 0.0033 | 0.0056 | 0.0038 | 0.0065 | 0.0000 | 0.0014 | 0.0009 | 0.0046 | 0.0024 | 0.0057 |
| Cr | 0.0062 | 0.0042 | 0.0052 | 0.0039 | 0.0033 | 0.0046 | 0.0051 | 0.0041 | 0.0030 | 0.0048 | 0.0035 | 0.0043 |
| Mn | 0.0272 | 0.0319 | 0.0263 | 0.0296 | 0.0218 | 0.0228 | 0.0231 | 0.0239 | 0.0213 | 0.0226 | 0.0246 | 0.0223 |
| Fe | 0.0000 | 0.0000 | 0.9128 | 0.5927 | 0.7501 | 0.7553 | 1.3622 | 1.3697 | 1.2143 | 1.4141 | 1.3462 | 1.3743 |
| Ba | 0.3109 | 0.3295 | 0.3408 | 0.4156 | 0.4659 | 0.4084 | 0.6255 | 0.6001 | 0.4261 | 0.9264 | 0.8286 | 0.5059 |
| Co | 0.0049 | 0.0050 | 0.0028 | 0.0039 | 0.0045 | 0.0048 | 0.0079 | 0.0093 | 0.0040 | 0.0078 | 0.0077 | 0.0055 |
| Ni | 0.0020 | 0.0006 | 0.0007 | 0.0023 | 0.0012 | 0.0019 | 0.0010 | 0.0011 | 0.0004 | 0.0011 | 0.0010 | 0.0013 |
| Cu | 0.0020 | 0.0000 | 0.0000 | 0.0009 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0016 | 0.0018 | 0.0006 |
| Zn | 0.0066 | 0.0044 | 0.0025 | 0.0044 | 0.0027 | 0.0016 | 0.0014 | 0.0031 | 0.0014 | 0.0024 | 0.0017 | 0.0001 |
| As | 0.0007 | 0.0003 | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 | 0.0004 | 0.0002 | 0.0002 | 0.0002 | 0.0001 |
| Pb | 0.0018 | 0.0009 | 0.0010 | 0.0013 | 0.0015 | 0.0013 | 0.0018 | 0.0024 | 0.0013 | 0.0014 | 0.0011 | 0.0009 |
| Th | 0.0008 | 0.0007 | 0.0006 | 0.0007 | 0.0006 | 0.0006 | 0.0006 | 0.0007 | 0.0006 | 0.0005 | 0.0005 | 0.0004 |
| Rb | 0.0074 | 0.0061 | 0.0048 | 0.0059 | 0.0035 | 0.0045 | 0.0000 | 0.0055 | 0.0044 | 0.0027 | 0.0024 | 0.0006 |
| U | 0.0000 | 0.0000 | 0.0003 | 0.0009 | 0.0000 | 0.0011 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sr | 0.0161 | 0.0072 | 0.0088 | 0.0221 | 0.0091 | 0.0136 | 0.0109 | 0.0223 | 0.0079 | 0.0242 | 0.0245 | 0.0201 |
| Y | 0.0057 | 0.0039 | 0.0029 | 0.0029 | 0.0032 | 0.0032 | 0.0005 | 0.0024 | 0.0037 | 0.0050 | 0.0051 | 0.0061 |
| Zr | 0.0247 | 0.0221 | 0.0185 | 0.0153 | 0.0096 | 0.0153 | 0.0065 | 0.0102 | 0.0078 | 0.0212 | 0.0269 | 0.0331 |
| Nb | 0.0013 | 0.0011 | 0.0009 | 0.0011 | 0.0009 | 0.0011 | 0.0007 | 0.0007 | 0.0007 | 0.0010 | 0.0010 | 0.0012 |
| Mo | 0.0019 | 0.0036 | 0.0038 | 0.0034 | 0.0084 | 0.0058 | 0.0113 | 0.0074 | 0.0082 | 0.0083 | 0.0083 | 0.0054 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0003 | 0.0004 | 0.0000 | 0.0004 | 0.0000 | 0.0005 | 0.0000 |

Table A.8 Raw PHXRF data for No.12 NC_HW1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #12 | | | Quadrant 2 #12 | | | Quadrant 3 #12 | | | Quadrant 4 #12 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.3948 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.8560 | 0.5989 | 0.6413 | 1.4518 | 1.5100 | 0.5766 | 0.7746 | 0.9091 | 0.6148 | 0.4918 | 0.5291 | 1.0475 |
| Si | 3.6077 | 3.2060 | 3.2779 | 5.8310 | 4.7980 | 4.3780 | 4.0433 | 4.0715 | 4.1113 | 3.9709 | 3.3641 | 5.5626 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0162 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| S | 0.5482 | 0.5356 | 0.4895 | 0.5149 | 0.4790 | 0.4712 | 0.4671 | 0.4649 | 0.5043 | 0.5090 | 0.5269 | 0.5034 |
| K | 0.4569 | 0.4303 | 0.4890 | 1.0025 | 0.7742 | 0.6645 | 0.6039 | 0.7115 | 0.6986 | 0.5428 | 0.5499 | 1.0747 |
| Ca | 3.7648 | 4.6001 | 5.0785 | 3.2713 | 2.9508 | 4.9314 | 6.5017 | 5.3591 | 4.8219 | 5.5750 | 5.2337 | 4.3580 |
| Ba | 0.5058 | 0.3381 | 0.0184 | 0.0396 | 0.0210 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0668 | 0.1119 | 0.0782 | 0.0535 | 0.0841 | 0.0845 | 0.0659 | 0.0678 | 0.0591 | 0.1025 |
| V | 0.0000 | 0.0000 | 0.0016 | 0.0065 | 0.0098 | 0.0013 | 0.0078 | 0.0029 | 0.0057 | 0.0075 | 0.0049 | 0.0020 |
| Cr | 0.0050 | 0.0047 | 0.0049 | 0.0082 | 0.0061 | 0.0051 | 0.0043 | 0.0055 | 0.0056 | 0.0043 | 0.0052 | 0.0062 |
| Mn | 0.0234 | 0.0249 | 0.0214 | 0.0276 | 0.0276 | 0.0225 | 0.0223 | 0.0229 | 0.0233 | 0.0239 | 0.0221 | 0.0246 |
| Fe | 0.0000 | 0.0000 | 1.0417 | 1.1449 | 0.7028 | 1.1110 | 1.3108 | 1.4571 | 0.9953 | 0.9878 | 1.0112 | 1.3700 |
| Ba | 0.0000 | 0.2053 | 0.2363 | 0.2588 | 0.0911 | 0.0319 | 0.0393 | 0.0000 | 0.0991 | 0.3096 | 0.0183 | 0.3853 |
| Co | 0.0008 | 0.0009 | 0.0008 | 0.0006 | 0.0005 | 0.0002 | 0.0011 | 0.0012 | 0.0009 | 0.0007 | 0.0006 | 0.0011 |
| Ni | 0.0034 | 0.0024 | 0.0028 | 0.0020 | 0.0012 | 0.0022 | 0.0023 | 0.0026 | 0.0027 | 0.0016 | 0.0015 | 0.0011 |
| Cu | 0.0048 | 0.0044 | 0.0061 | 0.0028 | 0.0042 | 0.0048 | 0.0036 | 0.0060 | 0.0048 | 0.0038 | 0.0038 | 0.0024 |
| Zn | 0.0056 | 0.0057 | 0.0044 | 0.0073 | 0.0048 | 0.0107 | 0.0051 | 0.0082 | 0.0063 | 0.0049 | 0.0063 | 0.0046 |
| As | 0.0005 | 0.0005 | 0.0004 | 0.0005 | 0.0004 | 0.0006 | 0.0005 | 0.0007 | 0.0005 | 0.0005 | 0.0004 | 0.0003 |
| Pb | 0.0011 | 0.0008 | 0.0012 | 0.0010 | 0.0008 | 0.0011 | 0.0019 | 0.0026 | 0.0006 | 0.0012 | 0.0007 | 0.0010 |
| Th | 0.0010 | 0.0010 | 0.0009 | 0.0010 | 0.0010 | 0.0010 | 0.0010 | 0.0011 | 0.0011 | 0.0012 | 0.0009 | 0.0010 |
| Rb | 0.0095 | 0.0101 | 0.0103 | 0.0085 | 0.0083 | 0.0097 | 0.0101 | 0.0109 | 0.0122 | 0.0133 | 0.0083 | 0.0114 |
| U | 0.0003 | 0.0000 | 0.0016 | 0.0000 | 0.0000 | 0.0000 | 0.0011 | 0.0001 | 0.0011 | 0.0014 | 0.0004 | 0.0000 |
| Sr | 0.0444 | 0.0393 | 0.0468 | 0.0359 | 0.0375 | 0.0320 | 0.0481 | 0.0416 | 0.0466 | 0.0409 | 0.0241 | 0.0331 |
| Y | 0.0015 | 0.0020 | 0.0017 | 0.0031 | 0.0032 | 0.0027 | 0.0016 | 0.0027 | 0.0012 | 0.0020 | 0.0027 | 0.0036 |
| Zr | 0.0156 | 0.0192 | 0.0203 | 0.0202 | 0.0206 | 0.0197 | 0.0182 | 0.0208 | 0.0202 | 0.0183 | 0.0173 | 0.0154 |
| Nb | 0.0010 | 0.0011 | 0.0011 | 0.0012 | 0.0013 | 0.0012 | 0.0012 | 0.0012 | 0.0012 | 0.0013 | 0.0012 | 0.0013 |
| Mo | 0.0003 | 0.0003 | 0.0000 | 0.0004 | 0.0007 | 0.0003 | 0.0007 | 0.0001 | 0.0000 | 0.0001 | 0.0012 | 0.0015 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0001 | 0.0002 | 0.0001 | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0002 |
| Sb | 0.0007 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0002 | 0.0000 | 0.0000 | 0.0000 | 0.0004 |

Table A.9 Raw PHXRF data for No.50 NC_HW1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #50 | | | Quadrant 2 #50 | | | Quadrant 3 #50 | | | Quadrant 4 #50 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.7543 | 0.8178 | 0.7400 | 1.1178 | 1.0736 | 0.9996 | 1.1781 | 1.0416 | 1.1298 | 0.9112 | 0.8516 | 0.9215 |
| Si | 4.4521 | 4.0874 | 3.9420 | 5.7087 | 4.6944 | 4.7703 | 5.5529 | 4.9997 | 5.9747 | 4.7389 | 4.7422 | 4.6946 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0064 | 0.0001 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| S | 0.5166 | 0.4773 | 0.5180 | 0.4773 | 0.4670 | 0.4719 | 0.5120 | 0.5283 | 0.5035 | 0.5161 | 0.5170 | 0.5148 |
| K | 0.6863 | 0.7635 | 0.6767 | 0.9271 | 0.8638 | 0.9155 | 1.1496 | 1.1740 | 1.3796 | 0.9783 | 0.9442 | 0.9731 |
| Ca | 4.8586 | 6.6942 | 4.4045 | 5.3374 | 4.8653 | 5.2052 | 4.8070 | 5.6185 | 5.1369 | 4.8208 | 4.9600 | 4.8299 |
| Ba | 0.0000 | 0.0000 | 0.0000 | 0.0089 | 0.0139 | 0.0099 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0891 | 0.0828 | 0.0666 | 0.1031 | 0.0748 | 0.0827 | 0.1088 | 0.0891 | 0.0864 | 0.1094 | 0.0781 | 0.0888 |
| V | 0.0050 | 0.0034 | 0.0057 | 0.0033 | 0.0046 | 0.0119 | 0.0059 | 0.0044 | 0.0150 | 0.0071 | 0.0073 | 0.0012 |
| Cr | 0.0064 | 0.0058 | 0.0048 | 0.0056 | 0.0053 | 0.0040 | 0.0051 | 0.0053 | 0.0033 | 0.0056 | 0.0052 | 0.0057 |
| Mn | 0.0237 | 0.0229 | 0.0232 | 0.0233 | 0.0240 | 0.0251 | 0.0246 | 0.0247 | 0.0254 | 0.0247 | 0.0230 | 0.0228 |
| Fe | 1.4878 | 1.3221 | 1.1182 | 1.5569 | 0.9255 | 1.1269 | 1.4307 | 1.4564 | 1.1926 | 1.4984 | 1.2625 | 1.2524 |
| Ba | 0.2746 | 0.2420 | 0.1837 | 0.1770 | 0.3999 | 1.1188 | 1.0496 | 1.2646 | 1.0144 | 0.2796 | 0.2614 | 0.3996 |
| Co | 0.0008 | 0.0027 | 0.0038 | 0.0044 | 0.0096 | 0.0166 | 0.0129 | 0.0170 | 0.0135 | 0.0031 | 0.0028 | 0.0024 |
| Ni | 0.0022 | 0.0015 | 0.0019 | 0.0017 | 0.0009 | 0.0000 | 0.0001 | 0.0009 | 0.0003 | 0.0014 | 0.0020 | 0.0017 |
| Cu | 0.0079 | 0.0082 | 0.0033 | 0.0020 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0027 | 0.0020 | 0.0020 |
| Zn | 0.0047 | 0.0045 | 0.0041 | 0.0034 | 0.0022 | 0.0005 | 0.0000 | 0.0009 | 0.0000 | 0.0034 | 0.0034 | 0.0040 |
| As | 0.0005 | 0.0028 | 0.0004 | 0.0004 | 0.0002 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0004 | 0.0003 | 0.0004 |
| Pb | 0.0011 | 0.0020 | 0.0012 | 0.0009 | 0.0015 | 0.0018 | 0.0014 | 0.0015 | 0.0016 | 0.0015 | 0.0019 | 0.0011 |
| Th | 0.0010 | 0.0011 | 0.0010 | 0.0011 | 0.0008 | 0.0006 | 0.0004 | 0.0005 | 0.0005 | 0.0010 | 0.0009 | 0.0010 |
| Rb | 0.0096 | 0.0109 | 0.0078 | 0.0123 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0108 | 0.0092 | 0.0112 |
| U | 0.0000 | 0.0009 | 0.0000 | 0.0040 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sr | 0.0421 | 0.0344 | 0.0382 | 0.0349 | 0.0126 | 0.0050 | 0.0035 | 0.0057 | 0.0051 | 0.0368 | 0.0314 | 0.0386 |
| Y | 0.0017 | 0.0018 | 0.0012 | 0.0001 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0026 | 0.0024 | 0.0024 |
| Zr | 0.0174 | 0.0136 | 0.0173 | 0.0165 | 0.0085 | 0.0055 | 0.0043 | 0.0054 | 0.0053 | 0.0137 | 0.0130 | 0.0140 |
| Nb | 0.0012 | 0.0012 | 0.0012 | 0.0015 | 0.0012 | 0.0008 | 0.0006 | 0.0007 | 0.0008 | 0.0010 | 0.0009 | 0.0010 |
| Mo | 0.0005 | 0.0018 | 0.0018 | 0.0015 | 0.0130 | 0.0142 | 0.0137 | 0.0147 | 0.0141 | 0.0030 | 0.0026 | 0.0019 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0003 | 0.0001 | 0.0012 | 0.0004 | 0.0011 | 0.0002 | 0.0009 | 0.0001 | 0.0006 | 0.0003 |

Table A.10 Raw PHXRF data for No.4 NC_CT2 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.5209 | 0.5036 | 0.4790 | 0.4834 | 0.4842 | 0.4716 | 0.3966 | 0.3951 | 0.4478 | 0.4071 | 0.3953 | 0.5279 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6941 | 0.6768 | 0.6319 | 0.3011 | 0.3572 | 0.2541 | 0.2681 | 0.2904 | 0.2588 | 0.2991 | 0.2902 | 0.4565 |
| Si | 1.9938 | 1.8787 | 2.0044 | 1.8516 | 1.9028 | 1.8237 | 1.7616 | 1.8083 | 1.7591 | 1.7011 | 1.7519 | 1.7868 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0008 | 0.0119 | 0.0233 | 0.0129 | 0.0270 | 0.0295 | 0.0188 | 0.0514 |
| S | 1.6604 | 1.8271 | 1.4765 | 1.4640 | 1.7064 | 1.2321 | 1.3217 | 1.5702 | 1.2874 | 1.6351 | 2.1067 | 2.2061 |
| K | 2.5057 | 2.6055 | 2.3982 | 2.6477 | 3.2013 | 2.2713 | 2.4336 | 3.1380 | 2.3994 | 2.6455 | 3.0321 | 2.9914 |
| Ca | 14.724 | 13.786 | 14.784 | 19.308 | 20.069 | 16.967 | 14.825 | 17.661 | 14.660 | 14.533 | 17.216 | 13.381 |
| Ba | 0.2589 | 0.2194 | 0.2603 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0480 | 0.0518 | 0.0509 | 0.0437 | 0.0507 | 0.0454 | 0.0432 | 0.0529 | 0.0389 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0025 | 0.0027 | 0.0031 | 0.0037 | 0.0032 | 0.0035 | 0.0044 | 0.0038 | 0.0043 |
| Cr | 0.0038 | 0.0040 | 0.0039 | 0.0034 | 0.0036 | 0.0036 | 0.0033 | 0.0034 | 0.0036 | 0.0033 | 0.0036 | 0.0036 |
| Mn | 0.0225 | 0.0226 | 0.0225 | 0.0204 | 0.0204 | 0.0205 | 0.0208 | 0.0204 | 0.0209 | 0.0206 | 0.0205 | 0.0210 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.8425 | 0.9148 | 0.7384 | 0.6519 | 0.9832 | 0.6849 | 0.7270 | 1.0980 | 0.6720 |
| Ba | 0.1685 | 0.0556 | 0.1967 | 0.1511 | 0.1848 | 0.1380 | 0.1675 | 0.1175 | 0.2085 | 0.9344 | 0.4725 | 0.2975 |
| Co | 0.0012 | 0.0013 | 0.0015 | 0.0013 | 0.0011 | 0.0033 | 0.0023 | 0.0042 | 0.0029 | 0.0018 | 0.0022 | 0.0037 |
| Ni | 0.0020 | 0.0019 | 0.0015 | 0.0018 | 0.0024 | 0.0019 | 0.0027 | 0.0019 | 0.0022 | 0.0027 | 0.0021 | 0.0021 |
| Cu | 0.0021 | 0.0017 | 0.0007 | 0.0017 | 0.0012 | 0.0005 | 0.0025 | 0.0000 | 0.0003 | 0.0174 | 0.0173 | 0.0000 |
| Zn | 0.0035 | 0.0029 | 0.0010 | 0.0029 | 0.0024 | 0.0021 | 0.0038 | 0.0010 | 0.0023 | 0.0061 | 0.0072 | 0.0014 |
| As | 0.0002 | 0.0003 | 0.0002 | 0.0003 | 0.0002 | 0.0003 | 0.0003 | 0.0002 | 0.0002 | 0.0005 | 0.0005 | 0.0002 |
| Pb | 0.0015 | 0.0009 | 0.0009 | 0.0007 | 0.0011 | 0.0013 | 0.0007 | 0.0008 | 0.0011 | 0.0006 | 0.0012 | 0.0013 |
| Th | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0003 |
| Rb | 0.0004 | 0.0011 | 0.0004 | 0.0008 | 0.0003 | 0.0013 | 0.0007 | 0.0000 | 0.0005 | 0.0000 | 0.0006 | 0.0002 |
| U | 0.0000 | 0.0011 | 0.0000 | 0.0005 | 0.0002 | 0.0009 | 0.0006 | 0.0000 | 0.0010 | 0.0000 | 0.0010 | 0.0004 |
| Sr | 0.0473 | 0.0471 | 0.0580 | 0.0414 | 0.0503 | 0.0356 | 0.0401 | 0.0341 | 0.0402 | 0.0301 | 0.0290 | 0.0323 |
| Y | 0.0018 | 0.0018 | 0.0019 | 0.0019 | 0.0017 | 0.0024 | 0.0017 | 0.0022 | 0.0016 | 0.0020 | 0.0017 | 0.0018 |
| Zr | 0.0100 | 0.0102 | 0.0093 | 0.0089 | 0.0079 | 0.0156 | 0.0092 | 0.0099 | 0.0067 | 0.0094 | 0.0087 | 0.0111 |
| Nb | 0.0002 | 0.0003 | 0.0003 | 0.0004 | 0.0004 | 0.0005 | 0.0005 | 0.0006 | 0.0005 | 0.0005 | 0.0004 | 0.0005 |
| Mo | 0.0012 | 0.0010 | 0.0004 | 0.0004 | 0.0001 | 0.0017 | 0.0004 | 0.0020 | 0.0006 | 0.0021 | 0.0024 | 0.0037 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 |
| Sb | 0.0002 | 0.0003 | 0.0007 | 0.0000 | 0.0002 | 0.0006 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0004 | 0.0000 |

Table A.11 Raw PHXRF data for No.12 NC_CT2 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #12 | | | Quadrant 2 #12 | | | Quadrant 3 #12 | | | Quadrant 4 #12 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.4543 | 0.1490 | 0.1230 | 0.0267 | 0.0708 | 0.0817 | 0.0654 | 0.0400 | 0.1221 | 0.1414 | 0.0764 | 0.1218 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 2.8748 | 2.7908 | 2.8669 | 2.9524 | 3.1256 | 3.1276 | 3.1191 | 3.0336 | 3.1134 | 3.0714 | 2.8873 | 2.8872 |
| Si | 6.3117 | 6.5043 | 6.7604 | 7.0053 | 7.4128 | 7.3068 | 7.3832 | 7.4506 | 7.3582 | 7.2987 | 6.9716 | 6.8433 |
| P | 0.0291 | 0.0289 | 0.0216 | 0.0215 | 0.0263 | 0.0237 | 0.0263 | 0.0231 | 0.0203 | 0.0202 | 0.0256 | 0.0252 |
| S | 0.7222 | 0.7238 | 0.7625 | 0.7802 | 0.7987 | 0.8093 | 0.8035 | 0.8142 | 0.8206 | 0.8030 | 0.7652 | 0.7597 |
| K | 1.1399 | 1.1904 | 1.2479 | 1.2799 | 1.3588 | 1.3339 | 1.3473 | 1.3680 | 1.3531 | 1.3355 | 1.2642 | 1.2453 |
| Ca | 0.9058 | 0.9423 | 0.9891 | 1.0034 | 1.0673 | 1.0932 | 1.0749 | 1.1056 | 1.0747 | 1.0685 | 1.0216 | 1.0039 |
| Ba | 0.4159 | 0.3631 | 0.2780 | 0.1917 | 0.1224 | 0.1302 | 0.1649 | 0.1101 | 0.1666 | 0.1696 | 0.2679 | 0.2943 |
| Ti | 0.4277 | 0.4694 | 0.5175 | 0.5580 | 0.6104 | 0.6048 | 0.5922 | 0.6166 | 0.5923 | 0.5869 | 0.5262 | 0.5153 |
| V | 0.0076 | 0.0097 | 0.0155 | 0.0163 | 0.0193 | 0.0213 | 0.0184 | 0.0205 | 0.0195 | 0.0183 | 0.0155 | 0.0126 |
| Cr | 0.0126 | 0.0123 | 0.0119 | 0.0119 | 0.0122 | 0.0114 | 0.0117 | 0.0119 | 0.0120 | 0.0119 | 0.0122 | 0.0121 |
| Mn | 0.0110 | 0.0107 | 0.0098 | 0.0082 | 0.0066 | 0.0072 | 0.0081 | 0.0071 | 0.0077 | 0.0068 | 0.0083 | 0.0087 |
| Fe | 1.5430 | 1.8380 | 2.2446 | 2.8499 | 3.3199 | 3.2597 | 3.0688 | 3.2057 | 3.0086 | 3.1389 | 2.6453 | 2.4610 |
| Ba | 0.6812 | 0.1600 | 0.2302 | 0.2277 | 0.4261 | 0.3135 | 1.0928 | 0.0807 | 0.1278 | 0.3063 | 0.1236 | 0.1333 |
| Co | 0.0051 | 0.0041 | 0.0034 | 0.0033 | 0.0029 | 0.0032 | 0.0022 | 0.0021 | 0.0022 | 0.0022 | 0.0021 | 0.0025 |
| Ni | 0.0029 | 0.0013 | 0.0013 | 0.0037 | 0.0031 | 0.0020 | 0.0040 | 0.0022 | 0.0022 | 0.0032 | 0.0027 | 0.0022 |
| Cu | 0.0041 | 0.0030 | 0.0048 | 0.0048 | 0.0031 | 0.0038 | 0.0036 | 0.0111 | 0.0070 | 0.0025 | 0.0043 | 0.0068 |
| Zn | 0.0023 | 0.0000 | 0.0002 | 0.0010 | 0.0000 | 0.0009 | 0.0000 | 0.0015 | 0.0005 | 0.0001 | 0.0002 | 0.0008 |
| As | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0002 |
| Pb | 0.0011 | 0.0007 | 0.0011 | 0.0014 | 0.0003 | 0.0016 | 0.0006 | 0.0012 | 0.0010 | 0.0009 | 0.0013 | 0.0011 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0003 | 0.0004 | 0.0003 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0005 | 0.0015 | 0.0005 | 0.0003 | 0.0000 | 0.0005 | 0.0007 | 0.0009 | 0.0003 | 0.0000 | 0.0000 | 0.0000 |
| U | 0.0006 | 0.0011 | 0.0009 | 0.0001 | 0.0003 | 0.0008 | 0.0010 | 0.0005 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sr | 0.0281 | 0.0263 | 0.0337 | 0.0272 | 0.0298 | 0.0304 | 0.0363 | 0.0513 | 0.0466 | 0.0304 | 0.0520 | 0.0380 |
| Y | 0.0019 | 0.0017 | 0.0014 | 0.0016 | 0.0020 | 0.0018 | 0.0022 | 0.0024 | 0.0024 | 0.0023 | 0.0023 | 0.0021 |
| Zr | 0.0082 | 0.0126 | 0.0107 | 0.0200 | 0.0233 | 0.0189 | 0.0097 | 0.0138 | 0.0138 | 0.0087 | 0.0154 | 0.0084 |
| Nb | 0.0005 | 0.0004 | 0.0005 | 0.0004 | 0.0005 | 0.0004 | 0.0007 | 0.0007 | 0.0006 | 0.0004 | 0.0005 | 0.0006 |
| Mo | 0.0052 | 0.0053 | 0.0040 | 0.0039 | 0.0012 | 0.0022 | 0.0019 | 0.0000 | 0.0000 | 0.0016 | 0.0000 | 0.0011 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0005 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0001 | 0.0000 | 0.0000 | 0.0000 |

Table A.12 Raw PHXRF data for No.50 NC_CT2 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #50 | | | Quadrant 2 #50 | | | Quadrant 3 #50 | | | Quadrant 4 #50 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.6959 | 0.6872 | 0.6838 | 0.6828 | 0.6838 | 0.6482 | 0.6675 | 0.6750 | 0.6773 | 0.6892 | 0.6882 | 0.6913 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6245 | 0.5494 | 0.5207 | 0.0727 | 0.0644 | 0.1877 | 0.0000 | 0.0494 | 0.0109 | 0.0052 | 0.0000 | 0.0000 |
| Si | 1.8760 | 1.9000 | 1.8598 | 1.6898 | 1.6777 | 1.6660 | 1.6956 | 1.7001 | 1.7265 | 1.7510 | 1.7683 | 1.8256 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0201 | 0.0182 | 0.0230 | 0.0223 | 0.0276 | 0.0469 | 0.0215 | 0.0989 | 0.0840 |
| S | 3.0985 | 2.7817 | 2.7606 | 2.4093 | 2.2609 | 2.6642 | 1.9592 | 2.0050 | 1.9712 | 1.9550 | 1.6033 | 1.8469 |
| K | 2.1508 | 1.8968 | 1.8759 | 1.7791 | 1.6148 | 2.3147 | 1.3978 | 1.4793 | 1.5111 | 1.4418 | 1.1193 | 1.2508 |
| Ca | 21.252 | 21.358 | 20.976 | 26.876 | 26.824 | 25.768 | 25.331 | 25.385 | 25.608 | 27.434 | 25.092 | 27.164 |
| Ba | 0.0042 | 0.0179 | 0.0216 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0180 | 0.0227 | 0.0195 | 0.0221 | 0.0219 | 0.0244 | 0.0271 | 0.0242 | 0.0265 |
| V | 0.0028 | 0.0022 | 0.0019 | 0.0037 | 0.0041 | 0.0042 | 0.0041 | 0.0037 | 0.0044 | 0.0040 | 0.0037 | 0.0038 |
| Cr | 0.0015 | 0.0015 | 0.0015 | 0.0012 | 0.0010 | 0.0015 | 0.0011 | 0.0012 | 0.0010 | 0.0012 | 0.0010 | 0.0010 |
| Mn | 0.0226 | 0.0225 | 0.0225 | 0.0224 | 0.0223 | 0.0224 | 0.0224 | 0.0223 | 0.0223 | 0.0225 | 0.0220 | 0.0219 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.4976 | 0.5432 | 0.5208 | 0.6024 | 0.6160 | 0.6414 | 0.7587 | 0.6205 | 0.7502 |
| Ba | 0.0646 | 0.1631 | 0.2330 | 0.2547 | 0.2539 | 0.2387 | 0.1864 | 0.0115 | 0.0000 | 0.1918 | 0.2527 | 0.1389 |
| Co | 0.0019 | 0.0020 | 0.0026 | 0.0032 | 0.0043 | 0.0070 | 0.0042 | 0.0012 | 0.0012 | 0.0012 | 0.0019 | 0.0025 |
| Ni | 0.0024 | 0.0009 | 0.0019 | 0.0018 | 0.0009 | 0.0016 | 0.0020 | 0.0019 | 0.0023 | 0.0022 | 0.0026 | 0.0015 |
| Cu | 0.0023 | 0.0001 | 0.0014 | 0.0011 | 0.0000 | 0.0000 | 0.0005 | 0.0020 | 0.0030 | 0.0010 | 0.0014 | 0.0005 |
| Zn | 0.0092 | 0.0011 | 0.0087 | 0.0014 | 0.0002 | 0.0000 | 0.0011 | 0.0028 | 0.0011 | 0.0015 | 0.0019 | 0.0012 |
| As | 0.0005 | 0.0001 | 0.0005 | 0.0002 | 0.0001 | 0.0001 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Pb | 0.0011 | 0.0013 | 0.0019 | 0.0009 | 0.0011 | 0.0009 | 0.0008 | 0.0009 | 0.0009 | 0.0009 | 0.0015 | 0.0010 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0004 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0004 |
| Rb | 0.0012 | 0.0018 | 0.0005 | 0.0006 | 0.0004 | 0.0000 | 0.0000 | 0.0010 | 0.0000 | 0.0009 | 0.0000 | 0.0000 |
| U | 0.0006 | 0.0009 | 0.0001 | 0.0011 | 0.0017 | 0.0010 | 0.0000 | 0.0004 | 0.0000 | 0.0002 | 0.0000 | 0.0000 |
| Sr | 0.0330 | 0.0279 | 0.0306 | 0.0305 | 0.0146 | 0.0169 | 0.0304 | 0.0331 | 0.0364 | 0.0320 | 0.0316 | 0.0307 |
| Y | 0.0020 | 0.0016 | 0.0019 | 0.0017 | 0.0011 | 0.0008 | 0.0019 | 0.0020 | 0.0019 | 0.0020 | 0.0021 | 0.0018 |
| Zr | 0.0099 | 0.0075 | 0.0060 | 0.0100 | 0.0112 | 0.0067 | 0.0094 | 0.0121 | 0.0121 | 0.0073 | 0.0102 | 0.0096 |
| Nb | 0.0005 | 0.0004 | 0.0006 | 0.0006 | 0.0005 | 0.0005 | 0.0007 | 0.0005 | 0.0006 | 0.0005 | 0.0006 | 0.0006 |
| Mo | 0.0020 | 0.0030 | 0.0021 | 0.0018 | 0.0102 | 0.0094 | 0.0026 | 0.0009 | 0.0006 | 0.0013 | 0.0012 | 0.0017 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0001 | 0.0005 | 0.0000 | 0.0000 | 0.0004 | 0.0014 | 0.0000 | 0.0008 | 0.0000 | 0.0004 | 0.0000 | 0.0000 |

Table A.13 Raw PHXRF data for Aggregate only NC_AP1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.2422 | 0.2578 | 0.2692 | 0.2392 | 0.2709 | 0.3772 | 0.2775 | 0.3371 | 0.3324 | 0.2747 | 0.2750 | 0.2403 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 1.6833 | 1.8697 | 1.6585 | 1.6205 | 1.6677 | 1.6410 | 1.6447 | 1.6875 | 1.8440 | 1.9298 | 1.8995 | 1.8594 |
| Si | 7.3887 | 7.6976 | 7.3464 | 7.2684 | 7.3268 | 6.8362 | 7.2749 | 7.1404 | 8.2606 | 8.5368 | 8.6342 | 8.5618 |
| P | 0.0702 | 0.0719 | 0.0787 | 0.0821 | 0.0895 | 0.0992 | 0.0891 | 0.1039 | 0.1097 | 0.1207 | 0.1139 | 0.1199 |
| S | 0.5105 | 0.5188 | 0.4837 | 0.4563 | 0.4497 | 0.4279 | 0.4583 | 0.4447 | 0.4550 | 0.4502 | 0.4595 | 0.4597 |
| K | 0.5011 | 0.5440 | 0.5724 | 0.6214 | 0.6071 | 0.5462 | 0.5840 | 0.5806 | 0.6240 | 0.6452 | 0.6091 | 0.6238 |
| Ca | 4.1126 | 4.3109 | 5.2745 | 5.3632 | 5.3920 | 4.9640 | 5.2242 | 5.1350 | 5.7911 | 5.6787 | 5.4288 | 5.3895 |
| Ba | 2.9886 | 3.1154 | 0.6291 | 0.5167 | 0.5008 | 0.4500 | 0.4325 | 0.4276 | 0.2400 | 0.2179 | 0.1814 | 0.1840 |
| Ti | 0.0000 | 0.0000 | 0.3417 | 0.3534 | 0.3546 | 0.3152 | 0.3817 | 0.3765 | 0.4875 | 0.4609 | 0.4686 | 0.4584 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0015 | 0.0014 | 0.0010 |
| Cr | 0.0075 | 0.0074 | 0.0039 | 0.0040 | 0.0045 | 0.0052 | 0.0041 | 0.0045 | 0.0040 | 0.0045 | 0.0046 | 0.0042 |
| Mn | 0.0413 | 0.0427 | 0.0464 | 0.0419 | 0.0428 | 0.0411 | 0.0447 | 0.0450 | 0.0455 | 0.0415 | 0.0443 | 0.0424 |
| Fe | 0.0000 | 0.0000 | 2.0233 | 2.4090 | 2.5326 | 2.1801 | 2.7820 | 2.5907 | 3.7464 | 3.8399 | 3.8929 | 3.7285 |
| Ba | 2.8805 | 2.5985 | 1.8068 | 2.0527 | 1.0000 | 0.7703 | 1.0704 | 0.5536 | 0.5716 | 0.6059 | 0.5000 | 0.7182 |
| Co | 0.0625 | 0.0537 | 0.0458 | 0.0484 | 0.0201 | 0.0189 | 0.0281 | 0.0148 | 0.0145 | 0.0175 | 0.0135 | 0.0173 |
| Ni | 0.0000 | 0.0000 | 0.0000 | 0.0004 | 0.0042 | 0.0047 | 0.0044 | 0.0051 | 0.0050 | 0.0049 | 0.0047 | 0.0049 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0048 | 0.0078 | 0.0076 | 0.0110 | 0.0027 | 0.0062 | 0.0049 | 0.0029 |
| Zn | 0.0013 | 0.0010 | 0.0014 | 0.0017 | 0.0061 | 0.0057 | 0.0044 | 0.0061 | 0.0053 | 0.0064 | 0.0060 | 0.0054 |
| As | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0004 | 0.0004 | 0.0003 | 0.0005 | 0.0004 | 0.0004 | 0.0004 | 0.0003 |
| Pb | 0.0006 | 0.0007 | 0.0008 | 0.0010 | 0.0009 | 0.0007 | 0.0010 | 0.0004 | 0.0004 | 0.0018 | 0.0007 | 0.0013 |
| Th | 0.0003 | 0.0002 | 0.0002 | 0.0003 | 0.0003 | 0.0004 | 0.0003 | 0.0004 | 0.0003 | 0.0003 | 0.0003 | 0.0004 |
| Rb | 0.0000 | 0.0008 | 0.0000 | 0.0000 | 0.0007 | 0.0010 | 0.0006 | 0.0018 | 0.0014 | 0.0018 | 0.0005 | 0.0010 |
| U | 0.0034 | 0.0045 | 0.0044 | 0.0032 | 0.0000 | 0.0000 | 0.0001 | 0.0003 | 0.0002 | 0.0010 | 0.0000 | 0.0007 |
| Sr | 0.0043 | 0.0035 | 0.0049 | 0.0047 | 0.0280 | 0.0287 | 0.0209 | 0.0303 | 0.0257 | 0.0222 | 0.0310 | 0.0279 |
| Y | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0025 | 0.0024 | 0.0022 | 0.0025 | 0.0025 | 0.0019 | 0.0026 | 0.0021 |
| Zr | 0.0040 | 0.0032 | 0.0040 | 0.0044 | 0.0091 | 0.0103 | 0.0067 | 0.0122 | 0.0060 | 0.0071 | 0.0065 | 0.0066 |
| Nb | 0.0006 | 0.0005 | 0.0007 | 0.0006 | 0.0005 | 0.0006 | 0.0005 | 0.0006 | 0.0005 | 0.0006 | 0.0006 | 0.0007 |
| Mo | 0.0132 | 0.0123 | 0.0129 | 0.0137 | 0.0028 | 0.0025 | 0.0051 | 0.0020 | 0.0024 | 0.0025 | 0.0015 | 0.0023 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0008 | 0.0010 | 0.0009 | 0.0004 | 0.0000 | 0.0000 | 0.0003 | 0.0001 | 0.0005 | 0.0000 | 0.0000 | 0.0001 |

Table A.14 Raw PHXRF data for Aggregate only NC_CT1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0717 | 0.0463 | 0.0928 | 0.1456 | 0.0545 | 0.0984 | 0.2330 | 0.1863 | 0.1884 | 0.1812 | 0.2236 | 0.1272 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.9011 | 0.9020 | 0.9514 | 0.9413 | 0.8729 | 0.8772 | 0.9222 | 1.0558 | 0.9892 | 0.9957 | 1.0377 | 0.9723 |
| Si | 7.7621 | 7.3768 | 7.8565 | 7.8666 | 7.9222 | 7.9421 | 7.8097 | 8.2931 | 7.5868 | 8.0328 | 8.2593 | 8.4129 |
| P | 0.0258 | 0.0303 | 0.0289 | 0.0209 | 0.0192 | 0.0191 | 0.0393 | 0.0330 | 0.0303 | 0.0314 | 0.0259 | 0.0340 |
| S | 0.4851 | 0.4731 | 0.4884 | 0.4981 | 0.5000 | 0.5039 | 0.4720 | 0.4865 | 0.4725 | 0.4748 | 0.5042 | 0.4795 |
| K | 0.3083 | 0.3069 | 0.3230 | 0.3236 | 0.2838 | 0.3009 | 0.3291 | 0.3395 | 0.3190 | 0.3528 | 0.3491 | 0.3482 |
| Ca | 4.1120 | 3.8979 | 4.2216 | 4.3377 | 4.4531 | 4.3028 | 5.0984 | 5.1665 | 4.8155 | 4.5823 | 4.5881 | 4.4733 |
| Ba | 0.0314 | 0.0323 | 0.0063 | 0.0185 | 0.0127 | 0.0240 | 0.0517 | 0.0141 | 0.0780 | 0.0523 | 0.0518 | 0.0514 |
| Ti | 0.2461 | 0.2334 | 0.2685 | 0.2487 | 0.2495 | 0.2417 | 0.2318 | 0.2561 | 0.2146 | 0.2386 | 0.2540 | 0.2532 |
| V | 0.0077 | 0.0074 | 0.0073 | 0.0101 | 0.0087 | 0.0097 | 0.0088 | 0.0084 | 0.0060 | 0.0080 | 0.0086 | 0.0108 |
| Cr | 0.0077 | 0.0085 | 0.0083 | 0.0084 | 0.0073 | 0.0075 | 0.0084 | 0.0080 | 0.0080 | 0.0081 | 0.0081 | 0.0072 |
| Mn | 0.0431 | 0.0421 | 0.0439 | 0.0455 | 0.0418 | 0.0429 | 0.0450 | 0.0472 | 0.0469 | 0.0428 | 0.0441 | 0.0450 |
| Fe | 4.7158 | 4.3362 | 5.0115 | 4.9048 | 4.5402 | 4.6245 | 4.6951 | 5.1270 | 4.1917 | 4.5358 | 4.5900 | 4.6817 |
| Ba | 0.6102 | 0.2697 | 0.1153 | 0.1394 | 0.1246 | 0.2198 | 0.1657 | 0.3487 | 0.3089 | 0.0875 | 0.1757 | 0.1347 |
| Co | 0.0122 | 0.0067 | 0.0062 | 0.0044 | 0.0029 | 0.0043 | 0.0041 | 0.0081 | 0.0122 | 0.0019 | 0.0046 | 0.0027 |
| Ni | 0.0050 | 0.0059 | 0.0064 | 0.0059 | 0.0063 | 0.0062 | 0.0070 | 0.0062 | 0.0064 | 0.0060 | 0.0061 | 0.0073 |
| Cu | 0.0035 | 0.0049 | 0.0049 | 0.0061 | 0.0101 | 0.0088 | 0.0144 | 0.0124 | 0.0059 | 0.0077 | 0.0086 | 0.0063 |
| Zn | 0.0061 | 0.0080 | 0.0072 | 0.0079 | 0.0076 | 0.0075 | 0.0065 | 0.0063 | 0.0071 | 0.0070 | 0.0078 | 0.0072 |
| As | 0.0004 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0004 |
| Pb | 0.0011 | 0.0009 | 0.0010 | 0.0018 | 0.0007 | 0.0008 | 0.0008 | 0.0012 | 0.0011 | 0.0009 | 0.0016 | 0.0012 |
| Th | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0004 | 0.0004 | 0.0003 | 0.0004 | 0.0003 | 0.0003 |
| Rb | 0.0022 | 0.0018 | 0.0015 | 0.0018 | 0.0016 | 0.0011 | 0.0017 | 0.0014 | 0.0009 | 0.0010 | 0.0020 | 0.0011 |
| U | 0.0005 | 0.0001 | 0.0000 | 0.0004 | 0.0006 | 0.0000 | 0.0006 | 0.0005 | 0.0000 | 0.0000 | 0.0016 | 0.0000 |
| Sr | 0.0309 | 0.0328 | 0.0332 | 0.0326 | 0.0309 | 0.0317 | 0.0314 | 0.0290 | 0.0295 | 0.0359 | 0.0261 | 0.0272 |
| Y | 0.0023 | 0.0028 | 0.0025 | 0.0024 | 0.0026 | 0.0026 | 0.0026 | 0.0027 | 0.0026 | 0.0032 | 0.0025 | 0.0026 |
| Zr | 0.0056 | 0.0063 | 0.0066 | 0.0066 | 0.0116 | 0.0102 | 0.0124 | 0.0113 | 0.0070 | 0.0066 | 0.0084 | 0.0078 |
| Nb | 0.0005 | 0.0006 | 0.0006 | 0.0006 | 0.0007 | 0.0006 | 0.0007 | 0.0007 | 0.0006 | 0.0007 | 0.0006 | 0.0005 |
| Mo | 0.0019 | 0.0008 | 0.0006 | 0.0006 | 0.0004 | 0.0008 | 0.0001 | 0.0009 | 0.0018 | 0.0007 | 0.0008 | 0.0006 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0000 | 0.0004 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0003 | 0.0001 | 0.0000 | 0.0006 |

Table A.15 Raw PHXRF data for Aggregate only NC_HW1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 1.7420 | 2.0233 | 1.7647 | 1.7541 | 1.7850 | 1.6999 | 1.5626 | 1.6265 | 1.5769 | 1.6034 | 1.4781 | 1.5590 |
| Si | 11.460 | 12.094 | 10.990 | 12.474 | 11.977 | 13.057 | 11.683 | 11.526 | 11.676 | 11.709 | 11.357 | 11.307 |
| P | 0.0123 | 0.0184 | 0.0154 | 0.0271 | 0.0308 | 0.0248 | 0.0188 | 0.0175 | 0.0248 | 0.0098 | 0.0167 | 0.0174 |
| S | 0.5835 | 0.5743 | 0.5637 | 0.5311 | 0.5161 | 0.5471 | 0.5382 | 0.5393 | 0.5267 | 0.5578 | 0.5367 | 0.5282 |
| K | 1.6546 | 1.6932 | 1.6860 | 2.1070 | 1.8247 | 2.0145 | 2.0043 | 1.9898 | 2.0176 | 1.9249 | 1.8375 | 1.7975 |
| Ca | 0.7454 | 0.8110 | 0.7432 | 1.0372 | 1.1515 | 1.2824 | 1.1402 | 1.2036 | 1.1823 | 1.2104 | 1.1031 | 1.0873 |
| Ba | 0.4692 | 0.4559 | 0.4716 | 0.0763 | 0.0759 | 0.0520 | 0.0268 | 0.0464 | 0.0158 | 0.0007 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0340 | 0.0508 | 0.0552 | 0.0630 | 0.0595 | 0.0754 | 0.0674 | 0.0599 | 0.0573 |
| V | 0.0061 | 0.0057 | 0.0019 | 0.0111 | 0.0102 | 0.0110 | 0.0122 | 0.0122 | 0.0127 | 0.0139 | 0.0131 | 0.0128 |
| Cr | 0.0095 | 0.0103 | 0.0097 | 0.0092 | 0.0088 | 0.0094 | 0.0076 | 0.0077 | 0.0079 | 0.0086 | 0.0081 | 0.0081 |
| Mn | 0.0382 | 0.0413 | 0.0397 | 0.0411 | 0.0430 | 0.0402 | 0.0438 | 0.0446 | 0.0458 | 0.0428 | 0.0432 | 0.0434 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.6093 | 0.7192 | 0.9127 | 1.0795 | 1.0465 | 1.1644 | 1.1890 | 0.9652 | 0.9558 |
| Ba | 0.2990 | 0.3734 | 0.3650 | 0.2889 | 0.3037 | 0.3627 | 0.3666 | 0.2628 | 0.2843 | 0.2815 | 0.2568 | 0.2242 |
| Co | 0.0062 | 0.0060 | 0.0075 | 0.0073 | 0.0062 | 0.0053 | 0.0054 | 0.0050 | 0.0037 | 0.0040 | 0.0044 | 0.0050 |
| Ni | 0.0009 | 0.0010 | 0.0004 | 0.0005 | 0.0012 | 0.0007 | 0.0007 | 0.0006 | 0.0004 | 0.0011 | 0.0009 | 0.0006 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Zn | 0.0029 | 0.0031 | 0.0018 | 0.0061 | 0.0047 | 0.0028 | 0.0051 | 0.0051 | 0.0050 | 0.0060 | 0.0039 | 0.0049 |
| As | 0.0002 | 0.0002 | 0.0002 | 0.0004 | 0.0003 | 0.0002 | 0.0003 | 0.0003 | 0.0003 | 0.0004 | 0.0003 | 0.0003 |
| Pb | 0.0010 | 0.0016 | 0.0011 | 0.0011 | 0.0014 | 0.0012 | 0.0016 | 0.0013 | 0.0011 | 0.0018 | 0.0010 | 0.0016 |
| Th | 0.0008 | 0.0008 | 0.0006 | 0.0008 | 0.0009 | 0.0008 | 0.0008 | 0.0009 | 0.0009 | 0.0009 | 0.0008 | 0.0008 |
| Rb | 0.0060 | 0.0081 | 0.0000 | 0.0088 | 0.0088 | 0.0080 | 0.0079 | 0.0089 | 0.0104 | 0.0094 | 0.0080 | 0.0074 |
| U | 0.0000 | 0.0000 | 0.0000 | 0.0016 | 0.0007 | 0.0019 | 0.0004 | 0.0010 | 0.0009 | 0.0013 | 0.0020 | 0.0023 |
| Sr | 0.0097 | 0.0100 | 0.0048 | 0.0114 | 0.0128 | 0.0115 | 0.0128 | 0.0126 | 0.0121 | 0.0128 | 0.0113 | 0.0116 |
| Y | 0.0039 | 0.0045 | 0.0018 | 0.0033 | 0.0040 | 0.0034 | 0.0040 | 0.0038 | 0.0044 | 0.0042 | 0.0030 | 0.0029 |
| Zr | 0.0140 | 0.0149 | 0.0073 | 0.0197 | 0.0212 | 0.0177 | 0.0213 | 0.0200 | 0.0153 | 0.0201 | 0.0192 | 0.0178 |
| Nb | 0.0011 | 0.0012 | 0.0007 | 0.0012 | 0.0014 | 0.0013 | 0.0013 | 0.0013 | 0.0013 | 0.0014 | 0.0013 | 0.0013 |
| Mo | 0.0070 | 0.0060 | 0.0133 | 0.0046 | 0.0043 | 0.0040 | 0.0037 | 0.0039 | 0.0034 | 0.0030 | 0.0034 | 0.0037 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0001 | 0.0002 | 0.0007 | 0.0000 | 0.0000 | 0.0000 | 0.0003 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |

Table A.16 Raw PHXRF data for Aggregate only NC_CT2 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.6063 | 0.6207 | 0.6198 | 0.6358 | 0.6276 | 0.6165 | 0.6296 | 0.6369 | 0.6447 | 0.6471 | 0.6434 | 0.6173 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Si | 2.8884 | 2.8265 | 2.9138 | 2.8238 | 2.8359 | 2.9216 | 2.7023 | 2.5082 | 2.4873 | 2.6839 | 2.8677 | 2.8292 |
| P | 0.0087 | 0.0193 | 0.0223 | 0.0338 | 0.0393 | 0.0319 | 0.0125 | 0.0181 | 0.0134 | 0.0397 | 0.0513 | 0.0375 |
| S | 0.1520 | 0.1477 | 0.1446 | 0.1240 | 0.1204 | 0.1182 | 0.1373 | 0.1186 | 0.1255 | 0.1192 | 0.1036 | 0.1259 |
| K | 0.1120 | 0.1155 | 0.1129 | 0.1569 | 0.1572 | 0.1273 | 0.1160 | 0.1246 | 0.1202 | 0.1284 | 0.1267 | 0.1219 |
| Ca | 22.067 | 22.282 | 22.424 | 22.719 | 22.800 | 22.897 | 22.816 | 22.990 | 22.652 | 23.371 | 22.736 | 22.459 |
| Ba | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0264 | 0.0264 | 0.0277 | 0.0330 | 0.0295 | 0.0233 | 0.0271 | 0.0400 | 0.0373 | 0.0242 | 0.0293 | 0.0310 |
| V | 0.0051 | 0.0056 | 0.0062 | 0.0056 | 0.0062 | 0.0066 | 0.0061 | 0.0055 | 0.0052 | 0.0059 | 0.0060 | 0.0059 |
| Cr | 0.0021 | 0.0021 | 0.0026 | 0.0022 | 0.0022 | 0.0024 | 0.0021 | 0.0015 | 0.0017 | 0.0021 | 0.0022 | 0.0017 |
| Mn | 0.0228 | 0.0230 | 0.0226 | 0.0230 | 0.0229 | 0.0229 | 0.0228 | 0.0230 | 0.0226 | 0.0227 | 0.0228 | 0.0229 |
| Fe | 0.3758 | 0.3706 | 0.3230 | 0.3366 | 0.3629 | 0.3589 | 0.3669 | 0.3426 | 0.3407 | 0.3623 | 0.3288 | 0.3407 |
| Ba | 0.2181 | 0.3926 | 0.3681 | 0.3908 | 0.1986 | 0.4577 | 0.3326 | 0.3049 | 0.2330 | 0.1500 | 0.0874 | 0.1611 |
| Co | 0.0027 | 0.0030 | 0.0034 | 0.0032 | 0.0032 | 0.0030 | 0.0033 | 0.0028 | 0.0019 | 0.0018 | 0.0015 | 0.0014 |
| Ni | 0.0021 | 0.0010 | 0.0017 | 0.0020 | 0.0024 | 0.0012 | 0.0014 | 0.0025 | 0.0031 | 0.0020 | 0.0027 | 0.0025 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Zn | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0001 | 0.0000 |
| As | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0000 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| Pb | 0.0011 | 0.0013 | 0.0010 | 0.0008 | 0.0013 | 0.0009 | 0.0006 | 0.0011 | 0.0010 | 0.0008 | 0.0011 | 0.0016 |
| Th | 0.0003 | 0.0002 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0010 | 0.0012 | 0.0009 | 0.0004 | 0.0003 | 0.0007 | 0.0003 | 0.0004 | 0.0001 | 0.0004 | 0.0006 | 0.0009 |
| U | 0.0006 | 0.0003 | 0.0001 | 0.0000 | 0.0000 | 0.0006 | 0.0000 | 0.0002 | 0.0000 | 0.0000 | 0.0002 | 0.0000 |
| Sr | 0.0264 | 0.0213 | 0.0235 | 0.0253 | 0.0251 | 0.0247 | 0.0286 | 0.0292 | 0.0331 | 0.0324 | 0.0296 | 0.0322 |
| Y | 0.0015 | 0.0017 | 0.0019 | 0.0015 | 0.0018 | 0.0015 | 0.0018 | 0.0016 | 0.0022 | 0.0020 | 0.0021 | 0.0017 |
| Zr | 0.0060 | 0.0057 | 0.0066 | 0.0088 | 0.0108 | 0.0056 | 0.0074 | 0.0079 | 0.0085 | 0.0108 | 0.0149 | 0.0123 |
| Nb | 0.0004 | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0005 | 0.0006 | 0.0005 |
| Mo | 0.0024 | 0.0060 | 0.0056 | 0.0055 | 0.0056 | 0.0049 | 0.0042 | 0.0037 | 0.0012 | 0.0006 | 0.0019 | 0.0004 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0009 | 0.0013 | 0.0007 | 0.0004 | 0.0009 | 0.0001 | 0.0004 | 0.0002 | 0.0003 | 0.0006 | 0.0001 | 0.0008 |

Table A.17 Raw PHXRF data for Mortar only NC_AP1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0358 | 0.3467 | 0.0811 | 0.0592 | 0.0462 | 0.1265 | 0.0000 | 0.0074 | 0.0000 | 0.0182 | 0.0181 | 0.0000 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.5531 | 0.7460 | 0.5905 | 0.5897 | 0.5310 | 0.5809 | 0.5099 | 0.5471 | 0.5217 | 0.5470 | 0.5356 | 0.5112 |
| Si | 8.6546 | 8.1964 | 9.5913 | 9.2700 | 9.5113 | 9.3735 | 8.7927 | 9.0673 | 9.0768 | 8.8573 | 8.8719 | 9.0726 |
| P | 0.0344 | 0.0521 | 0.0223 | 0.0297 | 0.0301 | 0.0346 | 0.0197 | 0.0148 | 0.0118 | 0.0130 | 0.0147 | 0.0157 |
| S | 0.6133 | 0.5666 | 0.6584 | 0.6492 | 0.6437 | 0.6412 | 0.6383 | 0.6425 | 0.6484 | 0.6412 | 0.6408 | 0.6423 |
| K | 0.1924 | 0.1839 | 0.2013 | 0.1881 | 0.1845 | 0.1928 | 0.1788 | 0.1829 | 0.1798 | 0.1896 | 0.1825 | 0.1799 |
| Ca | 8.0329 | 7.2924 | 8.8823 | 8.3239 | 8.4698 | 8.4071 | 7.8885 | 8.1212 | 8.1649 | 8.3348 | 8.3284 | 8.2000 |
| Ba | 0.0137 | 0.0307 | 0.0171 | 0.0215 | 0.0000 | 0.0290 | 0.0012 | 0.0000 | 0.0000 | 0.0073 | 0.0000 | 0.0037 |
| Ti | 0.1186 | 0.0945 | 0.1382 | 0.1203 | 0.1282 | 0.1254 | 0.1133 | 0.1212 | 0.1255 | 0.1250 | 0.1339 | 0.1246 |
| V | 0.0066 | 0.0068 | 0.0060 | 0.0065 | 0.0074 | 0.0072 | 0.0070 | 0.0067 | 0.0068 | 0.0065 | 0.0062 | 0.0063 |
| Cr | 0.0061 | 0.0064 | 0.0061 | 0.0061 | 0.0056 | 0.0062 | 0.0061 | 0.0060 | 0.0059 | 0.0059 | 0.0059 | 0.0061 |
| Mn | 0.0262 | 0.0263 | 0.0254 | 0.0264 | 0.0257 | 0.0260 | 0.0266 | 0.0262 | 0.0259 | 0.0265 | 0.0259 | 0.0261 |
| Fe | 1.0529 | 0.7871 | 1.3656 | 1.2095 | 1.3103 | 1.2377 | 1.1232 | 1.2401 | 1.2655 | 1.3308 | 1.3001 | 1.2359 |
| Ba | 0.4737 | 0.4146 | 0.4302 | 1.3913 | 1.0083 | 0.9951 | 1.1680 | 1.4329 | 0.3400 | 0.2688 | 0.1909 | 0.2453 |
| Co | 0.0088 | 0.0052 | 0.0091 | 0.0169 | 0.0138 | 0.0141 | 0.0148 | 0.0153 | 0.0040 | 0.0041 | 0.0039 | 0.0037 |
| Ni | 0.0020 | 0.0023 | 0.0016 | 0.0002 | 0.0007 | 0.0013 | 0.0004 | 0.0004 | 0.0027 | 0.0025 | 0.0025 | 0.0021 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0002 | 0.0012 | 0.0009 | 0.0014 |
| Zn | 0.0017 | 0.0028 | 0.0010 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0006 | 0.0008 | 0.0009 | 0.0011 |
| As | 0.0002 | 0.0002 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0000 | 0.0001 | 0.0002 | 0.0001 | 0.0002 |
| Pb | 0.0013 | 0.0007 | 0.0011 | 0.0016 | 0.0014 | 0.0013 | 0.0006 | 0.0012 | 0.0010 | 0.0012 | 0.0017 | 0.0014 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0003 | 0.0003 | 0.0002 | 0.0003 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0001 | 0.0000 | 0.0001 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0004 | 0.0003 | 0.0000 | 0.0006 |
| U | 0.0000 | 0.0000 | 0.0030 | 0.0047 | 0.0069 | 0.0055 | 0.0056 | 0.0047 | 0.0000 | 0.0000 | 0.0000 | 0.0002 |
| Sr | 0.0317 | 0.0405 | 0.0205 | 0.0046 | 0.0080 | 0.0082 | 0.0061 | 0.0047 | 0.0435 | 0.0455 | 0.0442 | 0.0453 |
| Y | 0.0020 | 0.0017 | 0.0002 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0021 | 0.0025 | 0.0026 | 0.0021 |
| Zr | 0.0082 | 0.0098 | 0.0070 | 0.0045 | 0.0052 | 0.0052 | 0.0049 | 0.0048 | 0.0125 | 0.0087 | 0.0095 | 0.0090 |
| Nb | 0.0005 | 0.0005 | 0.0006 | 0.0006 | 0.0007 | 0.0007 | 0.0008 | 0.0006 | 0.0005 | 0.0006 | 0.0006 | 0.0006 |
| Mo | 0.0062 | 0.0031 | 0.0101 | 0.0129 | 0.0128 | 0.0132 | 0.0138 | 0.0136 | 0.0030 | 0.0028 | 0.0026 | 0.0027 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0014 | 0.0011 | 0.0015 | 0.0015 | 0.0017 | 0.0011 | 0.0013 | 0.0017 | 0.0007 | 0.0011 | 0.0007 | 0.0008 |

Table A.18 Raw PHXRF data for Mortar only NC_CT1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0892 | 0.0275 | 0.1153 | 0.2845 | 0.1197 | 0.4576 | 0.1104 | 0.1903 | 0.1026 | 0.1385 | 0.1387 | 0.1826 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6913 | 0.7237 | 0.5360 | 0.6218 | 0.4724 | 0.7296 | 0.4903 | 0.5502 | 0.4728 | 0.5421 | 0.4863 | 0.5123 |
| Si | 9.1083 | 9.0889 | 8.3542 | 7.9433 | 8.4636 | 8.0032 | 9.0195 | 8.2071 | 8.6497 | 8.8737 | 9.1014 | 8.8149 |
| P | 0.0000 | 0.0000 | 0.0315 | 0.0508 | 0.0292 | 0.0586 | 0.0222 | 0.0483 | 0.0349 | 0.0490 | 0.0492 | 0.0461 |
| S | 0.7295 | 0.7398 | 0.6082 | 0.5624 | 0.6014 | 0.5508 | 0.6204 | 0.5568 | 0.5840 | 0.5823 | 0.6019 | 0.5869 |
| K | 0.2548 | 0.2600 | 0.2524 | 0.2600 | 0.2674 | 0.2590 | 0.2768 | 0.2613 | 0.2686 | 0.2729 | 0.2749 | 0.2665 |
| Ca | 7.6624 | 7.6536 | 8.8702 | 8.2923 | 8.9391 | 8.1564 | 9.2135 | 8.2440 | 8.8702 | 9.0205 | 9.3418 | 8.9681 |
| Ba | 0.6745 | 0.6846 | 0.1205 | 0.1057 | 0.1064 | 0.0758 | 0.0438 | 0.0448 | 0.0417 | 0.0240 | 0.0289 | 0.0163 |
| Ti | 0.0000 | 0.0000 | 0.0843 | 0.0742 | 0.0872 | 0.0816 | 0.1107 | 0.0925 | 0.1055 | 0.0998 | 0.1151 | 0.1045 |
| V | 0.0000 | 0.0000 | 0.0027 | 0.0032 | 0.0028 | 0.0043 | 0.0054 | 0.0055 | 0.0051 | 0.0060 | 0.0047 | 0.0056 |
| Cr | 0.0070 | 0.0066 | 0.0062 | 0.0061 | 0.0056 | 0.0063 | 0.0060 | 0.0061 | 0.0062 | 0.0062 | 0.0060 | 0.0063 |
| Mn | 0.0275 | 0.0274 | 0.0258 | 0.0258 | 0.0255 | 0.0255 | 0.0254 | 0.0259 | 0.0256 | 0.0259 | 0.0253 | 0.0254 |
| Fe | 0.0000 | 0.0000 | 0.5694 | 0.5024 | 0.6128 | 0.5618 | 0.9577 | 0.7067 | 0.9274 | 0.9292 | 1.0555 | 0.9484 |
| Ba | 0.2422 | 0.0000 | 0.2128 | 0.2563 | 0.3513 | 0.0911 | 0.1965 | 0.1818 | 0.1450 | 0.2186 | 0.1716 | 0.3234 |
| Co | 0.0032 | 0.0035 | 0.0033 | 0.0027 | 0.0048 | 0.0023 | 0.0017 | 0.0046 | 0.0023 | 0.0027 | 0.0037 | 0.0037 |
| Ni | 0.0014 | 0.0022 | 0.0016 | 0.0016 | 0.0016 | 0.0020 | 0.0024 | 0.0017 | 0.0017 | 0.0016 | 0.0017 | 0.0018 |
| Cu | 0.0031 | 0.0029 | 0.0026 | 0.0024 | 0.0019 | 0.0023 | 0.0024 | 0.0003 | 0.0025 | 0.0022 | 0.0019 | 0.0006 |
| Zn | 0.0061 | 0.0052 | 0.0051 | 0.0056 | 0.0051 | 0.0056 | 0.0065 | 0.0041 | 0.0056 | 0.0060 | 0.0054 | 0.0048 |
| As | 0.0005 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0005 | 0.0003 | 0.0004 | 0.0004 | 0.0004 | 0.0004 |
| Pb | 0.0018 | 0.0011 | 0.0019 | 0.0013 | 0.0010 | 0.0014 | 0.0012 | 0.0013 | 0.0014 | 0.0012 | 0.0009 | 0.0009 |
| Th | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0003 | 0.0003 | 0.0003 |
| Rb | 0.0014 | 0.0014 | 0.0014 | 0.0016 | 0.0017 | 0.0009 | 0.0016 | 0.0020 | 0.0014 | 0.0017 | 0.0013 | 0.0012 |
| U | 0.0000 | 0.0005 | 0.0004 | 0.0001 | 0.0012 | 0.0000 | 0.0001 | 0.0010 | 0.0005 | 0.0007 | 0.0009 | 0.0003 |
| Sr | 0.0270 | 0.0264 | 0.0273 | 0.0278 | 0.0253 | 0.0282 | 0.0287 | 0.0241 | 0.0275 | 0.0271 | 0.0263 | 0.0252 |
| Y | 0.0025 | 0.0027 | 0.0022 | 0.0026 | 0.0024 | 0.0026 | 0.0023 | 0.0020 | 0.0028 | 0.0022 | 0.0022 | 0.0026 |
| Zr | 0.0144 | 0.0125 | 0.0148 | 0.0148 | 0.0142 | 0.0152 | 0.0137 | 0.0119 | 0.0134 | 0.0130 | 0.0147 | 0.0138 |
| Nb | 0.0006 | 0.0007 | 0.0006 | 0.0007 | 0.0007 | 0.0006 | 0.0006 | 0.0006 | 0.0006 | 0.0006 | 0.0006 | 0.0006 |
| Mo | 0.0026 | 0.0026 | 0.0021 | 0.0024 | 0.0030 | 0.0014 | 0.0016 | 0.0026 | 0.0015 | 0.0018 | 0.0021 | 0.0019 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0017 | 0.0009 | 0.0009 | 0.0009 | 0.0012 | 0.0011 | 0.0013 | 0.0005 | 0.0010 | 0.0006 | 0.0012 | 0.0013 |

Table A.19 Raw PHXRF data for Mortar only NC_HW1 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.0586 | 0.0872 | 0.0490 | 0.1680 | 0.1407 | 0.1269 | 0.1075 | 0.0945 | 0.1205 | 0.1062 | 0.0666 | 0.0924 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 1.0620 | 1.0396 | 1.0807 | 1.0889 | 1.0734 | 1.1244 | 1.0947 | 1.1039 | 1.0814 | 1.1326 | 1.1583 | 1.1162 |
| Si | 7.6980 | 7.4904 | 7.9956 | 7.4066 | 7.6603 | 8.1659 | 8.2212 | 8.1663 | 7.8908 | 8.0544 | 8.2878 | 8.2025 |
| P | 0.0319 | 0.0444 | 0.0255 | 0.0453 | 0.0367 | 0.0324 | 0.0354 | 0.0234 | 0.0333 | 0.0348 | 0.0328 | 0.0286 |
| S | 0.5095 | 0.4848 | 0.5180 | 0.4775 | 0.4916 | 0.5051 | 0.5058 | 0.5187 | 0.5033 | 0.5129 | 0.5129 | 0.5153 |
| K | 1.3776 | 1.3912 | 1.4132 | 1.3346 | 1.3654 | 1.4222 | 1.4262 | 1.3924 | 1.3543 | 1.4049 | 1.4321 | 1.4355 |
| Ca | 9.4235 | 9.1294 | 9.7126 | 8.7023 | 9.0655 | 9.7672 | 9.6389 | 9.8116 | 9.4663 | 9.6034 | 9.8766 | 9.9435 |
| Ba | 0.0351 | 0.0320 | 0.0129 | 0.0275 | 0.0281 | 0.0226 | 0.0347 | 0.0192 | 0.0140 | 0.0174 | 0.0295 | 0.0226 |
| Ti | 0.1047 | 0.0936 | 0.1072 | 0.0834 | 0.0881 | 0.0984 | 0.0932 | 0.1004 | 0.0934 | 0.0995 | 0.1009 | 0.1012 |
| V | 0.0062 | 0.0060 | 0.0058 | 0.0074 | 0.0073 | 0.0064 | 0.0067 | 0.0074 | 0.0084 | 0.0067 | 0.0067 | 0.0066 |
| Cr | 0.0060 | 0.0065 | 0.0061 | 0.0068 | 0.0064 | 0.0063 | 0.0060 | 0.0060 | 0.0062 | 0.0066 | 0.0065 | 0.0065 |
| Mn | 0.0251 | 0.0252 | 0.0248 | 0.0246 | 0.0247 | 0.0239 | 0.0245 | 0.0250 | 0.0251 | 0.0249 | 0.0249 | 0.0246 |
| Fe | 1.3008 | 1.2053 | 1.4351 | 1.0486 | 1.2643 | 1.4257 | 1.4591 | 1.5519 | 1.3774 | 1.3911 | 1.5467 | 1.5319 |
| Ba | 0.1120 | 0.2651 | 0.3557 | 0.2327 | 0.2348 | 0.3303 | 0.2598 | 0.2994 | 0.2349 | 0.3054 | 0.3245 | 0.2845 |
| Co | 0.0035 | 0.0053 | 0.0097 | 0.0056 | 0.0057 | 0.0060 | 0.0024 | 0.0030 | 0.0050 | 0.0047 | 0.0045 | 0.0035 |
| Ni | 0.0018 | 0.0063 | 0.0005 | 0.0015 | 0.0011 | 0.0012 | 0.0023 | 0.0017 | 0.0010 | 0.0021 | 0.0025 | 0.0013 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0007 | 0.0000 | 0.0004 | 0.0005 | 0.0000 | 0.0009 | 0.0006 | 0.0000 |
| Zn | 0.0038 | 0.0029 | 0.0022 | 0.0044 | 0.0051 | 0.0031 | 0.0056 | 0.0045 | 0.0036 | 0.0047 | 0.0042 | 0.0053 |
| As | 0.0004 | 0.0003 | 0.0002 | 0.0003 | 0.0004 | 0.0003 | 0.0006 | 0.0005 | 0.0004 | 0.0005 | 0.0004 | 0.0004 |
| Pb | 0.0006 | 0.0017 | 0.0013 | 0.0014 | 0.0013 | 0.0013 | 0.0012 | 0.0016 | 0.0011 | 0.0014 | 0.0013 | 0.0010 |
| Th | 0.0010 | 0.0009 | 0.0007 | 0.0009 | 0.0010 | 0.0009 | 0.0011 | 0.0011 | 0.0010 | 0.0011 | 0.0011 | 0.0010 |
| Rb | 0.0105 | 0.0080 | 0.0036 | 0.0098 | 0.0104 | 0.0092 | 0.0112 | 0.0113 | 0.0105 | 0.0105 | 0.0106 | 0.0109 |
| U | 0.0021 | 0.0052 | 0.0000 | 0.0022 | 0.0032 | 0.0039 | 0.0011 | 0.0020 | 0.0025 | 0.0022 | 0.0024 | 0.0024 |
| Sr | 0.0437 | 0.0367 | 0.0257 | 0.0399 | 0.0408 | 0.0378 | 0.0468 | 0.0467 | 0.0431 | 0.0447 | 0.0452 | 0.0450 |
| Y | 0.0005 | 0.0000 | 0.0015 | 0.0007 | 0.0005 | 0.0000 | 0.0013 | 0.0012 | 0.0003 | 0.0007 | 0.0005 | 0.0008 |
| Zr | 0.0159 | 0.0154 | 0.0115 | 0.0158 | 0.0161 | 0.0152 | 0.0164 | 0.0176 | 0.0160 | 0.0176 | 0.0175 | 0.0176 |
| Nb | 0.0011 | 0.0014 | 0.0008 | 0.0011 | 0.0012 | 0.0011 | 0.0011 | 0.0013 | 0.0012 | 0.0013 | 0.0013 | 0.0012 |
| Mo | 0.0008 | 0.0029 | 0.0082 | 0.0016 | 0.0014 | 0.0024 | 0.0001 | 0.0008 | 0.0013 | 0.0006 | 0.0008 | 0.0007 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0012 | 0.0011 | 0.0018 | 0.0010 | 0.0009 | 0.0011 | 0.0012 | 0.0009 | 0.0012 | 0.0012 | 0.0013 | 0.0007 |

Table A.20 Raw PHXRF data for Mortar only NC_CT2 RCA sample

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.5642 | 0.5816 | 0.5408 | 0.5841 | 0.5630 | 0.5938 | 0.5943 | 0.6332 | 0.6247 | 0.5818 | 0.5984 | 0.6066 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Si | 3.3378 | 3.4460 | 3.4147 | 3.3396 | 3.4857 | 3.2969 | 3.2658 | 3.2201 | 3.1964 | 3.2536 | 3.4050 | 3.3912 |
| P | 0.0138 | 0.0047 | 0.0032 | 0.0276 | 0.0315 | 0.0088 | 0.0098 | 0.0227 | 0.0293 | 0.0102 | 0.0185 | 0.0265 |
| S | 0.2745 | 0.2853 | 0.2783 | 0.2807 | 0.2646 | 0.2903 | 0.2758 | 0.2679 | 0.2591 | 0.2759 | 0.2779 | 0.2676 |
| K | 0.1401 | 0.1488 | 0.1479 | 0.1501 | 0.1471 | 0.1477 | 0.1468 | 0.1482 | 0.1486 | 0.1345 | 0.1513 | 0.1331 |
| Ca | 21.403 | 21.106 | 21.373 | 21.087 | 20.872 | 20.880 | 21.344 | 22.068 | 21.811 | 21.373 | 21.418 | 21.284 |
| Ba | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0518 | 0.0431 | 0.0442 | 0.0425 | 0.0392 | 0.0389 | 0.0465 | 0.0550 | 0.0448 | 0.0412 | 0.0474 | 0.0415 |
| V | 0.0050 | 0.0056 | 0.0050 | 0.0056 | 0.0057 | 0.0056 | 0.0045 | 0.0045 | 0.0052 | 0.0058 | 0.0053 | 0.0055 |
| Cr | 0.0028 | 0.0026 | 0.0026 | 0.0039 | 0.0032 | 0.0037 | 0.0028 | 0.0025 | 0.0027 | 0.0028 | 0.0029 | 0.0030 |
| Mn | 0.0217 | 0.0212 | 0.0218 | 0.0220 | 0.0218 | 0.0215 | 0.0217 | 0.0213 | 0.0214 | 0.0212 | 0.0213 | 0.0216 |
| Fe | 0.6482 | 0.5953 | 0.6347 | 0.6097 | 0.6061 | 0.6140 | 0.6216 | 0.6464 | 0.6397 | 0.7423 | 0.6571 | 0.6098 |
| Ba | 0.3487 | 0.2566 | 0.2493 | 0.2058 | 0.2249 | 0.0817 | 0.4446 | 0.2817 | 0.5686 | 0.2763 | 0.3634 | 0.3268 |
| Co | 0.0034 | 0.0036 | 0.0035 | 0.0033 | 0.0032 | 0.0029 | 0.0036 | 0.0053 | 0.0056 | 0.0059 | 0.0025 | 0.0076 |
| Ni | 0.0016 | 0.0020 | 0.0014 | 0.0018 | 0.0015 | 0.0014 | 0.0016 | 0.0018 | 0.0003 | 0.0026 | 0.0021 | 0.0018 |
| Cu | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0005 | 0.0012 | 0.0000 | 0.0000 | 0.0000 | 0.0194 | 0.0000 | 0.0134 |
| Zn | 0.0055 | 0.0037 | 0.0054 | 0.0103 | 0.0171 | 0.0221 | 0.0018 | 0.0014 | 0.0014 | 0.1765 | 0.0101 | 0.1279 |
| As | 0.0003 | 0.0002 | 0.0003 | 0.0005 | 0.0009 | 0.0012 | 0.0002 | 0.0002 | 0.0002 | 0.0103 | 0.0015 | 0.0067 |
| Pb | 0.0013 | 0.0016 | 0.0008 | 0.0015 | 0.0007 | 0.0015 | 0.0013 | 0.0009 | 0.0014 | 0.0034 | 0.0014 | 0.0037 |
| Th | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0002 | 0.0004 | 0.0003 | 0.0003 |
| Rb | 0.0004 | 0.0005 | 0.0004 | 0.0003 | 0.0002 | 0.0004 | 0.0004 | 0.0004 | 0.0005 | 0.0000 | 0.0005 | 0.0000 |
| U | 0.0002 | 0.0001 | 0.0000 | 0.0000 | 0.0002 | 0.0006 | 0.0004 | 0.0015 | 0.0008 | 0.0000 | 0.0002 | 0.0040 |
| Sr | 0.0389 | 0.0357 | 0.0391 | 0.0383 | 0.0386 | 0.0389 | 0.0376 | 0.0232 | 0.0205 | 0.0357 | 0.0349 | 0.0172 |
| Y | 0.0019 | 0.0021 | 0.0021 | 0.0019 | 0.0013 | 0.0015 | 0.0016 | 0.0009 | 0.0010 | 0.0019 | 0.0019 | 0.0000 |
| Zr | 0.0082 | 0.0079 | 0.0079 | 0.0078 | 0.0069 | 0.0061 | 0.0070 | 0.0057 | 0.0058 | 0.0065 | 0.0084 | 0.0055 |
| Nb | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0005 | 0.0006 | 0.0003 | 0.0005 | 0.0005 | 0.0007 |
| Mo | 0.0028 | 0.0024 | 0.0021 | 0.0024 | 0.0023 | 0.0020 | 0.0021 | 0.0085 | 0.0093 | 0.0030 | 0.0022 | 0.0102 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 |
| Sb | 0.0003 | 0.0000 | 0.0008 | 0.0006 | 0.0008 | 0.0001 | 0.0001 | 0.0004 | 0.0013 | 0.0000 | 0.0003 | 0.0009 |

Table A.21 Raw PHXRF data for Cement/Fly ash mixture

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.5209 | 0.5036 | 0.4790 | 0.4834 | 0.4842 | 0.4716 | 0.3966 | 0.3951 | 0.4478 | 0.4071 | 0.3953 | 0.5279 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6941 | 0.6768 | 0.6319 | 0.3011 | 0.3572 | 0.2541 | 0.2681 | 0.2904 | 0.2588 | 0.2991 | 0.2902 | 0.4565 |
| Si | 1.9938 | 1.8787 | 2.0044 | 1.8516 | 1.9028 | 1.8237 | 1.7616 | 1.8083 | 1.7591 | 1.7011 | 1.7519 | 1.7868 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0008 | 0.0119 | 0.0233 | 0.0129 | 0.0270 | 0.0295 | 0.0188 | 0.0514 |
| S | 1.6604 | 1.8271 | 1.4765 | 1.4640 | 1.7064 | 1.2321 | 1.3217 | 1.5702 | 1.2874 | 1.6351 | 2.1067 | 2.2061 |
| K | 2.5057 | 2.6055 | 2.3982 | 2.6477 | 3.2013 | 2.2713 | 2.4336 | 3.1380 | 2.3994 | 2.6455 | 3.0321 | 2.9914 |
| Ca | 14.725 | 13.786 | 14.784 | 19.308 | 20.069 | 16.967 | 14.826 | 17.662 | 14.661 | 14.534 | 17.217 | 13.382 |
| Ba | 0.2589 | 0.2194 | 0.2603 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0480 | 0.0518 | 0.0509 | 0.0437 | 0.0507 | 0.0454 | 0.0432 | 0.0529 | 0.0389 |
| V | 0.0000 | 0.0000 | 0.0000 | 0.0025 | 0.0027 | 0.0031 | 0.0037 | 0.0032 | 0.0035 | 0.0044 | 0.0038 | 0.0043 |
| Cr | 0.0038 | 0.0040 | 0.0039 | 0.0034 | 0.0036 | 0.0036 | 0.0033 | 0.0034 | 0.0036 | 0.0033 | 0.0036 | 0.0036 |
| Mn | 0.0225 | 0.0226 | 0.0225 | 0.0204 | 0.0204 | 0.0205 | 0.0208 | 0.0204 | 0.0209 | 0.0206 | 0.0205 | 0.0210 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.8425 | 0.9148 | 0.7384 | 0.6519 | 0.9832 | 0.6849 | 0.7270 | 1.0980 | 0.6720 |
| Ba | 0.2757 | 0.1416 | 0.2068 | 0.0419 | 0.0754 | 0.0942 | 0.2305 | 0.0844 | 0.1683 | 0.0655 | 0.3048 | 0.0000 |
| Co | 0.0065 | 0.0050 | 0.0056 | 0.0027 | 0.0029 | 0.0025 | 0.0020 | 0.0033 | 0.0033 | 0.0031 | 0.0031 | 0.0019 |
| Ni | 0.0037 | 0.0038 | 0.0033 | 0.0035 | 0.0037 | 0.0034 | 0.0036 | 0.0028 | 0.0039 | 0.0042 | 0.0048 | 0.0038 |
| Cu | 0.0050 | 0.0058 | 0.0058 | 0.0068 | 0.0063 | 0.0071 | 0.0068 | 0.0055 | 0.0069 | 0.0062 | 0.0075 | 0.0079 |
| Zn | 0.0235 | 0.0263 | 0.0258 | 0.0262 | 0.0269 | 0.0269 | 0.0265 | 0.0252 | 0.0251 | 0.0269 | 0.0293 | 0.0284 |
| As | 0.0015 | 0.0017 | 0.0017 | 0.0017 | 0.0017 | 0.0017 | 0.0016 | 0.0016 | 0.0016 | 0.0016 | 0.0018 | 0.0022 |
| Pb | 0.0015 | 0.0016 | 0.0019 | 0.0015 | 0.0022 | 0.0010 | 0.0016 | 0.0023 | 0.0010 | 0.0014 | 0.0024 | 0.0013 |
| Th | 0.0007 | 0.0007 | 0.0007 | 0.0007 | 0.0007 | 0.0007 | 0.0006 | 0.0007 | 0.0007 | 0.0006 | 0.0007 | 0.0007 |
| Rb | 0.0064 | 0.0071 | 0.0062 | 0.0075 | 0.0082 | 0.0077 | 0.0064 | 0.0083 | 0.0065 | 0.0056 | 0.0064 | 0.0070 |
| U | 0.0022 | 0.0030 | 0.0013 | 0.0008 | 0.0022 | 0.0022 | 0.0004 | 0.0024 | 0.0014 | 0.0005 | 0.0015 | 0.0016 |
| Sr | 0.0623 | 0.0662 | 0.0672 | 0.0686 | 0.0669 | 0.0700 | 0.0667 | 0.0670 | 0.0670 | 0.0651 | 0.0733 | 0.0698 |
| Y | 0.0016 | 0.0016 | 0.0018 | 0.0021 | 0.0020 | 0.0020 | 0.0021 | 0.0021 | 0.0020 | 0.0024 | 0.0023 | 0.0024 |
| Zr | 0.0086 | 0.0084 | 0.0090 | 0.0084 | 0.0079 | 0.0081 | 0.0073 | 0.0077 | 0.0079 | 0.0079 | 0.0080 | 0.0080 |
| Nb | 0.0009 | 0.0009 | 0.0009 | 0.0009 | 0.0009 | 0.0009 | 0.0007 | 0.0009 | 0.0009 | 0.0008 | 0.0009 | 0.0008 |
| Mo | 0.0002 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0022 | 0.0016 | 0.0020 | 0.0021 | 0.0017 | 0.0019 | 0.0024 | 0.0020 | 0.0022 | 0.0021 | 0.0011 | 0.0025 |

## Table A.22 Raw PHXRF data for Class F Fly ash

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.4543 | 0.1490 | 0.1230 | 0.0267 | 0.0708 | 0.0817 | 0.0654 | 0.0400 | 0.1221 | 0.1414 | 0.0764 | 0.1218 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 2.8748 | 2.7908 | 2.8669 | 2.9524 | 3.1256 | 3.1276 | 3.1191 | 3.0336 | 3.1134 | 3.0714 | 2.8873 | 2.8872 |
| Si | 6.3117 | 6.5043 | 6.7604 | 7.0053 | 7.4128 | 7.3068 | 7.3832 | 7.4506 | 7.3582 | 7.2987 | 6.9716 | 6.8433 |
| P | 0.0291 | 0.0289 | 0.0216 | 0.0215 | 0.0263 | 0.0237 | 0.0263 | 0.0231 | 0.0203 | 0.0202 | 0.0256 | 0.0252 |
| S | 0.7222 | 0.7238 | 0.7625 | 0.7802 | 0.7987 | 0.8093 | 0.8035 | 0.8142 | 0.8206 | 0.8030 | 0.7652 | 0.7597 |
| K | 1.1399 | 1.1904 | 1.2479 | 1.2799 | 1.3588 | 1.3339 | 1.3473 | 1.3680 | 1.3531 | 1.3355 | 1.2642 | 1.2453 |
| Ca | 0.9058 | 0.9423 | 0.9891 | 1.0034 | 1.0673 | 1.0932 | 1.0749 | 1.1056 | 1.0747 | 1.0685 | 1.0216 | 1.0039 |
| Ba | 0.4159 | 0.3631 | 0.2780 | 0.1917 | 0.1224 | 0.1302 | 0.1649 | 0.1101 | 0.1666 | 0.1696 | 0.2679 | 0.2943 |
| Ti | 0.4277 | 0.4694 | 0.5175 | 0.5580 | 0.6104 | 0.6048 | 0.5922 | 0.6166 | 0.5923 | 0.5869 | 0.5262 | 0.5153 |
| V | 0.0076 | 0.0097 | 0.0155 | 0.0163 | 0.0193 | 0.0213 | 0.0184 | 0.0205 | 0.0195 | 0.0183 | 0.0155 | 0.0126 |
| Cr | 0.0126 | 0.0123 | 0.0119 | 0.0119 | 0.0122 | 0.0114 | 0.0117 | 0.0119 | 0.0120 | 0.0119 | 0.0122 | 0.0121 |
| Mn | 0.0110 | 0.0107 | 0.0098 | 0.0082 | 0.0066 | 0.0072 | 0.0081 | 0.0071 | 0.0077 | 0.0068 | 0.0083 | 0.0087 |
| Fe | 1.5430 | 1.8380 | 2.2446 | 2.8499 | 3.3199 | 3.2597 | 3.0688 | 3.2057 | 3.0086 | 3.1389 | 2.6453 | 2.4610 |
| Ba | 0.3096 | 0.3345 | 0.6123 | 0.5044 | 0.3601 | 0.3150 | 0.2881 | 0.1917 | 0.0000 | 0.5284 | 0.3762 | 0.4241 |
| Co | 0.0098 | 0.0087 | 0.0080 | 0.0072 | 0.0066 | 0.0064 | 0.0066 | 0.0060 | 0.0056 | 0.0084 | 0.0107 | 0.0094 |
| Ni | 0.0108 | 0.0110 | 0.0116 | 0.0114 | 0.0109 | 0.0111 | 0.0118 | 0.0120 | 0.0122 | 0.0104 | 0.0101 | 0.0109 |
| Cu | 0.0114 | 0.0107 | 0.0114 | 0.0119 | 0.0121 | 0.0110 | 0.0122 | 0.0112 | 0.0100 | 0.0115 | 0.0100 | 0.0117 |
| Zn | 0.0117 | 0.0127 | 0.0124 | 0.0118 | 0.0132 | 0.0120 | 0.0120 | 0.0127 | 0.0128 | 0.0128 | 0.0121 | 0.0125 |
| As | 0.0084 | 0.0087 | 0.0093 | 0.0087 | 0.0092 | 0.0087 | 0.0105 | 0.0104 | 0.0085 | 0.0096 | 0.0075 | 0.0095 |
| Pb | 0.0041 | 0.0041 | 0.0042 | 0.0041 | 0.0040 | 0.0036 | 0.0050 | 0.0036 | 0.0025 | 0.0045 | 0.0042 | 0.0041 |
| Th | 0.0010 | 0.0010 | 0.0010 | 0.0010 | 0.0011 | 0.0010 | 0.0011 | 0.0011 | 0.0011 | 0.0011 | 0.0010 | 0.0011 |
| Rb | 0.0107 | 0.0114 | 0.0110 | 0.0112 | 0.0113 | 0.0109 | 0.0118 | 0.0117 | 0.0114 | 0.0105 | 0.0113 | 0.0112 |
| U | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0001 | 0.0000 | 0.0000 | 0.0000 | 0.0010 | 0.0000 |
| Sr | 0.0631 | 0.0661 | 0.0674 | 0.0643 | 0.0668 | 0.0627 | 0.0666 | 0.0664 | 0.0677 | 0.0653 | 0.0632 | 0.0662 |
| Y | 0.0065 | 0.0062 | 0.0064 | 0.0068 | 0.0065 | 0.0065 | 0.0059 | 0.0061 | 0.0069 | 0.0062 | 0.0056 | 0.0060 |
| Zr | 0.0190 | 0.0195 | 0.0201 | 0.0201 | 0.0206 | 0.0191 | 0.0206 | 0.0200 | 0.0205 | 0.0200 | 0.0191 | 0.0198 |
| Nb | 0.0013 | 0.0013 | 0.0014 | 0.0014 | 0.0014 | 0.0012 | 0.0014 | 0.0014 | 0.0014 | 0.0014 | 0.0014 | 0.0014 |
| Mo | 0.0016 | 0.0013 | 0.0007 | 0.0007 | 0.0016 | 0.0013 | 0.0009 | 0.0011 | 0.0011 | 0.0011 | 0.0010 | 0.0011 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 | 0.0003 |
| Sb | 0.0029 | 0.0028 | 0.0023 | 0.0021 | 0.0028 | 0.0034 | 0.0031 | 0.0026 | 0.0025 | 0.0027 | 0.0024 | 0.0026 |

# Table A.23 Raw PHXRF data for Type I/II Cement

| MAJOR ELEMENTS | Quadrant 1 #4 | | | Quadrant 2 #4 | | | Quadrant 3 #4 | | | Quadrant 4 #4 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) | (R1) | (R2) | (R3) |
| Na | 0.6959 | 0.6872 | 0.6838 | 0.6828 | 0.6838 | 0.6482 | 0.6675 | 0.6750 | 0.6773 | 0.6892 | 0.6882 | 0.6913 |
| Mg | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Al | 0.6245 | 0.5494 | 0.5207 | 0.0727 | 0.0644 | 0.1877 | 0.0000 | 0.0494 | 0.0109 | 0.0052 | 0.0000 | 0.0000 |
| Si | 1.8760 | 1.9000 | 1.8598 | 1.6898 | 1.6777 | 1.6660 | 1.6956 | 1.7001 | 1.7265 | 1.7510 | 1.7683 | 1.8256 |
| P | 0.0000 | 0.0000 | 0.0000 | 0.0201 | 0.0182 | 0.0230 | 0.0223 | 0.0276 | 0.0469 | 0.0215 | 0.0989 | 0.0840 |
| S | 3.0985 | 2.7817 | 2.7606 | 2.4093 | 2.2609 | 2.6642 | 1.9592 | 2.0050 | 1.9712 | 1.9550 | 1.6033 | 1.8469 |
| K | 2.1508 | 1.8968 | 1.8759 | 1.7791 | 1.6148 | 2.3147 | 1.3978 | 1.4793 | 1.5111 | 1.4418 | 1.1193 | 1.2508 |
| Ca | 21.252 | 21.358 | 20.976 | 26.876 | 26.824 | 25.768 | 25.331 | 25.385 | 25.608 | 27.434 | 25.092 | 27.164 |
| Ba | 0.0042 | 0.0179 | 0.0216 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Ti | 0.0000 | 0.0000 | 0.0000 | 0.0180 | 0.0227 | 0.0195 | 0.0221 | 0.0219 | 0.0244 | 0.0271 | 0.0242 | 0.0265 |
| V | 0.0028 | 0.0022 | 0.0019 | 0.0037 | 0.0041 | 0.0042 | 0.0041 | 0.0037 | 0.0044 | 0.0040 | 0.0037 | 0.0038 |
| Cr | 0.0015 | 0.0015 | 0.0015 | 0.0012 | 0.0010 | 0.0015 | 0.0011 | 0.0012 | 0.0010 | 0.0012 | 0.0010 | 0.0010 |
| Mn | 0.0226 | 0.0225 | 0.0225 | 0.0224 | 0.0223 | 0.0224 | 0.0224 | 0.0223 | 0.0223 | 0.0225 | 0.0220 | 0.0219 |
| Fe | 0.0000 | 0.0000 | 0.0000 | 0.4976 | 0.5432 | 0.5208 | 0.6024 | 0.6160 | 0.6414 | 0.7587 | 0.6205 | 0.7502 |
| Ba | 0.0108 | 0.1106 | 0.1684 | 0.1407 | 0.3071 | 0.2734 | 0.2542 | 0.2770 | 0.2676 | 0.2808 | 0.1917 | 0.0865 |
| Co | 0.0043 | 0.0076 | 0.0064 | 0.0051 | 0.0051 | 0.0047 | 0.0049 | 0.0046 | 0.0050 | 0.0049 | 0.0042 | 0.0036 |
| Ni | 0.0025 | 0.0023 | 0.0018 | 0.0024 | 0.0023 | 0.0021 | 0.0017 | 0.0023 | 0.0023 | 0.0024 | 0.0023 | 0.0036 |
| Cu | 0.0061 | 0.0039 | 0.0043 | 0.0058 | 0.0061 | 0.0069 | 0.0065 | 0.0071 | 0.0058 | 0.0048 | 0.0077 | 0.0076 |
| Zn | 0.0221 | 0.0173 | 0.0191 | 0.0222 | 0.0220 | 0.0231 | 0.0232 | 0.0238 | 0.0222 | 0.0208 | 0.0241 | 0.0250 |
| As | 0.0021 | 0.0013 | 0.0016 | 0.0020 | 0.0016 | 0.0021 | 0.0018 | 0.0020 | 0.0018 | 0.0017 | 0.0024 | 0.0022 |
| Pb | 0.0026 | 0.0027 | 0.0028 | 0.0026 | 0.0025 | 0.0026 | 0.0026 | 0.0021 | 0.0023 | 0.0028 | 0.0030 | 0.0029 |
| Th | 0.0005 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 |
| Rb | 0.0023 | 0.0019 | 0.0008 | 0.0014 | 0.0015 | 0.0014 | 0.0016 | 0.0011 | 0.0025 | 0.0018 | 0.0022 | 0.0030 |
| U | 0.0000 | 0.0012 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0008 | 0.0000 | 0.0000 | 0.0007 |
| Sr | 0.0290 | 0.0203 | 0.0227 | 0.0265 | 0.0250 | 0.0270 | 0.0250 | 0.0263 | 0.0264 | 0.0264 | 0.0286 | 0.0290 |
| Y | 0.0025 | 0.0014 | 0.0021 | 0.0019 | 0.0020 | 0.0022 | 0.0020 | 0.0020 | 0.0024 | 0.0019 | 0.0024 | 0.0025 |
| Zr | 0.0083 | 0.0069 | 0.0077 | 0.0077 | 0.0069 | 0.0076 | 0.0074 | 0.0076 | 0.0077 | 0.0071 | 0.0082 | 0.0079 |
| Nb | 0.0006 | 0.0005 | 0.0006 | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0004 | 0.0005 | 0.0005 | 0.0005 | 0.0006 |
| Mo | 0.0000 | 0.0055 | 0.0024 | 0.0016 | 0.0016 | 0.0008 | 0.0009 | 0.0008 | 0.0005 | 0.0004 | 0.0000 | 0.0000 |
| Rh | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| Sn | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0002 |
| Sb | 0.0000 | 0.0004 | 0.0000 | 0.0011 | 0.0009 | 0.0003 | 0.0007 | 0.0004 | 0.0009 | 0.0005 | 0.0000 | 0.0000 |

APPENDIX B: SUPPLEMENTAL INFORMATION FOR ANOVA TEST

B.1 Size based (No.4) comparison of major and trace element concentrations across samples:

## Test for Equal Variances: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Si | 12 | 1.57047 | (1.02791, 3.03010) |
| DHG Si | 12 | 0.58909 | (0.38535, 1.13728) |
| Coastal Si | 12 | 0.65961 | (0.45413, 1.20989) |
| St Wooten Si | 12 | 1.12616 | (0.64574, 2.48024) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.008 |
| Levene | 4.18 | 0.011 |

## One-way ANOVA: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Si, DHG Si, Coastal Si, St Wooten Si |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 23.3668 | 16.55 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 40.46% | 36.40% | 29.14% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Si | 12 | 4.215 | 1.570 | (3.217, 5.213) |
| DHG Si | 12 | 3.835 | 0.589 | (3.461, 4.209) |
| Coastal Si | 12 | 2.353 | 0.660 | (1.933, 2.772) |
| St Wooten Si | 12 | 4.548 | 1.126 | (3.833, 5.264) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| St Wooten Si | 12 | 4.548 | A |
| airport Si | 12 | 4.215 | A |
| DHG Si | 12 | 3.835 | A |
| Coastal Si | 12 | 2.353 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport P, DHG P, Coastal P, St Wooten P

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport P | 12 | 0.0307505 | (0.0118189, 0.101037) |
| DHG P | 12 | 0.0105946 | (0.0053630, 0.026431) |
| Coastal P | 12 | 0.0190639 | (0.0110329, 0.041599) |
| St Wooten P | 12 | 0.0122317 | (0.0062612, 0.030177) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.300 |
| Levene | 1.24 | 0.307 |

## One-way ANOVA: airport P, DHG P, Coastal P, St Wooten P

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport P, DHG P, Coastal P, St Wooten P |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 0.001235 | 0.000412 | 1.05 | 0.381 |
| Error | 44 | 0.017280 | 0.000393 | | |
| Total | 47 | 0.018515 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0198172 | 6.67% | 0.31% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport P | 12 | 0.01897 | 0.03075 | ( 0.00744, 0.03050) |
| DHG P | 12 | 0.00693 | 0.01059 | (-0.00460, 0.01846) |
| Coastal P | 12 | 0.01881 | 0.01906 | ( 0.00728, 0.03034) |
| St Wooten P | 12 | 0.01173 | 0.01223 | ( 0.00020, 0.02325) |

Pooled StDev = 0.0198172

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport P | 12 | 0.01897 | A |
| Coastal P | 12 | 0.01881 | A |
| St Wooten P | 12 | 0.01173 | A |
| DHG P | 12 | 0.00693 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport S | 12 | 0.059882 | (0.0361373, 0.125312) |
| DHG S | 12 | 0.063582 | (0.0310821, 0.164251) |
| Coastal S | 12 | 0.046503 | (0.0317918, 0.085902) |
| St Wooten S | 12 | 0.111445 | (0.0545582, 0.287483) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | – | 0.213 |
| Levene | 1.22 | 0.313 |

## One-way ANOVA: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport S, DHG S, Coastal S, St Wooten S |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 0.7373 | 0.245783 | 44.26 | 0.000 |
| Error | 44 | 0.2443 | 0.005553 | | |
| Total | 47 | 0.9817 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0745168 | 75.11% | 73.41% | 70.38% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport S | 12 | 0.5025 | 0.0599 | (0.4591, 0.5458) |
| DHG S | 12 | 0.5628 | 0.0636 | (0.5194, 0.6061) |
| Coastal S | 12 | 0.2613 | 0.0465 | (0.2179, 0.3046) |
| St Wooten S | 12 | 0.5608 | 0.1114 | (0.5174, 0.6041) |

Pooled StDev = 0.0745168

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG S | 12 | 0.5628 | A |
| St Wooten S | 12 | 0.5608 | A |
| airport S | 12 | 0.5025 | A |
| Coastal S | 12 | 0.2613 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport K, DHG K, Coastal K, St Wooten K

Method

Null hypothesis All variances are equal
Alternative hypothesis At least one variance is different
Significance level α = 0.05

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport K | 12 | 0.140194 | (0.100500, 0.246971) |
| DHG K | 12 | 0.062202 | (0.027698, 0.176411) |
| Coastal K | 12 | 0.049716 | (0.032329, 0.096547) |
| St Wooten K | 12 | 0.143347 | (0.084992, 0.305317) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.002 |
| Levene | 4.56 | 0.007 |

## One-way ANOVA: airport K, DHG K, Coastal K, St Wooten K

Method

Null hypothesis All means are equal
Alternative hypothesis At least one mean is different
Significance level α = 0.05

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport K, DHG K, Coastal K, St Wooten K |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.8703 | 41.23 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 73.63% | 71.83% | 68.62% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport K | 12 | 0.2779 | 0.1402 | (0.1888, 0.3669) |
| DHG K | 12 | 0.2366 | 0.0622 | (0.1971, 0.2761) |
| Coastal K | 12 | 0.1108 | 0.0497 | (0.0792, 0.1424) |
| St Wooten K | 12 | 0.5808 | 0.1433 | (0.4897, 0.6718) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| St Wooten K | 12 | 0.5808 | A |
| airport K | 12 | 0.2779 | B |
| DHG K | 12 | 0.2366 | B |
| Coastal K | 12 | 0.1108 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ca | 12 | 1.57868 | (1.13145, 2.78165) |
| DHG Ca | 12 | 1.04066 | (0.55032, 2.48516) |
| Coastal Ca | 12 | 2.23084 | (1.56580, 4.01377) |
| St Wooten Ca | 12 | 1.86333 | (1.20823, 3.62896) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.034 |
| Levene | 2.09 | 0.116 |

## One-way ANOVA: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ca, DHG Ca, Coastal Ca, St Wooten Ca |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 669.6 | 223.186 | 74.25 | 0.000 |
| Error | 44 | 132.3 | 3.006 | | |
| Total | 47 | 801.8 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|

| | | | |
|---|---|---|---|
| 1.73377 | 83.50% | 82.38% | 80.37% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ca | 12 | 4.132 | 1.579 | ( 3.123, 5.140) |
| DHG Ca | 12 | 6.356 | 1.041 | ( 5.347, 7.365) |
| Coastal Ca | 12 | 13.388 | 2.231 | (12.379, 14.396) |
| St Wooten Ca | 12 | 4.473 | 1.863 | ( 3.465, 5.482) |

Pooled StDev = 1.73377

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| Coastal Ca | 12 | 13.388 | A |
| DHG Ca | 12 | 6.356 | B |
| St Wooten Ca | 12 | 4.473 | B C |
| airport Ca | 12 | 4.132 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ti | 12 | 0.119348 | (0.0631600, 0.284800) |
| DHG Ti | 12 | 0.031625 | (0.0191381, 0.065995) |
| Coastal Ti | 12 | 0.011261 | (0.0058757, 0.027255) |
| St Wooten Ti | 12 | 0.037297 | (0.0247488, 0.070981) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 7.62 | 0.000 |

## One-way ANOVA: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ti, DHG Ti, Coastal Ti, St Wooten Ti |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 20.5176 | 12.21 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 62.19% | 59.61% | 55.01% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ti | 12 | 0.2132 | 0.1193 | ( 0.1374, 0.2891) |
| DHG Ti | 12 | 0.02458 | 0.03162 | (0.00448, 0.04467) |
| Coastal Ti | 12 | 0.01922 | 0.01126 | (0.01207, 0.02638) |
| St Wooten Ti | 12 | 0.0519 | 0.0373 | ( 0.0282, 0.0756) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Ti | 12 | 0.2132 | A |
| St Wooten Ti | 12 | 0.0519 | B |
| DHG Ti | 12 | 0.02458 | B |
| Coastal Ti | 12 | 0.01922 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Cr | 12 | 0.0014793 | (0.0008970, 0.0030806) |
| DHG Cr | 12 | 0.0018295 | (0.0007944, 0.0053208) |
| Coastal Cr | 12 | 0.0006724 | (0.0003478, 0.0016417) |
| St Wooten Cr | 12 | 0.0008998 | (0.0005136, 0.0019910) |

Individual confidence level = 98.75%

Tests

Test

| Method | Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.088 |
| Levene | 1.61 | 0.201 |

## One-way ANOVA: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Cr, DHG Cr, Coastal Cr, St Wooten Cr |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 0.000043 | 0.000014 | 8.44 | 0.000 |
| Error | 44 | 0.000075 | 0.000002 | | |
| Total | 47 | 0.000118 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0013036 | 36.53% | 32.20% | 24.47% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Cr | 12 | 0.004656 | 0.001479 | (0.003898, 0.005415) |
| DHG Cr | 12 | 0.004533 | 0.001830 | (0.003774, 0.005291) |
| Coastal Cr | 12 | 0.002338 | 0.000672 | (0.001580, 0.003097) |
| St Wooten Cr | 12 | 0.004340 | 0.000900 | (0.003582, 0.005098) |

Pooled StDev = 0.00130358

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Cr | 12 | 0.004656 | A |
| DHG Cr | 12 | 0.004533 | A |
| St Wooten Cr | 12 | 0.004340 | A |
| Coastal Cr | 12 | 0.002338 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

Null hypothesis         All variances are equal
Alternative hypothesis  At least one variance is different
Significance level      α = 0.05

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Fe | 12 | 1.12331 | (0.562941, 2.83066) |
| DHG Fe | 12 | 0.40175 | (0.259111, 0.78666) |
| Coastal Fe | 12 | 0.23578 | (0.125402, 0.55984) |
| St Wooten Fe | 12 | 0.51982 | (0.271956, 1.25475) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.012 |
| Levene | 2.80 | 0.051 |

## One-way ANOVA: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Fe, DHG Fe, Coastal Fe, St Wooten Fe |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 24.46 | 8.1547 | 18.65 | 0.000 |
| Error | 44 | 19.24 | 0.4373 | | |
| Total | 47 | 43.70 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.661255 | 55.98% | 52.98% | 47.61% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Fe | 12 | 2.052 | 1.123 | ( 1.668, 2.437) |
| DHG Fe | 12 | 0.314 | 0.402 | ( -0.070, 0.699) |
| Coastal Fe | 12 | 0.2938 | 0.2358 | (-0.0909, 0.6785) |
| St Wooten Fe | 12 | 0.924 | 0.520 | ( 0.540, 1.309) |

Pooled StDev = 0.661255

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Fe | 12 | 2.052 | A |
| St Wooten Fe | 12 | 0.924 | B |
| DHG Fe | 12 | 0.314 | B |
| Coastal Fe | 12 | 0.2938 | B |

Means that do not share a letter are significantly different.

B.2 Size based (No.12) comparison of major and trace element concentrations across samples:

## Test for Equal Variances: airport Al, DHG Al, St Wooten Al

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Al | 12 | 0.168574 | (0.084817, 0.418542) |
| DHG Al | 12 | 0.222792 | (0.115576, 0.536497) |
| St Wooten Al | 12 | 0.345190 | (0.168298, 0.884450) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | – | 0.284 |
| Levene | 1.77 | 0.185 |

## One-way ANOVA: airport Al, DHG Al, St Wooten Al

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | airport Al, DHG Al, St Wooten Al |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 2.695 | 1.34750 | 20.50 | 0.000 |
| Error | 33 | 2.169 | 0.06574 | | |
| Total | 35 | 4.864 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.256391 | 55.40% | 52.70% | 46.93% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Al | 12 | 1.1205 | 0.1686 | (0.9699, 1.2711) |
| DHG Al | 12 | 0.4525 | 0.2228 | (0.3019, 0.6031) |
| St Wooten Al | 12 | 0.8335 | 0.3452 | (0.6829, 0.9840) |

Pooled StDev = 0.256391

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| airport Al | 12 | 1.1205 | A | | |
| St Wooten Al | 12 | 0.8335 | | B | |
| DHG Al | 12 | 0.4525 | | | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Si | 12 | 0.442081 | (0.297608, 0.82930) |
| DHG Si | 12 | 0.640592 | (0.342557, 1.51280) |
| Coastal Si | 12 | 0.259113 | (0.127398, 0.66553) |
| St Wooten Si | 12 | 0.847079 | (0.447044, 2.02699) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.037 |
| Levene | 2.56 | 0.067 |

## One-way ANOVA: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha$ = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Si, DHG Si, Coastal Si, St Wooten Si |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 37.66 | 12.5517 | 36.11 | 0.000 |
| Error | 44 | 15.30 | 0.3476 | | |
| Total | 47 | 52.95 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.589592 | 71.11% | 69.14% | 65.62% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Si | 12 | 4.338 | 0.442 | ( 3.995, 4.681) |
| DHG Si | 12 | 4.280 | 0.641 | ( 3.937, 4.623) |
| Coastal Si | 12 | 2.2261 | 0.2591 | (1.8831, 2.5692) |
| St Wooten Si | 12 | 4.185 | 0.847 | ( 3.842, 4.528) |

Pooled StDev = 0.589592

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping | |
|---|---|---|---|---|
| airport Si | 12 | 4.338 | A | |
| DHG Si | 12 | 4.280 | A | |
| St Wooten Si | 12 | 4.185 | A | |
| Coastal Si | 12 | 2.2261 | | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport P, DHG P, Coastal P, St Wooten P

Method

Null hypothesis          All variances are equal

Alternative hypothesis  At least one variance is different
Significance level  α = 0.05

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
| --- | --- | --- | --- |
| airport P | 12 | 0.0054230 | (0.0029371, 0.0126449) |
| DHG P | 12 | 0.0137650 | (0.0078810, 0.0303616) |
| Coastal P | 12 | 0.0155607 | (0.0065879, 0.0464158) |
| St Wooten P | 12 | 0.0046768 | (0.0009141, 0.0302193) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
| --- | --- | --- |
| Multiple comparisons | – | 0.046 |
| Levene | 2.52 | 0.070 |

## One-way ANOVA: airport P, DHG P, Coastal P, St Wooten P

Method

Null hypothesis  All means are equal
Alternative hypothesis  At least one mean is different
Significance level  α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
| --- | --- | --- |
| Factor | 4 | airport P, DHG P, Coastal P, St Wooten P |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
| --- | --- | --- | --- | --- | --- |
| Factor | 3 | 0.001249 | 0.000416 | 3.45 | 0.025 |
| Error | 44 | 0.005312 | 0.000121 | | |
| Total | 47 | 0.006560 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
| --- | --- | --- | --- |
| 0.0109874 | 19.03% | 13.51% | 3.64% |

Means

| Factor | N | Mean | StDev | 95% CI |
| --- | --- | --- | --- | --- |
| airport P | 12 | 0.00401 | 0.00542 | (-0.00238, 0.01040) |
| DHG P | 12 | 0.01469 | 0.01376 | ( 0.00829, 0.02108) |
| Coastal P | 12 | 0.00911 | 0.01556 | ( 0.00272, 0.01550) |
| St Wooten P | 12 | 0.00135 | 0.00468 | (-0.00504, 0.00774) |

Pooled StDev = 0.0109874

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG P | 12 | 0.01469 | A |
| Coastal P | 12 | 0.00911 | A B |
| airport P | 12 | 0.00401 | A B |
| St Wooten P | 12 | 0.00135 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport S | 12 | 0.0310916 | (0.0183906, 0.066381) |
| DHG S | 12 | 0.0479213 | (0.0307914, 0.094185) |
| Coastal S | 12 | 0.0969045 | (0.0699559, 0.169518) |
| St Wooten S | 12 | 0.0275076 | (0.0183879, 0.051967) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 11.53 | 0.000 |

## One-way ANOVA: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport S, DHG S, Coastal S, St Wooten S |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 23.3118 | 25.74 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 78.50% | 77.03% | 74.41% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport S | 12 | 0.49838 | 0.03109 | (0.47863, 0.51814) |
| DHG S | 12 | 0.5662 | 0.0479 | ( 0.5358, 0.5967) |
| Coastal S | 12 | 0.2855 | 0.0969 | ( 0.2239, 0.3471) |
| St Wooten S | 12 | 0.50118 | 0.02751 | (0.48370, 0.51865) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG S | 12 | 0.5662 | A |
| St Wooten S | 12 | 0.50118 | B |
| airport S | 12 | 0.49838 | B |
| Coastal S | 12 | 0.2855 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport K, DHG K, Coastal K, St Wooten K

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport K | 12 | 0.045825 | (0.018726, 0.141616) |
| DHG K | 12 | 0.110344 | (0.062071, 0.247719) |
| Coastal K | 12 | 0.035170 | (0.021640, 0.072184) |
| St Wooten K | 12 | 0.204114 | (0.103404, 0.508814) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.003 |
| Levene | 7.14 | 0.001 |

## One-way ANOVA: airport K, DHG K, Coastal K, St Wooten K

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport K, DHG K, Coastal K, St Wooten K |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.3675 | 40.21 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 74.13% | 72.36% | 69.21% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport K | 12 | 0.2708 | 0.0458 | (0.2417, 0.2999) |
| DHG K | 12 | 0.3005 | 0.1103 | (0.2304, 0.3706) |
| Coastal K | 12 | 0.1468 | 0.0352 | (0.1245, 0.1692) |
| St Wooten K | 12 | 0.6666 | 0.2041 | (0.5369, 0.7962) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| St Wooten K | 12 | 0.6666 | A | | |
| DHG K | 12 | 0.3005 | | B | |
| airport K | 12 | 0.2708 | | B | |
| Coastal K | 12 | 0.1468 | | | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ca | 12 | 0.64786 | (0.29632, 1.78874) |
| DHG Ca | 12 | 1.27328 | (0.83273, 2.45863) |
| Coastal Ca | 12 | 2.87143 | (1.78839, 5.82219) |
| St Wooten Ca | 12 | 1.00203 | (0.58678, 2.16090) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.001 |
| Levene | 7.14 | 0.001 |

## One-way ANOVA: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ca, DHG Ca, Coastal Ca, St Wooten Ca |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.7572 | 52.58 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 85.26% | 84.25% | 82.45% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ca | 12 | 4.430 | 0.648 | ( 4.019, 4.842) |
| DHG Ca | 12 | 7.331 | 1.273 | ( 6.522, 8.140) |
| Coastal Ca | 12 | 14.030 | 2.871 | (12.206, 15.854) |
| St Wooten Ca | 12 | 4.704 | 1.002 | ( 4.067, 5.341) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| Coastal Ca | 12 | 14.030 | A |
| DHG Ca | 12 | 7.331 | B |
| St Wooten Ca | 12 | 4.704 | C |
| airport Ca | 12 | 4.430 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ba, DHG Ba, Coastal Ba, St Wooten Ba

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ba | 12 | 0.677878 | (0.351234, 1.65219) |
| DHG Ba | 12 | 0.027470 | (0.005416, 0.17593) |
| Coastal Ba | 12 | 0.082786 | (0.016272, 0.53190) |
| St Wooten Ba | 12 | 0.165553 | (0.045424, 0.76198) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 6.59 | 0.001 |

## One-way ANOVA: airport Ba, DHG Ba, Coastal Ba, St Wooten Ba

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ba, DHG Ba, Coastal Ba, St Wooten Ba |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 19.7815 | 3.78 | 0.027 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 36.38% | 32.04% | 24.29% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ba | 12 | 0.624 | 0.678 | ( 0.193, 1.054) |
| DHG Ba | 12 | 0.00980 | 0.02747 | (-0.00766, 0.02725) |
| Coastal Ba | 12 | 0.0287 | 0.0828 | ( -0.0239, 0.0812) |
| St Wooten Ba | 12 | 0.0769 | 0.1656 | ( -0.0283, 0.1821) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Ba | 12 | 0.624 | A |

| | | | |
|---|---|---|---|
| St Wooten Ba | 12 | 0.0769 | A B |
| Coastal Ba | 12 | 0.0287 | B |
| DHG Ba | 12 | 0.00980 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ti | 12 | 0.102267 | (0.0879205, 0.150221) |
| DHG Ti | 12 | 0.030421 | (0.0207625, 0.056288) |
| Coastal Ti | 12 | 0.013161 | (0.0081996, 0.026678) |
| St Wooten Ti | 12 | 0.034582 | (0.0173241, 0.087179) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 49.22 | 0.000 |

## One-way ANOVA: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ti, DHG Ti, Coastal Ti, St Wooten Ti |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 21.3112 | 32.26 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 29.59% | 24.79% | 16.21% |

Means

| Factor | N | Mean | StDev | 95% CI |
| --- | --- | --- | --- | --- |
| airport Ti | 12 | 0.0970 | 0.1023 | ( 0.0321, 0.1620) |
| DHG Ti | 12 | 0.11086 | 0.03042 | (0.09153, 0.13018) |
| Coastal Ti | 12 | 0.01980 | 0.01316 | (0.01144, 0.02816) |
| St Wooten Ti | 12 | 0.06453 | 0.03458 | (0.04256, 0.08650) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
| --- | --- | --- | --- |
| DHG Ti | 12 | 0.11086 | A |
| airport Ti | 12 | 0.0970 | A B |
| St Wooten Ti | 12 | 0.06453 | B |
| Coastal Ti | 12 | 0.01980 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
| --- | --- |
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
| --- | --- | --- | --- |
| airport Cr | 12 | 0.0018603 | (0.0005248, 0.0083280) |
| DHG Cr | 12 | 0.0009147 | (0.0005169, 0.0020438) |
| Coastal Cr | 12 | 0.0006308 | (0.0002358, 0.0021304) |
| St Wooten Cr | 12 | 0.0010667 | (0.0003944, 0.0036436) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
| --- | --- | --- |
| Multiple comparisons | — | 0.504 |
| Levene | 1.18 | 0.328 |

## One-way ANOVA: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
| --- | --- |
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
| --- | --- | --- |
| Factor | 4 | airport Cr, DHG Cr, Coastal Cr, St Wooten Cr |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 0.000100 | 0.000033 | 22.92 | 0.000 |
| Error | 44 | 0.000064 | 0.000001 | | |
| Total | 47 | 0.000164 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0012076 | 60.98% | 58.31% | 53.56% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Cr | 12 | 0.006270 | 0.001860 | (0.005568, 0.006973) |
| DHG Cr | 12 | 0.003939 | 0.000915 | (0.003236, 0.004641) |
| Coastal Cr | 12 | 0.002487 | 0.000631 | (0.001785, 0.003190) |
| St Wooten Cr | 12 | 0.005427 | 0.001067 | (0.004724, 0.006129) |

Pooled StDev = 0.00120757

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Cr | 12 | 0.006270 | A |
| St Wooten Cr | 12 | 0.005427 | A |
| DHG Cr | 12 | 0.003939 | B |
| Coastal Cr | 12 | 0.002487 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Fe | 12 | 1.09415 | (0.884078, 1.71008) |
| DHG Fe | 12 | 0.42315 | (0.266457, 0.84862) |
| Coastal Fe | 12 | 0.23035 | (0.118290, 0.56648) |
| St Wooten Fe | 12 | 0.47705 | (0.211324, 1.35998) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 26.79 | 0.000 |

## One-way ANOVA: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Fe, DHG Fe, Coastal Fe, St Wooten Fe |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.3403 | 24.83 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 32.00% | 27.36% | 19.07% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Fe | 12 | 1.029 | 1.094 | ( 0.334, 1.724) |
| DHG Fe | 12 | 1.531 | 0.423 | ( 1.262, 1.799) |
| Coastal Fe | 12 | 0.3408 | 0.2304 | (0.1944, 0.4872) |
| St Wooten Fe | 12 | 0.928 | 0.477 | ( 0.625, 1.231) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG Fe | 12 | 1.531 | A |
| airport Fe | 12 | 1.029 | A B |
| St Wooten Fe | 12 | 0.928 | B |
| Coastal Fe | 12 | 0.3408 | B |

Means that do not share a letter are significantly different.

B.3 Size based (No.50) comparison of major and trace element concentrations across samples:

## Test for Equal Variances: airport Al, DHG Al, St Wooten Al

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Al | 12 | 0.072500 | (0.044896, 0.146253) |
| DHG Al | 12 | 0.055151 | (0.031224, 0.121689) |
| St Wooten Al | 12 | 0.150433 | (0.108496, 0.260562) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.002 |
| Levene | 10.53 | 0.000 |

## One-way ANOVA: airport Al, DHG Al, St Wooten Al

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | airport Al, DHG Al, St Wooten Al |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 20.0773 | 650.88 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 94.21% | 93.86% | 93.11% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Al | 12 | 1.4142 | 0.0725 | (1.3681, 1.4602) |
| DHG Al | 12 | 0.4540 | 0.0552 | (0.4190, 0.4891) |
| St Wooten Al | 12 | 0.9614 | 0.1504 | (0.8658, 1.0570) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping | |
|---|---|---|---|---|
| airport Al | 12 | 1.4142 | A | |
| St Wooten Al | 12 | 0.9614 | | B |

| | | | |
|---|---|---|---|
| DHG Al | 12 | 0.4540 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Si | 12 | 0.451285 | (0.308959, 0.83244) |
| DHG Si | 12 | 0.412302 | (0.276594, 0.77614) |
| Coastal Si | 12 | 0.459818 | (0.296039, 0.90193) |
| St Wooten Si | 12 | 0.614950 | (0.370420, 1.28925) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.527 |
| Levene | 0.20 | 0.894 |

## One-way ANOVA: airport Si, DHG Si, Coastal Si, St Wooten Si

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Si, DHG Si, Coastal Si, St Wooten Si |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 82.32 | 27.4405 | 113.95 | 0.000 |
| Error | 44 | 10.60 | 0.2408 | | |
| Total | 47 | 92.92 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|

| | | | |
|---|---|---|---|
| 0.490726 | 88.60% | 87.82% | 86.43% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Si | 12 | 5.125 | 0.451 | (4.840, 5.411) |
| DHG Si | 12 | 6.732 | 0.412 | (6.446, 7.017) |
| Coastal Si | 12 | 3.040 | 0.460 | (2.755, 3.326) |
| St Wooten Si | 12 | 4.863 | 0.615 | (4.578, 5.149) |

Pooled StDev = 0.490726

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG Si | 12 | 6.732 | A |
| airport Si | 12 | 5.125 | B |
| St Wooten Si | 12 | 4.863 | B |
| Coastal Si | 12 | 3.040 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport P, DHG P, Coastal P, St Wooten P

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport P | 12 | 0.0099174 | (0.0064663, 0.0190011) |
| DHG P | 12 | 0.0017813 | (0.0009232, 0.0042933) |
| Coastal P | 12 | 0.0000000 | ( *, *) |
| St Wooten P | 12 | 0.0018428 | (0.0003905, 0.0108642) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | – | 0.000 |
| Levene | 19.43 | 0.000 |

## One-way ANOVA: airport P, DHG P, St Wooten P

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |

Significance level      α = 0.05

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | airport P, DHG P, St Wooten P |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 19.7590 | 6.26 | 0.008 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 41.27% | 37.71% | 30.11% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport P | 12 | 0.01093 | 0.00992 | ( 0.00463, 0.01723) |
| DHG P | 12 | 0.001218 | 0.001781 | ( 0.000087, 0.002350) |
| St Wooten P | 12 | 0.000541 | 0.001843 | (-0.000630, 0.001712) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport P | 12 | 0.01093 | A |
| DHG P | 12 | 0.001218 | B |
| St Wooten P | 12 | 0.000541 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport S | 12 | 0.0318025 | (0.0236027, 0.054114) |
| DHG S | 12 | 0.0372836 | (0.0198319, 0.088517) |
| Coastal S | 12 | 0.0522946 | (0.0116504, 0.296433) |
| St Wooten S | 12 | 0.0217552 | (0.0141626, 0.042202) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.411 |
| Levene | 0.28 | 0.840 |

## One-way ANOVA: airport S, DHG S, Coastal S, St Wooten S

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport S, DHG S, Coastal S, St Wooten S |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 0.32177 | 0.107258 | 76.48 | 0.000 |
| Error | 44 | 0.06170 | 0.001402 | | |
| Total | 47 | 0.38348 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0374482 | 83.91% | 82.81% | 80.85% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport S | 12 | 0.50514 | 0.03180 | (0.48335, 0.52693) |
| DHG S | 12 | 0.5866 | 0.0373 | ( 0.5648, 0.6083) |
| Coastal S | 12 | 0.3591 | 0.0523 | ( 0.3373, 0.3809) |
| St Wooten S | 12 | 0.50165 | 0.02176 | (0.47986, 0.52344) |

Pooled StDev = 0.0374482

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| DHG S | 12 | 0.5866 | A |
| airport S | 12 | 0.50514 | B |
| St Wooten S | 12 | 0.50165 | B |
| Coastal S | 12 | 0.3591 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport K, DHG K, Coastal K, St Wooten K

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport K | 12 | 0.021336 | (0.013598, 0.042280) |
| DHG K | 12 | 0.065123 | (0.040239, 0.133100) |
| Coastal K | 12 | 0.023173 | (0.013960, 0.048579) |
| St Wooten K | 12 | 0.205377 | (0.114410, 0.465577) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 8.66 | 0.000 |

## One-way ANOVA: airport K, DHG K, Coastal K, St Wooten K

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport K, DHG K, Coastal K, St Wooten K |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.4699 | 132.02 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 90.18% | 89.51% | 88.31% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport K | 12 | 0.29666 | 0.02134 | (0.28311, 0.31022) |
| DHG K | 12 | 0.2588 | 0.0651 | ( 0.2174, 0.3001) |
| Coastal K | 12 | 0.14745 | 0.02317 | (0.13272, 0.16217) |
| St Wooten K | 12 | 0.9526 | 0.2054 | ( 0.8222, 1.0831) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| St Wooten K | 12 | 0.9526 | A |
| airport K | 12 | 0.29666 | B |
| DHG K | 12 | 0.2588 | B |
| Coastal K | 12 | 0.14745 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ca | 12 | 0.50885 | (0.230268, 1.42004) |
| DHG Ca | 12 | 0.92659 | (0.557396, 1.94519) |
| Coastal Ca | 12 | 1.21255 | (0.569211, 3.26196) |
| St Wooten Ca | 12 | 0.58175 | (0.191870, 2.22749) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.295 |
| Levene | 2.02 | 0.126 |

## One-way ANOVA: airport Ca, DHG Ca, Coastal Ca, St Wooten Ca

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ca, DHG Ca, Coastal Ca, St Wooten Ca |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 3 | 600.31 | 200.104 | 273.53 | 0.000 |
| Error | 44 | 32.19 | 0.732 | | |
| Total | 47 | 632.50 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.855307 | 94.91% | 94.56% | 93.94% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ca | 12 | 4.002 | 0.509 | ( 3.505, 4.500) |
| DHG Ca | 12 | 5.879 | 0.927 | ( 5.382, 6.377) |
| Coastal Ca | 12 | 13.023 | 1.213 | (12.526, 13.521) |
| St Wooten Ca | 12 | 5.128 | 0.582 | ( 4.631, 5.626) |

Pooled StDev = 0.855307

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| Coastal Ca | 12 | 13.023 | A |
| DHG Ca | 12 | 5.879 | B |
| St Wooten Ca | 12 | 5.128 | B |
| airport Ca | 12 | 4.002 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ba, DHG Ba, Coastal Ba, St Wooten Ba

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ba | 12 | 0.670213 | (0.572852, 0.990231) |
| DHG Ba | 12 | 0.152127 | (0.067110, 0.435489) |
| Coastal Ba | 12 | 0.035775 | (0.006992, 0.231157) |
| St Wooten Ba | 12 | 0.005062 | (0.001971, 0.016413) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | – | * |
| Levene | 129.86 | 0.000 |

## One-way ANOVA: airport Ba, DHG Ba, Coastal Ba, St Wooten Ba

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ba, DHG Ba, Coastal Ba, St Wooten Ba |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 18.5853 | 7.16 | 0.002 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 51.93% | 48.65% | 42.79% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ba | 12 | 0.823 | 0.670 | ( 0.397, 1.249) |
| DHG Ba | 12 | 0.0966 | 0.1521 | ( -0.0001, 0.1933) |
| Coastal Ba | 12 | 0.0103 | 0.0358 | ( -0.0124, 0.0331) |
| St Wooten Ba | 12 | 0.00273 | 0.00506 | (-0.00049, 0.00594) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| airport Ba | 12 | 0.823 | A |
| DHG Ba | 12 | 0.0966 | B |
| Coastal Ba | 12 | 0.0103 | B |
| St Wooten Ba | 12 | 0.00273 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Ti | 12 | 0.101714 | (0.0730764, 0.178786) |
| DHG Ti | 12 | 0.048547 | (0.0308225, 0.096561) |
| Coastal Ti | 12 | 0.015152 | (0.0074521, 0.038906) |
| St Wooten Ti | 12 | 0.013170 | (0.0080543, 0.027197) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 4.63 | 0.007 |

## One-way ANOVA: airport Ti, DHG Ti, Coastal Ti, St Wooten Ti

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Ti, DHG Ti, Coastal Ti, St Wooten Ti |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.3333 | 29.33 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 13.36% | 7.45% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Ti | 12 | 0.0812 | 0.1017 | ( 0.0166, 0.1458) |
| DHG Ti | 12 | 0.0701 | 0.0485 | ( 0.0393, 0.1010) |
| Coastal Ti | 12 | 0.03249 | 0.01515 | (0.02286, 0.04212) |
| St Wooten Ti | 12 | 0.08831 | 0.01317 | (0.07994, 0.09668) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| St Wooten Ti | 12 | 0.08831 | A |
| airport Ti | 12 | 0.0812 | A B |
| DHG Ti | 12 | 0.0701 | A B |
| Coastal Ti | 12 | 0.03249 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |

Significance level α = 0.05

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Cr | 12 | 0.0009124 | (0.0005511, 0.0019076) |
| DHG Cr | 12 | 0.0018914 | (0.0011365, 0.0039749) |
| Coastal Cr | 12 | 0.0007310 | (0.0003594, 0.0018773) |
| St Wooten Cr | 12 | 0.0008379 | (0.0003732, 0.0023755) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.057 |
| Levene | 5.11 | 0.004 |

## One-way ANOVA: airport Cr, DHG Cr, Coastal Cr, St Wooten Cr

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Cr, DHG Cr, Coastal Cr, St Wooten Cr |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 23.8188 | 29.21 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 51.57% | 48.27% | 42.37% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Cr | 12 | 0.006559 | 0.000912 | (0.005980, 0.007139) |
| DHG Cr | 12 | 0.005978 | 0.001891 | (0.004777, 0.007180) |
| Coastal Cr | 12 | 0.003443 | 0.000731 | (0.002978, 0.003907) |
| St Wooten Cr | 12 | 0.005171 | 0.000838 | (0.004639, 0.005704) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

Factor N Mean Grouping

| | | | |
|---|---|---|---|
| airport Cr | 12 | 0.006559 | A |
| DHG Cr | 12 | 0.005978 | A B |
| St Wooten Cr | 12 | 0.005171 | B |
| Coastal Cr | 12 | 0.003443 | C |

Means that do not share a letter are significantly different.

## Test for Equal Variances: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| airport Fe | 12 | 1.16750 | (0.743330, 2.31569) |
| DHG Fe | 12 | 0.52498 | (0.295235, 1.17887) |
| Coastal Fe | 12 | 0.18666 | (0.095114, 0.46260) |
| St Wooten Fe | 12 | 0.19095 | (0.116206, 0.39623) |

Individual confidence level = 98.75%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 3.92 | 0.015 |

## One-way ANOVA: airport Fe, DHG Fe, Coastal Fe, St Wooten Fe

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 4 | airport Fe, DHG Fe, Coastal Fe, St Wooten Fe |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 3 | 22.5923 | 40.66 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 19.77% | 14.30% | 4.52% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| airport Fe | 12 | 0.903 | 1.167 | ( 0.162, 1.645) |
| DHG Fe | 12 | 0.830 | 0.525 | ( 0.496, 1.163) |
| Coastal Fe | 12 | 0.4266 | 0.1867 | (0.3080, 0.5452) |
| St Wooten Fe | 12 | 1.3025 | 0.1909 | (1.1812, 1.4239) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| St Wooten Fe | 12 | 1.3025 | A |
| airport Fe | 12 | 0.903 | A B |
| DHG Fe | 12 | 0.830 | B |
| Coastal Fe | 12 | 0.4266 | B |

Means that do not share a letter are significantly different.

B.4 Size based (No.4, No.12, No.50) comparison of major and trace element concentrations within NC_AP1 (Airport):

## One-way ANOVA: No.4 Al Airport, No.12 Al Airport, No.50 Al Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Al Airport, No.12 Al Airport, No.50 Al Airport |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 17.0920 | 16.10 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 16.31% | 11.24% | 0.40% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Al Airport | 12 | 1.101 | 0.558 | ( 0.746, 1.455) |
| No.12 Al Airport | 12 | 1.1205 | 0.1686 | (1.0134, 1.2276) |
| No.50 Al Airport | 12 | 1.4142 | 0.0725 | (1.3681, 1.4602) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Al Airport | 12 | 1.4142 | A |
| No.12 Al Airport | 12 | 1.1205 | B |
| No.4 Al Airport | 12 | 1.101 | A B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Si Airport_1, No.12 Si Airport_1, No.50 Si Airport_1

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Si Airport_1, No.12 Si Airport_1, No.50 Si Airport_1 |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 20.0419 | 9.61 | 0.001 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 15.66% | 10.55% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Si Airport_1 | 12 | 4.215 | 1.570 | (3.217, 5.213) |
| No.12 Si Airport_1 | 12 | 4.338 | 0.442 | (4.057, 4.618) |
| No.50 Si Airport_1 | 12 | 5.125 | 0.451 | (4.839, 5.412) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Si Airport_1 | 12 | 5.125 | A |
| No.12 Si Airport_1 | 12 | 4.338 | B |
| No.4 Si Airport_1 | 12 | 4.215 | A B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 P Airport, No.12 P Airport, No.50 P Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 P Airport, No.12 P Airport, No.50 P Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.001345 | 0.000673 | 1.88 | 0.169 |
| Error | 33 | 0.011807 | 0.000358 | | |
| Total | 35 | 0.013152 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0189152 | 10.23% | 4.79% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 P Airport | 12 | 0.01897 | 0.03075 | ( 0.00786, 0.03008) |
| No.12 P Airport | 12 | 0.00401 | 0.00542 | (-0.00710, 0.01512) |
| No.50 P Airport | 12 | 0.01093 | 0.00992 | (-0.00018, 0.02204) |

Pooled StDev = 0.0189152

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 P Airport | 12 | 0.01897 | A |
| No.50 P Airport | 12 | 0.01093 | A |
| No.12 P Airport | 12 | 0.00401 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 S Airport, No.12 S Airport, No.50 S Airport

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 S Airport, No.12 S Airport, No.50 S Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000278 | 0.000139 | 0.07 | 0.928 |
| Error | 33 | 0.061204 | 0.001855 | | |
| Total | 35 | 0.061481 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0430657 | 0.45% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 S Airport | 12 | 0.5025 | 0.0599 | ( 0.4772, 0.5277) |
| No.12 S Airport | 12 | 0.49838 | 0.03109 | (0.47309, 0.52368) |
| No.50 S Airport | 12 | 0.50514 | 0.03180 | (0.47985, 0.53043) |

Pooled StDev = 0.0430657

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 S Airport | 12 | 0.50514 | A |
| No.4 S Airport | 12 | 0.5025 | A |
| No.12 S Airport | 12 | 0.49838 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 k Airport, No.12 k Airport, No.50 k Airport

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were not assumed for the analysis.

Factor Information

Factor  Levels  Values

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 k Airport, No.12 k Airport, No.50 k Airport |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 17.4465 | 1.57 | 0.236 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 1.72% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 k Airport | 12 | 0.2779 | 0.1402 | ( 0.1888, 0.3669) |
| No.12 k Airport | 12 | 0.2708 | 0.0458 | ( 0.2417, 0.2999) |
| No.50 k Airport | 12 | 0.29666 | 0.02134 | (0.28311, 0.31022) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 k Airport | 12 | 0.29666 | A |
| No.4 k Airport | 12 | 0.2779 | A |
| No.12 k Airport | 12 | 0.2708 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ca Airport, No.12 Ca Airport, No.50 Ca Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ca Airport, No.12 Ca Airport, No.50 Ca Airport |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 19.9564 | 1.57 | 0.233 |

Model Summary

R-sq  R-sq(adj)  R-sq(pred)

| | | |
|---|---|---|
| 3.21% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ca Airport | 12 | 4.132 | 1.579 | (3.129, 5.135) |
| No.12 Ca Airport | 12 | 4.430 | 0.648 | (4.019, 4.842) |
| No.50 Ca Airport | 12 | 4.002 | 0.509 | (3.679, 4.326) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 Ca Airport | 12 | 4.430 | A |
| No.4 Ca Airport | 12 | 4.132 | A |
| No.50 Ca Airport | 12 | 4.002 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ba Airport, No.12 Ba Airport, No.50 Ba Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ba Airport, No.12 Ba Airport, No.50 Ba Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 1.192 | 0.5962 | 1.63 | 0.212 |
| Error | 33 | 12.079 | 0.3660 | | |
| Total | 35 | 13.271 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.605003 | 8.99% | 3.47% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ba Airport | 12 | 0.378 | 0.435 | (0.022, 0.733) |
| No.12 Ba Airport | 12 | 0.624 | 0.678 | (0.268, 0.979) |
| No.50 Ba Airport | 12 | 0.823 | 0.670 | (0.467, 1.178) |

Pooled StDev = 0.605003

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Ba Airport | 12 | 0.823 | A |
| No.12 Ba Airport | 12 | 0.624 | A |
| No.4 Ba Airport | 12 | 0.378 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ti Airport, No.12 Ti Airport, No.50 Ti Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ti Airport, No.12 Ti Airport, No.50 Ti Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.1247 | 0.06235 | 5.34 | 0.010 |
| Error | 33 | 0.3855 | 0.01168 | | |
| Total | 35 | 0.5102 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.108086 | 24.44% | 19.86% | 10.08% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ti Airport | 12 | 0.2132 | 0.1193 | (0.1497, 0.2767) |
| No.12 Ti Airport | 12 | 0.0970 | 0.1023 | (0.0336, 0.1605) |
| No.50 Ti Airport | 12 | 0.0812 | 0.1017 | (0.0177, 0.1447) |

Pooled StDev = 0.108086

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping | |
|---|---|---|---|---|
| No.4 Ti Airport | 12 | 0.2132 | A | |
| No.12 Ti Airport | 12 | 0.0970 | | B |
| No.50 Ti Airport | 12 | 0.0812 | | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Cr Airport, No.12 Cr  Airport, No.50 Cr Airport

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Cr Airport, No.12 Cr  Airport, No.50 Cr Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000025 | 0.000013 | 5.84 | 0.007 |
| Error | 33 | 0.000071 | 0.000002 | | |
| Total | 35 | 0.000097 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0014698 | 26.15% | 21.67% | 12.11% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Cr Airport | 12 | 0.004656 | 0.001479 | (0.003793, 0.005519) |
| No.12 Cr  Airport | 12 | 0.006270 | 0.001860 | (0.005407, 0.007133) |
| No.50 Cr Airport | 12 | 0.006559 | 0.000912 | (0.005696, 0.007423) |

Pooled StDev = 0.00146984

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Cr Airport | 12 | 0.006559 | A |
| No.12 Cr  Airport | 12 | 0.006270 | A |
| No.4 Cr Airport | 12 | 0.004656 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Mn Airport, No.12 Mn Airport, No.50 Mn Airport

Method

Null hypothesis         All means are equal

Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Mn Airport, No.12 Mn Airport, No.50 Mn Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000005 | 0.000003 | 0.15 | 0.864 |
| Error | 33 | 0.000617 | 0.000019 | | |
| Total | 35 | 0.000622 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0043231 | 0.88% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Mn Airport | 12 | 0.02845 | 0.00540 | ( 0.02591, 0.03099) |
| No.12 Mn Airport | 12 | 0.02754 | 0.00410 | ( 0.02500, 0.03008) |
| No.50 Mn Airport | 12 | 0.028239 | 0.003179 | (0.025700, 0.030778) |

Pooled StDev = 0.00432311

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 Mn Airport | 12 | 0.02845 | A |
| No.50 Mn Airport | 12 | 0.028239 | A |
| No.12 Mn Airport | 12 | 0.02754 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Fe Airport, No.12 Fe Airport, No.50 Fe Airport

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Fe Airport, No.12 Fe Airport, No.50 Fe Airport |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 9.531 | 4.765 | 3.74 | 0.034 |
| Error | 33 | 42.042 | 1.274 | | |
| Total | 35 | 51.573 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 1.12872 | 18.48% | 13.54% | 2.98% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Fe Airport | 12 | 2.052 | 1.123 | (1.389, 2.715) |
| No.12 Fe Airport | 12 | 1.029 | 1.094 | (0.366, 1.692) |
| No.50 Fe Airport | 12 | 0.903 | 1.167 | (0.241, 1.566) |

Pooled StDev = 1.12872

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 Fe Airport | 12 | 2.052 | A |
| No.12 Fe Airport | 12 | 1.029 | A B |
| No.50 Fe Airport | 12 | 0.903 | B |

Means that do not share a letter are significantly different.

B.5 Size based (No.4, No.12, No.50) comparison of major and trace element concentrations within NC_CT1 (D.H. Griffin):

## One-way ANOVA: No.4 Al DHG, No.12 Al DHG, No.50 Al DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Al DHG, No.12 Al DHG, No.50 Al DHG |

Welch’s Test

DF

| Source | Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 16.5522 | 1.89 | 0.182 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 9.44% | 3.95% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Al DHG | 12 | 0.3400 | 0.1917 | (0.2182, 0.4619) |
| No.12 Al DHG | 12 | 0.4525 | 0.2228 | (0.3110, 0.5941) |
| No.50 Al DHG | 12 | 0.4540 | 0.0552 | (0.4190, 0.4891) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Al DHG | 12 | 0.4540 | A |
| No.12 Al DHG | 12 | 0.4525 | A |
| No.4 Al DHG | 12 | 0.3400 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Si DHG, No.12 Si DHG, No.50 Si DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Si DHG, No.12 Si DHG, No.50 Si DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 58.39 | 29.1973 | 94.45 | 0.000 |
| Error | 33 | 10.20 | 0.3091 | | |
| Total | 35 | 68.60 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.555992 | 85.13% | 84.23% | 82.30% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Si DHG | 12 | 3.835 | 0.589 | (3.508, 4.161) |
| No.12 Si DHG | 12 | 4.280 | 0.641 | (3.954, 4.607) |

| | | | | |
|---|---|---|---|---|
| No.50 Si DHG | 12 | 6.732 | 0.412 | (6.405, 7.058) |

Pooled StDev = 0.555992

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Si DHG | 12 | 6.732 | A |
| No.12 Si DHG | 12 | 4.280 | B |
| No.4 Si DHG | 12 | 3.835 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 P DHG, No.12 P DHG, No.50 P DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 P DHG, No.12 P DHG, No.50 P DHG |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 15.3041 | 6.92 | 0.007 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 24.64% | 20.07% | 10.32% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 P DHG | 12 | 0.00693 | 0.01059 | ( 0.00020, 0.01366) |
| No.12 P DHG | 12 | 0.01469 | 0.01376 | ( 0.00594, 0.02343) |
| No.50 P DHG | 12 | 0.001218 | 0.001781 | (0.000087, 0.002350) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 P DHG | 12 | 0.01469 | A |
| No.4 P DHG | 12 | 0.00693 | A B |
| No.50 P DHG | 12 | 0.001218 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 S DHG, No.12 S DHG, No.50 S DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 S DHG, No.12 S DHG, No.50 S DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.003971 | 0.001985 | 0.77 | 0.471 |
| Error | 33 | 0.085021 | 0.002576 | | |
| Total | 35 | 0.088991 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0507581 | 4.46% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 S DHG | 12 | 0.5628 | 0.0636 | (0.5329, 0.5926) |
| No.12 S DHG | 12 | 0.5662 | 0.0479 | (0.5364, 0.5960) |
| No.50 S DHG | 12 | 0.5866 | 0.0373 | (0.5568, 0.6164) |

Pooled StDev = 0.0507581

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 S DHG | 12 | 0.5866 | A |
| No.12 S DHG | 12 | 0.5662 | A |
| No.4 S DHG | 12 | 0.5628 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 K DHG, No.12 K DHG, No.50 K DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 K DHG, No.12 K DHG, No.50 K DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.02525 | 0.012624 | 1.87 | 0.171 |
| Error | 33 | 0.22315 | 0.006762 | | |
| Total | 35 | 0.24839 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0822314 | 10.16% | 4.72% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 K DHG | 12 | 0.2366 | 0.0622 | (0.1883, 0.2849) |
| No.12 K DHG | 12 | 0.3005 | 0.1103 | (0.2522, 0.3488) |
| No.50 K DHG | 12 | 0.2588 | 0.0651 | (0.2105, 0.3071) |

Pooled StDev = 0.0822314

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 K DHG | 12 | 0.3005 | A |
| No.50 K DHG | 12 | 0.2588 | A |
| No.4 K DHG | 12 | 0.2366 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ca DHG, No.12 Ca DHG, No.50 Ca DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ca DHG, No.12 Ca DHG, No.50 Ca DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 13.15 | 6.576 | 5.54 | 0.008 |
| Error | 33 | 39.19 | 1.188 | | |

```
Total   35   52.34
```

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 1.08977 | 25.13% | 20.59% | 10.90% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ca DHG | 12 | 6.356 | 1.041 | (5.716, 6.996) |
| No.12 Ca DHG | 12 | 7.331 | 1.273 | (6.691, 7.971) |
| No.50 Ca DHG | 12 | 5.879 | 0.927 | (5.239, 6.519) |

Pooled StDev = 1.08977

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 Ca DHG | 12 | 7.331 | A |
| No.4 Ca DHG | 12 | 6.356 | A B |
| No.50 Ca DHG | 12 | 5.879 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ba DHG, No.12 Ba DHG, No.50 Ba DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ba DHG, No.12 Ba DHG, No.50 Ba DHG |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 15.3707 | 7.61 | 0.005 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 27.26% | 22.85% | 13.43% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| No.4 Ba DHG | 12 | 0.2238 | 0.2087 | ( 0.0912, 0.3564) |
| No.12 Ba DHG | 12 | 0.00980 | 0.02747 | (-0.00766, 0.02725) |
| No.50 Ba DHG | 12 | 0.0966 | 0.1521 | ( -0.0001, 0.1933) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 Ba DHG | 12 | 0.2238 | A |
| No.50 Ba DHG | 12 | 0.0966 | A B |
| No.12 Ba DHG | 12 | 0.00980 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ti DHG, No.12 Ti DHG, No.50 Ti DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ti DHG, No.12 Ti DHG, No.50 Ti DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.04471 | 0.022356 | 15.66 | 0.000 |
| Error | 33 | 0.04711 | 0.001427 | | |
| Total | 35 | 0.09182 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0377816 | 48.70% | 45.59% | 38.94% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ti DHG | 12 | 0.02458 | 0.03162 | (0.00239, 0.04677) |
| No.12 Ti DHG | 12 | 0.11086 | 0.03042 | (0.08867, 0.13305) |
| No.50 Ti DHG | 12 | 0.0701 | 0.0485 | ( 0.0479, 0.0923) |

Pooled StDev = 0.0377816

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| No.12 Ti DHG | 12 | 0.11086 | A | | |
| No.50 Ti DHG | 12 | 0.0701 | | B | |
| No.4 Ti DHG | 12 | 0.02458 | | | C |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 V DHG, No.12 V DHG, No.50 V DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 V DHG, No.12 V DHG, No.50 V DHG |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 20.3376 | 3.29 | 0.058 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 23.84% | 19.22% | 9.36% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 V DHG | 12 | 0.001260 | 0.001985 | (-0.000001, 0.002521) |
| No.12 V DHG | 12 | 0.001855 | 0.002251 | ( 0.000424, 0.003285) |
| No.50 V DHG | 12 | 0.00554 | 0.00534 | ( 0.00215, 0.00893) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 V DHG | 12 | 0.00554 | A |
| No.12 V DHG | 12 | 0.001855 | A |
| No.4 V DHG | 12 | 0.001260 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Mn DHG, No.12 Mn DHG, No.50 Mn DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Mn DHG, No.12 Mn DHG, No.50 Mn DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000212 | 0.000106 | 4.96 | 0.013 |
| Error | 33 | 0.000705 | 0.000021 | | |
| Total | 35 | 0.000917 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0046223 | 23.11% | 18.45% | 8.49% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Mn DHG | 12 | 0.02226 | 0.00711 | ( 0.01954, 0.02497) |
| No.12 Mn DHG | 12 | 0.024296 | 0.002307 | (0.021581, 0.027011) |
| No.50 Mn DHG | 12 | 0.028112 | 0.002858 | (0.025398, 0.030827) |

Pooled StDev = 0.00462234

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Mn DHG | 12 | 0.028112 | A |
| No.12 Mn DHG | 12 | 0.024296 | A B |
| No.4 Mn DHG | 12 | 0.02226 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Cr DHG, No.12 Cr DHG, No.50 Cr DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Cr DHG, No.12 Cr DHG, No.50 Cr DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000026 | 0.000013 | 5.11 | 0.012 |
| Error | 33 | 0.000085 | 0.000003 | | |
| Total | 35 | 0.000112 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0016084 | 23.63% | 19.00% | 9.12% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Cr DHG | 12 | 0.004533 | 0.001830 | (0.003588, 0.005477) |
| No.12 Cr DHG | 12 | 0.003939 | 0.000915 | (0.002994, 0.004883) |
| No.50 Cr DHG | 12 | 0.005978 | 0.001891 | (0.005034, 0.006923) |

Pooled StDev = 0.00160842

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Cr DHG | 12 | 0.005978 | A |
| No.4 Cr DHG | 12 | 0.004533 | A B |
| No.12 Cr DHG | 12 | 0.003939 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Fe DHG, No.12 Fe DHG, No.50 Fe DHG

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Fe DHG, No.12 Fe DHG, No.50 Fe DHG |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 8.945 | 4.4725 | 21.78 | 0.000 |
| Error | 33 | 6.777 | 0.2054 | | |
| Total | 35 | 15.722 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.453160 | 56.90% | 54.28% | 48.70% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Fe DHG | 12 | 0.314 | 0.402 | (0.048, 0.580) |
| No.12 Fe DHG | 12 | 1.531 | 0.423 | (1.264, 1.797) |

```
No.50 Fe DHG  12  0.830  0.525  (0.563, 1.096)

Pooled StDev = 0.453160
```

### Tukey Pairwise Comparisons

```
Grouping Information Using the Tukey Method and 95% Confidence

Factor         N   Mean  Grouping
No.12 Fe DHG  12  1.531  A
No.50 Fe DHG  12  0.830    B
No.4 Fe DHG   12  0.314      C

Means that do not share a letter are significantly different.
```

B.6 Size based (No.4, No.12, No.50) comparison of major and trace element concentrations within NC_CT2 (Coastal):

### One-way ANOVA: No.4 Si Coastal, No.12 Si Coastal, No.50 Si Coastal

```
Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were not assumed for the analysis.


Factor Information

Factor  Levels  Values
Factor       3  No.4 Si Coastal, No.12 Si Coastal, No.50 Si Coastal


Welch's Test

         DF
Source  Num   DF Den  F-Value  P-Value
Factor    2  19.3092    13.85    0.000


Model Summary

  R-sq  R-sq(adj)  R-sq(pred)
36.97%     33.15%      24.99%


Means

Factor             N    Mean   StDev       95% CI
No.4 Si Coastal   12   2.353   0.660  ( 1.933,  2.772)
No.12 Si Coastal  12  2.2261  0.2591  (2.0615, 2.3908)
No.50 Si Coastal  12   3.040   0.460  ( 2.748,  3.332)
```

### Games-Howell Pairwise Comparisons

```
Grouping Information Using the Games-Howell Method and 95% Confidence

Factor             N    Mean  Grouping
No.50 Si Coastal  12   3.040  A
```

| | | | |
|---|---|---|---|
| No.4 Si Coastal | 12 | 2.353 | B |
| No.12 Si Coastal | 12 | 2.2261 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 S Coastal, No.12 S Coastal, No.50 S Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 S Coastal, No.12 S Coastal, No.50 S Coastal |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 20.7907 | 11.55 | 0.000 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 28.38% | 24.04% | 14.76% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 S Coastal | 12 | 0.2613 | 0.0465 | (0.2317, 0.2908) |
| No.12 S Coastal | 12 | 0.2855 | 0.0969 | (0.2239, 0.3471) |
| No.50 S Coastal | 12 | 0.3591 | 0.0523 | (0.3259, 0.3923) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 S Coastal | 12 | 0.3591 | A |
| No.12 S Coastal | 12 | 0.2855 | A B |
| No.4 S Coastal | 12 | 0.2613 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 K Coastal, No.12 K Coastal, No.50 K Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 K Coastal, No.12 K Coastal, No.50 K Coastal |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 20.1866 | 2.72 | 0.090 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 18.46% | 13.52% | 2.96% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 K Coastal | 12 | 0.1108 | 0.0497 | ( 0.0792, 0.1424) |
| No.12 K Coastal | 12 | 0.1468 | 0.0352 | ( 0.1245, 0.1692) |
| No.50 K Coastal | 12 | 0.14745 | 0.02317 | (0.13272, 0.16217) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 K Coastal | 12 | 0.14745 | A |
| No.12 K Coastal | 12 | 0.1468 | A |
| No.4 K Coastal | 12 | 0.1108 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ca Coastal, No.12 Ca Coastal, No.50 Ca Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ca Coastal, No.12 Ca Coastal, No.50 Ca Coastal |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 19.3589 | 0.65 | 0.535 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 3.72% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ca Coastal | 12 | 13.388 | 2.231 | (11.970, 14.805) |
| No.12 Ca Coastal | 12 | 14.030 | 2.871 | (12.206, 15.854) |
| No.50 Ca Coastal | 12 | 13.023 | 1.213 | (12.253, 13.794) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 Ca Coastal | 12 | 14.030 | A |
| No.4 Ca Coastal | 12 | 13.388 | A |
| No.50 Ca Coastal | 12 | 13.023 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ti Coastal, No.12 Ti Coastal, No.50 Ti Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ti Coastal, No.12 Ti Coastal, No.50 Ti Coastal |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 21.6837 | 3.27 | 0.058 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 18.81% | 13.89% | 3.38% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ti Coastal | 12 | 0.01922 | 0.01126 | (0.01207, 0.02638) |
| No.12 Ti Coastal | 12 | 0.01980 | 0.01316 | (0.01144, 0.02816) |
| No.50 Ti Coastal | 12 | 0.03249 | 0.01515 | (0.02286, 0.04212) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Ti Coastal | 12 | 0.03249 | A |
| No.12 Ti Coastal | 12 | 0.01980 | A |
| No.4 Ti Coastal | 12 | 0.01922 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Ti Coastal, No.12 Ti Coastal, No.50 Ti Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Ti Coastal, No.12 Ti Coastal, No.50 Ti Coastal |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.001350 | 0.000675 | 3.82 | 0.032 |
| Error | 33 | 0.005826 | 0.000177 | | |
| Total | 35 | 0.007175 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0132868 | 18.81% | 13.89% | 3.38% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Ti Coastal | 12 | 0.01922 | 0.01126 | (0.01142, 0.02703) |
| No.12 Ti Coastal | 12 | 0.01980 | 0.01316 | (0.01199, 0.02760) |
| No.50 Ti Coastal | 12 | 0.03249 | 0.01515 | (0.02469, 0.04029) |

Pooled StDev = 0.0132868

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Ti Coastal | 12 | 0.03249 | A |
| No.12 Ti Coastal | 12 | 0.01980 | A |
| No.4 Ti Coastal | 12 | 0.01922 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Cr Coastal, No.12 Cr Coastal, No.50 Cr Coastal

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Cr Coastal, No.12 Cr Coastal, No.50 Cr Coastal |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000009 | 0.000004 | 9.34 | 0.001 |
| Error | 33 | 0.000015 | 0.000000 | | |
| Total | 35 | 0.000024 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0006793 | 36.14% | 32.27% | 24.01% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Cr Coastal | 12 | 0.002338 | 0.000672 | (0.001939, 0.002737) |
| No.12 Cr Coastal | 12 | 0.002487 | 0.000631 | (0.002088, 0.002886) |
| No.50 Cr Coastal | 12 | 0.003443 | 0.000731 | (0.003044, 0.003842) |

Pooled StDev = 0.000679288

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Cr Coastal | 12 | 0.003443 | A |
| No.12 Cr Coastal | 12 | 0.002487 | B |
| No.4 Cr Coastal | 12 | 0.002338 | B |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 V Coastal, No.12 V Coastal, No.50 V Coastal

Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 V Coastal, No.12 V Coastal, No.50 V Coastal |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000017 | 0.000008 | 2.99 | 0.064 |
| Error | 33 | 0.000092 | 0.000003 | | |
| Total | 35 | 0.000108 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0016663 | 15.32% | 10.19% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 V Coastal | 12 | 0.004894 | 0.001015 | (0.003915, 0.005872) |
| No.12 V Coastal | 12 | 0.003751 | 0.001494 | (0.002772, 0.004730) |
| No.50 V Coastal | 12 | 0.005368 | 0.002251 | (0.004389, 0.006346) |

Pooled StDev = 0.00166630

**Tukey Pairwise Comparisons**

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 V Coastal | 12 | 0.005368 | A |
| No.4 V Coastal | 12 | 0.004894 | A |
| No.12 V Coastal | 12 | 0.003751 | A |

Means that do not share a letter are significantly different

## One-way ANOVA: No.4 Mn Coastal, No.12 Mn Coastal, No.50 Mn Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Mn Coastal, No.12 Mn Coastal, No.50 Mn Coastal |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000009 | 0.000005 | 2.57 | 0.092 |
| Error | 33 | 0.000060 | 0.000002 | | |
| Total | 35 | 0.000070 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0013536 | 13.49% | 8.24% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Mn Coastal | 12 | 0.021454 | 0.000610 | (0.020659, 0.022249) |
| No.12 Mn Coastal | 12 | 0.022640 | 0.002209 | (0.021845, 0.023435) |
| No.50 Mn Coastal | 12 | 0.022398 | 0.000493 | (0.021603, 0.023193) |

Pooled StDev = 0.00135363

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 Mn Coastal | 12 | 0.022640 | A |
| No.50 Mn Coastal | 12 | 0.022398 | A |
| No.4 Mn Coastal | 12 | 0.021454 | A |

Means that do not share a letter are significantly different.

## One-way ANOVA: No.4 Fe Coastal, No.12 Fe Coastal, No.50 Fe Coastal

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 Fe Coastal, No.12 Fe Coastal, No.50 Fe Coastal |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.1088 | 0.05440 | 1.14 | 0.333 |
| Error | 33 | 1.5785 | 0.04783 | | |
| Total | 35 | 1.6873 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.218705 | 6.45% | 0.78% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 Fe Coastal | 12 | 0.2938 | 0.2358 | (0.1654, 0.4223) |
| No.12 Fe Coastal | 12 | 0.3408 | 0.2304 | (0.2123, 0.4692) |
| No.50 Fe Coastal | 12 | 0.4266 | 0.1867 | (0.2982, 0.5551) |

Pooled StDev = 0.218705

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 Fe Coastal | 12 | 0.4266 | A |
| No.12 Fe Coastal | 12 | 0.3408 | A |
| No.4 Fe Coastal | 12 | 0.2938 | A |

Means that do not share a letter are significantly different.

B.7 Size based (No.4, No.12, No.50) comparison of major and trace element concentrations within NC_HW1 (S.T Wooten):

## Test for Equal Variances: No.4 STW Al, No.12 STW Al, No.50 STW Al

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Al | 12 | 0.249062 | (0.146907, 0.527485) |
| No.12 STW Al | 12 | 0.345190 | (0.168298, 0.884450) |
| No.50 STW Al | 12 | 0.150433 | (0.108496, 0.260562) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.201 |
| Levene | 1.62 | 0.213 |

## One-way ANOVA: No.4 STW Al, No.12 STW Al, No.50 STW Al

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Al, No.12 STW Al, No.50 STW Al |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.8589 | 0.42947 | 6.32 | 0.005 |
| Error | 33 | 2.2420 | 0.06794 | | |
| Total | 35 | 3.1009 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.260652 | 27.70% | 23.32% | 13.96% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Al | 12 | 0.5891 | 0.2491 | (0.4360, 0.7421) |
| No.12 STW Al | 12 | 0.8335 | 0.3452 | (0.6804, 0.9865) |
| No.50 STW Al | 12 | 0.9614 | 0.1504 | (0.8083, 1.1145) |

Pooled StDev = 0.260652

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW Al | 12 | 0.9614 | A |
| No.12 STW Al | 12 | 0.8335 | A B |
| No.4 STW Al | 12 | 0.5891 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Si, No.12 STW Si, No.50 STW Si

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Si | 12 | 1.12616 | (0.665061, 2.38219) |
| No.12 STW Si | 12 | 0.84708 | (0.462404, 1.93849) |
| No.50 STW Si | 12 | 0.61495 | (0.380528, 1.24145) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.204 |
| Levene | 1.67 | 0.203 |

## One-way ANOVA: No.4 STW Si, No.12 STW Si, No.50 STW Si

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Si, No.12 STW Si, No.50 STW Si |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 2.762 | 1.3812 | 1.75 | 0.189 |
| Error | 33 | 26.003 | 0.7880 | | |
| Total | 35 | 28.766 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.887683 | 9.60% | 4.12% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Si | 12 | 4.548 | 1.126 | (4.027, 5.070) |
| No.12 STW Si | 12 | 4.185 | 0.847 | (3.664, 4.707) |
| No.50 STW Si | 12 | 4.863 | 0.615 | (4.342, 5.385) |

Pooled StDev = 0.887683

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW Si | 12 | 4.863 | A |
| No.4 STW Si | 12 | 4.548 | A |
| No.12 STW Si | 12 | 4.185 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW P, No.12 STW P, No.50 STW P

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW P | 12 | 0.0122317 | (0.0064865, 0.0288137) |
| No.12 STW P | 12 | 0.0046768 | (0.0009955, 0.0274484) |
| No.50 STW P | 12 | 0.0018428 | (0.0003905, 0.0108642) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.001 |

| | | |
|---|---|---|
| Levene | 11.57 | 0.000 |

## One-way ANOVA: No.4 STW P, No.12 STW P, No.50 STW P

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW P, No.12 STW P, No.50 STW P |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 16.8735 | 4.79 | 0.023 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 32.67% | 28.59% | 19.88% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW P | 12 | 0.01173 | 0.01223 | ( 0.00395, 0.01950) |
| No.12 STW P | 12 | 0.00135 | 0.00468 | ( -0.00162, 0.00432) |
| No.50 STW P | 12 | 0.000541 | 0.001843 | (-0.000630, 0.001712) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 STW P | 12 | 0.01173 | A |
| No.12 STW P | 12 | 0.00135 | B |
| No.50 STW P | 12 | 0.000541 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW S, No.12 STW S, No.50 STW S

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW S | 12 | 0.111445 | (0.0566531, 0.273863) |

| | | | |
|---|---|---|---|
| No.12 STW S | 12 | 0.027508 | (0.0187879, 0.050311) |
| No.50 STW S | 12 | 0.021755 | (0.0144906, 0.040802) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.006 |
| Levene | 5.14 | 0.011 |

## One-way ANOVA: No.4 STW S, No.12 STW S, No.50 STW S

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW S, No.12 STW S, No.50 STW S |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 19.5137 | 1.61 | 0.226 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 15.80% | 10.70% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW S | 12 | 0.5608 | 0.1114 | ( 0.4900, 0.6316) |
| No.12 STW S | 12 | 0.50118 | 0.02751 | (0.48370, 0.51865) |
| No.50 STW S | 12 | 0.50165 | 0.02176 | (0.48783, 0.51547) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 STW S | 12 | 0.5608 | A |
| No.50 STW S | 12 | 0.50165 | A |
| No.12 STW S | 12 | 0.50118 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW K, No.12 STW K, No.50 STW K

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW K | 12 | 0.143347 | (0.087383, 0.293757) |
| No.12 STW K | 12 | 0.204114 | (0.107184, 0.485570) |
| No.50 STW K | 12 | 0.205377 | (0.118010, 0.446502) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.528 |
| Levene | 0.62 | 0.544 |

## One-way ANOVA: No.4 STW K, No.12 STW K, No.50 STW K

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW K, No.12 STW K, No.50 STW K |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.9100 | 0.45498 | 13.08 | 0.000 |
| Error | 33 | 1.1483 | 0.03480 | | |
| Total | 35 | 2.0583 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.186539 | 44.21% | 40.83% | 33.61% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW K | 12 | 0.5808 | 0.1433 | (0.4712, 0.6903) |
| No.12 STW K | 12 | 0.6666 | 0.2041 | (0.5570, 0.7761) |
| No.50 STW K | 12 | 0.9526 | 0.2054 | (0.8431, 1.0622) |

Pooled StDev = 0.186539

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW K | 12 | 0.9526 | A |
| No.12 STW K | 12 | 0.6666 | B |
| No.4 STW K | 12 | 0.5808 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Ca, No.12 STW Ca, No.50 STW Ca

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Ca | 12 | 1.86333 | (1.23647, 3.50781) |
| No.12 STW Ca | 12 | 1.00203 | (0.60368, 2.07774) |
| No.50 STW Ca | 12 | 0.58175 | (0.20336, 2.07893) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.003 |
| Levene | 5.42 | 0.009 |

## One-way ANOVA: No.4 STW Ca, No.12 STW Ca, No.50 STW Ca

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Ca, No.12 STW Ca, No.50 STW Ca |

Welch's Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 18.9767 | 1.24 | 0.311 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 4.76% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Ca | 12 | 4.473 | 1.863 | (3.289, 5.657) |
| No.12 STW Ca | 12 | 4.704 | 1.002 | (4.067, 5.341) |
| No.50 STW Ca | 12 | 5.128 | 0.582 | (4.759, 5.498) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW Ca | 12 | 5.128 | A |
| No.12 STW Ca | 12 | 4.704 | A |
| No.4 STW Ca | 12 | 4.473 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Ba, No.12 STW Ba, No.50 STW Ba

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Ba | 12 | 0.100490 | (0.0319963, 0.394260) |
| No.12 STW Ba | 12 | 0.165553 | (0.0486061, 0.704403) |
| No.50 STW Ba | 12 | 0.005062 | (0.0020715, 0.015451) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.000 |
| Levene | 1.34 | 0.276 |

## One-way ANOVA: No.4 STW Ba, No.12 STW Ba, No.50 STW Ba

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

Factor  Levels  Values

Factor 3 No.4 STW Ba, No.12 STW Ba, No.50 STW Ba

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.03354 | 0.01677 | 1.34 | 0.276 |
| Error | 33 | 0.41285 | 0.01251 | | |
| Total | 35 | 0.44639 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.111851 | 7.51% | 1.91% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Ba | 12 | 0.0480 | 0.1005 | ( -0.0177, 0.1137) |
| No.12 STW Ba | 12 | 0.0769 | 0.1656 | ( 0.0112, 0.1426) |
| No.50 STW Ba | 12 | 0.00273 | 0.00506 | (-0.06296, 0.06842) |

Pooled StDev = 0.111851

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 STW Ba | 12 | 0.0769 | A |
| No.4 STW Ba | 12 | 0.0480 | A |
| No.50 STW Ba | 12 | 0.00273 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Ti, No.12 STW Ti, No.50 STW Ti

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | $\alpha = 0.05$ |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Ti | 12 | 0.0372968 | (0.0252969, 0.0686934) |
| No.12 STW Ti | 12 | 0.0345824 | (0.0179678, 0.0831481) |
| No.50 STW Ti | 12 | 0.0131703 | (0.0082676, 0.0262089) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.005 |
| Levene | 3.41 | 0.045 |

## One-way ANOVA: No.4 STW Ti, No.12 STW Ti, No.50 STW Ti

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were not assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Ti, No.12 STW Ti, No.50 STW Ti |

Welch’s Test

| Source | DF Num | DF Den | F-Value | P-Value |
|---|---|---|---|---|
| Factor | 2 | 17.8540 | 6.58 | 0.007 |

Model Summary

| R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|
| 21.30% | 16.54% | 6.35% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Ti | 12 | 0.0519 | 0.0373 | ( 0.0282, 0.0756) |
| No.12 STW Ti | 12 | 0.06453 | 0.03458 | (0.04256, 0.08650) |
| No.50 STW Ti | 12 | 0.08831 | 0.01317 | (0.07994, 0.09668) |

## Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW Ti | 12 | 0.08831 | A |
| No.12 STW Ti | 12 | 0.06453 | A B |
| No.4 STW Ti | 12 | 0.0519 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW V, No.12 STW V, No.50 STW V

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW V | 12 | 0.0020651 | (0.0014434, 0.0036909) |
| No.12 STW V | 12 | 0.0033015 | (0.0023233, 0.0058606) |
| No.50 STW V | 12 | 0.0038255 | (0.0017081, 0.0107028) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.151 |
| Levene | 1.20 | 0.314 |

## One-way ANOVA: No.4 STW V, No.12 STW V, No.50 STW V

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW V, No.12 STW V, No.50 STW V |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000056 | 0.000028 | 2.80 | 0.075 |
| Error | 33 | 0.000328 | 0.000010 | | |
| Total | 35 | 0.000384 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0031517 | 14.53% | 9.35% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW V | 12 | 0.003286 | 0.002065 | (0.001435, 0.005137) |
| No.12 STW V | 12 | 0.004165 | 0.003301 | (0.002314, 0.006016) |
| No.50 STW V | 12 | 0.00625 | 0.00383 | ( 0.00440, 0.00810) |

Pooled StDev = 0.00315167

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW V | 12 | 0.00625 | A |
| No.12 STW V | 12 | 0.004165 | A |
| No.4 STW V | 12 | 0.003286 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Cr, No.12 STW Cr, No.50 STW Cr

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Cr | 12 | 0.0008998 | (0.0005291, 0.0019118) |
| No.12 STW Cr | 12 | 0.0010667 | (0.0004155, 0.0034208) |
| No.50 STW Cr | 12 | 0.0008379 | (0.0003895, 0.0022519) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.876 |
| Levene | 0.14 | 0.869 |

## One-way ANOVA: No.4 STW Cr, No.12 STW Cr, No.50 STW Cr

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Cr, No.12 STW Cr, No.50 STW Cr |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000008 | 0.000004 | 4.39 | 0.020 |
| Error | 33 | 0.000029 | 0.000001 | | |
| Total | 35 | 0.000037 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0009398 | 21.00% | 16.21% | 5.98% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Cr | 12 | 0.004340 | 0.000900 | (0.003788, 0.004892) |
| No.12 STW Cr | 12 | 0.005427 | 0.001067 | (0.004875, 0.005979) |
| No.50 STW Cr | 12 | 0.005171 | 0.000838 | (0.004619, 0.005723) |

Pooled StDev = 0.000939786

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.12 STW Cr | 12 | 0.005427 | A |
| No.50 STW Cr | 12 | 0.005171 | A B |
| No.4 STW Cr | 12 | 0.004340 | B |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Mn, No.12 STW Mn, No.50 STW Mn

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Mn | 12 | 0.0033293 | (0.0016524, 0.0083797) |
| No.12 STW Mn | 12 | 0.0020076 | (0.0010385, 0.0048485) |
| No.50 STW Mn | 12 | 0.0009171 | (0.0006730, 0.0015614) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | — | 0.039 |
| Levene | 3.02 | 0.062 |

## One-way ANOVA: No.4 STW Mn, No.12 STW Mn, No.50 STW Mn

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Mn, No.12 STW Mn, No.50 STW Mn |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 0.000006 | 0.000003 | 0.59 | 0.562 |
| Error | 33 | 0.000176 | 0.000005 | | |
| Total | 35 | 0.000182 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0023062 | 3.44% | 0.00% | 0.00% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Mn | 12 | 0.024794 | 0.003329 | (0.023439, 0.026148) |
| No.12 STW Mn | 12 | 0.023866 | 0.002008 | (0.022511, 0.025220) |
| No.50 STW Mn | 12 | 0.023962 | 0.000917 | (0.022607, 0.025316) |

Pooled StDev = 0.00230620

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.4 STW Mn | 12 | 0.024794 | A |
| No.50 STW Mn | 12 | 0.023962 | A |
| No.12 STW Mn | 12 | 0.023866 | A |

Means that do not share a letter are significantly different.

## Test for Equal Variances: No.4 STW Fe, No.12 STW Fe, No.50 STW Fe

Method

| | |
|---|---|
| Null hypothesis | All variances are equal |
| Alternative hypothesis | At least one variance is different |
| Significance level | α = 0.05 |

95% Bonferroni Confidence Intervals for Standard Deviations

| Sample | N | StDev | CI |
|---|---|---|---|
| No.4 STW Fe | 12 | 0.519819 | (0.281428, 1.19943) |
| No.12 STW Fe | 12 | 0.477051 | (0.220580, 1.28885) |
| No.50 STW Fe | 12 | 0.190946 | (0.119313, 0.38174) |

Individual confidence level = 98.3333%

Tests

| Method | Test Statistic | P-Value |
|---|---|---|
| Multiple comparisons | – | 0.069 |
| Levene | 2.70 | 0.082 |

## One-way ANOVA: No.4 STW Fe, No.12 STW Fe, No.50 STW Fe

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Factor | 3 | No.4 STW Fe, No.12 STW Fe, No.50 STW Fe |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Factor | 2 | 1.134 | 0.5671 | 3.18 | 0.054 |
| Error | 33 | 5.877 | 0.1781 | | |
| Total | 35 | 7.011 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.421999 | 16.18% | 11.10% | 0.24% |

Means

| Factor | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| No.4 STW Fe | 12 | 0.924 | 0.520 | ( 0.676, 1.172) |
| No.12 STW Fe | 12 | 0.928 | 0.477 | ( 0.680, 1.176) |
| No.50 STW Fe | 12 | 1.3025 | 0.1909 | (1.0547, 1.5504) |

Pooled StDev = 0.421999

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Factor | N | Mean | Grouping |
|---|---|---|---|
| No.50 STW Fe | 12 | 1.3025 | A |
| No.12 STW Fe | 12 | 0.928 | A |
| No.4 STW Fe | 12 | 0.924 | A |

Means that do not share a letter are significantly different.

APPENDIX C: SUPPLEMENTAL INFORMATION FOR STEPWISE REGRESSION

# NO.4

## Regression Analysis: Mortar Conte versus NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, ...

Stepwise Selection of Terms

Candidate terms: NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, BaLa1, TiKa1, V Ka1, CrKa1,
MnKa1, FeKa1, Tr Ba, CoKa1, NiKa1, CuKa1, ZnKa1, AsKa1, PbLa1, ThLa1, RbKa1, U La1,
SrKa1, Y Ka1, ZrKa1, NbKa1, MoKa1, SnKa1, SbKa1

| | ----Step 1---- | | -----Step 2----- | |
|---|---|---|---|---|
| | Coef | P | Coef | P |
| Constant | 34.35 | | 54.52 | |
| V Ka1 | 4014 | 0.048 | 3787 | 0.018 |
| PbLa1 | | | -16911 | 0.051 |
| | | | | |
| S | | 3.40299 | | 0.385057 |
| R-sq | | 90.72% | | 99.94% |
| R-sq(adj) | | 86.08% | | 99.82% |
| R-sq(pred) | | 66.62% | | 98.47% |

α to enter = 0.15, α to remove = 0.15

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Regression | 2 | 249.479 | 124.740 | 841.31 | 0.024 |
| V Ka1 | 1 | 195.409 | 195.409 | 1317.94 | 0.018 |
| PbLa1 | 1 | 23.012 | 23.012 | 155.21 | 0.051 |
| Error | 1 | 0.148 | 0.148 | | |
| Total | 3 | 249.627 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.385057 | 99.94% | 99.82% | 98.47% |

Coefficients

| Term | Coef | SE Coef | T-Value | P-Value | VIF |
|---|---|---|---|---|---|
| Constant | 54.52 | 1.65 | 33.08 | 0.019 | |
| V Ka1 | 3787 | 104 | 36.30 | 0.018 | 1.03 |
| PbLa1 | -16911 | 1357 | -12.46 | 0.051 | 1.03 |

Regression Equation

Mortar Content = 54.52 + 3787 V Ka1 - 16911 PbLa1

# NO.12

## Regression Analysis: Mortar Conte versus NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, ...

Stepwise Selection of Terms

α to enter = 0.15, α to remove = 0.15

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Regression | 2 | 249.223 | 124.611 | 308.05 | 0.040 |
| P Ka1 | 1 | 20.856 | 20.856 | 51.56 | 0.088 |
| PbLa1 | 1 | 233.607 | 233.607 | 577.51 | 0.026 |
| Error | 1 | 0.405 | 0.405 | | |
| Total | 3 | 249.627 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.636012 | 99.84% | 99.51% | 97.00% |

Coefficients

| Term | Coef | SE Coef | T-Value | P-Value | VIF |
|---|---|---|---|---|---|
| Constant | 189.97 | 6.22 | 30.52 | 0.021 | |
| P Ka1 | -524.0 | 73.0 | -7.18 | 0.088 | 1.37 |
| PbLa1 | -124820 | 5194 | -24.03 | 0.026 | 1.37 |

Regression Equation

Mortar Content = 189.97 - 524.0 P Ka1 - 124820 PbLa1

## Regression Analysis: Mortar Conte versus NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, ...

Stepwise Selection of Terms

Candidate terms: NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, BaLa1, TiKa1, V Ka1, CrKa1,
MnKa1, FeKa1, Tr Ba, CoKa1, NiKa1, CuKa1, ZnKa1, AsKa1, PbLa1, ThLa1, RbKa1, U La1,
SrKa1, Y Ka1, ZrKa1, NbKa1, MoKa1, SnKa1, SbKa1

| | -----Step 1----- | | ------Step 2----- | |
|---|---|---|---|---|
| | Coef | P | Coef | P |
| Constant | 164.1 | | 189.97 | |
| PbLa1 | -105438 | 0.044 | -124820 | 0.026 |
| P Ka1 | | | -524.0 | 0.088 |
| | | | | |
| S | | 3.26041 | | 0.636012 |
| R-sq | | 91.48% | | 99.84% |
| R-sq(adj) | | 87.22% | | 99.51% |
| R-sq(pred) | | 74.59% | | 97.00% |

α to enter = 0.15, α to remove = 0.15

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Regression | 2 | 249.223 | 124.611 | 308.05 | 0.040 |
| P Ka1 | 1 | 20.856 | 20.856 | 51.56 | 0.088 |
| PbLa1 | 1 | 233.607 | 233.607 | 577.51 | 0.026 |
| Error | 1 | 0.405 | 0.405 | | |
| Total | 3 | 249.627 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.636012 | 99.84% | 99.51% | 97.00% |

Coefficients

| Term | Coef | SE Coef | T-Value | P-Value | VIF |
|---|---|---|---|---|---|
| Constant | 189.97 | 6.22 | 30.52 | 0.021 | |
| P Ka1 | -524.0 | 73.0 | -7.18 | 0.088 | 1.37 |
| PbLa1 | -124820 | 5194 | -24.03 | 0.026 | 1.37 |

Regression Equation

Mortar Content = 189.97 - 524.0 P Ka1 - 124820 PbLa1

## Regression Analysis: Mortar Conte versus NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, ...

Forward Selection of Terms

Candidate terms: NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, BaLa1, TiKa1, V Ka1, CrKa1, MnKa1, FeKa1, Tr Ba, CoKa1, NiKa1, CuKa1, ZnKa1, AsKa1, PbLa1, ThLa1, RbKa1, U La1, SrKa1, Y Ka1, ZrKa1, NbKa1, MoKa1, SnKa1, SbKa1

| | -----Step 1----- | | ------Step 2----- | |
|---|---|---|---|---|
| | Coef | P | Coef | P |
| Constant | 164.1 | | 189.97 | |
| PbLa1 | -105438 | 0.044 | -124820 | 0.026 |
| P Ka1 | | | -524.0 | 0.088 |
| S | | 3.26041 | | 0.636012 |
| R-sq | | 91.48% | | 99.84% |
| R-sq(adj) | | 87.22% | | 99.51% |
| R-sq(pred) | | 74.59% | | 97.00% |

α to enter = 0.15

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Regression | 2 | 249.223 | 124.611 | 308.05 | 0.040 |
| P Ka1 | 1 | 20.856 | 20.856 | 51.56 | 0.088 |
| PbLa1 | 1 | 233.607 | 233.607 | 577.51 | 0.026 |
| Error | 1 | 0.405 | 0.405 | | |
| Total | 3 | 249.627 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.636012 | 99.84% | 99.51% | 97.00% |

Coefficients

| Term | Coef | SE Coef | T-Value | P-Value | VIF |
|---|---|---|---|---|---|
| Constant | 189.97 | 6.22 | 30.52 | 0.021 | |
| P Ka1 | -524.0 | 73.0 | -7.18 | 0.088 | 1.37 |
| PbLa1 | -124820 | 5194 | -24.03 | 0.026 | 1.37 |

Regression Equation

Mortar Content = 189.97 - 524.0 P Ka1 - 124820 PbLa1

# NO.50

## Regression Analysis: Mortar Conte versus NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, ...

Forward Selection of Terms

Candidate terms: NaKa1, AlKa1, SiKa1, P Ka1, S Ka1, K Ka1, CaKa1, BaLa1, TiKa1, V Ka1, CrKa1,
MnKa1, FeKa1, Tr Ba, CoKa1, NiKa1, CuKa1, ZnKa1, AsKa1, PbLa1, ThLa1, RbKa1, U La1,
SrKa1, Y Ka1, ZrKa1, NbKa1, MoKa1, SnKa1, SbKa1

| | ----Step 1---- | |
|---|---|---|
| | Coef | P |
| Constant | 78.66 | |
| CrKa1 | -6588 | 0.021 |
| | | |
| S | | 2.28695 |
| R-sq | | 95.81% |
| R-sq(adj) | | 93.71% |
| R-sq(pred) | | 86.77% |

α to enter = 0.15

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Regression | 1 | 239.17 | 239.167 | 45.73 | 0.021 |
| CrKa1 | 1 | 239.17 | 239.167 | 45.73 | 0.021 |
| Error | 2 | 10.46 | 5.230 | | |
| Total | 3 | 249.63 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 2.28695 | 95.81% | 93.71% | 86.77% |

Coefficients

| Term | Coef | SE Coef | T-Value | P-Value | VIF |
|---|---|---|---|---|---|

| | | | | | |
|---|---|---|---|---|---|
| Constant | 78.66 | 5.28 | 14.91 | 0.004 | |
| CrKa1 | -6588 | 974 | -6.76 | 0.021 | 1.00 |

Regression Equation

Mortar Content = 78.66 - 6588 CrKa1

APPENDIX D: SIMPLE LINEAR REGRESSION ACTUAL MORTAR WEIGHT % VS WHOLE ROCK ANALYSIS WEIGHT % PLOTS

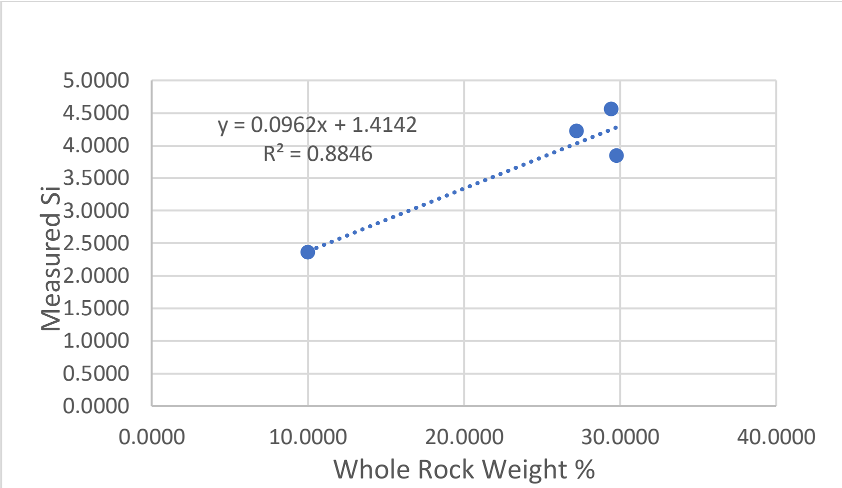


Figure D.1 Whole Rock Weight % Vs Measured Si (No.4)

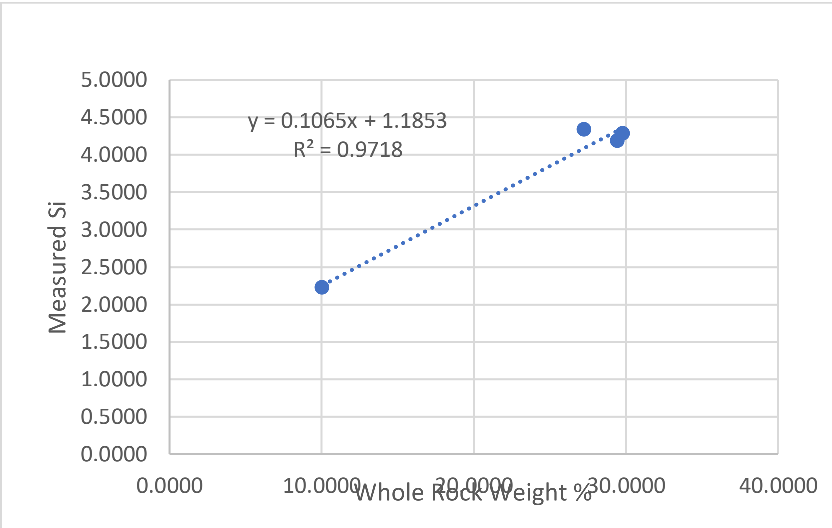


Figure D.2 Whole Rock Weight % Vs Measured Si (No.12)

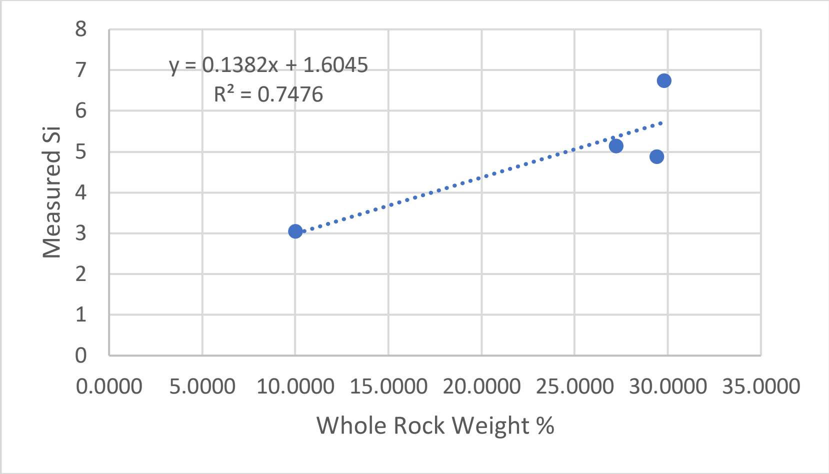


Figure D.3 Whole Rock Weight % Vs Measured Si (No.50)

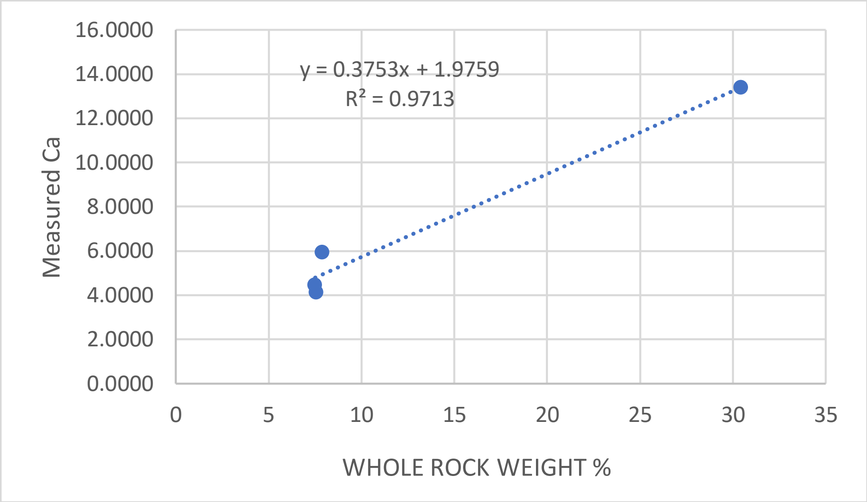


Figure D.4 Whole Rock Weight % Vs Measured Ca (No.4)

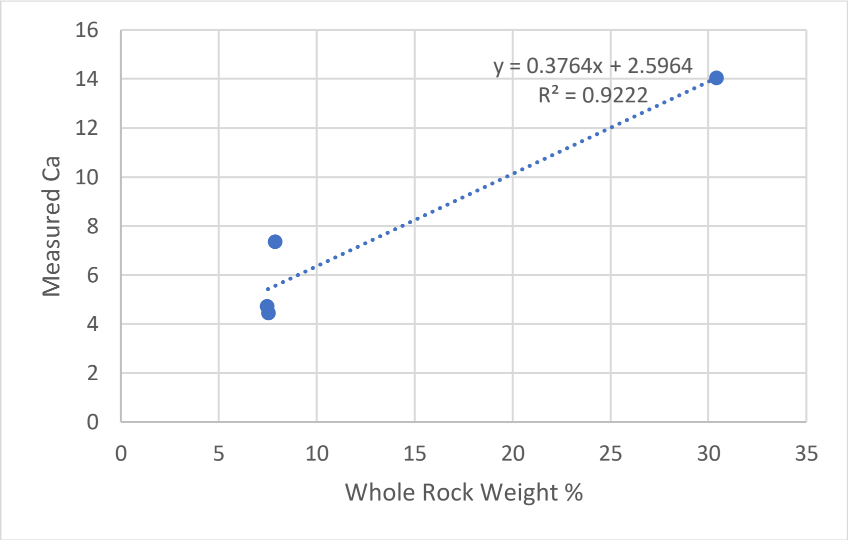


Figure D.5 Whole Rock Weight % Vs Measured Ca (No.12)

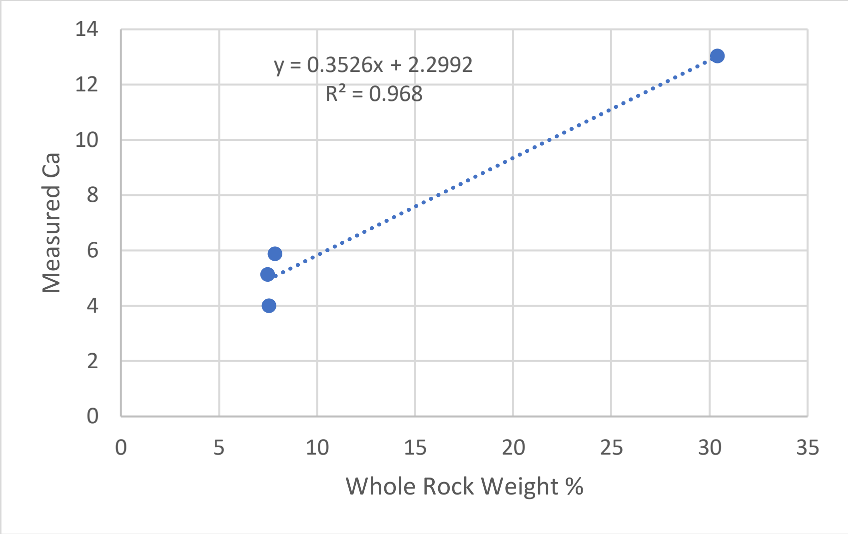


Figure D.6 Whole Rock Weight % Vs Measured Ca (No.50)

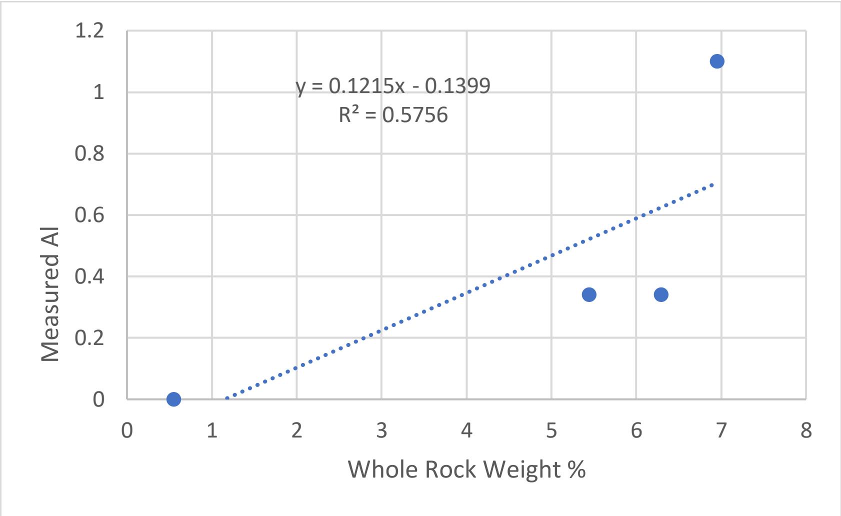


Figure D.7 Whole Rock Weight % Vs Measured Al (No.4)

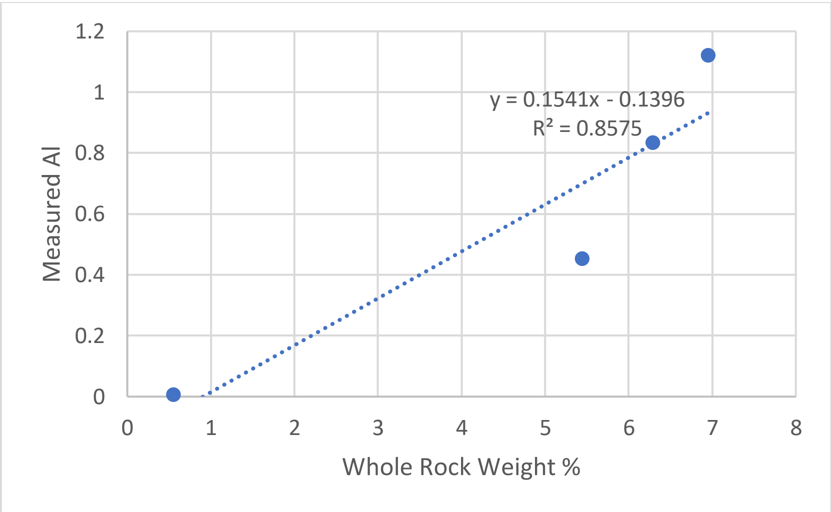


Figure D.8 Whole Rock Weight % Vs Measured Al (No.12)

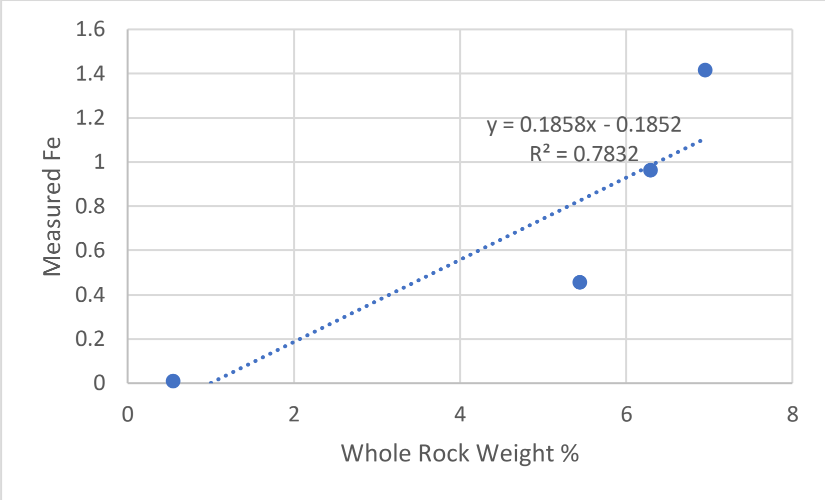


Figure D.9 Whole Rock Weight % Vs Measured Al (No.50)

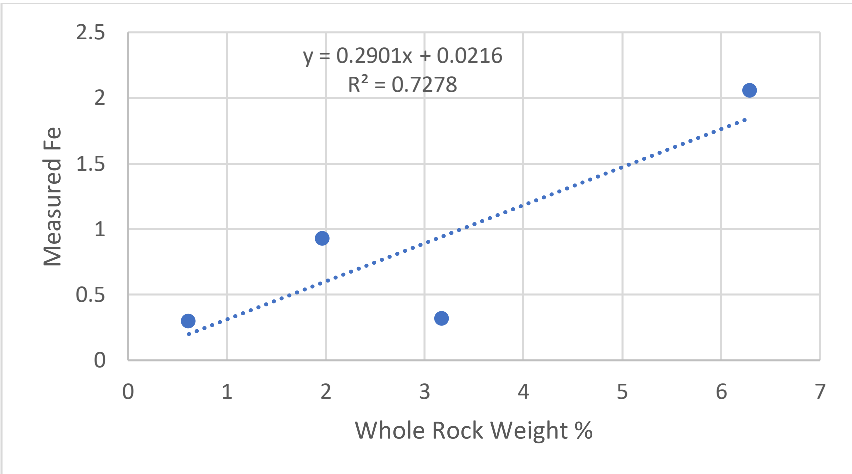


Figure D.10 Whole Rock Weight % Vs Measured Fe (No.4)

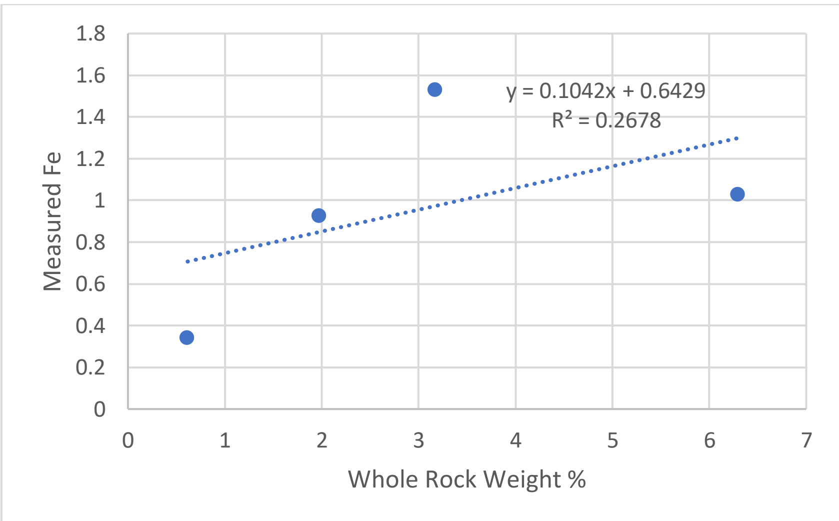


Figure D.11 Whole Rock Weight % Vs Measured Fe (No.12)

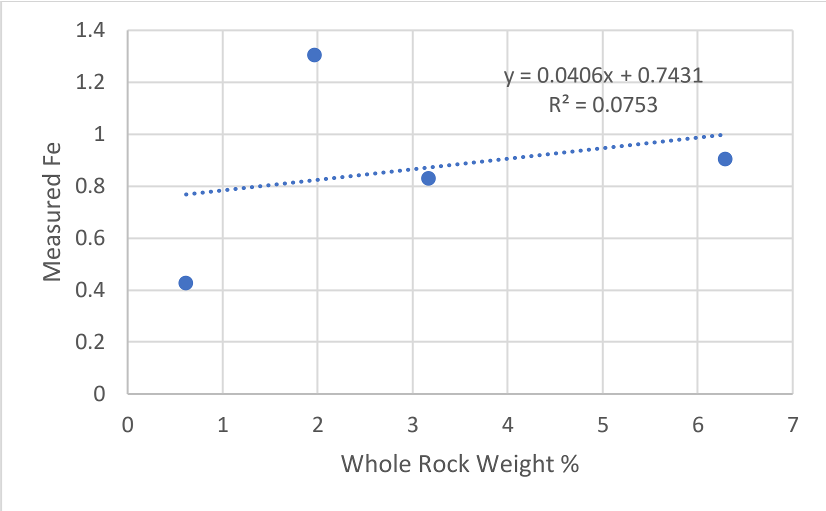


Figure D.12 Whole Rock Weight % Vs Measured Fe (No.50)

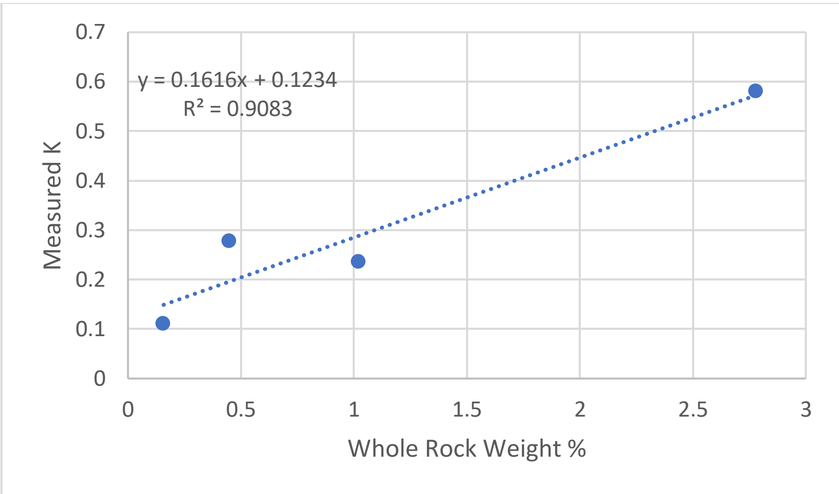


Figure D.13 Whole Rock Weight % Vs Measured K (No.4)

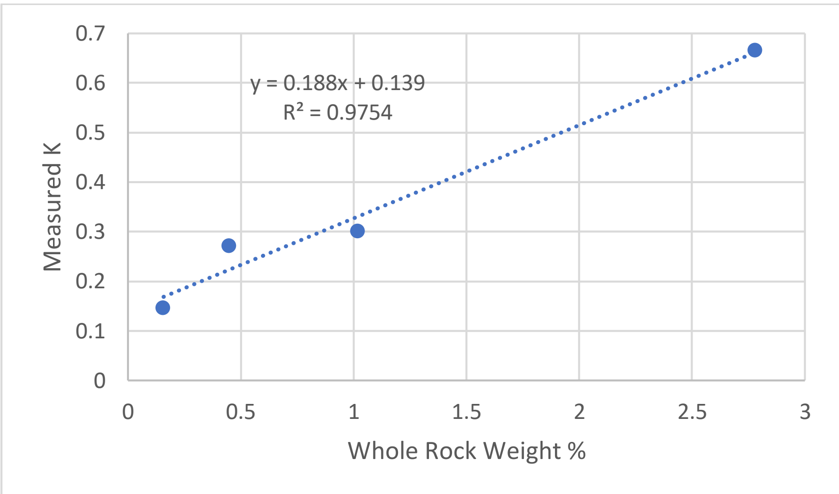


Figure D.14 Whole Rock Weight % Vs Measured K (No.12)

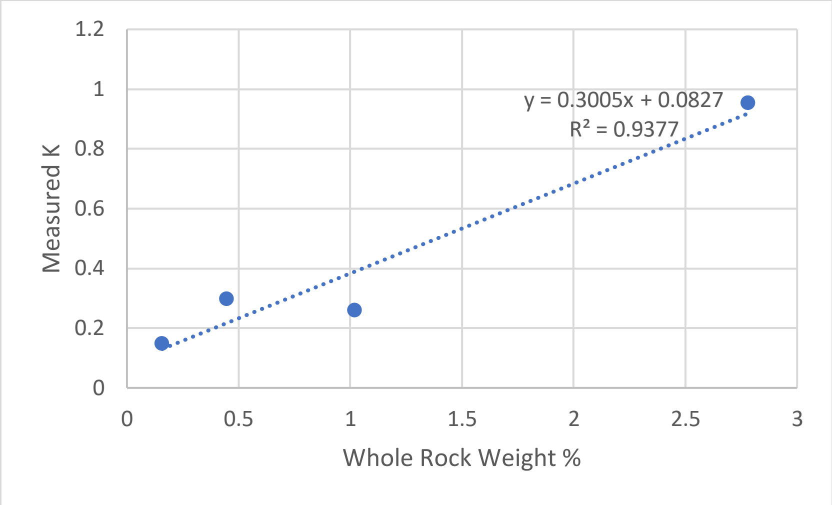

Figure D.15 Whole Rock Weight % Vs Measured K (No.50)

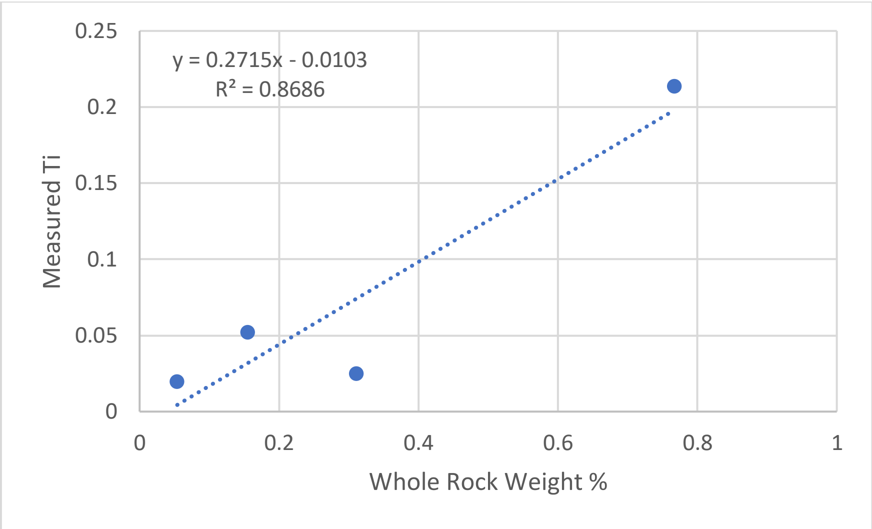

Figure D.16 Whole Rock Weight % Vs Measured Ti (No.4)

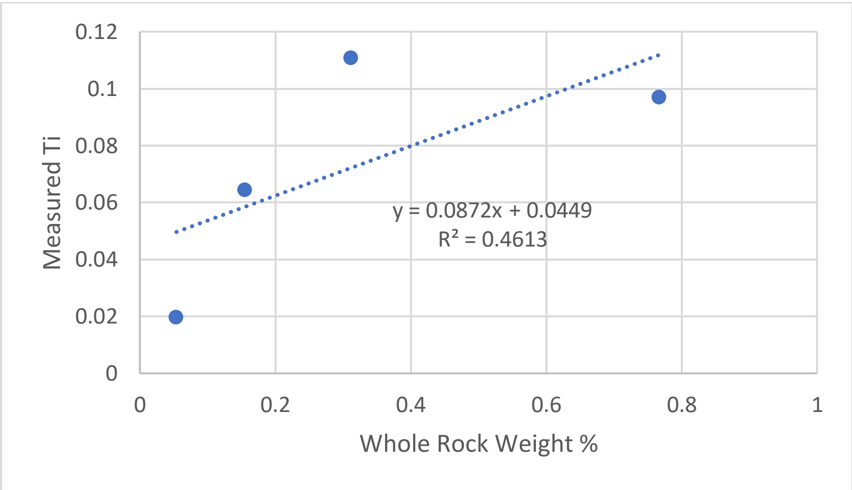

Figure D.17 Whole Rock Weight % Vs Measured Ti (No.12)

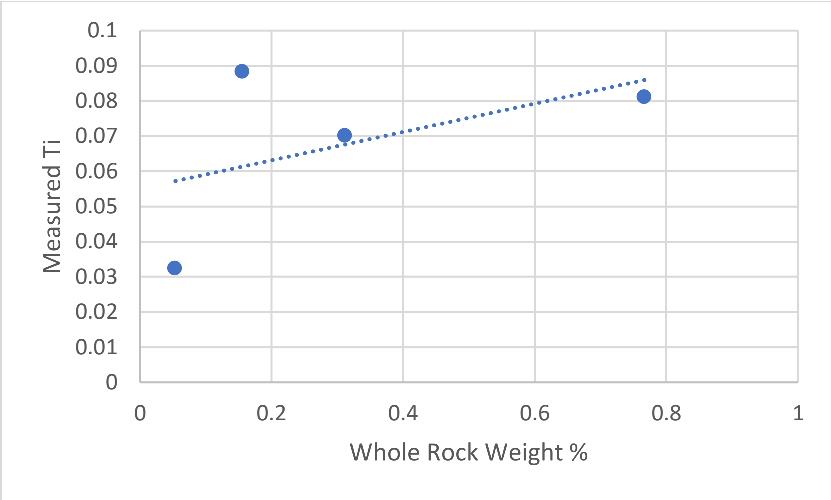


Figure D.18 Whole Rock Weight % Vs Measured Ti (No.50)

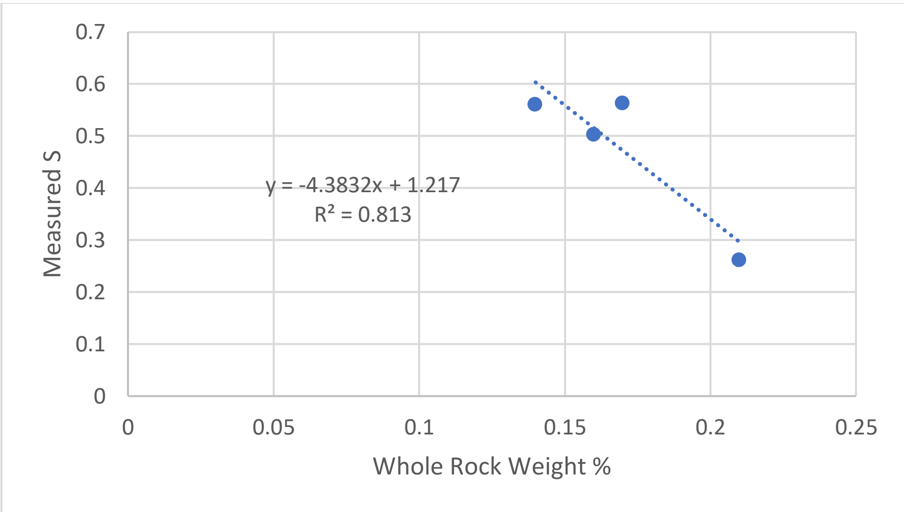


Figure D.19 Whole Rock Weight % Vs Measured S (No.4)

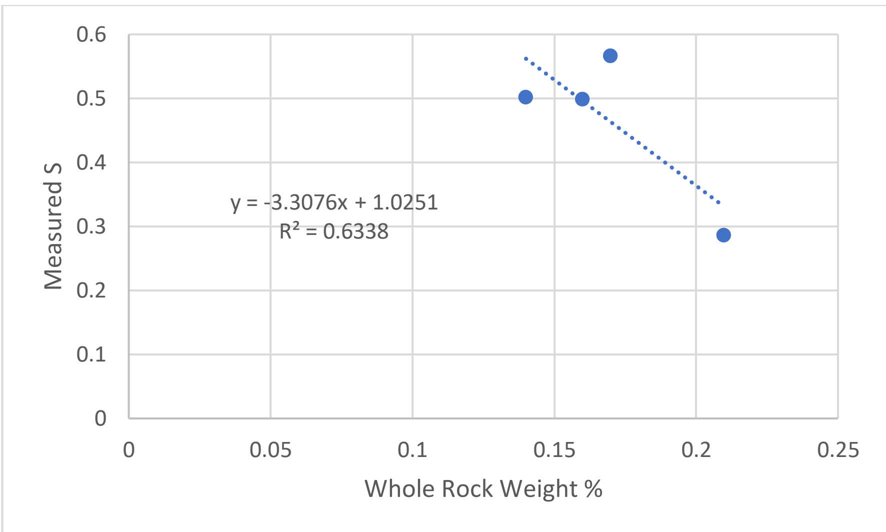


Figure D.20 Whole Rock Weight % Vs Measured S (No.12)

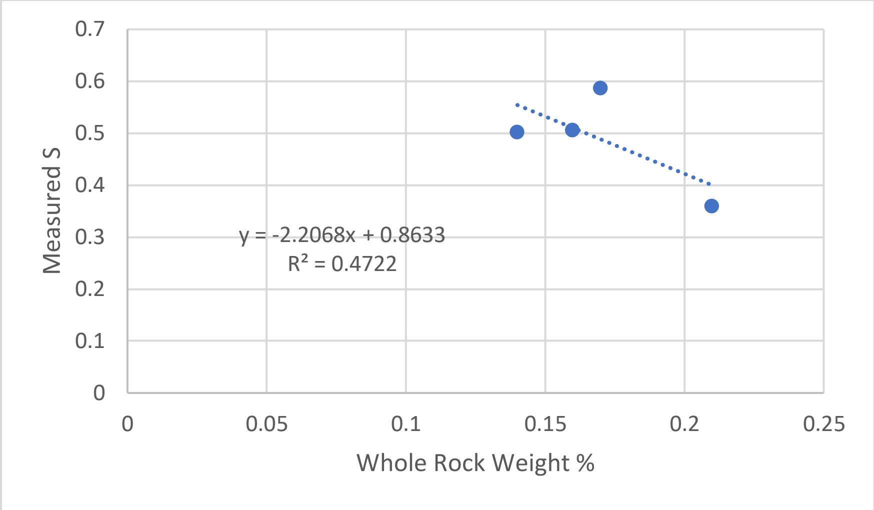


Figure D.21 Whole Rock Weight % Vs Measured S (No.50)

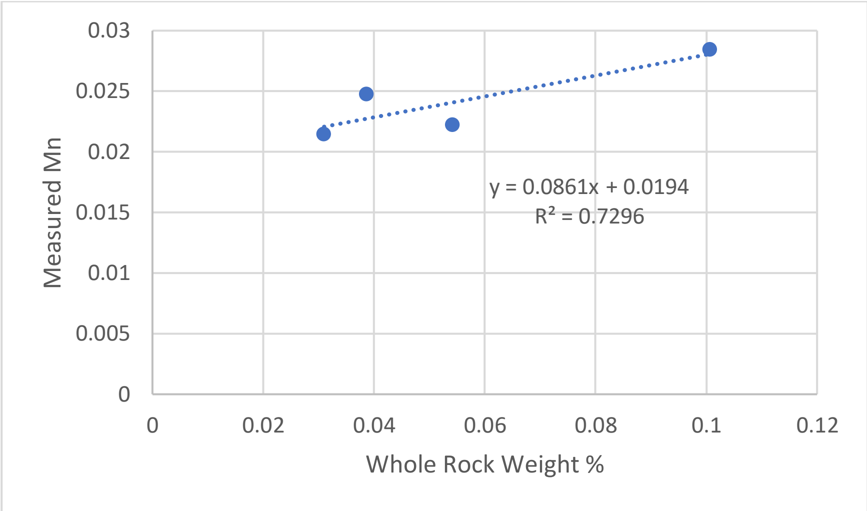


Figure D.22 Whole Rock Weight % Vs Measured Mn (No.4)

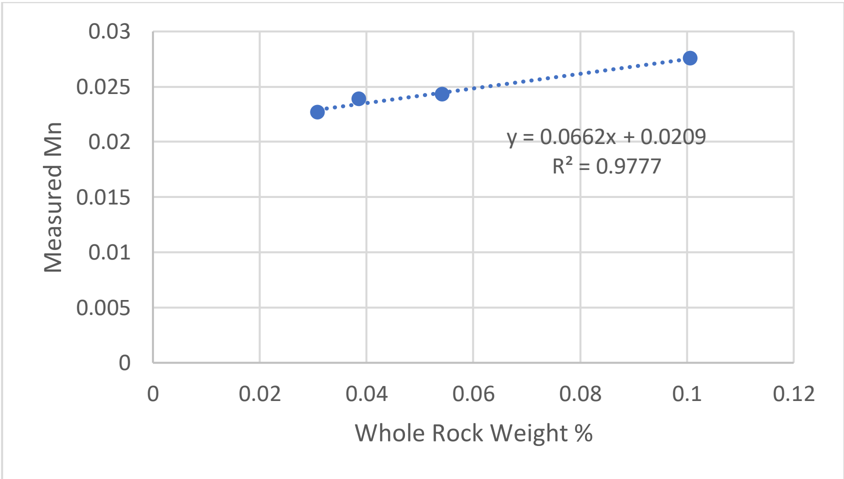


Figure D.23 Whole Rock Weight % Vs Measured Mn (No.12)

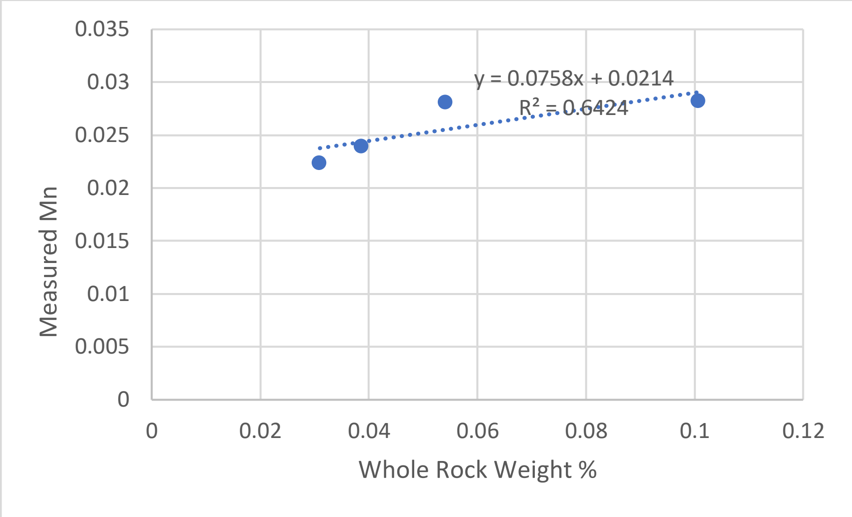


Figure D.24 Whole Rock Weight % Vs Measured Mn (No.50)

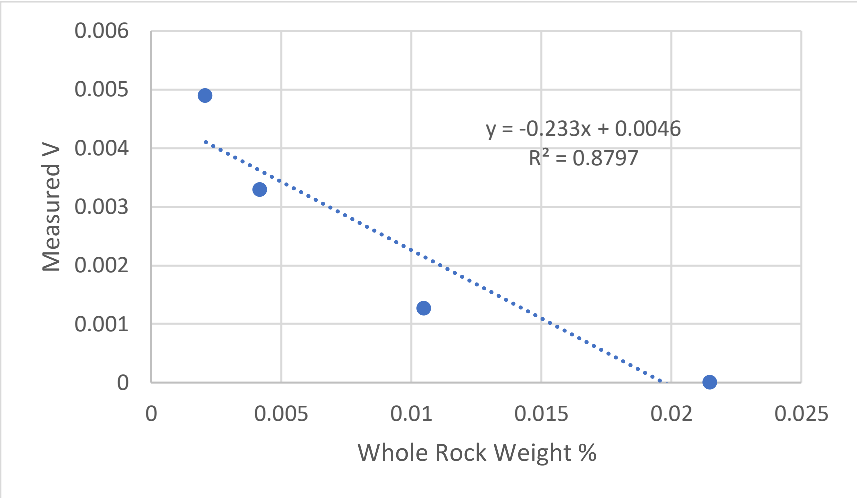


Figure D.25 Whole Rock Weight % Vs Measured V (No.4)

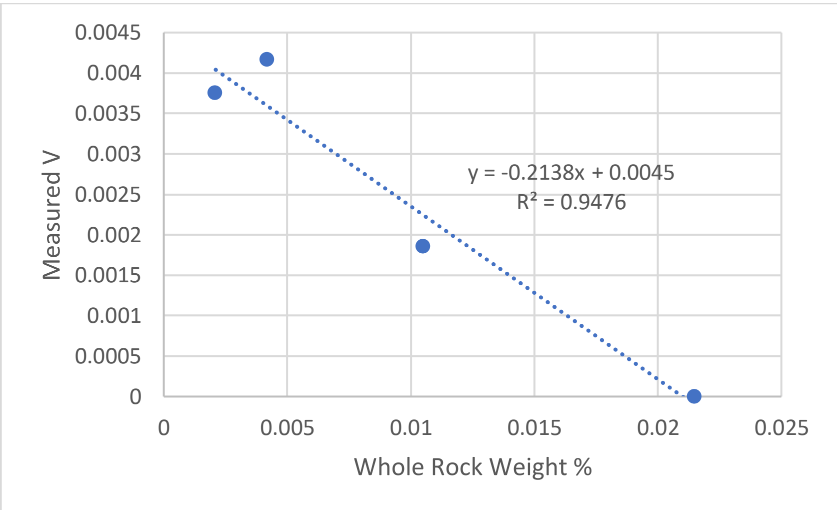


Figure D.26 Whole Rock Weight % Vs Measured V (No.12)

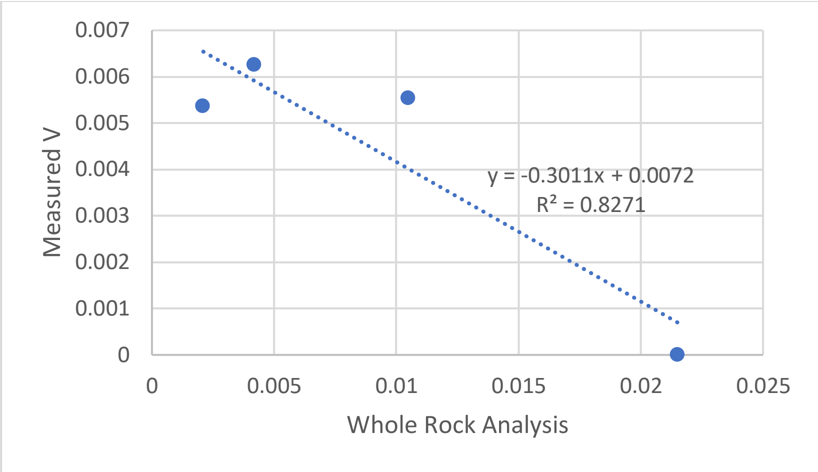

Figure D.27 Whole Rock Weight % Vs Measured V (No.50)

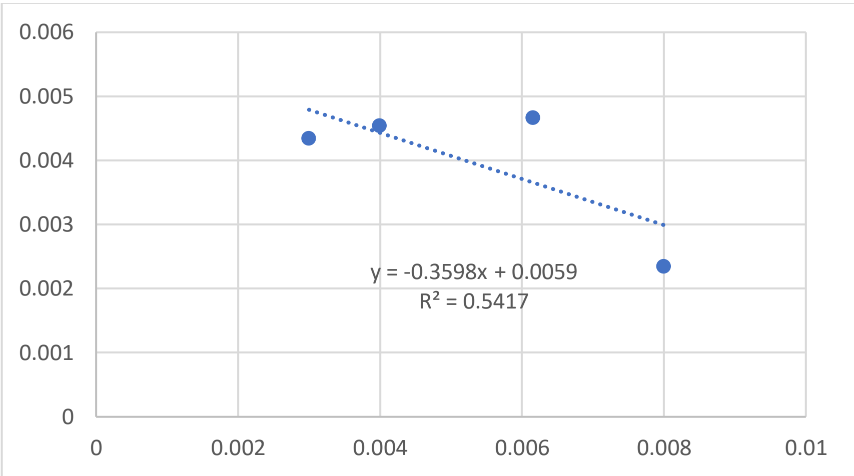

Figure D.28 Whole Rock Weight % Vs Measured Cr (No.4)

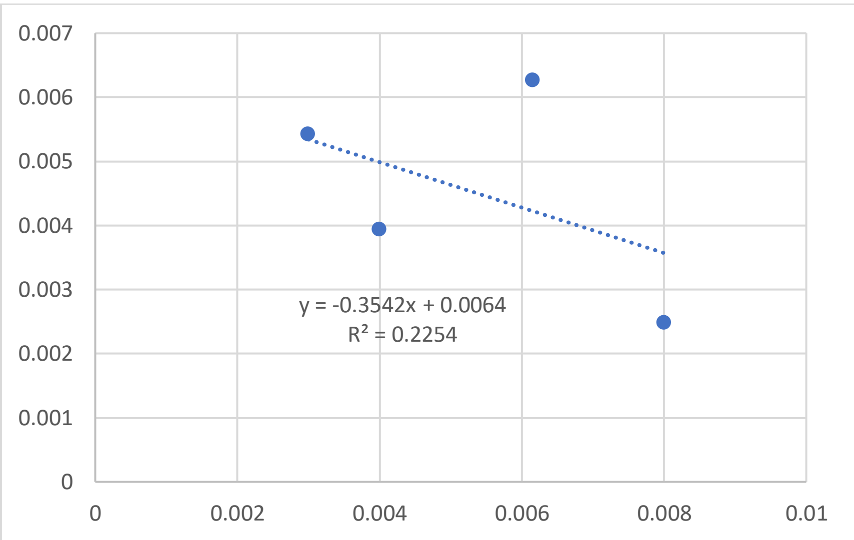

Figure D.29 Whole Rock Weight % Vs Measured Cr (No.12)

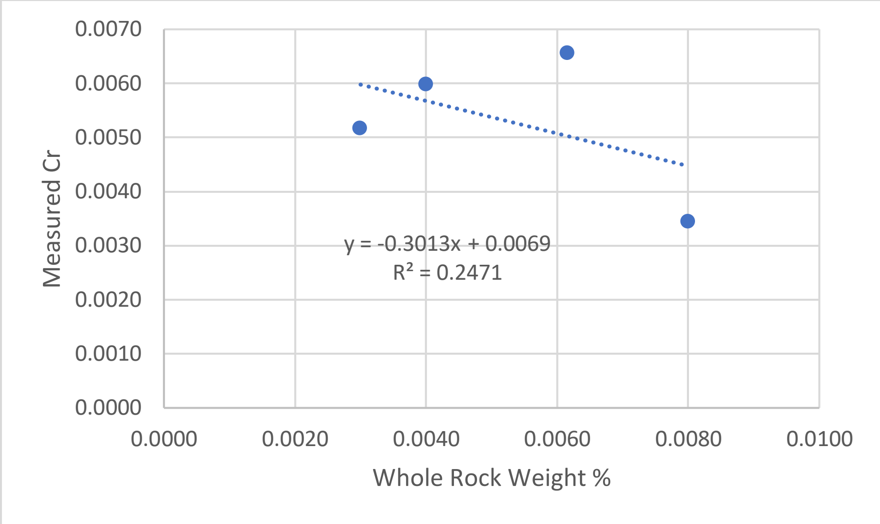


Figure D.30 Whole Rock Weight % Vs Measured Cr (No.50)

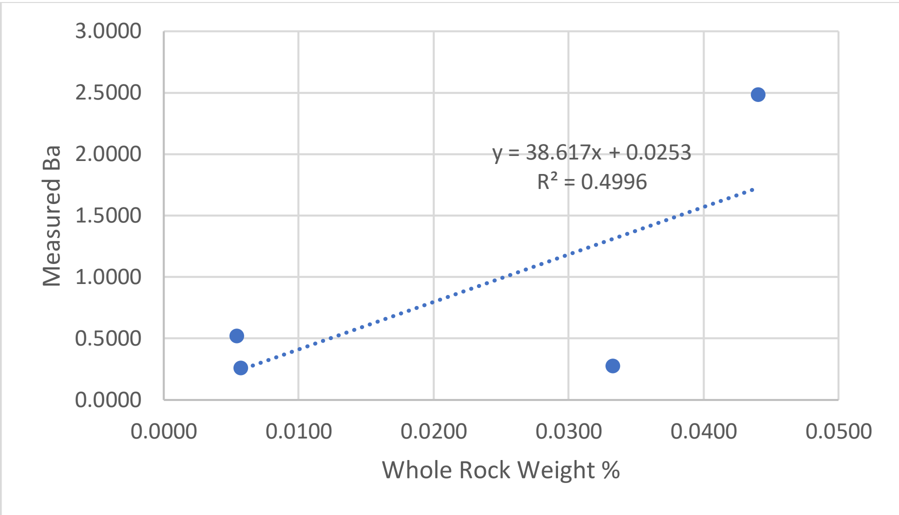


Figure D.31 Whole Rock Weight % Vs Measured Ba (No.4)

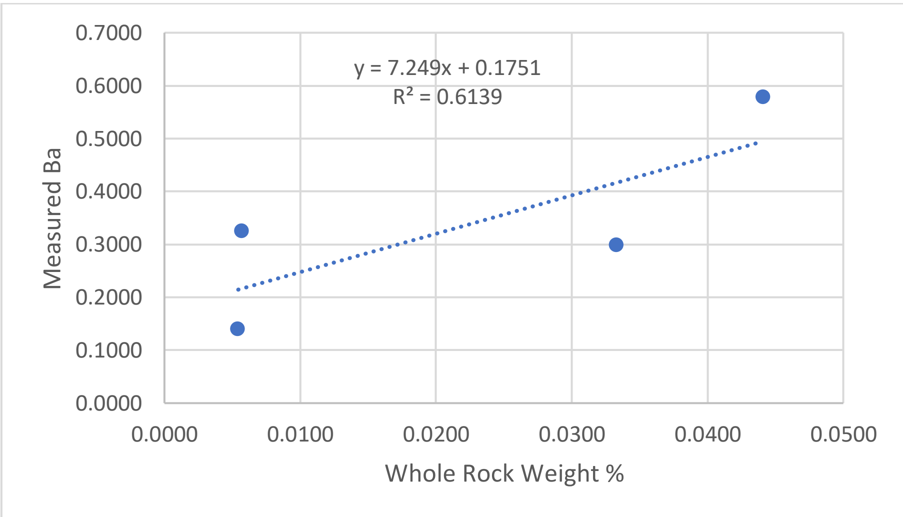

Figure D.32 Whole Rock Weight % Vs Measured Ba (No.12)

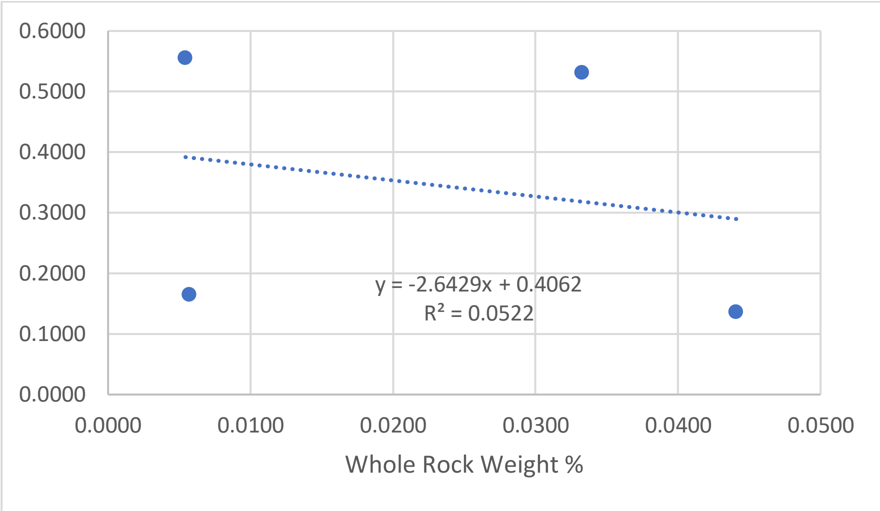


Figure D.33 Whole Rock Weight % Vs Measured Ba (No.50)

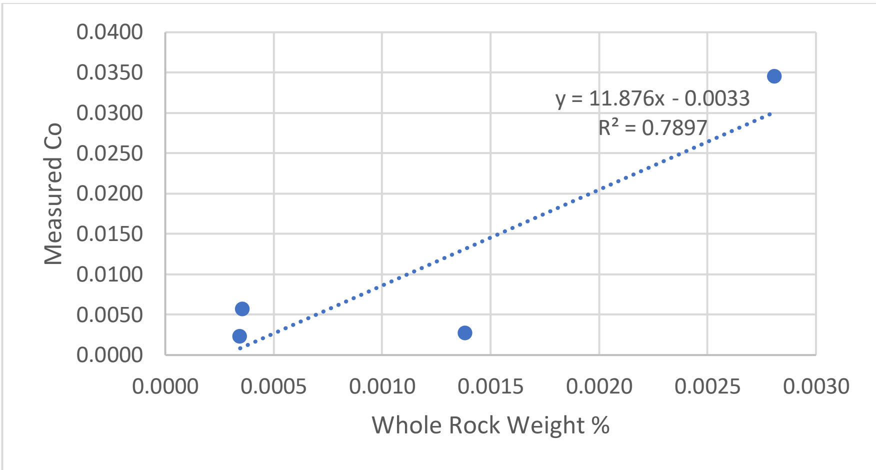


Figure D.34 Whole Rock Weight % Vs Measured Co (No.4)

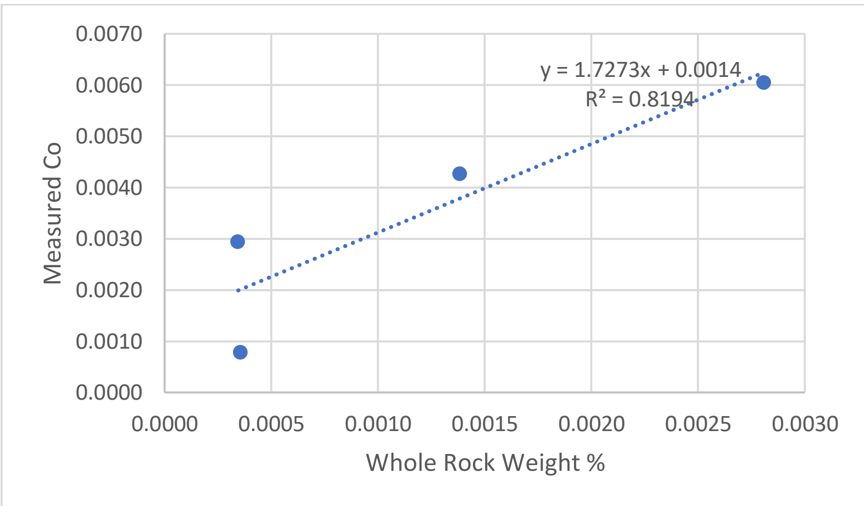


Figure D.35 Whole Rock Weight % Vs Measured Co (No.12)

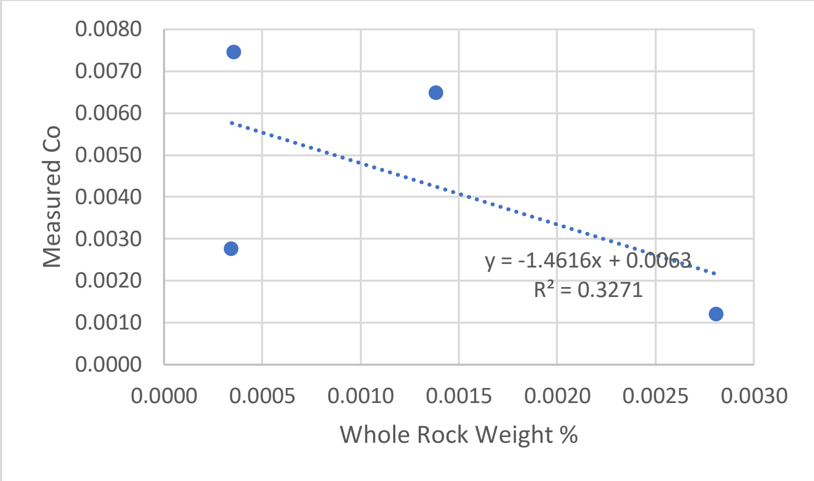


Figure D.36 Whole Rock Weight % Vs Measured Co (No.50)

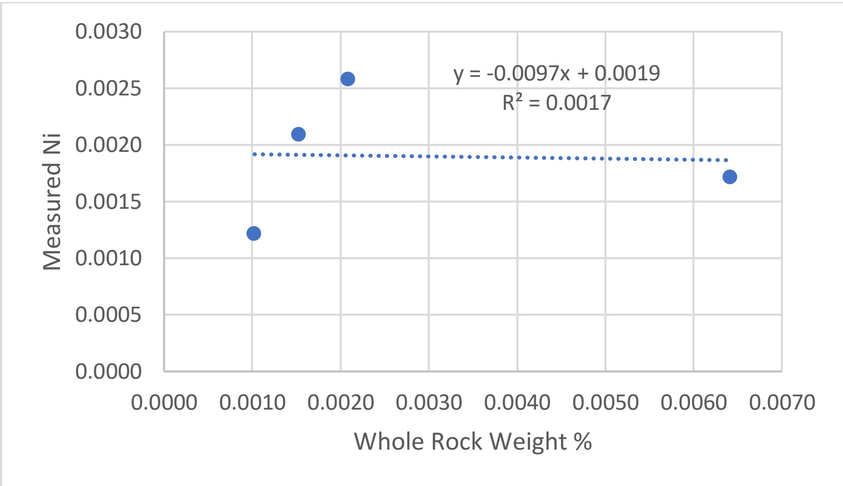


Figure D.37 Whole Rock Weight % Vs Measured Ni (No.4)

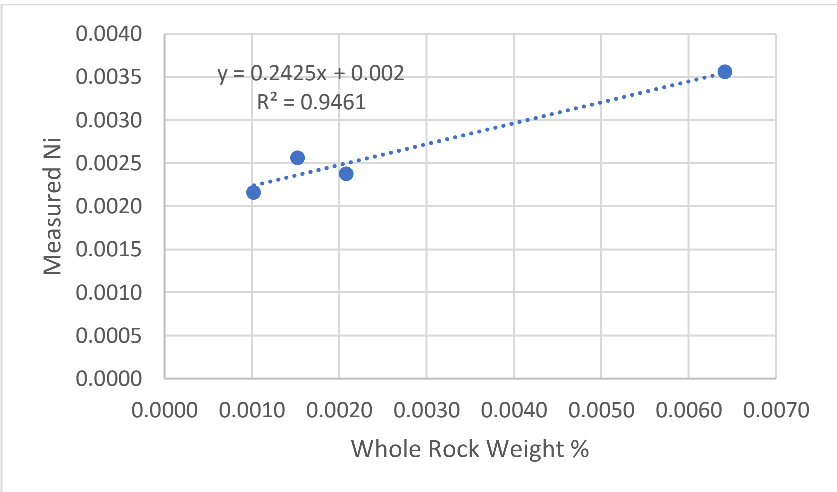


Figure D.38 Whole Rock Weight % Vs Measured Ni (No.12)

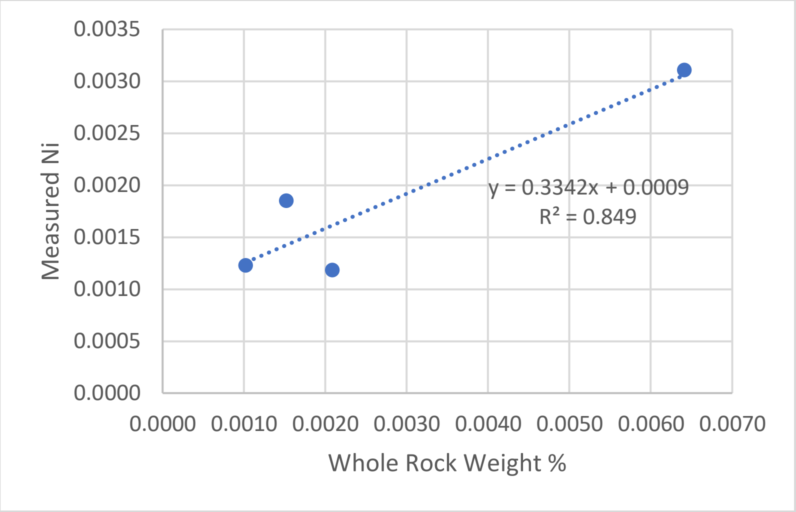


Figure D.39 Whole Rock Weight % Vs Measured Ni (No.50)

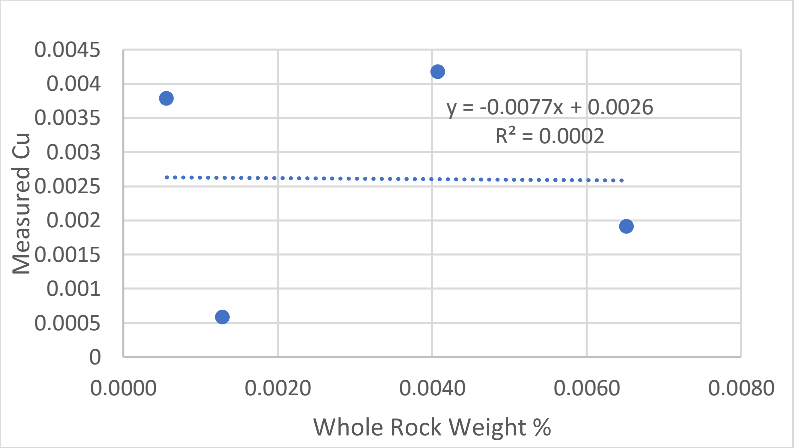


Figure D.40 Whole Rock Weight % Vs Measured Cu (No.4)

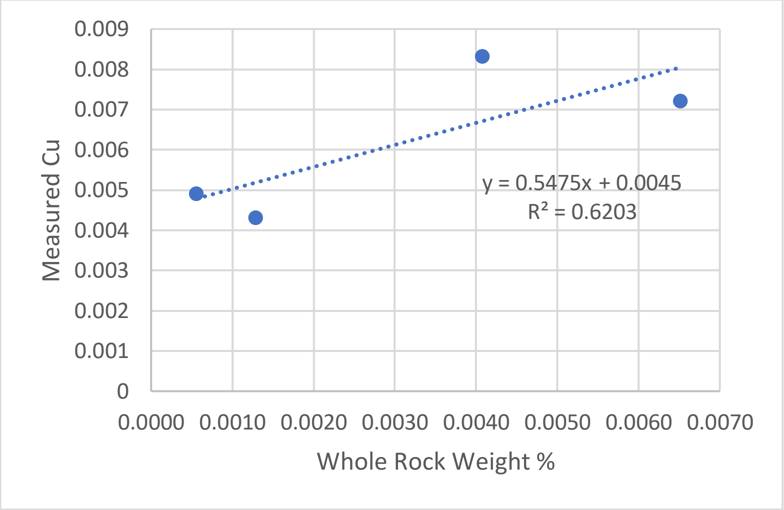


Figure D.41 Whole Rock Weight % Vs Measured Cu (No.12)

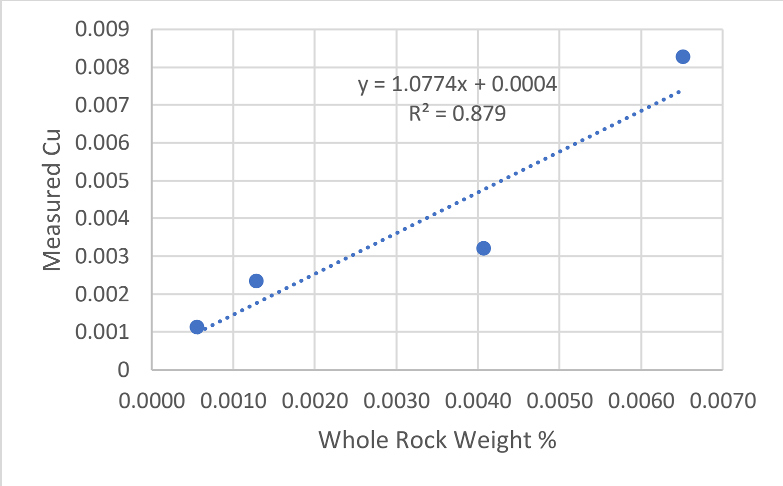


Figure D.42 Whole Rock Weight % Vs Measured Cu (No.50)

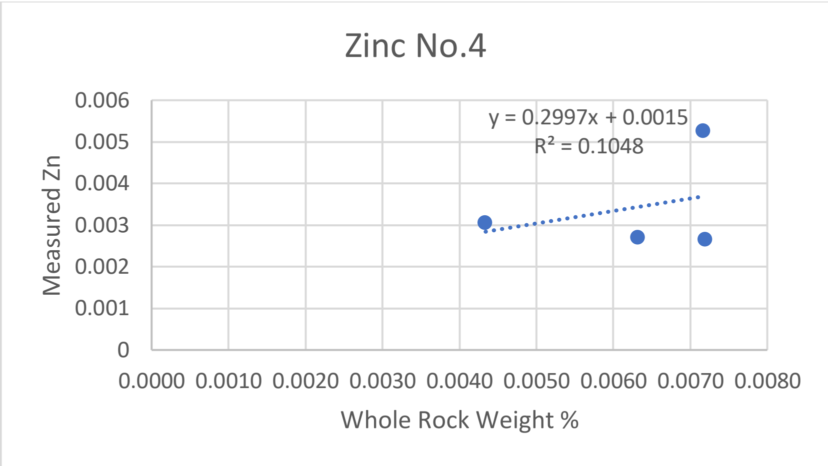


Figure D.43 Whole Rock Weight % Vs Measured Zn (No.4)

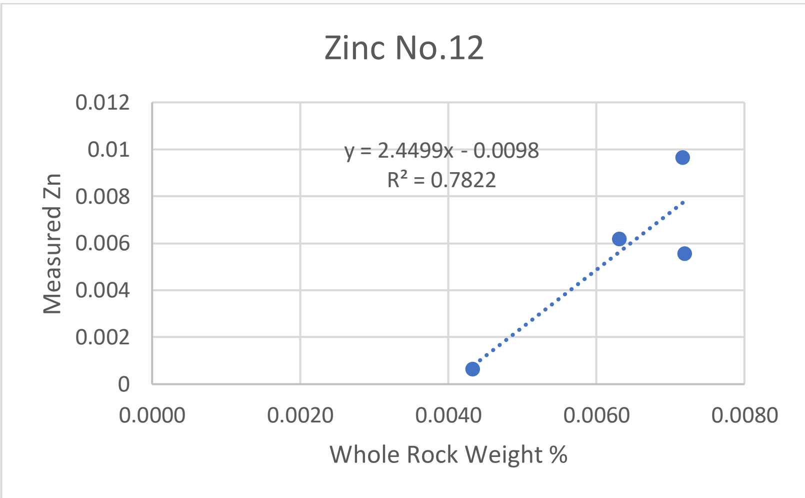


Figure D.44 Whole Rock Weight % Vs Measured Zn (No.12)

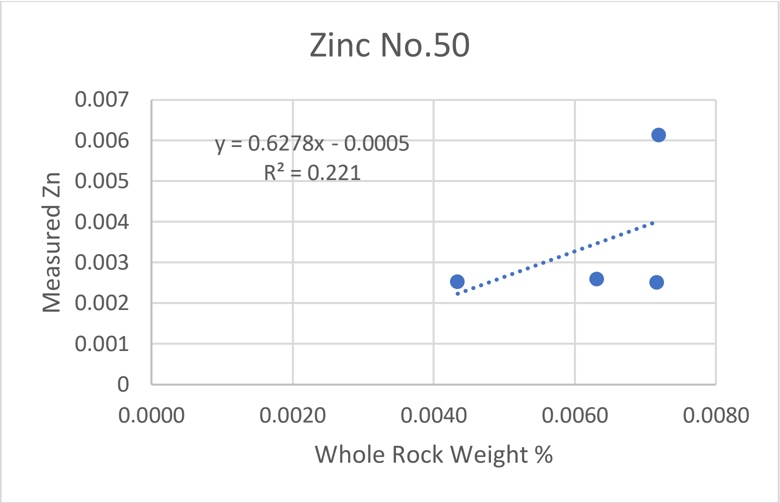


Figure D.45 Whole Rock Weight % Vs Measured Zn (No.50)

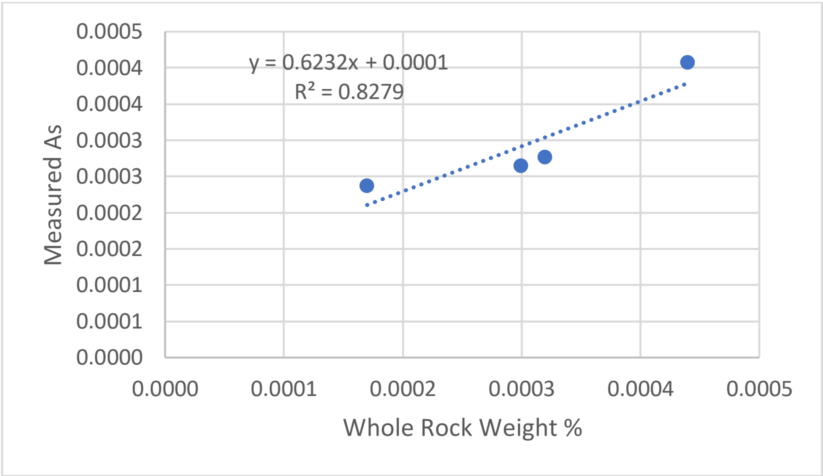


Figure D.46 Whole Rock Weight % Vs Measured As (No.4)

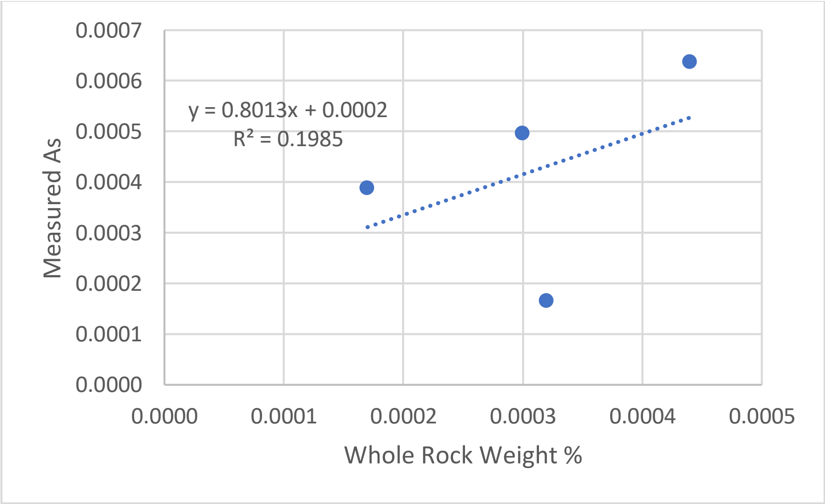

Figure D.47 Whole Rock Weight % Vs Measured As (No.12)

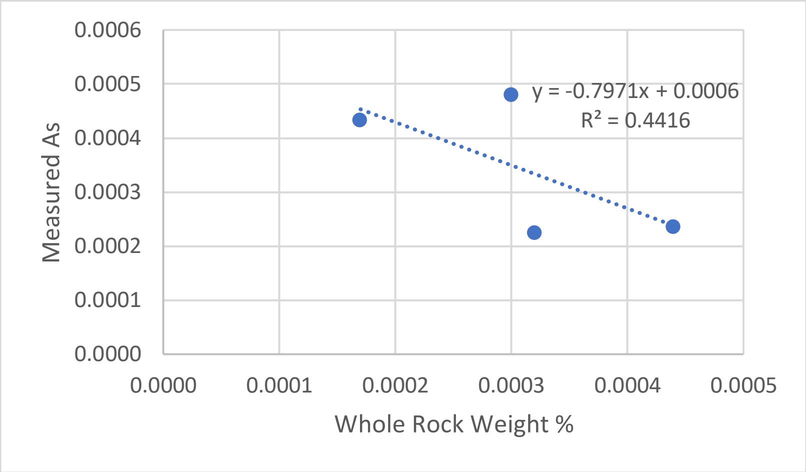


Figure D.48 Whole Rock Weight % Vs Measured As (No.50)

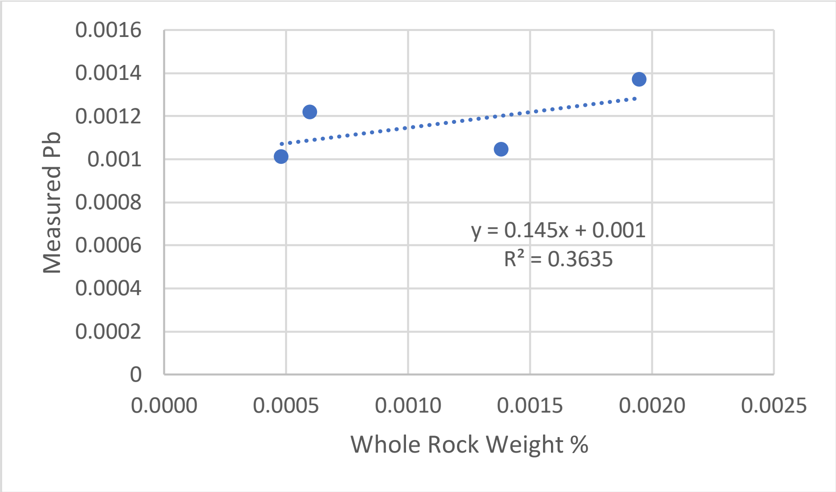


Figure D.49 Whole Rock Weight % Vs Measured Pb (No.4)

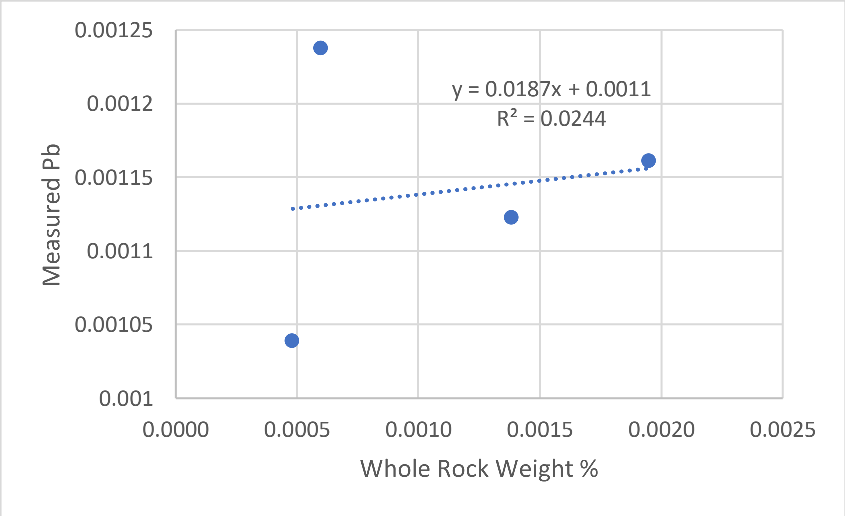


Figure D.50 Whole Rock Weight % Vs Measured Pb (No.12)

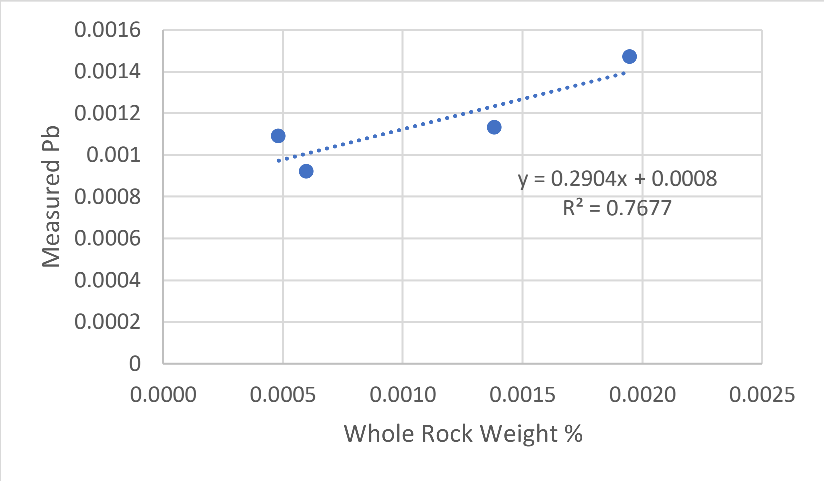


Figure D.51 Whole Rock Weight % Vs Measured Pb (No.50)

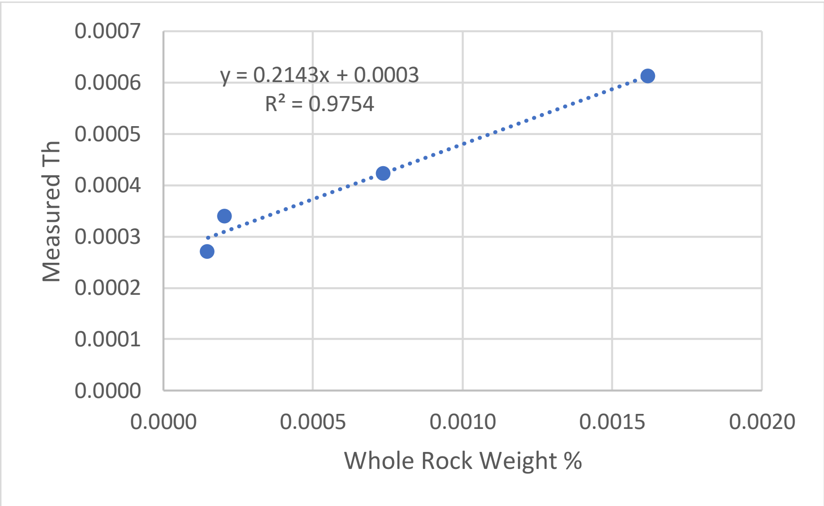


Figure D.52 Whole Rock Weight % Vs Measured Th (No.4)

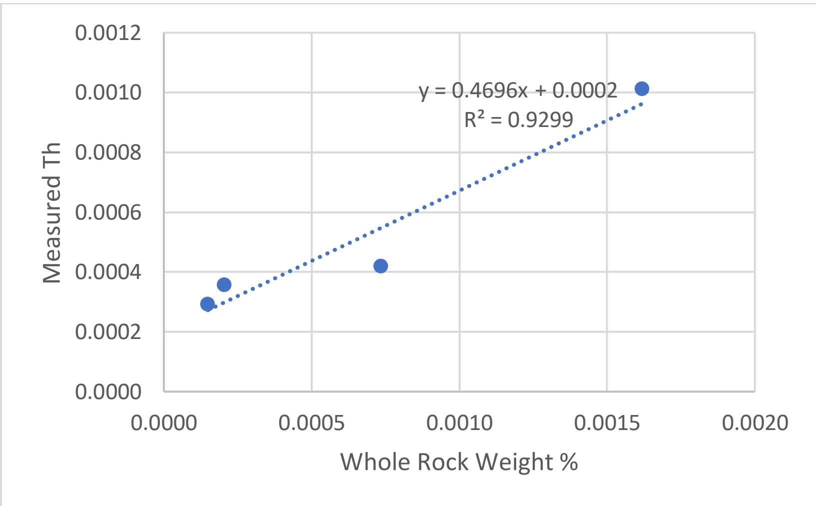


Figure D.53 Whole Rock Weight % Vs Measured Th (No.12)

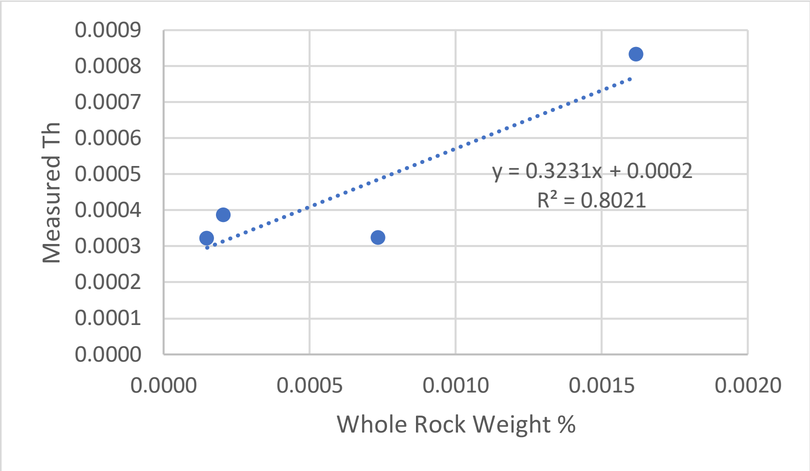


Figure D.54 Whole Rock Weight % Vs Measured Th (No.50)

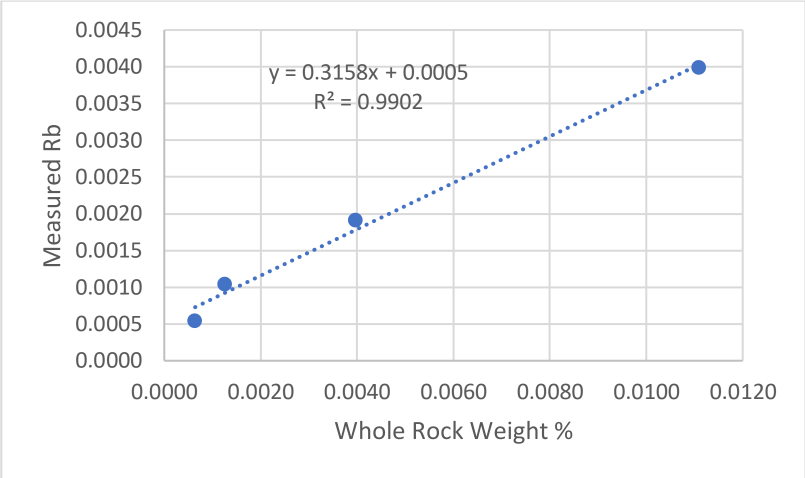


Figure D.55 Whole Rock Weight % Vs Measured Rb (No.4)

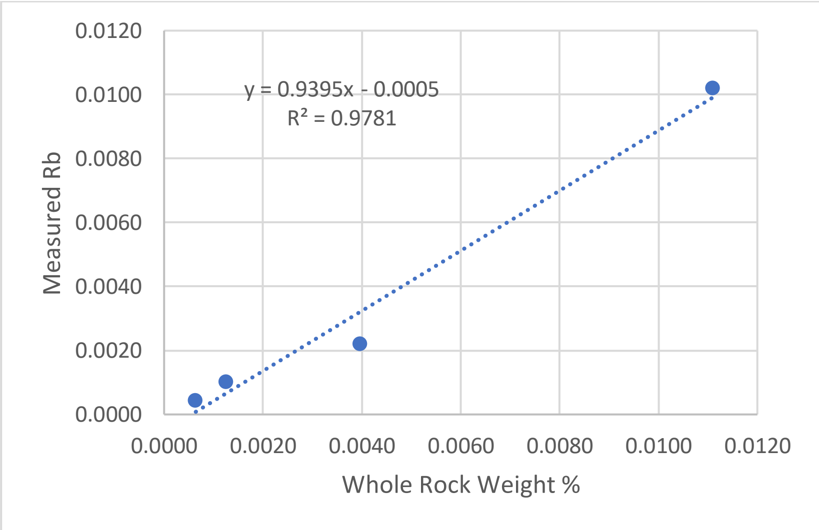


Figure D.56 Whole Rock Weight % Vs Measured Rb (No.12)

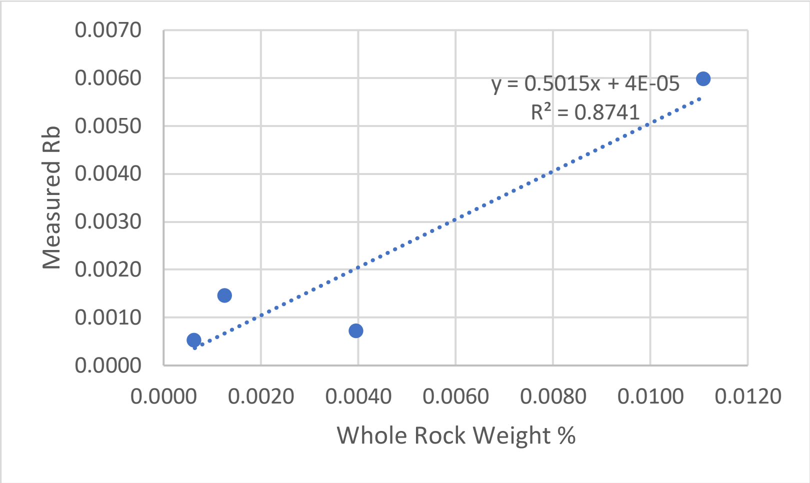


Figure D.57 Whole Rock Weight % Vs Measured Rb (No.50)

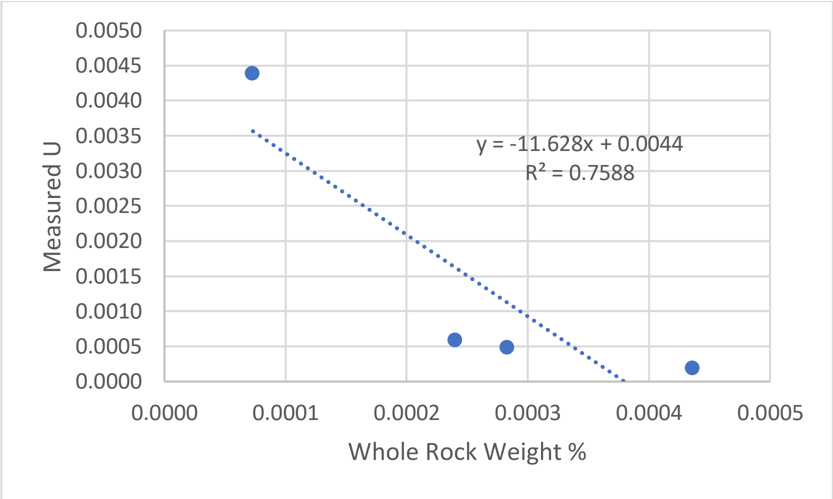


Figure D.58 Whole Rock Weight % Vs Measured U (No.4)

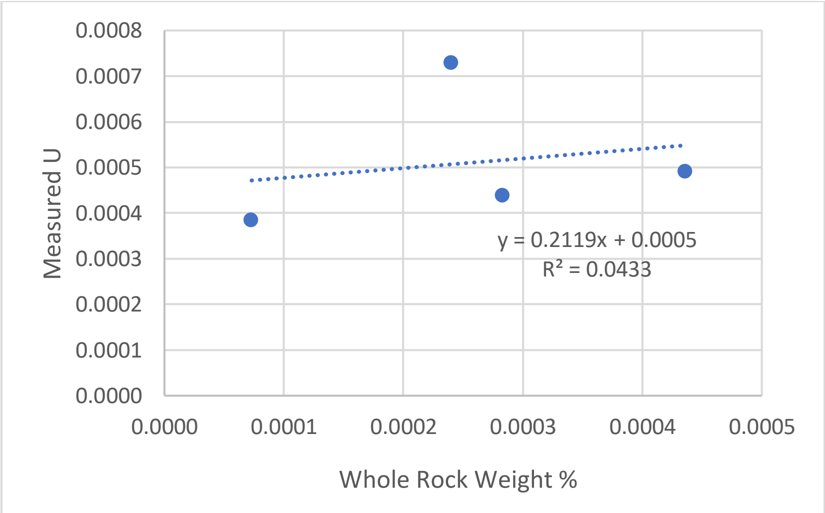

Figure D.59 Whole Rock Weight % Vs Measured U (No.12)

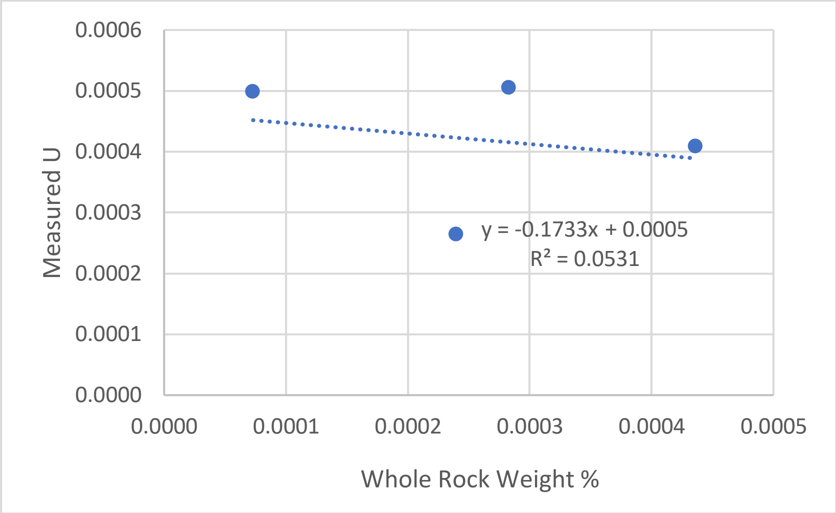


Figure D.60 Whole Rock Weight % Vs Measured U (No.50)

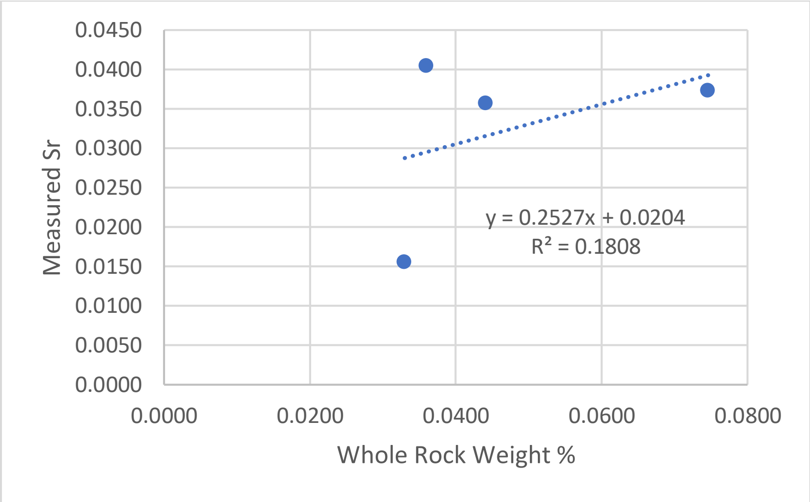


Figure D.61 Whole Rock Weight % Vs Measured Sr (No.4)

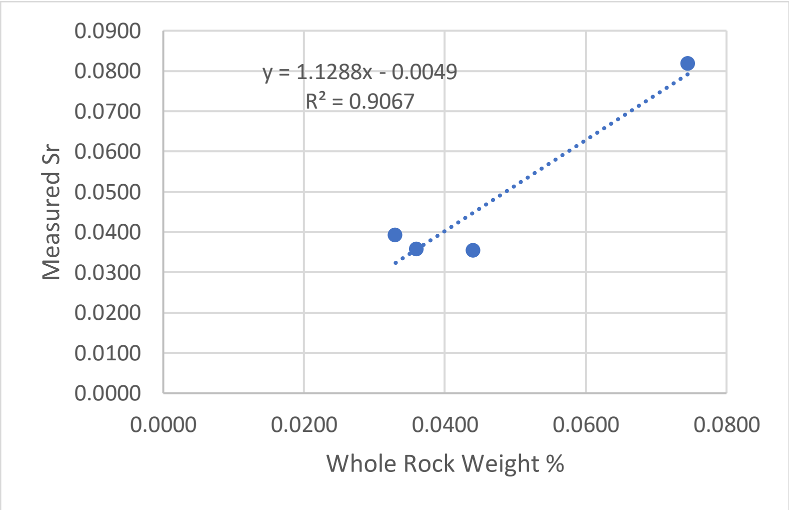

Figure D.62 Whole Rock Weight % Vs Measured Sr (No.12)

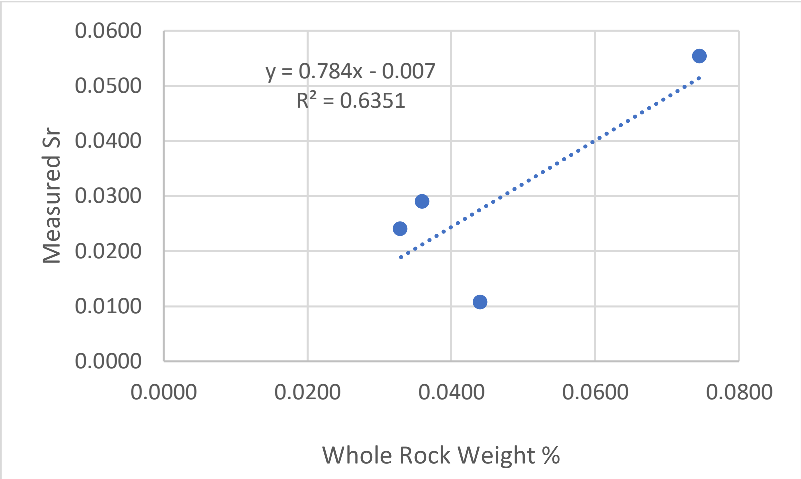


Figure D.63 Whole Rock Weight % Vs Measured Sr (No.50)

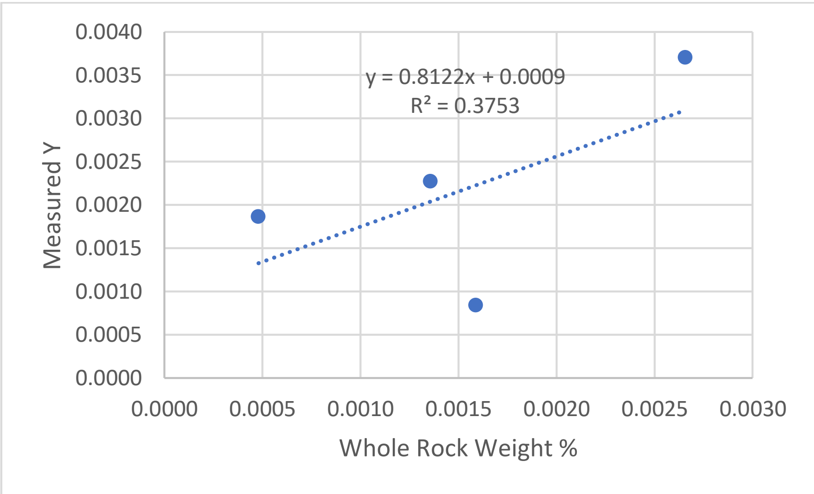


Figure D.64 Whole Rock Weight % Vs Measured Y (No.4)

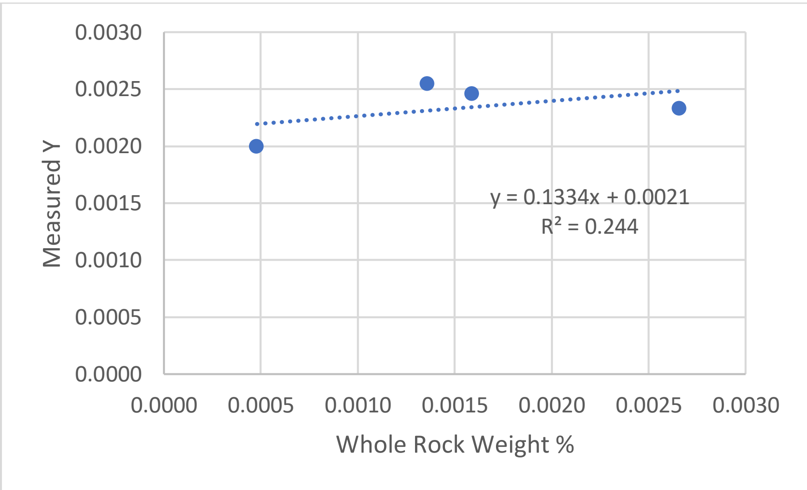

Figure D.65 Whole Rock Weight % Vs Measured Y (No.12)

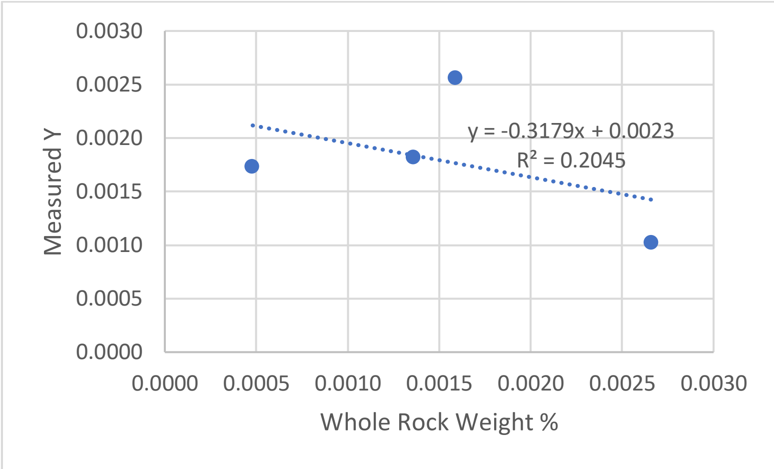


Figure D.66 Whole Rock Weight % Vs Measured Y (No.50)

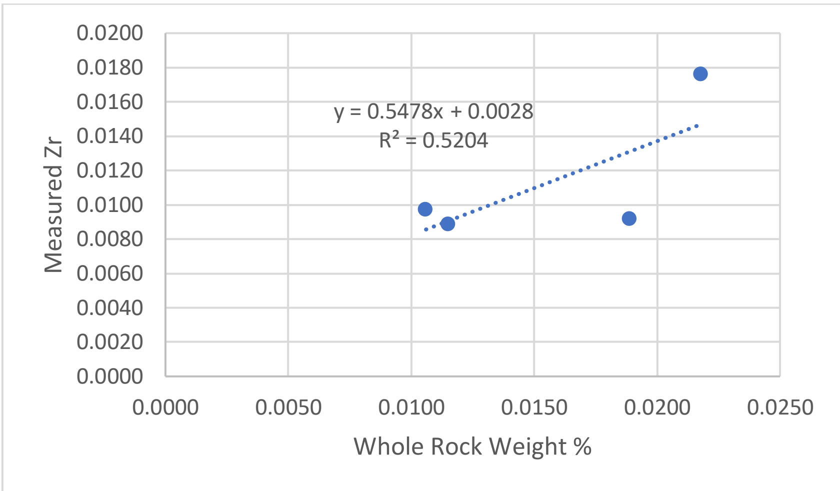


Figure D.67 Whole Rock Weight % Vs Measured Zr (No.4)

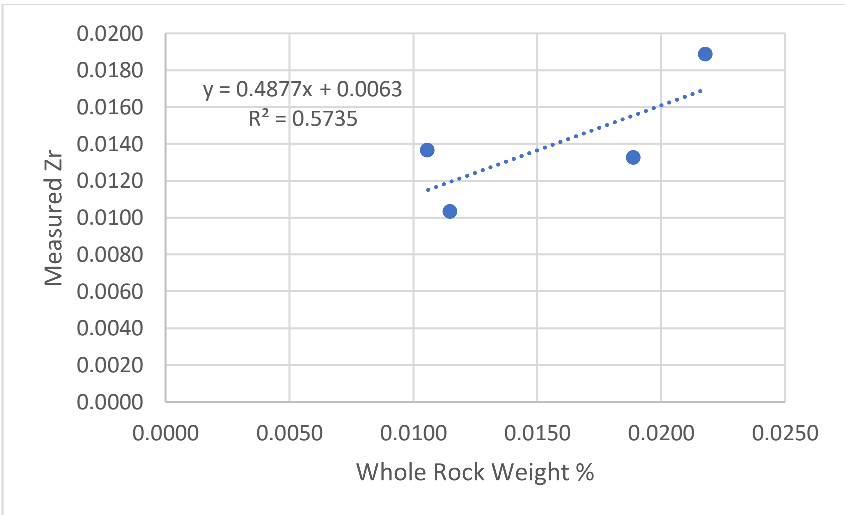

Figure D.68 Whole Rock Weight % Vs Measured Zr (No.12)

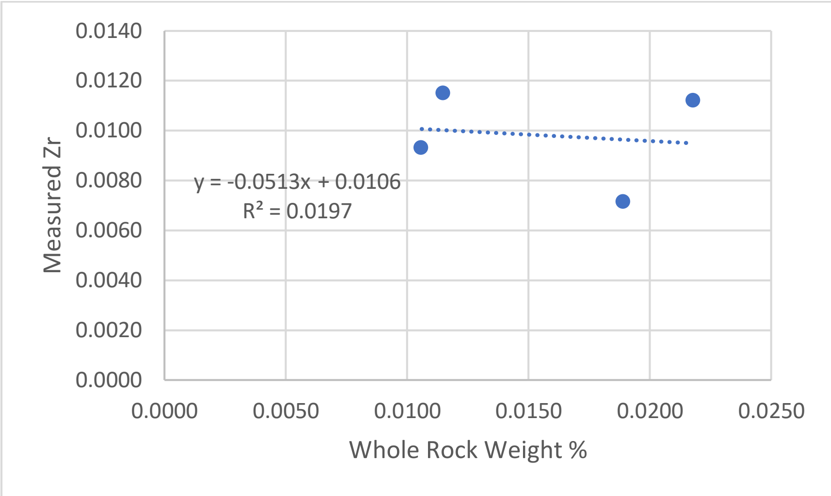


Figure D.69 Whole Rock Weight % Vs Measured Zr (No.50)

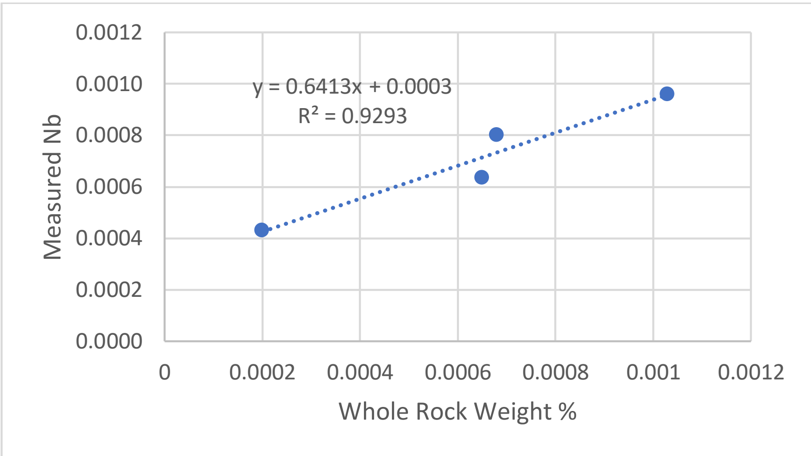


Figure D.70 Whole Rock Weight % Vs Measured Nb (No.4)

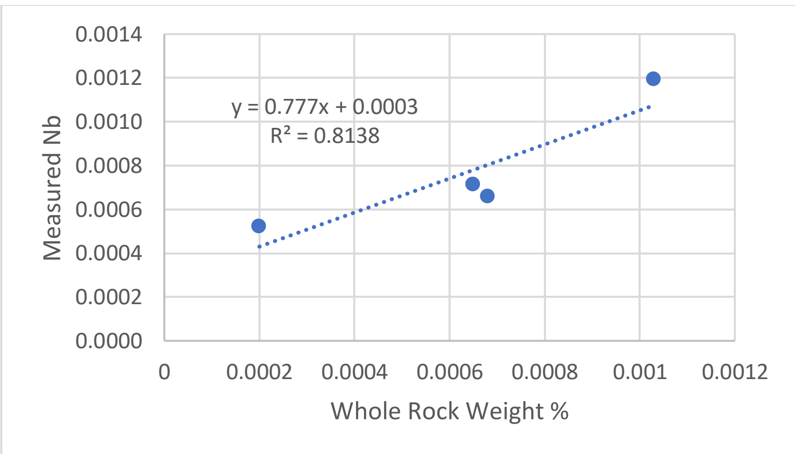


Figure D.71 Whole Rock Weight % Vs Measured Nb (No.12)

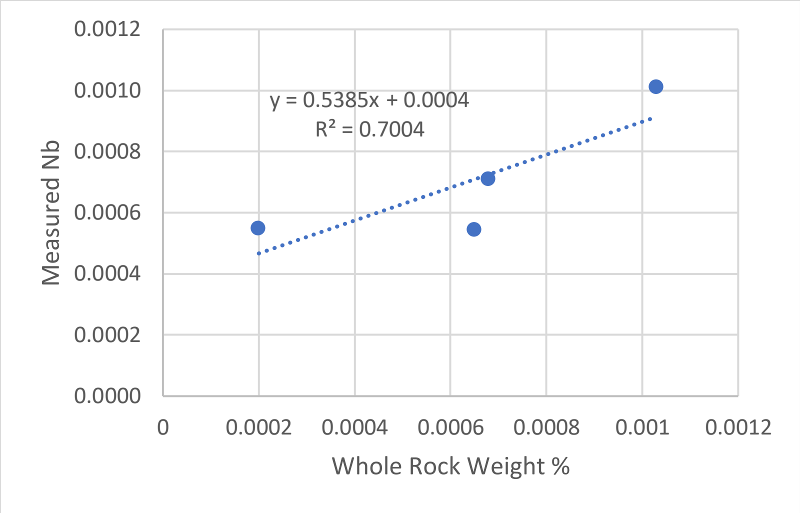


Figure D.72 Whole Rock Weight % Vs Measured Nb (No.50)

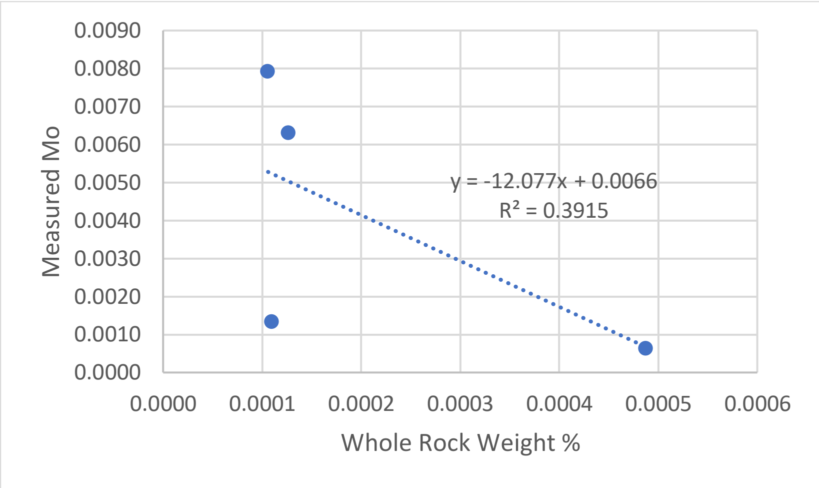


Figure D.73 Whole Rock Weight % Vs Measured Mo (No.4)

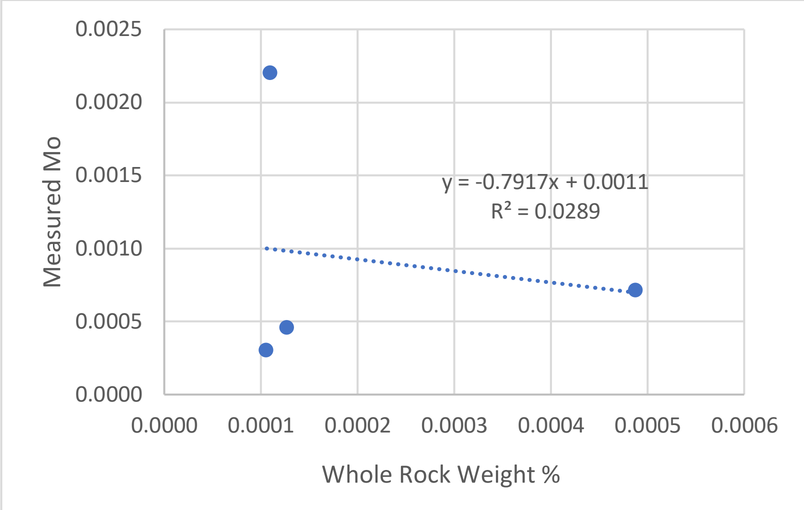


Figure D.74 Whole Rock Weight % Vs Measured Mo (No.12)

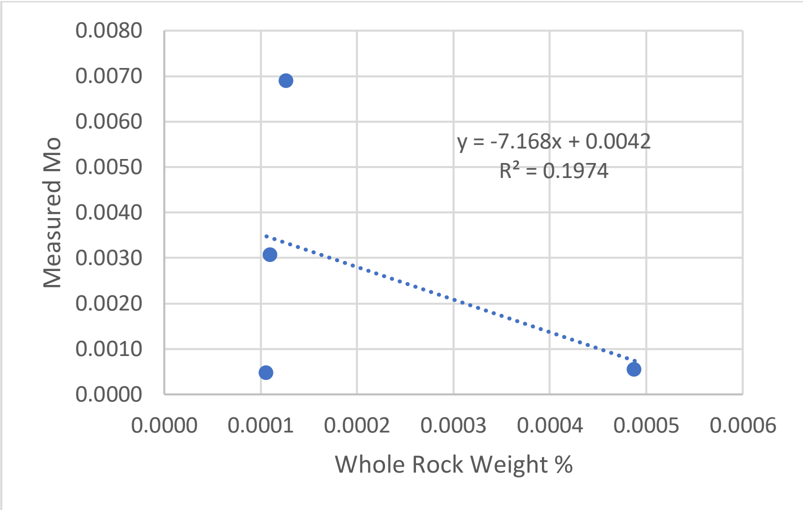


Figure D.75 Whole Rock Weight % Vs Measured Mo (No.50)

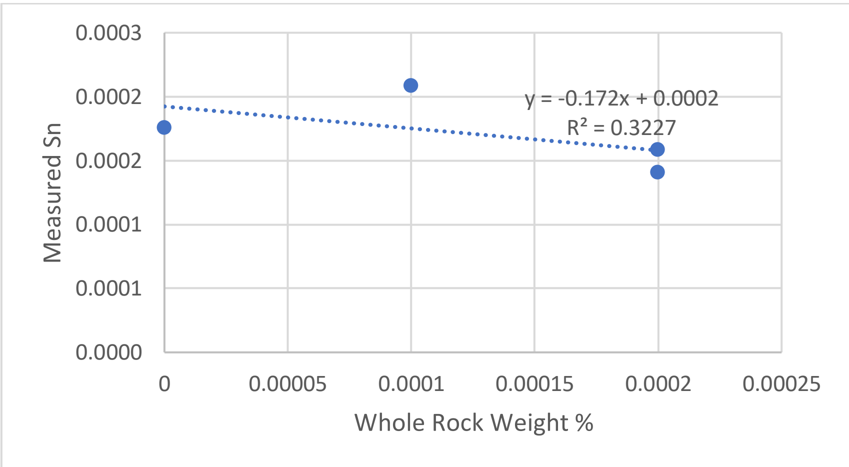


Figure D.76 Whole Rock Weight % Vs Measured Sn (No.4)

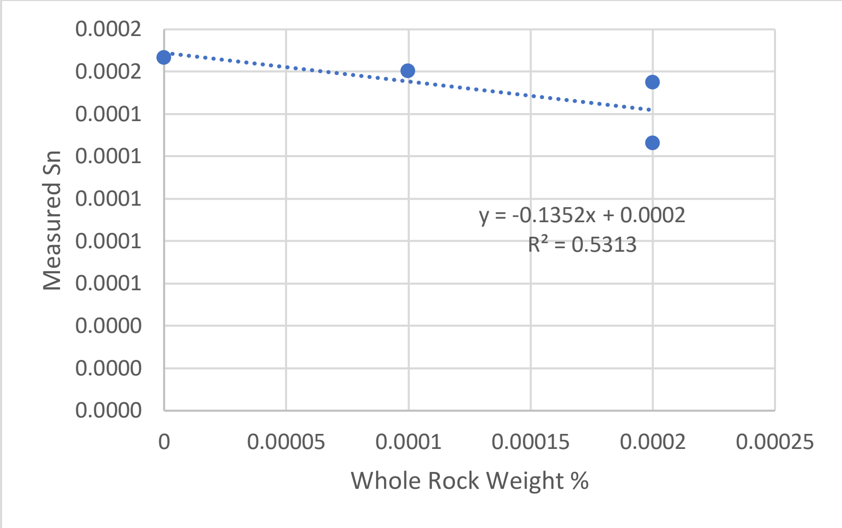


Figure D.77 Whole Rock Weight % Vs Measured Sn (No.12)

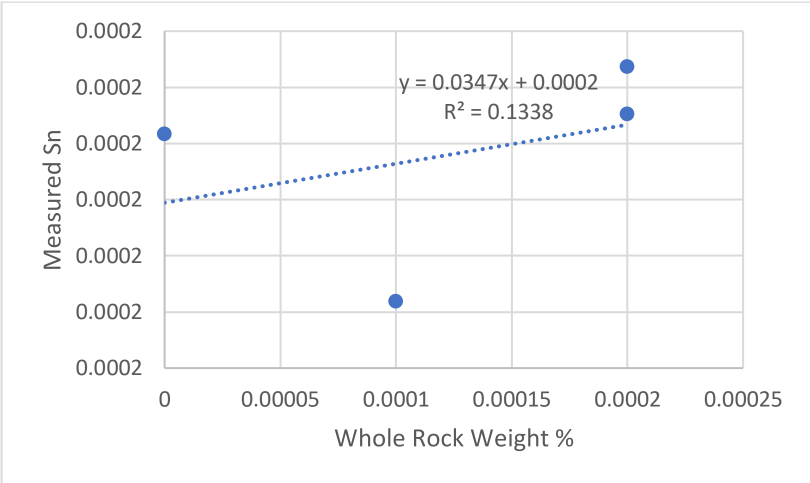


Figure D.78 Whole Rock Weight % Vs Measured Sn (No.50)

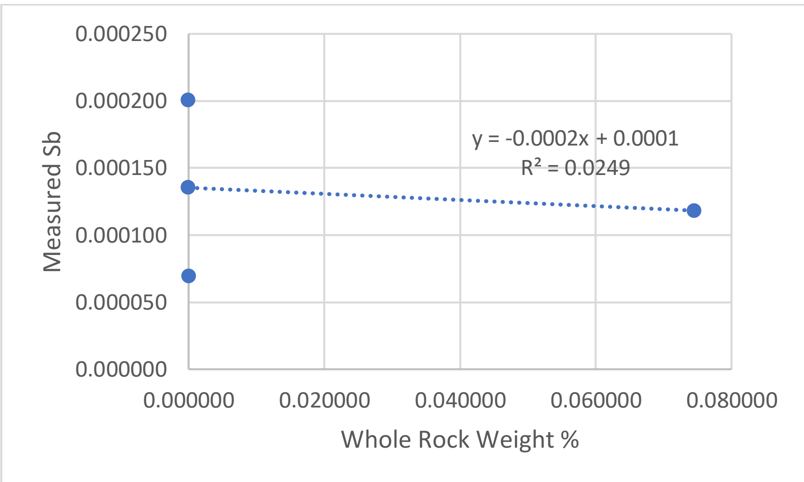


Figure D.79 Whole Rock Weight % Vs Measured Sb (No.4)

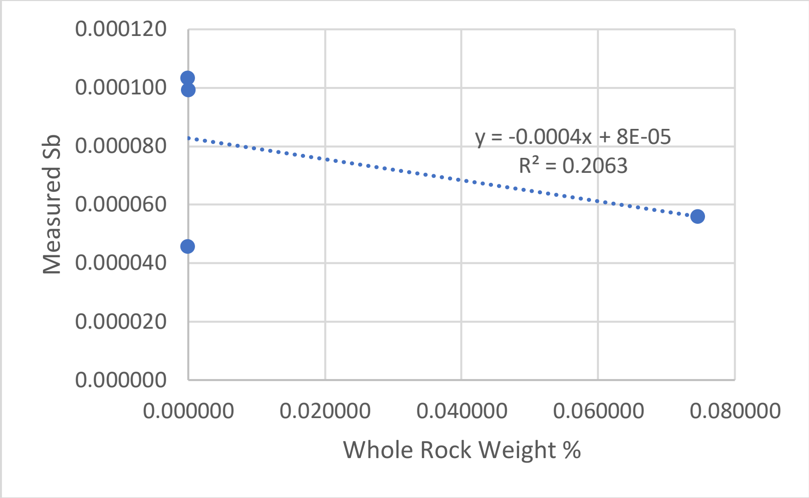


Figure D.80 Whole Rock Weight % Vs Measured Sb (No.12)

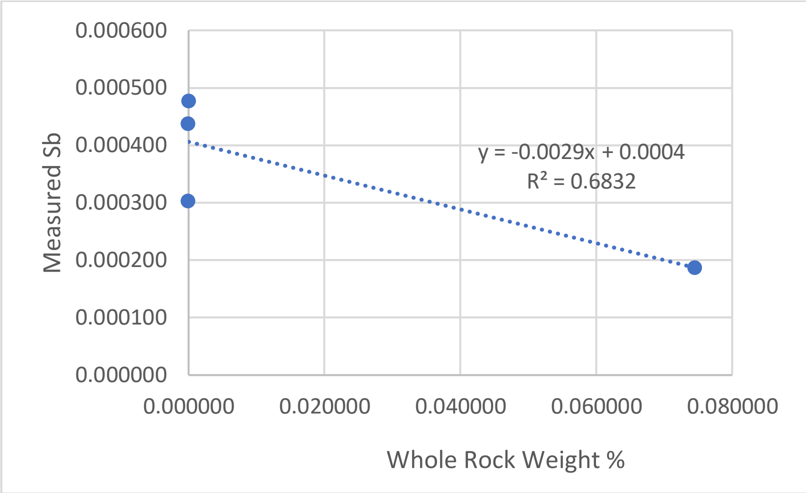


Figure D.81 Whole Rock Weight % Vs Measured Sb (No.50)

# APPENDIX E: LABORATORY REPORT FOR WHOLE ROCK ANALYSIS

ALS USA Inc.
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www.alsglobal.com/geochemistry

To: UNIVERSITY OF NORTH CAROLINA AT CHARLOTTE
9201 UNIVERSITY CITY BLVD
CHARLOTTE NV 28223

Page: 1
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 1-MAY-2020
Account: CHANOR

**CERTIFICATE RE20082600**

Project: Recycled Concrete Aggregate

This report is for 3 Rock samples submitted to our lab in Reno, NV, USA on 13-APR-2020.

The following have access to data associated with this certificate:
TARA CAVALLINE

**SAMPLE PREPARATION**

| ALS CODE | DESCRIPTION |
|---|---|
| WEI-21 | Received Sample Weight |
| LOG-22 | Sample login - Rcd w/o BarCode |
| SND-ALS | Send samples to internal laboratory |
| PUL-31 | Pulverize up to 250g 85% <75 um |

**ANALYTICAL PROCEDURES**

| ALS CODE | DESCRIPTION | INSTRUMENT |
|---|---|---|
| C-IR07 | Total Carbon (IR Spectroscopy) | LECO |
| S-IR08 | Total Sulphur (IR Spectroscopy) | LECO |
| ME-MS81 | Lithium Borate Fusion ICP-MS | ICP-MS |
| ME-MS42 | Up to 34 elements by ICP-MS | ICP-MS |
| ME-MS61L | Super Trace Lowest DL 4A by ICP-MS | |
| ME-ICP06 | Whole Rock Package - ICP-AES | ICP-AES |
| TOT-ICP06 | Total Calculation for ICP06 | |
| OA-GRA05 | Loss on Ignition at 1000C | WST-SEQ |

The results of this assay were based solely upon the content of the sample submitted. Any decision to invest should be made only after the potential investment value of the claim 'or deposit has been determined based on the results of assays of multiple samples of geological materials collected by the prospective investor or by a qualified person selected by him/her and based on an evaluation of all engineering data which is available concerning any proposed project. Statement required by Nevada State Law NRS 519

This is the Final Report and supersedes any preliminary report with this certificate number. Results apply to samples as submitted. All pages of this report have been checked and approved for release.
***** See Appendix Page for comments regarding this certificate *****
Comments: full project: Recycled Concrete Aggregate Analysis

Signature:
Saa Traxler, General Manager, North Vancouver

Figure E.1 Whole Rock Analysis laboratory report page:1

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Page: 2 - A
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 1-MAY-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

**CERTIFICATE OF ANALYSIS RE20082600**

| Sample Description | Method / Analyte / Units / LOD | WEI-21 Recvd Wt. kg 0.02 | ME-ICP06 SiO2 % 0.01 | ME-ICP06 Al2O3 % 0.01 | ME-ICP06 Fe2O3 % 0.01 | ME-ICP06 CaO % 0.01 | ME-ICP06 MgO % 0.01 | ME-ICP06 Na2O % 0.01 | ME-ICP06 K2O % 0.01 | ME-ICP06 Cr2O3 % 0.002 | ME-ICP06 TiO2 % 0.01 | ME-ICP06 MnO % 0.01 | ME-ICP06 P2O5 % 0.01 | ME-ICP06 SrO % 0.01 | ME-ICP06 BaO % 0.01 | TOT-ICP06 Total % 0.01 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NC_CT1 | | 0.02 | 63.8 | 10.30 | 4.54 | 11.05 | 2.09 | 1.85 | 1.23 | 0.007 | 0.52 | 0.07 | 0.14 | 0.05 | 0.04 | 99.62 |
| NC_HW1 | | 0.02 | 63.0 | 11.90 | 2.82 | 10.50 | 0.62 | 3.06 | 3.35 | 0.005 | 0.26 | 0.05 | 0.11 | 0.04 | 0.09 | 101.17 |
| NC-AP1 | | 0.02 | 58.3 | 13.15 | 9.00 | 10.60 | 4.16 | 2.18 | 0.54 | 0.009 | 1.28 | 0.13 | 0.30 | 0.08 | 0.05 | 101.97 |

Figure E.2 Whole Rock Analysis laboratory report page:2-A

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Page: 2 - B
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 1-MAY-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20082600

| Sample Description | Method | OA-GRA05 | C-IR07 | S-IR08 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Analyte | LOI | C | S | Ba | Ce | Cr | Cs | Dy | Er | Eu | Ga | Gd | Ge | Hf | Ho |
| | Units | % | % | % | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm |
| | LOD | 0.01 | 0.01 | 0.01 | 0.5 | 0.1 | 10 | 0.01 | 0.05 | 0.03 | 0.02 | 0.1 | 0.05 | 5 | 0.1 | 0.01 |
| NC_CT1 | | 3.93 | 0.66 | 0.17 | 333 | 46.9 | 40 | 0.80 | 2.38 | 1.38 | 0.82 | 15.1 | 3.00 | <5 | 4.6 | 0.49 |
| NC_HW1 | | 5.36 | 0.83 | 0.14 | 783 | 77.1 | 30 | 2.55 | 5.23 | 3.05 | 1.16 | 19.2 | 5.16 | <5 | 6.0 | 0.99 |
| NC-AP1 | | 2.19 | 0.35 | 0.16 | 441 | 37.2 | 60 | 0.56 | 3.14 | 1.63 | 1.55 | 16.0 | 3.29 | <5 | 2.8 | 0.59 |

Comments: full project: Recycled Concrete Aggregate Analysis

Figure E.3 Whole Rock Analysis laboratory report page:2-B

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Page: 2 - C
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 1-MAY-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20082600

| Sample Description | Method | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Analyte | La | Lu | Nb | Nd | Pr | Rb | Sm | Sn | Sr | Ta | Tb | Th | Tm | U | V |
| | Units | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm |
| | LOD | 0.1 | 0.01 | 0.1 | 0.1 | 0.02 | 0.2 | 0.03 | 1 | 0.1 | 0.1 | 0.01 | 0.05 | 0.01 | 0.05 | 5 |
| NC_CT1 | | 23.3 | 0.22 | 6.5 | 20.2 | 5.20 | 39.7 | 3.91 | 2 | 441 | 0.5 | 0.43 | 7.37 | 0.18 | 2.40 | 105 |
| NC_HW1 | | 38.5 | 0.42 | 10.3 | 34.1 | 9.01 | 111.0 | 6.39 | 2 | 330 | 0.8 | 0.79 | 16.20 | 0.40 | 4.36 | 42 |
| NC-AP1 | | 16.4 | 0.22 | 6.8 | 21.7 | 4.82 | 12.6 | 3.88 | 1 | 746 | 0.4 | 0.51 | 2.06 | 0.24 | 0.73 | 215 |

Comments: full project: Recycled Concrete Aggregate Analysis

Figure E.4 Whole Rock Analysis laboratory report page:2-C

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**Page: 2 - D**
**Total # Pages: 2 (A - E)**
**Plus Appendix Pages**
**Finalized Date: 1-MAY-2020**
**Account: CHANOR**

Project: Recycled Concrete Aggregate

**CERTIFICATE OF ANALYSIS RE20082600**

| Sample Description | Method | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS81 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS42 | ME-MS61L | ME-MS61L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Analyte | W | Y | Yb | Zr | As | Bi | In | Hg | Re | Sb | Se | Te | Tl | Ag | Cd |
| | Units | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm |
| | LOD | 1 | 0.1 | 0.03 | 2 | 0.1 | 0.01 | 0.005 | 0.005 | 0.001 | 0.05 | 0.2 | 0.01 | 0.02 | 0.002 | 0.005 |
| NC_CT1 | | 1 | 13.6 | 1.46 | 189 | 4.4 | 0.09 | 0.022 | <0.005 | 0.002 | 0.48 | 0.6 | 0.01 | 0.16 | 0.053 | 0.146 |
| NC_HW1 | | 1 | 26.6 | 2.86 | 218 | 3.0 | 0.06 | 0.023 | <0.005 | 0.001 | 0.22 | 0.2 | 0.01 | 0.17 | 0.029 | 0.070 |
| NC-AP1 | | 1 | 15.9 | 1.53 | 115 | 1.7 | 0.04 | 0.015 | <0.005 | 0.001 | 0.19 | 0.4 | 0.02 | 0.10 | 0.034 | 0.095 |

Comments: full project: Recycled Concrete Aggregate Analysis

Figure E.5 Whole Rock Analysis laboratory report page:2-D

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**Page: 2 - E**
**Total # Pages: 2 (A - E)**
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**Finalized Date: 1-MAY-2020**
**Account: CHANOR**

Project: Recycled Concrete Aggregate

**CERTIFICATE OF ANALYSIS RE20082600**

| Sample Description | Method | ME-MS61L | ME-MS61L | ME-MS61L | ME-MS61L | ME-MS61L | ME-MS61L | ME-MS61L | ME-MS61L |
|---|---|---|---|---|---|---|---|---|---|
| | Analyte | Co | Cu | Li | Mo | Ni | Pb | Sc | Zn |
| | Units | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm |
| | LOD | 0.005 | 0.02 | 0.2 | 0.02 | 0.08 | 0.01 | 0.01 | 0.2 |
| NC_CT1 | | 13.85 | 40.8 | 16.7 | 4.88 | 20.9 | 13.85 | 12.20 | 71.7 |
| NC_HW1 | | 3.59 | 12.90 | 29.6 | 1.27 | 10.25 | 19.50 | 5.10 | 63.2 |
| NC-AP1 | | 28.1 | 65.2 | 10.7 | 1.06 | 64.2 | 6.00 | 23.2 | 72.0 |

Comments: full project: Recycled Concrete Aggregate Analysis

Figure E.6 Whole Rock Analysis laboratory report page:2-E

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**Page: 1**
**Total # Pages: 2 (A - E)**
**Plus Appendix Pages**
**Finalized Date: 26-APR-2020**
**Account: CHANOR**

**CERTIFICATE RE20080994**

Project: Recycled Concrete Aggregate

This report is for 1 Rock sample submitted to our lab in Reno, NV, USA on 10-APR-2020.

The following have access to data associated with this certificate:
TARA CAVALLINE

**SAMPLE PREPARATION**

| ALS CODE | DESCRIPTION |
|---|---|
| WEI-21 | Received Sample Weight |
| LOG-22 | Sample login - Rcd w/o BarCode |
| SND-ALS | Send samples to internal laboratory |
| PUL-31mp | Manual Pulverization w/o PUL-QC |
| CRU-31 | Fine crushing - 70% <2mm |

**ANALYTICAL PROCEDURES**

| ALS CODE | DESCRIPTION | INSTRUMENT |
|---|---|---|
| S-IR08 | Total Sulphur (IR Spectroscopy) | LECO |
| ME-MS81 | Lithium Borate Fusion ICP-MS | ICP-MS |
| ME-MS42 | Up to 34 elements by ICP-MS | ICP-MS |
| ME-MS61L | Super Trace Lowest DL 4A by ICP-MS | |
| ME-ICP06 | Whole Rock Package - ICP-AES | ICP-AES |
| TOT-ICP06 | Total Calculation for ICP06 | |
| OA-GRA05 | Loss on Ignition at 1000C | WST-SEQ |
| C-IR07 | Total Carbon (IR Spectroscopy) | LECO |

The results of this assay were based solely upon the content of the sample submitted. Any decision to invest should be made only after the potential investment value of the claim 'or deposit has been determined based on the results of assays of multiple samples of geological materials collected by the prospective investor or by a qualified person selected by him/her and based on an evaluation of all engineering data which is available concerning any proposed project. Statement required by Nevada State Law NRS 519

This is the Final Report and supersedes any preliminary report with this certificate number. Results apply to samples as submitted. All pages of this report have been checked and approved for release.

***** See Appendix Page for comments regarding this certificate *****

Comments: full Project name: Recycled Concrete Aggregate Analysis

**Signature:**
Saa Traxler, General Manager, North Vancouver

Figure E.7 Whole Rock Analysis laboratory report FOR NC_CT2 page:2

ALS USA Inc.
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To: **UNIVERSITY OF NORTH CAROLINA AT CHARLOTTE**
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**CHARLOTTE NV 28223**

**Page: 2 - A**
**Total # Pages: 2 (A - E)**
**Plus Appendix Pages**
**Finalized Date: 26-APR-2020**
**Account: CHANOR**

Project: Recycled Concrete Aggregate

**CERTIFICATE OF ANALYSIS RE20080994**

| Sample Description | Method / Analyte / Units / LOD | WEI-21 Recvd Wt. kg 0.02 | ME-ICP06 SiO2 % 0.01 | ME-ICP06 Al2O3 % 0.01 | ME-ICP06 Fe2O3 % 0.01 | ME-ICP06 CaO % 0.01 | ME-ICP06 MgO % 0.01 | ME-ICP06 Na2O % 0.01 | ME-ICP06 K2O % 0.01 | ME-ICP06 Cr2O3 % 0.002 | ME-ICP06 TiO2 % 0.01 | ME-ICP06 MnO % 0.01 | ME-ICP06 P2O5 % 0.01 | ME-ICP06 SrO % 0.01 | ME-ICP06 BaO % 0.01 | TOT-ICP06 Total % 0.01 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NC_CT2 | | 0.02 | 21.5 | 1.05 | 0.88 | 42.6 | 0.82 | 0.12 | 0.19 | 0.013 | 0.09 | 0.04 | 0.33 | 0.05 | 0.01 | 98.09 |

Comments: full Project name: Recycled Concrete Aggregate Analysis

Figure E.8 Whole Rock Analysis laboratory report FOR NC_CT2 page:2A

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Page: 2 - B
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 26-APR-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20080994

| Sample Description | Method / Analyte / Units / LOD | OA-GRA05 LOI % 0.01 | C-IR07 C % 0.01 | S-IR08 S % 0.01 | ME-MS81 Ba ppm 0.5 | ME-MS81 Ce ppm 0.1 | ME-MS81 Cr ppm 10 | ME-MS81 Cs ppm 0.01 | ME-MS81 Dy ppm 0.05 | ME-MS81 Er ppm 0.03 | ME-MS81 Eu ppm 0.02 | ME-MS81 Ga ppm 0.1 | ME-MS81 Gd ppm 0.05 | ME-MS81 Ge ppm 5 | ME-MS81 Hf ppm 0.1 | ME-MS81 Ho ppm 0.01 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NC_CT2 | | 30.4 | 8.28 | 0.21 | 57.4 | 9.6 | 80 | 0.29 | 0.97 | 0.50 | 0.21 | 1.6 | 0.88 | <5 | 2.4 | 0.16 |

Comments: full Project name: Recycled Concrete Aggregate Analysis

Figure E.9 Whole Rock Analysis laboratory report FOR NC_CT2 page:2B

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Page: 2 - C
Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 26-APR-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20080994

| Sample Description | Method / Analyte / Units / LOD | ME-MS81 La ppm 0.1 | ME-MS81 Lu ppm 0.01 | ME-MS81 Nb ppm 0.1 | ME-MS81 Nd ppm 0.1 | ME-MS81 Pr ppm 0.02 | ME-MS81 Rb ppm 0.2 | ME-MS81 Sm ppm 0.03 | ME-MS81 Sn ppm 1 | ME-MS81 Sr ppm 0.1 | ME-MS81 Ta ppm 0.1 | ME-MS81 Tb ppm 0.01 | ME-MS81 Th ppm 0.05 | ME-MS81 Tm ppm 0.01 | ME-MS81 U ppm 0.05 | ME-MS81 V ppm 5 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NC_CT2 | | 5.6 | 0.07 | 2.0 | 5.4 | 1.34 | 6.4 | 1.06 | <1 | 360 | 0.1 | 0.14 | 1.49 | 0.07 | 2.83 | 21 |

Comments: full Project name: Recycled Concrete Aggregate Analysis

Figure E.10 Whole Rock Analysis laboratory report FOR NC_CT2 page:2C

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Total # Pages: 2 (A - E)
Plus Appendix Pages
Finalized Date: 26-APR-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20080994

| Sample Description | Method / Analyte / Units / LOD | ME-MS81 W ppm 1 | ME-MS81 Y ppm 0.1 | ME-MS81 Yb ppm 0.03 | ME-MS81 Zr ppm 2 | ME-MS42 As ppm 0.1 | ME-MS42 Bi ppm 0.01 | ME-MS42 In ppm 0.005 | ME-MS42 Hg ppm 0.005 | ME-MS42 Re ppm 0.001 | ME-MS42 Sb ppm 0.05 | ME-MS42 Se ppm 0.2 | ME-MS42 Te ppm 0.01 | ME-MS42 Tl ppm 0.02 | ME-MS61L Ag ppm 0.002 | ME-MS61L Cd ppm 0.005 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NC_CT2 | | 2 | 4.8 | 0.55 | 106 | 3.2 | 0.04 | 0.005 | 0.098 | 0.003 | 0.28 | 0.3 | 0.03 | 0.03 | 0.019 | 0.376 |

Comments: full Project name: Recycled Concrete Aggregate Analysis

Figure E.11 Whole Rock Analysis laboratory report FOR NC_CT2 page:2D

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Plus Appendix Pages
Finalized Date: 26-APR-2020
Account: CHANOR

Project: Recycled Concrete Aggregate

CERTIFICATE OF ANALYSIS RE20080994

| Sample Description | Method / Analyte / Units / LOD | ME-MS61L Co ppm 0.005 | ME-MS61L Cu ppm 0.02 | ME-MS61L Li ppm 0.2 | ME-MS61L Mo ppm 0.02 | ME-MS61L Ni ppm 0.08 | ME-MS61L Pb ppm 0.01 | ME-MS61L Sc ppm 0.01 | ME-MS61L Zn ppm 0.2 |
|---|---|---|---|---|---|---|---|---|---|
| NC_CT2 | | 3.45 | 5.62 | 4.6 | 1.10 | 15.30 | 4.82 | 1.17 | 43.4 |

Comments: full Project name: Recycled Concrete Aggregate Analysis

Figure E.12 Whole Rock Analysis laboratory report FOR NC_CT2 page:2E