Characteristic Analysis of Acid Mine Drainage Precipitates for the Optimization of Rare Earth Extraction Processes

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Academic Abstract

Acid mine drainage (AMD) forms when sulfur bearing rocks such as pyrite, are exposed to air and water. The oxidation of these minerals leads to the generation of sulfuric acid, which in turn mobilizes metals such as iron, aluminum, manganese, and others. If left untreated, AMD can cause severe harm to the surrounding ecosystem. By law, mining companies are required to treat AMD, often by oxidizing the contaminated water, raising the pH with a chemical additive, and precipitating the metals out of solution. Recent studies at West Virginia University and Virginia Tech have shown that AMD and the treatment precipitates (AMDp) are enriched in rare earth elements (REEs). Given the importance of REEs to modern technology, as well as potential supply restrictions, subsequent research has attempted to identify promising methods to extract and recovery REEs from AMD and AMDp. Prior studies have shown that the physical characteristics of AMDp can vary considerably from site to site, and a robust processes scheme must account for any site-specific disparities.

To better understand the inherent variability of AMDp, a scientific study was commissioned to investigate a standard method of characterizing AMDp for the optimization of rare earth extraction processes. The tests developed in this work define the total acid dose needed to dissolve AMDp at various target pH points. Through the course of the study, over 150 unique AMDp samples were evaluated, and comparative analyses were conducted on samples from different sites as well as replicate samples from the same sites. The resultant dataset was analyzed using an empirical model, and a statistical analysis was conducted to correlate the model parameters and other AMDp physical properties. Relationships between elemental assays, moisture, and fitting parameters of the empirical models were found. These results ultimately led to a recommendation for future treatment of AMD and prospective sites.
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General Audience Abstract

Acid mine drainage (AMD) is a longstanding environmental issue that is caused when sulfur-bearing rocks are exposed to the environment during the mining process. By law, companies are required to treat AMD prior to discharging the water back into the environment. This treatment process creates a waste byproduct, AMD precipitate, that largely consists of metal hydroxides, including iron, aluminum, manganese, and others. Historically, AMD precipitate has been considered an undesirable waste that must be carefully disposed either in old mine workings or in permanent storage cells. However, recent research has shown that AMD precipitate contains elevated concentrations of rare earth elements (REEs). REEs are a group of chemically similar elements that are well known for their use in several modern technologies, including magnets, catalysts, glasses, light-weight metal alloys, and other high-tech uses. REEs are often sparsely concentrated in nature and rarely form ore deposits of a commercial grade. As a result, several private companies and government agencies have sought alternative sources of REEs. Prior research has shown that AMD precipitate may be a suitable alternative source; however, the chemical and physical nature of AMD varies considerably between different sites. This research seeks to utilize a standard characterization test to identify the characteristics of AMD precipitate that ultimately dictate process amenability. Throughout the course of the study, over 150 unique AMD precipitate samples were analyzed, and the cumulative results show which class of sites constitute the most promising prospecting targets.
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Chapter 1: Introduction

1.1 Preface

Acid mine drainage (AMD) is a longstanding environmental issue that is caused when sulfide bearing rocks, such as pyrite, are exposed to air and water. Oxidation of these minerals leads to the generation of sulfuric acid, which tends to mobilize heavy metals, such as iron, aluminum, manganese, and others. If left uncontrolled, AMD can migrate to local watersheds and cause serious damage to aquatic ecosystem. While AMD is common to many mining districts, the problem is particularly noteworthy in Northern and Central Appalachia, where the coal measures often contain elevated concentrations of pyrite.

Per Section 402 of the Clean Water Act, active mine operations are required to obtain permits that specify pollutant discharge limits for effluents leaving the mine property. In order to comply with these permits, most mines utilize treatment systems that consist of mechanical oxidation, chemical acid neutralization, and solids precipitation to collectively remove transition metals and reduce the acidity (Ziemkiewicz et al., 2016). The chemical treatment of AMD tends to produce an amorphous hydroxide residue of metal precipitates. This waste material is often denoted AMD precipitate (AMDp), AMD solids, or AMD sludge. AMDp can occur in various forms and consistencies based on the composition of the metal species as well as specific treatment method that was used. A picture of a typical AMDp sample is shown in Figure 1. While several groups have attempted to identify suitable end uses for AMDp, few if any have found commercial success. Today, most of this treatment byproduct is either pumped back into open mine voids or dried in above-ground storage cells prior to permanent disposal in a landfill (Skousen et al., 2000; Johnson and Hallberg, 2004).
The treatment of AMD and subsequent production of AMDp tend to capture and remove numerous major and minor metals through either direct precipitation and/or adsorption to the precipitated species. In recent studies, researchers at West Virginia University and Virginia Tech have found that AMDp tends to be particularly enriched in rare earth elements (REEs), a group of 17 chemically similar metallic elements that include the lanthanide series, scandium, and yttrium (Figure 2) (Ziemkiewicz et al., 2016). At these AMD treatment sites, the concentrations of REEs in AMDp were found to be 1 to 4 times greater than that of the surrounding rock (Stewart, 2016) and nearly 2000 times greater than that of raw AMD (Ziemkiewicz et al., 2016). Ziemkiewicz et al (2016) showed that the REEs precipitate alongside the metal hydroxides during AMD treatment and theorized that the REEs have a solubility similar to other transition metals found in AMD.
Figure 2. Periodic table depicting those elements that constitute the rare earth elements (REEs). REEs are often categorized into light and heavy fractions, as well as those deemed "critical" by the US Department of Energy.

1.2 Motivation

The concentrated occurrence of REEs in AMDp is particularly noteworthy given the current global market conditions as well the future market projections for these elements. REEs are crucial for several high-tech markets; however, the current supply of REEs is dominated by Chinese sources. The USGS estimated in 2009 that China controlled 43% of the world’s REE reserves and supplied more than 95% of all consumed REEs in the world (USGS). With China’s growing domestic needs for REEs, trade policies have resulted in strict export quotas that limit the amount of REE that are exported to other countries (Zhang, 2015). This large trade imbalance (depicted in Figure 3 and Figure 4) has created tremendous market pressure to identify alternative REE sources, particularly in those countries that are heavily reliant on imports (e.g. Japan and USA).
Despite their name, REEs are moderately abundant in the earth’s crust, with an average crustal concentration similar to that of other major industrial metals, such as lead, tin, molybdenum, and tungsten
REEs are found in trace amounts in a variety of minerals including silicates, oxides, carbonates, phosphates and halides; however, few natural processes lead to concentrated REE ore deposits (Jordens et al., 2013). The low concentrations tend to increase the difficulty and cost of the extraction process, and the chemical similarity of the elements requires sophisticated (and costly) separation technologies.

In recent years, the US Department of Energy – National Energy Technology Laboratory has identified coal and coal byproducts as a potentially promising source of REEs (Stewart et al., 2016; Honaker et al., 2017; Ziemkiewicz et al., 2016; Zhang et al., 2015). Several studies have shown that the coal measures tend to be enriched in REEs, and as a result, several studies were commissioned to identify and develop technologies that can effectively extract and separate REEs from coal-based feedstocks. Given the elevated concentrations of REEs in the coal measures as well as the prominence of AMD in mining districts, Ziemkiewicz and his coworkers conducted a preliminary study to develop a process that can extract and concentrate REEs from AMD and AMDp (Ziemkiewicz et al., 2018). In this process, the raw AMD acts as a “natural” heap leach reactor that extracts REEs from the host rock. The requisite AMD treatment plant concentrates this “leach liquor” and produces AMDp as feedstock for an REE recovery process. A simplified schematic of the process developed by Ziemkiewicz et al. is shown in Figure 5. This process follows a traditional hydrometallurgical processing route whereby the AMDp is first leached in acid, and the leach solution is then purified and concentrated by solvent extraction. The final REE products are produced via precipitation.
As is typical for most hydrometallurgical flowsheets, the cost of the AMDp processing route is largely driven by consumable costs, particularly the acid costs in the leaching stage. This sensitivity is particularly noteworthy for AMDp processing, as by definition, AMDp is not a natural and consistent material, but rather an amorphous mix that can vary considerably in both composition and consistency based on numerous site-specific factors. Depending on the aqueous chemistry and the degree of overtreatment, the natural pH of AMDp can vary from very acidic (pH 3 to 4) to highly alkaline (pH 12+). This degree of variability creates challenges in both standardizing the approach of the AMDp leaching step as well as projecting costs and payback for a potential commercial operation.

1.3 Objectives

Given uncertainties and variability associated with the acid leaching of AMDp, a scientific study was commissioned to investigate the acid consumption needed to fully dissolve AMDp and clarify the material characteristics that lead to greater or smaller acid consumption. The scope of this study was quite extensive, as over 150 individual AMDp samples were collected from the Northern and Central Appalachian coal basins. This particular work on acid consumption represents one component of a larger
body of work seeking to both define the REE resource in AMDp and develop a processing technology that can effectively and efficiently produce REEs. Given this context, the three primary objectives of this research include:

- Determine the moisture content and major metal constituents of a large suite of AMD precipitate samples (n ~ 150) representing a range of different geographic regions, treatment processes, elemental compositions, and morphological characteristics.
- Characterize the relationship between acid addition and resultant pH for to gain information on the characteristic acid consumption sample for each AMDp sample.
- Interpret the acid consumption data using an empirical modeling approach and identify if any statistical correlations show a relationship between acid consumption and other intrinsic sample properties.

1.4 Organization

This thesis will be organized into five separate chapters. Chapter 1 includes a description of background information and the motivation for this project as well as objectives and organization. This information provides an overview of AMD treatment, REE sources and markets, and the process currently being developed to extract REEs from AMDp.

Chapter 2 is a literature review composed of chemical and physical processing of REEs, and the current research that is being done by the National Energy Technology Laboratory (NETL) and other groups on REE extractions from acid mine drainage and coal fly ash. Following this is a detailed discussion on AMD including the causes and chemistry, the legal responsibility to treat AMD, mitigation techniques, different treatment options including oxidation and pH adjustment, AMDp as a waste product, current AMDp disposal options, why AMDp is enriched in REEs, and any other prior characterization studies that have been done on AMDp.

Chapter 3 includes the materials and methods needed to test and characterize each of the AMDp samples. A discussion on the two-phase sampling process as well as details on the characterization studies
(XRF and moisture) are provided. The standard testing procedure is described, and the experimental design along with the data analysis and modeling is fully examined.

Chapter 4 reviews the results of the testing series and discussion on the various characterization and acid consumption tests. A detailed analysis of the empirical modeling and statistical analyses are also reviewed.

Chapter 5 summarizes the key findings of this work and provides recommendations for future research and development.

The thesis includes a detailed Appendix with the raw test data from each of the experimental series.
Chapter 2: Literature Review

2.1 Acid Mine Drainage

2.1.1 Causes and Chemistry

Acid mine drainage (AMD) refers to the production of sulfuric acid due to the oxidation of freshly liberated sulfide minerals. The central and northern Appalachian coal mining regions contain several metal and iron sulfides that cause AMD; however, the most prevalent are pyrite and marcasite (both forms of FeS$_2$). As these sulfides oxidize, sulfuric acid is produced, and the mining process increases the rate of oxidation (Hedin 2006). This process is described chemically by the following reactions:

$$2FeS_2 + 7O_2 + 2H_2O \rightarrow 2Fe^{2+} + 4SO_4^{2-} + 4H^+ \quad (2.1)$$

Here, the sulfur is oxidized to form hydrogen ions and sulfate, which ultimately are components required to make sulfuric acid (USEPA 1994).

$$4Fe^{2+} + O_2 + 4H^+ \rightarrow 4Fe^{3+} + H_2O \quad (2.2)$$

In Reaction 2.2, the soluble iron byproduct (Fe$^{2+}$) is left in solution which in turn converts the ferrous iron materials to ferric ions at low pH values (USEPA 1994).

$$2FeS_2 + 14Fe^{3+} + 8H_2O \rightarrow 15Fe^{2+} + 2SO_4^{2-} + 16H^+ \quad (2.3)$$

Reaction 2.3 takes into account the presence of a bacteria which, at pH values less than 3.5, will allow pyrite to be dissolved if in contact of a ferric ion (USEPA 1994).

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3(s) + 3H^+ \quad (2.4)$$

Finally, Reaction 2.4 depicts ferric iron precipitates from the AMD as hydrated iron oxide. This reaction causes the hydrated iron oxide to precipitate, which in turn creates the orange staining on stream bottoms commonly seen throughout Appalachia (USEPA 1994).
The sulfuric acid produced from this process tends to mobilize certain major metals including: \( \text{Fe}^{2+}, \text{Fe}^{3+}, \text{Al}^{3+}, \text{Mn}^{2+} \), as well \( \text{RE}^{3+} \). The mine water acidity is then further increased as these metals undergo hydrolysis reactions, which in turn reduces the pH of the AMD. The acidity-producing hydrolysis reactions are shown in reactions (2.5 – 2.9) (Hedin, 2006):

\[
\begin{align*}
\text{Fe}^{2+} + \frac{1}{4} O_2 + \frac{5}{2} H_2O & \rightarrow \text{Fe(OH)}_3 + 2H^+ \quad (2.5) \\
\text{Fe}^{3+} + 3H_2O & \rightarrow \text{Fe(OH)}_3 + 3H^+ \quad (2.6) \\
\text{Al}^{3+} + 3H_2O & \rightarrow \text{Al(OH)}_3 + 3H^+ \quad (2.7) \\
\text{Mn}^{2+} + \frac{1}{2} O_2 + H_2O & \rightarrow \text{MnO}_2 + 2H^+ \quad (2.8) \\
\text{RE}^{3+} + 3H_2O & \rightarrow \text{RE(OH)}_3 + 3H^+ \quad (2.9)
\end{align*}
\]

As a result, the net acidity can be estimated by the AMD pH as well as the concentration of major metals in solution, i.e.:

\[
\text{Acid}^{\text{calc}} = 50 \times \left( 2 \times \frac{\text{Fe}^{2+}}{56} + 3 \times \frac{\text{Fe}^{3+}}{56} + 3 \times \frac{\text{Al}}{27} + 2 \times \frac{\text{Mn}}{55} + 1000 \times 10^{-pH} \right) - \text{Alkalinity} \quad (2.10)
\]

where acidity and alkalinity are mg/L CaCO\(_3\) and metals are mg/L. Alkalinity is primarily in the form of bicarbonate ions, which consumes free protons. Reaction 2.11 depicts this acid consumption:

\[
\text{HCO}_3^- + H^+ \rightarrow H_2O + CO_2 \quad (2.11)
\]

2.1.2 Legal Responsibility

The National Pollution Discharge Elimination System (NPDES), per Section 402 of the Clean Water Act requires permitting of all point source discharges. This program requires that all water, processed or storm water, must leave through a monitored NPDES outlet. Water at these outlets, or monitoring points, must meet or exceed the quality parameters (or effluent standards) described in the NPDES permit.
Examples of these parameters include levels of iron (Fe), manganese (Mn), aluminum (Al), total suspended solids (TSS) and pH.

Because of these standards, the mine water rarely if ever meets these requirements naturally so treatment of the discharge must occur. However, due to the restricting terrain all throughout the Appalachian region, different types of contaminants present in the water at different sites, and different effluent limit parameters treatment of these mine influenced waters varies from site to site.

2.1.3 Treatment Options

Several AMD treatment techniques have been implemented to maintain effluent limit compliance per government regulation (USEPA 1994; Akcil, 2005; Skousen et al. 2000). The two main strategies to combat AMD, passive and active treatment operations. Passive treatments typically rely on naturally occurring biological, geochemical, and physical processes. The two most popular passive treatment types can be divided into just biological and geochemical systems. Biological systems are an engineered ecosystem that provides the required oxidation reduction, acid neutralization, and precipitate settling functions needed to conform to effluent standards. Examples of biological systems include aerobic and anaerobic wetlands (AeWs and AnWs), vertical flow wetlands (VFWs), bioreactors (SRB), and manganese removal beds (MRBs). Geochemical systems incorporate the use of an alkaline material, most commonly lime or caustic soda, in order to leach the mine influenced water to achieve the effluent standards. Geochemical system examples include anoxic limestone drains (ALDs), open limestone channels (OLCs), limestone leach beds (LLBs), and steel slag leach beds (SLBs) (Skousen et al., 2017).

Active treatment refers to the addiction of chemicals to the mine influenced water in order to treat the water to effluent standards. This treatment typically only is used if the available resources warrant it, such as land restrictions, high flow rates, and short time periods where the impacted water is present. In addition to the usage of chemicals, the presence of oxygen is a significant factor when designing an active treatment system. Dissolved oxygen in AMD helps adjust the pH of the water and promotes the precipitation of metal hydroxides. Because of this precipitation, additional chemicals such as flocculants,
coagulants, and oxidizing agents are added, and in some cases mechanical aerators are implemented to increase oxygen content in the water. Common active treatments have continuous additions of alkaline chemical reagent such as lime (CaO), hydrated lime (Ca(OH)\(_2\)), anhydrous ammonia (NH\(_3\)), or sodium hydroxide (NaOH) to neutralize acidity (Skousen 2017).

2.1.4 pH Adjustment Techniques

Chemical treatment of AMD sites is primarily driven by pH adjustment techniques in order to reduce the amount of dissolved metals in the acidic water. Skousen et al. (2000) states that there are six alkaline chemicals used to treat AMD by pH adjustment: (1) limestone (CaCO\(_3\)), (2) hydrated lime (Ca(OH)\(_2\)), (3) pebble quicklime (CaO), (4) soda ash (Na\(_2\)CO\(_3\)), (5) caustic soda (NaOH), and (6) ammonia (NH\(_3\) or NH\(_4\)OH). Chemical choice for treatment depends both on technical and environmental factors. Technical factors consist of the concentration of metals in the water, flow rate of the mine influenced water, and acidity levels. Economic factors includes the cost of the reagents, labor, machinery and equipment, as well as the number of years the treatment will be needed plus the interest rate. Given the chemistry of AMD and AMDp, limestone, pebble quicklime, and soda ash would be more beneficial in the treatment of AMD as opposed to the other treatments of hydrated lime, caustic soda, and ammonia. Below is an example comparing the chemical reaction of typical AMD species (hydrogen sulfate and Fe\(^{2+}\)) with both pebble quicklime (2.12) and caustic soda (2.13).

\[
Fe^{2+} + 2HSO_4^- + 2CaO \rightarrow FeO + 2CaSO_4 + H_2O \quad (2.12)
\]

\[
Fe^{2+} + 2HSO_4^- + 4NaOH \rightarrow FeOH_2 + Na_2SO_4 + 2H_2O \quad (2.13)
\]

The pebble quicklime consumes the same amount of acid with half as much base than the caustic soda. This is due to the nature of the anions present in limestone, pebble quicklime, and soda ash, making them more efficient chemically.

Figure 6 depicts the theoretical solubilities of common metal hydroxides found in AMD. The pH needed to precipitate most metals out of the water typically ranges from pH 6 to 9 (with the exception of
Fe$^{3+}$, which precipitates out at a pH ≥3.5). Depending on if oxygen is present or not also effects the precipitation of these ions. If oxygen if present, Fe$^{2+}$ oxidizes to Fe$^{3+}$ which after the addition of a neutralizing agent bringing the pH up, it becomes Fe(OH)$_3$ (or more commonly known as, “yellow boy”). This then precipitates out at a pH ≥3.5. On the other hand, in an oxygen poor environment, these metal hydroxides would need enough alkalinity added to raise the solution pH to 8.5 in order to precipitate. Therefore, it is beneficial to introduce aeration to the water before adding the neutralizing chemical agent in order to reduce the amounts of neutralizing agents needed to precipitate out the Fe from AMD (Skousen et al. 2000).

![Theoretical Solubilities](image)

*Figure 6 Theoretical Solubilites of AMD (Skousen 2015)*

### 2.1.5 AMDp Disposal

Although the different options for treatment of AMD provide a great solution for treating mine water, it creates a considerable amount of AMDp that needs to be disposed of properly. There are several ways in which AMDp disposal is typically handled: (1) leaving the AMDp submerged in a pond
indefinitely, (2) pumping or hauling AMDp from ponds to abandoned mines or old surface mine pits, and (3) dumping the AMDp into refuse piles (Skousen et al., 2000). It is important to note that the aging of AMDp created more stable AMDp, decreasing the likelihood of metals being released (Watzlaf and Casson 1990). This stayed true even when the AMDp was reintroduced into acidic environments. Lastly, if the AMDp is allowed to be aged in a dry environment it results in better stability than those aged underwater (Skousen et al. 2000).

2.2 Rare Earth Processing

2.2.1 Major Ore Deposits

Currently, REEs are extracted by two primary sources; mineral deposits and ion-exchangeable clays. The Mountain Pass bastnasite mine, located in the United States, is one of the world’s largest open pit deposits of rare-earth minerals. The mining and extraction practices at this mine use standard techniques; however, the floatation and beneficiation of the rare earth ore are extremely complex. (Krishnamurthy and Gupta 2016; Pradip, 1991) In order to process the ore, a multistage conditioning procedure involving soda ash, lignin sulfonate, and steam with a fatty acid collector must be used.

However, the largest of all REE deposits is not even primarily a REE mine, but rather it is an open pit iron ore mine. Bayan Obo deposit in China was found to also be host to one of the largest REE deposits in the world. In 2005, Bayan Obo produced 45% of the world’s REEs, mostly due to the deposit being close to the surface which in turn made mining and processing costs extremely low (Chi Ruan 2005; Jordens 2013). The other type of REE deposit are REE-bearing clays or simply ion-adsorption clays. These deposits occur all throughout southern China, and are often mined and processed using in-situ leaching with an ionic liquid.

2.2.2 Physical and Chemical Processing

Generally, mineral-based rare earth deposits are beneficiated through combinations of gravity concentration, magnetic separation, electrostatic separation, and froth flotation. Froth flotation, however,
is the most crucial and heavily researched process for the physical beneficiation of rare earth mineral deposits. The popularity of flotation is primarily due to its capacity and adaptability, i.e. flotation can be tailored to the mineralogy of different deposits as well as being able to process a wide range of fine particles. These flotation techniques are both used to beneficiate in the two largest rare-earth deposits: Mountain Pass and Bayan Obo (Krishnamurthy and Gupta 2016).

Although the Mountain Pass and Bayan Obo mines use similar beneficiate techniques, the main difference lies within the amount of conditioning treatments each deposit ore goes through. A simplified flow sheet of the Mountain Pass floatation technique is shown in Figure 7 (Krishnamurthy and Gupta 2016; Pradip, 1991), while Figure 8 shows the process for the Bayan Obo ore. The conditioning process for Mountain Pass is much more extensive, and as a result, the processing cost is much higher than that of the Bayan Obo mine (Krishnamurthy and Gupta 2016; Jordens 2013).

Following physical separations, REEs are usually refined using a conventional hydrometallurgical processing route that includes acid or alkaline leaching, solvent extraction, and oxalate precipitation (Krishnamurthy and Gupta 2016).

As stated previously, Southern China is host to a unique class of REE-rich clay deposits. To extract the REEs from these clay deposits, an in-situ mining technique was developed by Jiangxi South Rare Earth Hi-Tech (SREC). The method involves drilling holes into the deposit, and then filling the hole with a reagent (either ammonium sulfate or some salt based solution). After extraction, oxalic acid is then added to precipitate the REEs as oxalates. The oxalates are then filtered and roasted then converted to rare earth oxides (REOs). This results in the concentrates of REO content of more than 90 percent. It is estimated that 90 percent of rare earths are recovered by leaching the ore with either an ammonium or salt-based solution (British Geological Survey, 2017).
Figure 7 Simplified flow sheet for recovery of REOs from Mountain Pass ore (Krishnamurthy and Gupta 2016).

Figure 8 Schemes for physical beneficiation of Bayan Obo ore. (Krishnamurthy and Gupta 2016)
2.2.3 Alternative REE Resources

Over the past decade, private industry, government agencies, and researchers have attempted to identify alternative sources of REEs. One potentially promising resource that contains elevated concentrations of rare early elements is coal and coal products. Seredin and Dai (2012) showed several coal basins throughout the world that have REE concentrations comparable to those of conventional economic ores and enriched clay deposits. The REE-enriched coal is both enriched in the surrounding coal seams and mineralogy, which in turn makes it possible to recover these REEs through different stages of coal extraction such as overburden removal, coal mining and the oxidation of the newly exposed material, and refuse from coal processing. Table 1 describes several coal ashes along with the respective rare earth content (ash basis).

Table 1 REE content of select coal ashes throughout the World (Seredin and Dai, 2012).

<table>
<thead>
<tr>
<th>Country</th>
<th>Region</th>
<th>Deposit</th>
<th>Mine</th>
<th>Seam</th>
<th>REE (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Pavlovka</td>
<td>Pavlovka-2</td>
<td>I/a</td>
<td>4551</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Pavlovka</td>
<td>Pavlovka-2</td>
<td>I/b</td>
<td>8426</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Pavlovka</td>
<td>Pavlovka-2</td>
<td>I/c</td>
<td>4879</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Pavlovka</td>
<td>Spetzugli</td>
<td>III-low</td>
<td>972</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Pavlovka</td>
<td>Luzanovka</td>
<td>III3</td>
<td>1294</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Ravkovka</td>
<td>Northern</td>
<td>III/a</td>
<td>1235</td>
</tr>
<tr>
<td>Russia</td>
<td>RFE</td>
<td>Ravkovka</td>
<td>Southern</td>
<td>III</td>
<td>1144</td>
</tr>
<tr>
<td>Mongolia</td>
<td>N/A</td>
<td>Aduunchuluun</td>
<td>Aduunchuluun</td>
<td>Lower</td>
<td>2263</td>
</tr>
<tr>
<td>Tadjikistan</td>
<td>N/A</td>
<td>Nazar-Ailok</td>
<td>N/A</td>
<td>N/A</td>
<td>950</td>
</tr>
<tr>
<td>China</td>
<td>Sichuan</td>
<td>Sichuan Basin</td>
<td>Changhe</td>
<td>K8</td>
<td>1401</td>
</tr>
<tr>
<td>China</td>
<td>SW China</td>
<td>Songzao</td>
<td>Songzao</td>
<td>12</td>
<td>1636</td>
</tr>
<tr>
<td>China</td>
<td>Inner Mongolia</td>
<td>Daqingshan</td>
<td>Adaohai</td>
<td>CP2</td>
<td>721</td>
</tr>
<tr>
<td>China</td>
<td>Guizhou</td>
<td>Luizhi</td>
<td>Dacaoba</td>
<td>14</td>
<td>2491</td>
</tr>
<tr>
<td>China</td>
<td>Yunnan</td>
<td>Weining</td>
<td>Mahe</td>
<td>C6b</td>
<td>1234</td>
</tr>
<tr>
<td>China</td>
<td>Guangxi</td>
<td>Fusui</td>
<td>Guanglong</td>
<td>I/a</td>
<td>1180</td>
</tr>
<tr>
<td>China</td>
<td>Guangxi</td>
<td>Fusui</td>
<td>Guanglong</td>
<td>I/b</td>
<td>1010</td>
</tr>
</tbody>
</table>

While coal ash tends to be greatly enriched in REE, researchers have recently shown that average REE abundance in the actual coal material is only 62 ppm (Zhang et al., 2015). However, in the Appalachian Region of the United States in certain coal beds, REE abundance has been measured to have
a range as high as 500 to 4000 ppm. Other recent work showed that REE coal deposits of high concentration (>700 ppm) were densely distributed in Northern Appalachian, Central Appalachian, and Black Warrior basins (Zhang et al. 2015). These REEs were discovered to have a correlation with the inorganic material in coal. Honaker et al (2017) found that REEs in coal samples from Appalachia are mainly in the form of REE-rich phosphate, typically denoted as monazite. The monazite grains (<2 micron) infill the cracks in the clays in which the coal is surrounded by (Zhang et al., 2015).

2.2.4 REE Enrichment in AMD and AMDp

AMD in the Appalachian coal region has been shown to have elevated concentrations of REEs. This results is primarily due to three contributing factors, which have been explained in prior sections, namely; (1) the mild solubility of REEs in acid, (2) the enrichment of REEs in the Appalachian coal measures, and (3) the prevalence of acid mine drainage in Appalachian coal mines. Previous studies have indicated that pH and the presence of colloids play a major role in REE concentrations in natural water ways (Zhao, 2007; Protano and Riccobono 2002; Astrom and Corin 2003). During one study by Zhao (2007), observations of the AMD from the Sitai coal mine in northern China had higher than usual concentrations of $\text{SO}_4^{2-}$. These sulfate ions lead to the formation of $\text{RE}($$\text{SO}_4^+$$)_3$, resulting in higher concentrations of REEs in AMD. The concentrations of Fe, Al, Mn, and $\text{RE}($$\text{SO}_4^+$$)_3$ in AMD samples were discovered to decrease with increasing pH. This result implied that when the coagulation and precipitation of Fe-Al-Mn oxide/hydroxide colloids, the REE ions are likely adsorbed in these colloids. The REEs then precipitate along with other hydroxides, ultimately reporting to the AMDp. When the solid precipitates come out of solution from the AMD, the REEs were found to have higher concentrations in the AMDp. This is due to the surrounding mineralogy of the mining activity and the $\text{RE(OH)}_3$ that forms during the treatment process of AMD adheres to the same pH gradient as its Fe counterpart (Soyol-Erdene et al., 2018; Zhao, 2007).

It is important to note that precipitation of metals is directly correlated to pH adjustment. Zhao (2007) discovered during the formation of AMD, REE ions become adsorbed in the Fe-Al-Mn oxide/hydroxide colloid. This then meant that these newly formed REE-colloids follow the same
precipitation patterns as the metal oxides/hydroxides. Ziemkiewicz et al., (2018) identified that those target REE-colloids or rare earth hydroxides, RE(OH)₃, and other common metal hydroxides precipitate in a similar pH gradient, especially Fe³⁺.

2.2.5 Prior Characterization Studies

Several studies have characterized the composition and formation of REE-rich coal (Dai et al., 2016; Zhao, 2007; Soyol-Erdene et al., 2018) and coal by-products (Zhang et al., 2017; Franus et al., 2014; Gupta et al., 2014). The common conclusion for the characterization of the REE-rich coal was that the surrounding mineralogy including the composition of neighboring eroded material, volcanic ashes, and peat formations all have significant contributions to the REE distribution in AMD and other coal by-products, more specifically fly ash. However, there is a lack of a discussion about the technical aspects of the leaching and acid consumption of the AMDp. As a result, a significant research opportunity exists in order to have a better scientific understanding of the characteristics of AMDp for the optimization of rare earth extraction processes.
Chapter 3: Materials & Methods

3.1 Experimental Objectives

While several prior studies have characterized the form and composition of AMDp, few (if any) have evaluated the extent of acid consumption during AMDp leaching. Given this lack of technical data and scientific understanding, an experimental study was commissioned to evaluate the acid consumption needed to reach specific target pH points for various AMDp samples. This work was conducted in three distinct phases: (1) sample collection and characterization; (2) acid consumption method development and parametric testing; and (3) data reduction, modeling, and interpretation. Materials, methods, and protocols for each of the three phases is described in more detail below.

3.2 Sample Collection & Characterization

3.2.1 Sampling

The samples used in this experimental study were collected as part of a larger effort to characterize the potential REE resource in AMDp storage ponds throughout Northern and Central Appalachia (NAPP and CAPP, respectively). The broader effort conducted by other members of the project team included onsite sampling of both raw AMD and AMDp, REE and major metal analysis for AMDp samples, and XRD analysis of AMDp samples. The author of this study did not participate in the sampling effort; however, the pertinent details are summarized here since they are relevant to the author’s work. Further details on the sampling effort, the REE characterization, and other studies are given by Ziemkiewicz et al. (2018).

The objective of the sampling effort was to obtain a diverse mix of AMDp materials that represent a range of different raw AMD characteristics (strongly acidic to net alkaline), major metal constituents (iron rich, aluminum rich, and silica rich), and AMD treatment systems (caustic, anhydrous ammonia, hydrated lime, etc.). In total, 155 AMDp samples were obtained from 140 different treatments sites in four different states, including West Virginia, Pennsylvania, Maryland, and Ohio. Approximately 75% of the samples were obtained from NAPP, and 25% were obtained from CAPP (with Roanoke, WV being the approximate
delineation between NAPP and CAPP). As part of the broader project objectives, the sampling was conducted in two distinct phases: an exploratory phase that prioritized the sheer number of sites and a detailed phase that prioritized a more comprehensive evaluation of a few select sites. During the initial phase, only one to three replicate samples were obtained from each site to obtain a general idea of the REE content at each location. Pending the results of this campaign, 20 promising sites were selected for a more detailed evaluation, and no less than 10 replicate samples were recovered. This approach provides the capability to determine both variability between different sites as well as the variability within a single site.

At each treatment site, AMDp samples were collected into one-gallon HDPE sample buckets and sealed with a simple bucket lid. The sampling approach varied considerably at each site, depending on the site-specific conditions; however, considerable effort was made to ensure that the samples were representative of the areal extent of the storage pond. Given safety concerns and other constraints, only limited attempts were made to sample the storage ponds at depth. Nevertheless, sampling efforts did seek to minimize recovery of tramp material, including plants, brush, other organic waste, unreacted lime, dirt, and general debris. These materials were further screened out in the laboratory prior to any downstream analysis. Multiple samples were collected at specific sites if the site conditions suggested that the AMDp material was of a different composition (e.g. more than one treatment system was being used). This practice explains the slight discrepancy between the number of AMDp samples (155) and the number of sites samples (140). These numbers do not include the replicate samples collected as part of the detailed evaluation.

After receiving the samples in the laboratory, the materials were retained in the sealed one-gallon buckets at ambient room conditions, until they were ready for testing. A small split (<5 grams) was first recovered for REE analysis, while other splits were obtained for moisture determination, XRF, and XRD analysis. Finally, larger splits (up to 100 grams) were recovered for acid consumption tests. As mentioned above, the procedures and results for the XRD and REE analysis are given by Ziemkiewicz et al., (2018).
3.2.2 Moisture Determination

In order to express the results of the acid consumption tests on a dry mass basis, the AMDp moisture was determined for each AMDp sample. These analyses were conducted concurrently with the actual acid consumption tests (no more than 1 hour discrepancy between the two tests) to ensure that measured moisture values accurately reflected the moisture of the acid consumption test specimens (i.e. no inadvertent moisture loss due to evaporation or poor sample splitting). For each moisture analysis, three unique splits of 80 to 120 grams (wet weight) were recovered from the bulk AMDp sample. The wet weight each specimen was measured, and the specimens were then dried overnight in a laboratory oven at 80°C. After fully desiccating the samples, the dry weight was determined, and the moisture content (%) was determined via mass loss. The dried samples were then stored in a sample bag and retained for further analysis.

3.2.3 X-Ray Florescence (XRF) Analysis

Following the moisture analysis, the dried AMDp samples were then analyzed for major metal content using a Niton XL2 handheld X-Ray Florescence (XRF) analyzer. A picture of the XRF unit is shown in Figure 9. Each dry AMDp specimen was first ground by hand using a mortar and pestle and then placed into a sample cup. These cups were assembled by placing the film on top of the sample cup collar, then the sample cup cylinder was pressed into the collar so that the film was pulled tightly between the collar and cylinder. Next, the grounded sample was placed into the cup until the material reached half the height of the sample cup. A cotton ball was then placed on top of the sample material to ensure that the material was closely packed and did not move during handling. The cap was then placed on the cup and labeled accordingly. Pictures of this process are shown in Figure 10.
The XRF scan used the “Mining Cu/Zn” mode with a 60 second overall scan time. Elements included in the analysis were: Ba, Sb, Sn, Mo, Nb, Zr, Sr, Rb, Bi, As, Pb, W, Zn, Cu, Ni, Co, Fe, Mn, Cr, V, Ti, Ca, K, Al, P, Si, Cl, S, and Mg; however, the major elements prioritized in this study included: Fe, Ca, Al, Si, Mn, and S.
3.3 Acid Consumption Testing

3.3.1 General Approach

The acid consumption tests (also denoted acid titration or acid leaching tests) were conducted on each AMDp sample to identify the amount of acid (normalized to mass of acid per unit dry weight AMDp) needed to reach various target pH values. Each test was conducted using a standardized protocol, and several “test series” were conducted to fully evaluate the influence of specific independent variables. The raw results were analyzed using standard statistical approaches, and a non-linear empirical model was fit to the experimental data to provide another approach to the analysis. The results provide quantitative metrics to assess the amenability of acid leaching for various AMDp samples.

3.3.2 Apparatus

A standard test apparatus was used for all experiments. The apparatus includes a multi-position stirrer hot plate and specialized glassware that includes ports for sampling and pH measurement. Initially, a total reflux condenser was added to the reaction vessel to ensure that acid evaporation would not distort the acid consumption measurements; however, visual observations during the preliminary trials showed that no acid was condensing during the tests. This result was not unexpected, since all tests were performed at ambient temperature. As a result, the reflux condenser was later removed and not used for the majority of the test series. Comparative test runs showed no substantive difference in acid consumption between the tests run with and without the reflux condenser. Pictures of the initial and final test apparatus are shown in Figure 11 and Figure 12.

The Oakton laboratory pH meter was calibrated daily using three pH buffer solutions, namely pH 7, 4 and 2. For most test runs, this experimental setup was replicated so that up to three test runs could be completed simultaneously. All testing was done in a certified fume hood and in ambient room temperatures.
Figure 11. Initial test apparatus with reflux condenser attached to reaction vessel.

Figure 12. Final test apparatus with no reflux condenser.
3.3.3 **Chemicals & Reagents**

Diluted acid solutions were generated by mixing stock acid and deionized water at a ratio needed to create the desired acid concentration for testing, typically 3 mol/L. The acids utilized in the leaching tests included hydrochloric (37%), nitric (70%), and sulfuric (95%). All reagents were provided by standard laboratory chemical vendors, including Sigma Aldrich (St. Louis, MO), Fisher Scientific (Hampton, NH), and others. Diluted solutions were stored for the duration of the project, which did not exceed 18 months. Technical data for the acids are shown in Table 2.

<table>
<thead>
<tr>
<th>Acid</th>
<th>Density (g/mL)</th>
<th>Formula Weight (g/mol)</th>
<th>Stock Concentration (% w/w)</th>
<th>Test Molarity (mol/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl</td>
<td>1.2</td>
<td>34.46</td>
<td>37.0</td>
<td>3</td>
</tr>
<tr>
<td>HNO₃</td>
<td>1.413</td>
<td>63.01</td>
<td>70.0</td>
<td>3</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>1.84</td>
<td>98.08</td>
<td>95.0</td>
<td>3</td>
</tr>
</tbody>
</table>

Because the major metal hydroxides are insoluble, acid consumption is necessary to dissolve the metals. Below are the acid consumption reactions used in this study:

\[
RE(OH)₃(s) + 3HNO₃(aq) \rightarrow RE(NO₃)₃(aq) + 3H₂O (l) \tag{3.1}
\]

\[
2RE(OH)₃(s) + 3H₂SO₄(aq) \rightarrow RE(SO₄)₃(aq) + 6H₂O (l) \tag{3.2}
\]

\[
RE(OH)₃(s) + 3HCl(aq) \rightarrow RE(Cl)₃(aq) + 3H₂O (l) \tag{3.3}
\]

3.3.4 **Standard Test Protocol**

A standardized test protocol was employed to ensure consistency and reproducibility between test runs. All steps were conducted under an approved chemical hygiene plan (CHP). Key safety elements included a laboratory fume hood and with sufficient personal protective equipment, including but not limited to acid-resistant gloves, safety glasses or goggles, a suitable lab coat, closed-toed shoes, and other equipment as needed. Throughout the test program, the mixing intensity, acid concentration, leaching
temperature, and leaching time were all held constant, while cumulative acid dose was controlled as the primary independent variable for each test run. The resultant pH was then measured as the primary experimental response.

During the test sequence, approximately 100 grams of wet AMDp was first recovered from the desired one-gallon sample container. This material placed into the leaching vessel and weighted to determine the wet AMDp mass. Next, 100 mL of distilled water was mixed into the leaching vessel, and the reaction vessel was mounted on the magnetic stirrer plate. The stirrer rate was set to 300 PRM, and the solution was fully mixed for two minutes. After mixing, the initial pH (i.e. with no acid addition) was recorded. Acid was then added following an exponential sequence starting with 1 mL and doubling with each subsequent acid addition (i.e. 2 mL, 4 mL, 8 mL, 16 mL, etc.). After each acid addition, the solution was mixed for 5 minutes, and the pH was then recorded after the reaction was complete. Acid addition continued until the steady state pH reached a value less than 0.5, which typically required six to nine acid addition steps depending on the specific test specimen. After reaching the target pH point, the test was stopped, and the leachate was disposed following the guidelines of the CHP. The raw data first recorded in a laboratory notebook and later logged using a standard test database sheet in Microsoft Excel. Each test was repeated at least three times using representative splits from the same bucket. Data from the three replicate runs were averaged to produce a single result representative of the bucket sample.

The standard experimental parameters for the acid leaching tests are given in Table 3. Preliminary tests and prior knowledge have confirmed that these values are suitable for the given experimental procedure. Likewise, a screenshot of the Excel datasheet is shown in Figure 13.
Table 3. Acid Consumption Test Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid Type</td>
<td>--</td>
<td>Nitric, Sulfuric, and Hydrochloric</td>
</tr>
<tr>
<td>Acid Concentration</td>
<td>Mol/L</td>
<td>3</td>
</tr>
<tr>
<td>Acid Addition Increments</td>
<td>mL</td>
<td>1, 2, 4, 8, 16, 32, 64, and 128</td>
</tr>
<tr>
<td>Time Increment between Additions</td>
<td>Min.</td>
<td>5</td>
</tr>
<tr>
<td>Final Leach pH</td>
<td>--</td>
<td>&lt; 0.5</td>
</tr>
<tr>
<td>Leach Temperature</td>
<td>--</td>
<td>Ambient</td>
</tr>
<tr>
<td>Solid to liquid ratio (wet solids)</td>
<td>--</td>
<td>0.5:1, 1:1, 1.5:1</td>
</tr>
<tr>
<td>AMDp mass</td>
<td>grams</td>
<td>50, 100, 150</td>
</tr>
<tr>
<td>Stirrer Speed</td>
<td>RPM</td>
<td>300</td>
</tr>
<tr>
<td>Replicate Trials</td>
<td>--</td>
<td>3 to 10</td>
</tr>
</tbody>
</table>

Note: **Bold** entries indicate standard values. Others evaluated as part of experimental design.

Figure 13. Standard Excel data entry sheet.
3.3.5 Experimental Design

The experimental efforts were organized into five primary test series (and one preliminary series) to validate the test method and evaluate several sources of variation in the experimental results. Table 4 summarizes the objectives and samples used in each test series. Please note, the discrepancy between the total number of AMDp samples collected (155) and the total number of samples evaluated in Series 5 (153) is due to lack of sample. Two of the AMDp samples had very little solids (<1% solids), and the amount of material was not sufficient for acid consumption testing.

Table 4. Experimental Design Summary

<table>
<thead>
<tr>
<th>Series ID</th>
<th>Series Name</th>
<th>Number of Test Runs</th>
<th>Samples Evaluated</th>
<th>Objective</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Preliminary Tests</td>
<td>3</td>
<td>SL-1-10 SL-3-1</td>
<td>Validate experimental protocols.</td>
</tr>
<tr>
<td>1</td>
<td>Test Repeatability</td>
<td>10</td>
<td>SL-3-1</td>
<td>Evaluate variability of the test results on a single sample.</td>
</tr>
<tr>
<td>2</td>
<td>Solids Concentration</td>
<td>3</td>
<td>SL-21-5</td>
<td>Validate the applicability of dry mass normalization approach.</td>
</tr>
<tr>
<td>3</td>
<td>Acid Comparison</td>
<td>9</td>
<td>SL-40-1 SL-51-1 SL-67-1</td>
<td>Evaluate the difference between nitric, sulfuric, and hydrochloric acid.</td>
</tr>
<tr>
<td>4</td>
<td>Intra-Site Variation</td>
<td>38</td>
<td>SL-40-1 SL-67-1 SL-51-1 SL-66-1</td>
<td>Assess the variability of the test results on samples taken from multiple locations at the same site.</td>
</tr>
<tr>
<td>5</td>
<td>Site-to-Site Variation</td>
<td>153</td>
<td>All (x153)</td>
<td>Assess one sample from each site.</td>
</tr>
</tbody>
</table>
3.4 Data Analysis and Modeling

3.4.1 Data Reductions

The raw experimental data was pre-processed by first normalizing the acid addition (measured in mL) to the mass of acid per unit dry weight AMDp (given in g/g). Throughout the testing and analysis, this normalization was shown to produce the most consistent results, while providing a “fair” comparison between samples of different moisture content. The calculation used to normalize the data is given by:

\[ d = \frac{V_a w C}{M_w (1 - u)} \left( \frac{1}{1000} \right) \]  

[Eq. 1]

where \( d \) is the normalized acid dose (in g/g), \( V_a \) is the cumulative volume of acid added (in mL), \( w \) is the molecular weight of the acid (in g/mol), \( C \) is the acid concentration (in mol/L), \( M_w \) is the wet mass of the AMDp recorded before the test, and \( u \) is the AMDp moisture content. The conversion factor [1/1000] accounts for the conversion from mL to L needed in the numerator.

3.4.2 Empirical Modeling

After normalization, the data was found to be best presented as measured pH versus normalized acid dose on a semi-log x plot. This approach thus creates a unique acid consumption curve for each AMDp sample. To facilitate a quantitative statistical evaluation of the data, a non-linear empirical model was derived to simplify each curve to a number of fitting parameters. Several two and three factor models were evaluated; however, the model found to provide the best balance between fitting accuracy and fitting robustness is given by:

\[ pH(d) = pH_{Max} - \left( \frac{1}{1 + \exp \left( \alpha \left( 1 - \log \left( \frac{d}{d_{50}} \right) \right) \right)} \right) pH_{Max} \]  

[Eq. 2]

where \( pH(d) \) is the measured pH at a given acid dose, \( d \) [in g/g]. \( pH_{max} \), \( \alpha \), and \( d_{50} \) are fitting parameters for the empirical equation.
This model form is a slight modification to a standard “partition curve” that is often used for the analysis and modeling of particulate separations, such as screens, cyclones, and dense medium vessels (King, 2001). This specific model was selected over competing models because the fitting parameters directly refer to geometric intricacies of the acid consumption curve. Specifically, \( \text{pH}_{\text{max}} \) is the \( y \)-axis position of the high asymptote, or approximately the starting pH of the AMDp in an aqueous solution. \( \alpha \) is related to the slope of the curve at the inflection point. High \( \alpha \) values indicate a fast transition from high pH to low pH while lower \( \alpha \) values indicate a shallower slope. Lastly, \( d_{50} \) is related to the \( x \)-axis position of the inflection point. Low \( d_{50} \) values refer to a lower pH can be achieved at smaller acid dosages, while higher \( d_{50} \) values indicate a higher dosage is needed to produce low pH values.

Following the empirical modeling, several standard statistical methods (e.g. Pearson’s coefficient) were used to evaluate the correlation between the model fitting parameters (i.e. the acid consumption curve) and the inherent characteristics of the AMDp material. Moreover, this data was used to conduct a simple “process amenability” analysis to show which samples are able to reach a target pH point at some breakeven acid dose. While overall processing economics are beyond the scope of this study, this approach can be integrated into that analysis, as the breakeven acid dose can be calculated by comparing the acid cost to REE content of each sample (i.e. cost vs. benefit analysis).
Chapter 4: Results

4.1 Sample Characterization

4.1.1 X-Ray Fluorescence (XRF)

Initially, the handheld XRF scanner was used to determine the elemental content for 150 unique AMDp samples that were later evaluated in various leaching tests. Using the appropriate analytical settings, the XRF produced measurable assays for 29 different elements, including Ba, Sb, Sn, Mo, Nb, Zr, Sr, Rb, Bi, As, Pb, W, Zn, Cu, Ni, Co, Fe, Mn, Cr, V, Ti, Ca, K, Al, P, Si, Cl, S, and Mg. Figure 14 shows the average elemental distribution for all samples (only the top 5 elements are shown), while Figure 15 shows the maximum assay recorded from the population of AMDp samples for each element. In all the figures in this study that describe the XRF data, it should be noted that the section labeled as, “other” is just the summation of all the other elements that were deemed negligible on their own as well as the hydroxides and other anions that could not be processed by the XRF. As shown in these figures, iron was the predominant species, with an average content of 16.4% and a maximum of 62.2% in one specific specimen (SL-30-R). As expected, other major species include Ca, S, Si, Al, Mn, Mg, Cl, and K. The XRF also detected trace amounts of other elements which are also noted in Figure 15.

![Average of All Samples (n = 150)](image)

*Figure 14. Average assay for all AMDp samples, as determined by handheld XRF.*

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Figure 15. Maximum elemental concentration determined from full population of AMDp samples (n = 150).

This particular XRF instrument does not have the sensitivity needed to perform REE analysis, and thus, those elements are notably missing from the characterization results. Data from Ziemkiewicz et al., 2018 shows that the overall average REE content for this population of AMDp samples is 708.5 g/t.
Since the XRF analysis was conducted using a simple packed powder sample preparation technique, the results are less precise than that of more sophisticated methods, such as digestion followed by ICP-MS. Nevertheless, the XRF results can be used for comparative analysis between specimens as well as classification of the AMDp samples based on the primary elemental constituent. This classification was then subsequently used to interpret and analyze the other test results, including moisture and acid consumption.

The results of this primary element classification are shown in Figure 16 while Figure 17 shows the elemental distribution for the samples in each classification group. As indicated here, iron is the dominant species in approximately one third of the samples, and this specific group of samples has a much smaller fraction of the remaining major elements (<15% silica, calcium, aluminum, and magnesium combined). Likewise, another third of the samples are calcium-rich, while about 20% are rich in silica. The remaining 10% are classified as either aluminum-, manganese-, or sulfur-rich. While the calcium-rich and silica-rich samples have a large difference between the primary species and the next closest species, the aluminum-rich samples are much more uniformly dispersed. Altogether, these XRF results coincide with expectations, as iron, silica, and aluminum are routinely listed as the predominant major metals in AMD and AMDp. The calcium-rich samples are likely products of gross overtreatment, as the high calcium content is associated with unreacted lime.
Figure 16. Distribution of samples according to primary elemental species.

Figure 17. Average assay for AMDp samples, classified by primary species classification group.
4.1.2 Moisture Content

The moisture content was determined for each of the AMDp samples to express the acid consumption test results on a dry mass basis. A frequency distribution of the moisture data (Figure 188) shows that the plurality of samples are extremely high in moisture, with 37% of the samples exceeding 90% moisture and more than 60% of the samples exceeding 80% moisture.

![Moisture Content of Sludge Samples](image)

*Figure 18. Distribution of AMDp moisture results.*

Further analysis of the moisture data was conducted to determine the distribution of moisture values classified by primary elemental species. Figure 19 shows the results of this analysis. While the silica, iron, and calcium-rich specimens follow a similar range of moisture values, the aluminum-rich samples clearly show an elevated moisture content relative to the other groups. This result may be distorted due to the relatively small sample size (n = 14) of aluminum-rich specimens when compared to the other groups that have 30 to 50 samples. The distribution of the other samples appears to be reduced due to a couple anomalously low points, which are not present in the aluminum-rich population. In either case, the data
shows that the moisture content does not vary considerably between the different classifications of AMDp specimens.

![Figure 19. AMDp moisture data categorized by major elemental species.](image)

### 4.2 Acid Consumption Testing

Over the course of the experimental study, over 600 individual leaching test runs were completed (including replicate runs). As described in Section 3.3.5, these test runs were organized into five primary test series as well as a preliminary training test series. The results in this section have been organized by those test series delimiters. Moreover, the raw results from all acid consumption tests is included in an Appendix.

#### 4.2.1 Test Series 0: Preliminary Tests

The initial test series was used to assist in the method development and serve as a training exercise for the future test runs. For these initial tests, two arbitrary samples were evaluated (SL-1-10 and SL-3-1), and the acid addition was not conducted in a controlled manner as specified in Section 3.3.4. Rather, the acid was added in a single step in an attempt to reach a target pH point (pH = 2 or pH = 0.5) depending on
the test. After adding the acid, the solution as mixed for 30 minutes to ensure that the equilibrium pH was achieved. The results from this test are shown in Table 5.

Table 5. Experimental Results for Test Series 0: Preliminary Tests

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Sample ID</th>
<th>Acid</th>
<th>Acid at pH = 2 (kg/tonne)</th>
<th>Acid at pH = 0.5 (kg/tonne)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0a</td>
<td>SL-1-10</td>
<td>3M Nitric</td>
<td>98.8</td>
<td>--</td>
</tr>
<tr>
<td>0b</td>
<td>SL-1-10</td>
<td>3M Nitric</td>
<td>--</td>
<td>162.0</td>
</tr>
<tr>
<td>0c</td>
<td>SL-3-1</td>
<td>3M Nitric</td>
<td>--</td>
<td>107.5</td>
</tr>
<tr>
<td>1</td>
<td>SL-3-1</td>
<td>3M Nitric</td>
<td>286.6</td>
<td>367.8</td>
</tr>
</tbody>
</table>

Several lessons were learned from this initial test series that significantly influenced the method development and experimental design for future test series. First, the initial test protocol called for 30 minutes of reaction time after the acid addition. Observations during these four tests showed that the acid consumption reached steady state quite rapidly, generally within two minutes of introducing the acid. Moreover, the response of acid dose to pH was observed to be nonlinear in many cases, and thus the single acid dose was deemed to be of little value when compared to that of a controlled sweep of acid doses. Thus, the final experimental protocol dictated that acid be dosed in an exponential series with continuous pH measurements after each subsequent acid dose. Nevertheless, this test series confirmed the sufficiency of other experimental parameters, including the mixing intensity, acid concentration, acid type, and reaction temperature.

4.2.2 Test Series 1: Test Repeatability

After finalizing the test protocol that is described in Section 3.3.4, the first true experimental series was conducted to assess the reproducibility of the actual test protocol. This test series included 10 repeat test runs over three days on a single bucket of sample collected from a single site (SL-3-1). Figure 20 summarizes the results from this test series. The left panel shows the test results using the standard format (i.e. measured pH versus mass-normalized acid dose on a semi-log x axis), while the right panel shows the
standard deviation of the measured pH (as a percent of the mean) plotted against the normalized acid dose. In addition, a pie chart showing the XRF results is included as an inset. This data indicates that the test is fairly reproducible, particularly for acid doses less than 1 g/g and corresponding pH values greater 1.0. The variability in the test, as indicated by the standard deviation, increases considerably when the conditions exceed these thresholds. Nevertheless, it should be noted that the absolute standard deviation (c.f. the standard deviation normalized to the mean) stays relatively consistent across all pH and acid dose values. Given these results, all subsequent test series were conducted using three replicate tests for each desired condition. The corresponding results thus represent the average of the three replicates.

![Figure 20. Acid consumption test results for Test Series #1: Test Repeatability.]

### 4.2.3 Test Series 2: Influence of Solids Concentration

As part of the standard test procedure (Section 3.3.4), a wet solid to liquid ratio of 1:1 was used for most tests. Test Series #2 evaluated this standard parameter by determining the influence of solids concentration on acid consumption for a single test specimen (SL-21-5). These results are shown in Figure 21. The left panel shows the measured pH as a function of the raw acid dose, while the right panel shows the results after normalizing the x-axis to the dry AMDp mass, per [Eq. 1]. While the 1.5:1 sample showed some variability at the very low acid doses, these two plots otherwise clearly show the impact of the dry
mass normalization procedure in correcting for different solids concentrations. The position of the sharp pH drop varies considerably with respect to raw acid dose (left panel, recall the x-axis is on a log scale), with a low value of ~40 mL for an S:L ratio of 0.5:1 and a high value of ~130 mL for an S:L ratio of 1.5:1. However, the position of the sharp pH drop coincides to a value of ~1.8 g/g when the data is mass normalized. Altogether, this result confirms that any solid to liquid ratio is suitable for routine analysis, provided that the data is normalized to the dry AMDp mass. Moreover, this result proves that the moisture content is a significant value that must be accurately measured and integrated into the results. As a result of this test series, all subsequent tests were conducted using a wet solid to liquid ratio of 1:1, and the moisture content was determined concurrently with each test run.

![Figure 21. Acid consumption test results for Test Series #2: Influence of Solids Concentration.](image)

### 4.2.4 Test Series 3: Influence of Acid Type

The third parametric test series assessed the difference in acid consumption with respect to the acid type, namely considering hydrochloric, nitric, and sulfuric acid. These comparisons were repeated for three different test specimens, including two aluminum-rich samples and one manganese-rich sample (SL-51-1, SL-67-1, and SL-40-1). Overall, the results (Figure 22) show some variability between the various samples; however, the nature of the variation is not consistent between the test specimens. For example, in the SL-
51-1 tests, all three acids produced very similar results with a slight vertical offset between the three acid types. While nitric and hydrochloric acid fall within the same error bounds, sulfuric acid produced a slightly lower pH across all acid doses. A similar pattern is observed for SL-40-1; however, in this case, the difference between sulfuric and the other two acids is much more pronounced. For the final sample, SL-67-1, sulfuric and nitric largely overlap, but hydrochloric is notably elevated.

These results remain inconclusive in suggesting that one particular acid is universally superior to the other two; however, in all cases, nitric acid was not anomalously high or low. This results thus confirms that nitric acid is most suitable for subsequent test series.

Figure 22. Acid consumption test results for Test Series #3: Influence of Acid Type.
4.2.5 Test Series 4: Intra-Site Variation

The sample collection activities of this study were conducted in two phases: an exploratory phase that emphasized a large number of AMD treatment sites and a detailed phase that emphasized a more focused assessment on a smaller number of promising sites (see Section 3.2.1). In the detailed campaign, study, multiple buckets of sample, often 8 to 12, were recovered from select sites to better understand the variation of AMDp composition within storage ponds at a single site. Four specific sites from this detailed campaign (SL-66-1, SL-51-1, SL-67-1, and SL-40-1) were selected for a detailed acid consumption study. The results of these “Intra-Site Variation” acid leaching tests (Series #4) are shown in Figure 23. Please note that the XRF insets are omitted from this figure, as XRF analysis was not performed on each individual site subsample.

Figure 23. Acid consumption test results for Test Series #4: Intra-Site Variation.
With few exceptions, this data largely indicates that material from the same site is fairly consistent. Data from SL-66-1, SL-51-1, and SL-67-1 follow similar trends and typically only have one or two outlier samples that deviate from mean. SL-51-1 also had one sample (SL-51-1f) that had considerably low test reproducibility and thus resulted in large error bars as shown on the plot. Despite this high test uncertainty, the sample averages still fall within the bounds of the other data sets.

Contrary to the other samples in this study, SL-40-1 did not show the same degree of consistency. Many of these tests had considerably high error bars and the amount of acid needed to reach a target pH value spans more than one order of magnitude for some specific points. For example, to reach a target pH of 3.0, the amount of acid varied from less than 0.1 g/g to nearly 1.0 g/g depending on the specific test specimen. This result can be explained by the field sampling approach, as the subsamples were recovered from two distinct zones in the water treatment system. Samples a through i were recovered from a downstream storage pond, while j through l were recovered further upstream on the AMDp flow path. This difference in location likely contributes to the large discrepancy in acid consumption, as the AMDp materials likely have different composition and different forms depending upon the location within the treatment process.

While intra-site variation was not evaluated for other sites, this specific test series does provide some quantitative assessment of variability that can be used in rigorous error propagation calculations in the future. Furthermore, this data indicates that the site location alone does not mitigate the test variability, particularly in cases like SL-40-1 where AMDp is samples from multiple zones.

4.2.6 Test Series 5: Site-to-Site Variation

The principal outcome from the overall acid consumption study is the site variation data collected in Test Series #5. In this series, the standard acid leaching test (which by convention includes 3 replicates) was performed on one sample from each of the 153 sites sampled in broader effort. Data from each individual test is included in the Appendix, and summary data is included here for analysis and discussion. The results from all tests are shown in Figure 24. While individual results cannot be interpreted from this
plot, it does indicate the range of values recorded throughout the course of testing. For example, while most samples have starting pH (i.e. asymptotic y-intercept) that is near neutral, a second cohort has a starting pH value of 12.0 to 13.0. The high starting pH samples are mostly resultant from overdosed treatment and have considerable amounts of unreacted lime. The acid consumption in these samples shows a very consistent pattern that includes a very steep decline in pH after that free lime is presumably depleted. Moreover, another group of samples has a very low starting pH (<4.0) and minimal response to additional acid addition.

Figure 24. Acid consumption test results for Test Series #5: Site-to-Site Variation.

Figure 25 shows these same results classified by primary elemental species as determined by the handheld XRF. The categorization does show that there are unique trends observed for each AMDp classification; however, the data also indicates primary species alone cannot account for all the variation in the data set. The most significant difference is observed between the iron-rich and calcium-rich samples.
None of the calcium-rich samples have a starting pH (i.e. a pH value at 0 acid addition) below 6.0, and the only samples with a starting pH above 12.0 are in the calcium-rich group. The iron-rich samples, on the other hand, tended to have a lower starting pH, and most of the samples with a starting pH lower than 4.0 were in the iron-rich group. Interestingly, the silica-rich group shows some similarities between both the iron-rich and calcium-rich groups, and it should be noted that, on average, samples in the silica-rich group had equal parts iron and calcium (Figure 17). A further analysis of the influence of composition on starting pH is shown in Figure 26, which depicts the distribution of starting pH values classified by primary elemental species.

Figure 25. Acid consumption test results for Test Series #5: Site-to-Site Variation, classified by major elemental species.
4.3 Modeling & Statistical Analysis

4.3.1 Empirical Modeling

Given the magnitude of the data set and difficulty in direct quantitative comparisons of acid consumption curves, an empirical modeling approach was used to simplify the dataset by providing a limited number of fitting parameters that could be compared directly. A visual inspection of the typical acid consumption curves shows that three geometric features are needed to adequately define the shape of
the curve: (1) the y-axis location of the upper asymptote, (2) the x-axis location of the steep decent in measured pH (i.e. the inflection point of the cure), and (3) the slope of the curve at the inflection point. After evaluating several mathematical functions, [Eq. 2] was selected as the best model that could capture these three geometric features for each acid consumption curve. All 153 test runs in Test Series #5 as well as the 38 test runs in Test Series #4 were fit to the empirical model by using a nonlinear optimizer (Excel Solver) to minimize the sum of the squared error (SSQE) between the predicted and experimental values. The results from each fitting exercise are shown in the Appendix. Summary data is included here for discussion and analysis.

Overall, the empirical model provided exceptional fit to the majority of the dataset. Example of the best fit and the worst fit (as indicated by SSQE) are shown in Figure 27. The poor fit in the SL-36-1 sample can be explained by the shape of the acid consumption curve. This data set contains two inflection points (likely due to two major buffering ions), which is difficult to model using [Eq. 2]. Nevertheless, the overall trend is still captured, and more importantly, this double-asymptote behavior was only present in less than 10 the acid consumption curves. The gross majority of the data sets showed exceptional fit to the empirical mode.

---

**Figure 27.** Example of best (left panel) and worst (right panel) data fits from empirical modeling of all test results.
One advantage to this particular fitting equation is that the three model parameters directly correspond to geometric features of the curve. This benefit is depicted in Figure 28, which shows relatively extreme values for each of the fitting parameters. Recall, $pH_{\text{Max}}$ indicates the position of the high asymptote, $\alpha$ is the slope of the transition, and $d_{50}$ is the x-axis location of the inflection point.

Low Value

<table>
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<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
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<td>$pH_{\text{Max}}$</td>
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<tr>
<td>$\alpha$</td>
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</tr>
<tr>
<td>$d_{50}$</td>
<td>2.29E-4</td>
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</table>

High Value

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$pH_{\text{Max}}$</td>
<td>12.31</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>6.18</td>
</tr>
<tr>
<td>$d_{50}$</td>
<td>0.09</td>
</tr>
</tbody>
</table>

Figure 28. Examples of extreme values (and the resultant curves) for each of the three fitting parameters.
Initially, the model parameters were used to quantitatively assess the variation in the Test Series #4: Intra-Site Variation data set (originally shown in Figure 23). The distribution of model parameters from each site sub-sample are shown in Figure 29. These results provide some quantitative basis for the visual trends observed in the raw data. For example, all curves in the SL-66-1 set have very similar parameters, except for one low and one high outlier. These outliers are shown and quantified in the $pH_{Max}$ and $d_{50}$ datasets. These model fits also confirm large variability in other parameters, including $\alpha$ in SL-40-1 and $pH_{Max}$ in SL-51-1. Interestingly, the model data does indicate substantial overlap between the different sites, suggesting that, at least for these samples, the intra-site variation is just as significant as the site-to-site variation.

![Figure 29. Distribution of model fitting parameters determined from Test Series #4: Intra-Site Variation.](image)
A similar analysis was performed on the Test Series #5: Site-to-Site Variation dataset and categorized by primary elemental species. These results are shown in Figure 30 and largely confirm the visual observations from the categorized raw data in Figure 24 and Figure 25.

As a specific example, the distribution of $pH_{\text{max}}$ values has significant overlap between the silica, iron, and calcium-rich specimens; however, of these three, the iron-rich samples were the only group to include low values, < 5.0, and the calcium-rich samples were the only to include high values > 11.0. The aluminum-rich and other samples have relatively wide distributions, likely due to the low number of samples within these classifications. With respect to the $\alpha$ and $d_{50}$ parameters, most elemental groups cover a very similar range of values, approximately 1.0 to 4.0 for $\alpha$ and 0 to 0.02 for $d_{50}$. The calcium-rich samples are clear exceptions to this trend, as both the $\alpha$ value and the $d_{50}$ value are elevated above the other sample
classifications. This elevated $d_{50}$ value indicates that calcium-rich samples tend to require a high acid dose before a noticeable pH drop. Likewise, the high $\alpha$ value indicates that the rate of the pH drop is quite high. Fundamentally, this empirical result is further evidence that the calcium-rich sample correspond to AMD that has been over-treated and thus has significant free lime that must be neutralized during acid leaching.

It should also be noted that there were some anomalous outputs for the fitting parameters of Al-rich samples. In Figure 30, the pH max versus the primary elemental species plot shows that a large majority of the starting pH values for Al-rich samples were greater than 15, with some even greater than 20. Upon further investigation of the test and fitting data, it was discovered that some of the Al-rich samples had expected pH max values of 37, 41, and 98. In Figure 31, a comparison analysis was performed with one set of data including all the estimated pH max values while the other only including pH max points than had a value less than 15.

![Figure 31 Al-rich Outliers versus No Outliers](image)

This led to the conclusion that while most pH max values for this data set should fall between 6.5 and 7.5, however the model still produced anomalously high values $>15$. SL-90-1, an example empirical model (Figure 32) of an anomalously high pHmax, was then analyzed.
Upon further investigation, there was a lack of a clear y-asymptote as well as the initial slope at low acid doses causes the model fit to produce very high pH max values, often > 20 (or as high as 98 in this case). Visual observation of the plot also suggests that empirical model isn’t capturing all the geometric features of this particular curve. A careful review of the full dataset shows that this particular curve geometry (as indicated by anomalously high pHmax values) is typically associated with aluminum-rich samples, though not all aluminum-rich samples have this geometry.

4.3.2 Statistical Analysis

Attempts to directly correlate the model parameters to observable AMDp parameters largely proved unsuccessful. To this end, Figure 33 shows the three fitting parameters plotted individually against the elemental assays of Fe, Ca, Si, and Al. Similarly

Table 6 shows a matrix of Pearson’s coefficients for the fitting parameters, moisture, and major elemental assays. These data show that most data series have little or no correlation to the other series. One exception is Ca, which has a significant positive correlation to both α (Pearson’s = 0.64) and d$_{50}$
(Pearson’s = 0.72). These results are expected, given the plurality of supporting evidence in Figure 25 and Figure 30. Furthermore, these results suggest that the acid consumption curve is difficult to predict from raw AMDp data alone. With the current state of knowledge, the acid consumption data is easier to determine experimentally than to predict fundamentally.

Figure 33. Cross-plots showing the three fitting parameters ($pH_{Max}$, $\alpha$, and $d_{50}$) plotted against Fe, Ca, Si, and Al assay values.
Table 6. Pearson’s Correlation Coefficient for Fitting Parameters and AMDp Elemental Assays

<table>
<thead>
<tr>
<th></th>
<th>pHMax</th>
<th>alpha</th>
<th>d50</th>
<th>Moisture</th>
<th>Fe</th>
<th>Ca</th>
<th>Si</th>
<th>Mn</th>
<th>Mg</th>
<th>Al</th>
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<td>0.27</td>
<td>-0.45</td>
<td>-0.41</td>
<td>0.53</td>
<td>0.24</td>
<td>-0.26</td>
<td>1.00</td>
</tr>
</tbody>
</table>

One interesting observation is the relationship between the measured starting pH measured in the lab (prior to any acid addition), and the pHMax model value (shown as a parody plot in Figure 34). While the model parameter is intended to be indicative of the starting pH, it is not a direct equivalency, as Equation 2 is undefined at an acid dose of 0 g/g. Nevertheless, Figure 34 shows that there is fair agreement between the measured pH and the model parameter, with the model parameter slightly overestimating the measured pH in most cases. One group of samples showed a relatively low measured pH (4 to 7) but a relatively high model pHMax (9 to 13). This outcome is likely an artifact of the model fit for a particular shape of data.
4.4 Discussion & Implications

The cumulative results of the acid leaching tests provide a quantitative evaluation of the acid doses needed to reach target leach pH values and also provide some indication on the variability that can be expected from site to site. While the variation due to test reproducibility, solid concentration, and acid type are small and manageable, the intra-site variation (particularly on sites processing different types of material) can be significant and should be assessed empirically in future studies. Nevertheless, the site-to-site variation and the variation between AMDp samples with different elemental compositions proved to be the most significant in the analysis. While the full acid consumption curve can be adequately modeled with a three-parameter non-linear equation, the actual fitting parameters cannot be readily predicted from
the composition and moisture data. A more fundamental assessment may provide a pathway to this end, but that activity is out of the scope of the current study.

The models and analysis from these tests can be used to guide future exploration efforts by deemphasizing AMDp classes that are high acid consumers and are likely cost prohibitive to process by acid leaching. For example, one method to utilize and apply the current data set is to assign a target acid dose (perhaps based on an economic or process design metric), use the model parameters to solve for the output pH for each AMDp sample, and classify the resultant pH data based on primary elemental species.

An example of this analysis is shown in Figure 35 for arbitrary acid doses of 0.1 g/g (left panel) and 0.5 g/g (right panel). The results are depicted as the distribution of output pH values, categorized by primary species. If one assumes a target pH of < 3.0, the results in Figure 35 indicate that the majority of iron-rich samples are able to reach that target at the 0.1 g/g acid dose. A smaller number of silica and aluminum-rich samples meet this target; however, only the outlier calcium-rich samples qualify. At the higher acid dose of 0.5 g/g, nearly all silica, iron, and aluminum-rich samples meet the pH < 3.0 target, as well as approximately 50% of the calcium-rich samples. With the full population of sample data and model parameters, this analysis can easily be repeated using different acid doses and target pH values as dictated by the recovery process design and process economics. In any case, this data shows that future exploration targets should prioritize iron-rich samples and deemphasize calcium-rich samples based on the relative level of acid consumption between these two groups.
Figure 35. Distribution of output pH values categorized by primary elemental species. Output pH was predicted [Eq. 2] at an arbitrary acid dose of 0.1 g/g (left panel) and 0.5 g/g (right panel). All other fitting parameters were derived from the raw data for each AMDp sample.
Chapter 5: Conclusion & Recommendations

5.1 Conclusions

Over 600 leaching tests were conducted on AMDp specimens recovered from both the preliminary and intensive sampling campaigns. In addition to the leaching tests, a handheld XRF was used to determine the concentration of major elemental species in each AMDp sample, as well as performing a moisture analyses to correct the values to a dry weight basis.

Altogether, these tests characterized the response of pH with respect to increasing acid dose, and a variety of test series evaluated the variation in these results with respect to: test reproducibility, solids concentration, acid type, intra-site variation, and site-to-site variation. While the variation due to test reproducibility, solid concentration, and acid type are small and manageable, the intra-site variation (particularly on sites processing different types of material) can be significant and should be assessed empirically in future studies. Nevertheless, the site-to-site variation and the variation between AMDp samples with different elemental compositions proved to be the most significant in the analysis.

Lastly, a three-parameter empirical model was found to robustly and accurately fit the acid consumption curves. While this model does provide a mechanism for analyzing and assessing the data, the actual fitting parameters cannot be readily predicted from the composition and moisture data. A more fundamental assessment may provide a pathway to this end, but that activity is out of the scope of the current study. A procedure for utilizing the model to assess process amenability has been proposed and demonstrated.

5.2 Recommendations for Future Study

While this research has provided a mechanism for analyzing and assessing AMDp data, further work is required to determine more accurate predictive models. The author of this thesis recommends the following areas in which additional research may be continued:
1. Further assessment of the data in order to provide more accurate predictions for fitting parameters for AMDp data fundamentally. The model parameters in this study could not be readily predicted from the elemental assays, the moisture data, and fitting parameters. Therefore, a more rigorous analysis could yield promising predictive results, and in turn, create a way to predict acid consumption data of AMDp fundamentally rather than experimentally.

2. Further investigation on the different treatment options for REE-rich AMD. Many of the samples in this study were over saturated by AMD treatment resulting in high amounts of acid needed in order to dissolve the AMDp. This could lead to the stop of over treatment of AMD, be less expensive to extract, and produce more prospective sites.

3. Further analysis of the elemental composition of the AMDp sample and correlations that could be yielded from that. This includes which specific REEs are present (and most frequent) in the prospective samples and the environment in which they were produced.
References


Brian W. Stewart, Rosemary C. Capo, Benjamin C. Hedin, Robert S. Hedin. 2016. “Rare Earth Element Resources in Coal Mine Drainage and Treatment Precipitates in the Appalachian Basin, USA.”

Chi Ruan, Tian Jun Li Zhongjun Peng Cui Wu Tuaxin Li Shirong Wang Cunwen Zhou Zhiang. 2005. “Existing State and Partioning of Rare Earth on Weathered Ores.” *Journal of Rare Earths*.


National Environmental Technology Laboratory. www.netl.doe.gov/. 2018

Paul Ziemkiewicz, Tom He, Aaron Noble, Xinbo Lie. 2016. “Recovery of Rare Earth Elements (REEs) from Coal Mine Drainage.”


Skousen, Jeff. Reclamation of Disturbed Soils. 2015.


Ziemkiewicz, Paul, et al. REE Identification and Characterization of Coal and Coal By-Products Containing High Rare Earth Element Concentrations. Department of Energy, 2018, pp. 1–104, REE Identification and Characterization of Coal and Coal By-Products Containing High Rare Earth Element Concentrations
Appendix

The following appendix contains the experimental data from all acid consumption tests categorized by test series. The appendix also contains the fitting results for the empirical modeling exercises.

Fitting Tests SL-1-1 through SL-140-2
Site Variation Test Results Including XRF Data

### General Test Information
- **Test No.**
- **Sample ID**
- **Pre-processing**
- **Sample Desc.**
- **Stirrer Speed**
- **Temperature**
- **Test Notes**

### Initial Test Data
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample** (g)
- **Moisture 1: Dry sample** (g)
- **Acid Concentration** (mol/L)
- **Sludge Moisture 1** (g)
- **Sludge Moisture 2** (g)
- **Sludge Moisture 3** (g)

### Moisture Data
- **Moisture - 2: Wet Sample** (g)
- **Moisture - 2: Dry sample** (g)
- **Time**
- **Acid Type**
- **Acid molar mass** (g/mol)
- **Sludge Wet Mass** (g)
- **Sludge Dry Mass** (g)
- **Sludge Volume** (ml)
- **Water Added** (ml)
- **ml**
- **Chloride**

### Analytical Reports
- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**

### Calculations
- **Acid**
- **Measured**
- **Average**
- **Cumu. Acid Added** (g)
- **Acid / Unit Mass Sludge** (g/g)

### Data Entry

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHF for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 8 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHF.

### Legend
- **Data Input**
- **Concentrate**
- **Calculation**

### Key Result
- **Description Text**

### Diagram
- **SL-3-1**
- **Fe**, **Al**, **Si**, **Ca**, **Other**
- **Acid / Unit Mass Sludge** (g/g) vs. **pH**

**Notes:**
- Include in Avg
- **x**
**General Test Information**

**Initial Test Data**

- **Test No.**
- **S:L Mass Ratio** -- 1:1
- **Moisture**
  - **1: Wet Sample**
  - **g** 100.93
- **Test by**
  - **Acid Type**
  - **Nitric**
- **Moisture**
  - **1: Dry Sample**
  - **g** 39.9

**Analytical Reports**

- **Date**
- **Acid Concentration**
  - **mol/L** 3
- **Sludge Moisture**
  - **1**
  - **--**
  - **0.60**
- **Time**
- **Acid Molar Mass**
  - **g/mol** 63.01
- **Moisture**
  - **2: Wet Sample**
  - **g** 100.18
- **Sample ID**
- **Temperature**
  - **°C** 22
- **Moisture**
  - **2: Dry Sample**
  - **g** 45.33
- **Sample Desc.**
- **Stirrer Speed**
  - **RPM**
- **Moisture**
  - **3: Wet Sample**
  - **g** 100.75
- **Sample ID**
- **Temperature**
  - **°C**
- **Moisture**
  - **3: Dry Sample**
  - **g** 52.39

**Pre-processing**

- **Sample ID**
- **Sample Desc.**
  - **Black, Grey Sludge**
  - **Directly sampled from bucket, returned frozen**

**Data Entry**

<table>
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<tr>
<th>Sludge Acid Titration Test - Experimental Data Entry Sheet</th>
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<tr>
<td><strong>General Test Information</strong></td>
</tr>
<tr>
<td><strong>Initial Test Data</strong></td>
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<tr>
<td><strong>Analytical Reports</strong></td>
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<td><strong>Pre-processing</strong></td>
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**Calculations**

<table>
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<th>Calculations</th>
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</thead>
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<tr>
<td><strong>Decimetal Number</strong></td>
</tr>
<tr>
<td>-----------------------</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

**Legend**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Descriptive Text**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Perform moisture analysis on at least 3 feed sludge samples.
4. Add feed sludge to reaction vessel. Record volume and mass.
5. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Measure initial pH of solution.
8. Add desired volume of acid, wait until reaction is complete, measure and record pH.
9. Repeat step 8 until endpoint pH is reached (pH ≤ 7)
10. Dispose of any waste according to CHP.
**General Test Information**

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<tr>
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**Initial Test Data**

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<th>0.82</th>
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</table>

**Procedure:**

1. Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Result
- Calculation
- Description Text

---

**Data Entry**

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<th>Measured 3</th>
<th>Measured 4</th>
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<th>Sl. Dev</th>
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<td>0.14</td>
<td>0.12</td>
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**Calculations**

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<th>Cumulative Acid Added</th>
<th>Acid Added</th>
<th>Sludge Dry Mass (g/g)</th>
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<td>0.70</td>
<td>0.70</td>
<td>0.70</td>
</tr>
<tr>
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<td>0.30</td>
<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Legend**

- Acid / Unit Mass Sludge (g/g)
- Other
- Al, K, Na, Mg

---

**Chart**

- SL-1-1

---

**Table**

<table>
<thead>
<tr>
<th>Analytical Reports</th>
<th>Test by</th>
<th>Acid Type</th>
<th>Nitric</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
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</table>

<table>
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<th>Sludge Moisture 1</th>
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</tr>
</thead>
</table>

| Sludge Moisture 1 | -- | 0.82 |

<table>
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<tr>
<th>Time</th>
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<th>g/mol</th>
<th>Sludge Moisture 1</th>
<th>--</th>
<th>0.83</th>
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</thead>
</table>

| Sludge Moisture 1 | -- | 0.83 |

---

**Sludge Volume**

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<tr>
<th>Sample ID</th>
<th>Temperature</th>
<th>rpm</th>
<th>Stirrer Speed</th>
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</table>

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**Additional Calculations**

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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added</th>
<th>Acid Added</th>
<th>Sludge Dry Mass (g/g)</th>
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</thead>
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<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>2</td>
<td>0.15</td>
<td>0.25</td>
<td>0.25</td>
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</tr>
<tr>
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<td>0.20</td>
<td>0.45</td>
<td>0.45</td>
<td>0.45</td>
</tr>
<tr>
<td>4</td>
<td>0.25</td>
<td>0.70</td>
<td>0.70</td>
<td>0.70</td>
</tr>
<tr>
<td>5</td>
<td>0.30</td>
<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Note:** Include in Avg x x x x x
### General Test Information

- **Test No.** 1
- **Date** 2017.5.1
- **Sample ID** SL-1-5
- **Sample Desc.** Black, Grey Sludge
- **Pre-processing** Black, grey sample from bucket, removed Tramp

### Initial Test Data

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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
<th>Moisture 1: Dry sample</th>
<th>g</th>
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</table>

### Analytical Reports

- **Acid Type:** Nitric
- **Acid Concentration:** mol/L
- **Acid Molar Mass:** g/mol
- **Sludge Moisture 1**:
  - g: 0.79

### Test Notes

- **Sample ID**
- **Temperature** c
- **Stirrer Speed** RPM
- **Time Step** min

### Data Entry

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<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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</table>

### Calculations

- **Cumu. Acid Added**
- **Acid Added**
- **Acid Added / Sludge Dry Mass (g/g)**

---

**Legend**

- **Test Type**
- **Sample ID**
- **Sample Desc.**
- **Pre-processing**
General Test Information

Test No. 1

Sample ID 1-01

Sludge Moisture 1

Sludge Moisture 1: Wet Sample

Moisture 1: Wet Sample

g 100.38

Moisture 1: Dry Sample

g 68.61

Date 2017.5.2

Acid Type Nitric

Analytical Reports

Pre-processing Sample directly sampled from bucket, removed Tramp

Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Legend

Test Input

Calculation

Descriptive Text

Data Entry

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<tr>
<th>Increment</th>
<th>Acid Added</th>
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<th>Measured Volume</th>
<th>Measured Volume</th>
<th>Measured Volume</th>
<th>Average</th>
<th>SL Dry Mass</th>
</tr>
</thead>
<tbody>
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<td>(mL)</td>
<td>(mL)</td>
<td>(mL)</td>
<td>(mL)</td>
<td>(mL)</td>
<td>(mL)</td>
<td>(g)</td>
</tr>
<tr>
<td>1</td>
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<td>2.00</td>
<td>4.00</td>
<td>6.00</td>
<td>8.00</td>
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<td>12.00</td>
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Calculations

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<th>Average</th>
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<th>Cumus. Acid Added (g)</th>
<th>Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g / g)</th>
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</thead>
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<tr>
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<td>(mL)</td>
<td>(mL)</td>
<td>(g)</td>
<td>(g / g)</td>
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Calculations

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SL-23-1

Legend

Test Input

Calculation

Descriptive Text

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Include in Avg
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### Test Notes

- Include in Avg
- x

### Data Entry

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### Calculations

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</table>

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- Red
- Orange
- Sludge
- N/A

### Graph

- SL-24-2

---

80
### General Test Information
- Test No. 1
- Date 4.5.17
- Sample ID S:L 21-5

### Initial Test Data
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio** -- 1:01
  - **Moisture 1: Wet Sample** g 100.96

### Acids
- **Acid** Nitric
- **Acid Concentration** mol/L 3
- **Acid molar mass** g/mol 63.01
- **Sludge Moisture 1** -- 0.33
- **Time**
- **Acid Concentration** mol/L 3
- **Acid Molar Mass** g/mol 63.01
- **Moisture 2: Wet Sample** g 100.93
- **Sample ID**
- **Sample Description** Wet Sludge

### Pre-processing
- **Location**
- **Type**
- **Sample ID**
- **Sample Description**
- **Date** 4.5.17

### Test Notes
- **Stirrer Speed** RPM
- **Temperature** c 22
- **Sample ID**
- **Sample Description**
- **Date** 4.5.17

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### Data Entry

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### Calculations

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### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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10. Record step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend
- **Test ID**
- **Sample ID**
- **Sample Description**
- **Sample Description**
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
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**Procedure:**
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**
- Test Equal
- Waist Disposal
- Calculated
- Key Result
- Description Text

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**Data Entry**

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**Calculations**

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**Legend:**
- Water Added
- Initial Volume
- pH
- Sludge pH
- Sludge Dry Mass
- Sludge Moisture 1
- Sludge Moisture 2
- Sludge Moisture 3
- Sludge Volume
- Test Notes
- Test No.
- Acid Type
- Date
- Acid Concentration
- Acid molar mass
- Temperature
- Stirrer Speed
- Sample ID
- Sample Desc.
- S:L Mass Ratio
- Moisture 1: Wet Sample
- Moisture 1: Dry Sample
- Moisture 2: Wet Sample
- Moisture 2: Dry Sample
- Moisture 3: Wet Sample
- Moisture 3: Dry Sample
- Rep 1
- Rep 2
- Rep 3
- Rep 4
- Rep 5
- Increment
- Number
- pH
- pH Added
**General Test Information**

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**Initial Test Data**

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**Moisture Data**

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**Analytical Reports**

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**Calculations**

- **Increment**
  - Acid Added (ml)
  - Measured pH
  - Measured Unit Mass Sludge (g/g)
- **Average**
  - Cumulative Acid Added (g)
  - Acidity Added / Unit Mass Sludge (g/g)

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
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<th>Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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<td></td>
<td></td>
<td>ml</td>
<td>g</td>
<td>(g / g)</td>
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**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Initial
- Calculation
- Key Result
- Description Text

---

**Graph**

- Title: Acid Unit Mass Sludge (g/g) vs. pH
- Axes: x-axis: Acid Unit Mass Sludge (g/g), y-axis: pH
- Data points and trend line indicating decrease in pH with increase in acid concentration.
**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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11. Dispose of any waste according to CHP.

**Legend:**

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text
# General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration %</th>
<th>Acid Molar Mass g/mol</th>
<th>Temperature °C</th>
<th>Stirrer Speed RPM</th>
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# Moisture Data

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# Test Notes

- Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- Add dilute acid solution at desired acid concentration. Record acid concentration.
- Calibrate pH meter if necessary.
- Perform moisture analysis on at least 3 feed sludge samples.
- Add feed sludge to reaction vessel. Record volume and mass.
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- Set desired temperature and stirrer speed. Thoroughly mix.
- Measure and record initial pH of solution.
- Add desired volume of acid, wait until reaction is complete, measure and record pH.
- Repeat step 9 until endpoint pH is reached (<0.5).
- Dispose of any waste according to CHP.
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**
- **Date**
- **Acid Type**
- **Acid Concentration**
- **Acid Moles**
- **Acid Moles/mL**
- **Acid Moles/L**
- **Temperature**
- **Reactor Speed**
- **Moisture 2: Wet Sample**
- **Moisture 2: Dry Sample**
- **Time**
- **Acid Molar Mass**
- **Sludge Moisture 1**
- **Sludge Moisture 2**
- **Sample ID**
- **Sample Desc.**
- **Sample Type**
- **Pre-processing**
- **Analytical Reports**

**Analytical Reports**

- **Nitric**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**
- **Sample ID**
- **Sample Desc.**
- **Sample Type**
- **Pre-processing**
- **Analytical Reports**

**Sludge Moisture 1**

- **Sludge Moisture 1**
- **Sludge Moisture 2**
- **Sample ID**
- **Sample Desc.**
- **Sample Type**
- **Pre-processing**
- **Analytical Reports**

**Calculations**

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**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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---

**Legend**

- Test Entry
- Data Entry
- Descriptive Text
- Calculations
- Key Result
- Description Text

---

**Sludge Acid Titration Test - Experimental Data Entry Sheet**

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<th>Increment Number</th>
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<th>Result (g)</th>
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---

**Graph:**

- **SL-18-1**
- **pH vs. Acid Added**
- **Acid Added vs. pH**

---

**Data Entry**

- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**

---

**Calculation**

- **Acid Added**
- **Sludge Dry Mass**
- **Sludge Wet Mass**
- **Average Moisture**
- **Water Added**

---

**Legend**

- Black, Aqueous Sludge
- N/A
- Directly sampled from bucket, removed Tramp
- SL-18-1

---

**Note:**

Include in Avg "x"
**General Test Information**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration mol/L</th>
<th>Acid Molar Mass g/mol</th>
<th>Temperature °C</th>
<th>Stirrer Speed RPM</th>
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**Analytical Reports**

- **Pre-processing**: Sludge (from bucket) removed Tramp
- **Sample Description**: Directly sampled from bucket, removed Tramp

**Test Notes**

- **Sample ID**: SL-24-1
- **Temperature**: 22 °C
- **Time Step**: 5 min
- **Analytical Reports**: Nitric acid

**Calculations**

<table>
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<tr>
<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
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**Legend**

- **Test Type**: pH
- **Key Result**: pH
- **Description Text**: pH
**General Test Information**

- **Test No.**: 14
- **Sample Desc.**: Red Sludge
- **Sample ID**: SL-22-5
- **Date**: 2017.5.11

**Moisture Data**

| Test No. | S:L Mass Ratio | Moisture 1: Wet Sample | g |
|----------|----------------|------------------------|
| 1:01     |                | 100.13                 |
|          |                | 61.62                  |

**Test by Acid Type**

- **Nitric Acid**
- **Acid Concentration**: 3 mol/L
- **Sludge Moisture 1**: 0.39 g
- **Temperature**: 22 c
- **Stirrer Speed**: RPM
- **Date**: 2017.5.11

**Additional Test Information**

- **Moisture 2**: Wet Sample - 100.13 g
- **Moisture 3**: Wet Sample - 100.18 g
- **Moisture 2**: Dry Sample - 57.98 g
- **Sludge Moisture 1**: 0.39 g
- **Sludge Moisture 1**: 0.42 g

---

**Procedure**

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3. Calibrate pH meter if necessary.
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

---

**Legend**

- **Rep 1**: Increment 1
- **Rep 2**: Increment 2
- **Rep 3**: Increment 3
- **Rep 4**: Increment 4
- **Rep 5**: Increment 5

**Calculations**

<table>
<thead>
<tr>
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<th>Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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**Data Entry**

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</table>

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**Diagram**

- **Sl-22-5**

---

**Note:** Include in Avg x
### General Test Information

**Test No.**
SL-10-1

**Initial Test Data**

**Moisture Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>Moisture</th>
<th>g</th>
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<tbody>
<tr>
<td>1</td>
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**Date**
2017.5.16

**Acid Type**
Nitric

**Acid Concentration**
mol/L
3

**Moisture 1: Wet Sample**
g
100.13

**Acid Molar Mass**
g/mol
63.01

**Nitric Acid**

**Moisture 2: Wet Sample**
g
100.18

**Acid Volume**
l
150.0

**Moisture 2: Dry Sample**
g
13.86

**Sample ID**
SL-10-1

**Sample Desc.**
Red Sludge, Fudge-Like

**Pre-processing**

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<thead>
<tr>
<th>Step</th>
<th>Time</th>
<th>Stirrer Speed</th>
<th>RPM</th>
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**Analytical Reports**

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**Data Entry**

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<th>Acid Added (g)</th>
<th>Measured pH</th>
<th>Measured Slush Dry Mass (g)</th>
<th>Average Slush Dry Mass (g)</th>
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**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Equal**
- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.
**General Test Information**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
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<tr>
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<td>0.87</td>
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**Test by**

<table>
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<th>Acid Type</th>
<th>Moisture 1: Dry Sample (g)</th>
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<tr>
<td>Nitric</td>
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**Initial Test Data**

<table>
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<tr>
<th>Date</th>
<th>Acid Concentration (mol/L)</th>
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**Moisture Data**

<table>
<thead>
<tr>
<th>Time</th>
<th>Acid Moles (g)</th>
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<tbody>
<tr>
<td>Sludge</td>
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**Analytical Reports**

<table>
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<th>pH</th>
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**Pre-processing**

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**Test Notes**

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<th>Stirrer Speed (RPM)</th>
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<td>med</td>
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**Calculations**

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<th>Average pH</th>
<th>Cumulative Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>St. Dev</th>
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<td>1.00</td>
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</table>

**Legend**

- **Key Result**: Test Result
- **Description Text**: Test Notes
- **Data Entry**: Table of Data Entry
- **Calculations**: Table of Calculations
- **Procedure**: Experimental Procedure

**Procedure**

1. Gather adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 fresh sludge samples.
5. Add fresh sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio. Record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
# General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture Data</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>1:01</td>
<td>Moisture 1: Wet Sample g 100.54</td>
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<tr>
<td></td>
<td></td>
<td>Moisture 2: Wet Sample g 100.54</td>
</tr>
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<td>Moisture 5: Wet Sample g 100.54</td>
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<td>Moisture 10: Wet Sample g 100.54</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture Data</th>
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<tbody>
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<td>1:01</td>
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<tr>
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<td>Moisture 10: Dry Sample g 27.93</td>
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</table>

# Analytical Report

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

# Legend

- **Test Type**
- **Pre-processing**
- **Analysis Reports**
- **Calculations**
- **Key Result**
- **Test Notes**

---

### Data Entry

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added (ml)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>Sl. Dry Mass (g)</th>
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### Calculations

<table>
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<th>Increment</th>
<th>Average pH</th>
<th>Cum. Acid Added (ml)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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---

### Procedure

1. Determine the mass of sludge and water needed for the reaction.
2. Set the desired temperature and stirrer speed.
3. Mix the sludge and water thoroughly.
4. Measure the initial pH of the solution.
5. Add the desired volume of acid to the solution.
6. Wait until the reaction is complete.
7. Measure and record the final pH of the solution.
8. Repeat steps 5-7 until the endpoint pH is reached (<0.5).
9. Dispose of any waste according to CHP.

---

### Feasibility

- **Note:** Include in Avg

---

### Sludge Wet Mass

- **SL-2-2**
- Fe, 25.1
- Al, 8.1
- Si, 7.6
- S, 2.1
- Other, 57.1
General Test Information

Test No. 3
Test No. 2
Test No. 1
Sample ID. SL-8-1
Sample Desc. Light Brown, Gravy-Like Sludge
Pre-processing. Directly sampled from bucket, removed Tramp

Analytical Reports

Test Notes

Initial Test Data

Moisture Data

Test No. S:L Mass Ratio -- 1:01 Moisture 1: Wet Sample g 100.88
Test by Acid Type Nitric Moisture 1: Dry sample g 39.1
Date 2017.5.19 AM
Acid Concentration mol/L 3
Sludge Moisture 1 -- 0.61
Acid molar mass g/mol 63.01
Sludge Moisture 1 -- 0.68
Acid / Unit Mass Sludge (g/g) SL-8-1 Si, 12.2 Ca, 11.6 Fe, 8.4 Al, 5.9 Other, 61.8

Moisture Data

Moisture 1: Wet sample g 100.0
Moisture 2: Wet sample g 100.0
Moisture 3: Wet sample g 100.0
Moisture 1: Dry sample g 39.1
Moisture 2: Dry sample g 29.85
Moisture 3: Dry sample g 31.83

Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Data Entry

Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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Legend

Key Result Description Text

Test Notes

Inc. pH Measured Measured Measured Measured Average SL. Dev.
1 | 6.86 | 6.86 | 6.86 | 6.86 | 6.86 | 6.86 |
2 | 7.54 | 7.54 | 7.54 | 7.54 | 7.54 | 7.54 |
3 | 8.18 | 8.18 | 8.18 | 8.18 | 8.18 | 8.18 |
4 | 8.82 | 8.82 | 8.82 | 8.82 | 8.82 | 8.82 |
5 | 9.41 | 9.41 | 9.41 | 9.41 | 9.41 | 9.41 |
6 | 10.08 | 10.08 | 10.08 | 10.08 | 10.08 | 10.08 |

Note: Include in Avg "x"
Sludge Acid Titration Test - Experimental Data Entry Sheet

### General Test Information
- **Test No.**
- **Sample ID.**
- **Date.**
- **Sample Description.**

### Initial Test Data
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

### Physical Properties
- **Sample Processing**
- **Sample Collect From**

### Analytical Reports
- **Moisture 2: Wet Sample**
- **Moisture 2: Dry Sample**
- **Moisture 3: Wet Sample**
- **Moisture 3: Dry Sample**

### Test Notes
- **Sample ID.**
- **Temperature**
- **Stirrer Speed**

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations:
- **Incubation Number**
- **Acid Added (mL)**
- **Cumu Acid (mL)**
- **Acid Added / Unit Mass Sludge (g/g)**

### Key Results:
- **pH**

---

### Legend:
- **Test No.**
- **Sample ID.**
- **Sample Description.**
- **Date.**
- **Sample Processing.**
- **Sample Collect From.**

---

### Sludge Acid Titration Test

<table>
<thead>
<tr>
<th>Incubation</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</table>

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### Graph:
- **Acid / Unit Mass Sludge (g/g)**
- **pH**
**General Test Information**

**Initial Test Data**

- **Sample ID**: SI-27-1
- **Sample Description**: Red Aqueous Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

**Moisture Data**

- **Test No.**: S:L Mass Ratio
  - **1:01**
- **Moisture 1: Wet Sample**: 101.59 g
- **Moisture 1: Dry Sample**: 3.25 g
- **Date**: 2017.5.23
- **Acid Concentration**: 3 mol/L
- **Acid Molar Mass**: 63.01 g/mol
- **Sludge Moisture 1**: 0.97
- **Time**: 5 min
- **Stirrer Speed**: 12 RPM
- **Initial Volume**: 200.0 mL

**Calculations**

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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumus. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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<td>(mL)</td>
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<td></td>
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</tbody>
</table>

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**
- **Increment**
- **Acid**
- **Measured**
- **Average**
- **SL Dry Mass**
- **Initial Volume**

**Notes**

- Include in Avg: 
  - **x**
  - **y**

**Graph**

- **SL-27-1**

**Data Entry**
### General Test Information
- **Test No.**: 21.1
- **Sample ID**: 295-1
- **Sample Desc.**: Red Extremely Aqueous Sludge
- **Sample Size**: Directly sampled from bucket, removed Tramp
- **Pre-processing**: Sample was stirred for 3 hours, Homogenized
- **Reduction**: Sample was reduced to 100 ml

### Initial Test Data

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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture</th>
<th>Temperature</th>
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<th>Initial Test Data</th>
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### Analytical Reports

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<th>Temperature</th>
<th>Sludge Moisture 1</th>
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<tbody>
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</table>

### Test Notes
- Sludge Wet Mass: 100.0 g
- Sludge Dry Mass: 1.6 g
- Water Added: 100.0 ml
- Initial Volume: 100.0 ml

### Test Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average pH</th>
<th>Cum. Acid Added (mol)</th>
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### Legend
- **Key Result**: Accuracy
- **Description Text**: N/A

---

**Note:**
Include in Avg any values removed from calculation.

---

**Graph:**
- SL-27-1
- **Y-axis**: Avg pH
- **X-axis**: Acid / Unit Mass Sludge (g/g)

---

**Data Entry**

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<tr>
<th>Data Entry</th>
<th>Rep 1</th>
<th>Rep 2</th>
<th>Rep 3</th>
<th>Rep 4</th>
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**Legend**
- **Text Input**: N/A
- **Numeric Input**: N/A
- **Calculation**: N/A
- **Key Result**: Accuracy

---

**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
### General Test Information

- **Test No.**
- **Sample Desc.**
- **Sample ID**

### Initial Test Data

- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample**

### Test by Acid Type

- **Nitric**
  - **Moisture 1: Dry sample**
  - **Date**
  - **Acid Concentration**
  - **Acid molar mass**
  - **Sludge Moisture 1**
  - **Time**
  - **Sludge Moisture 1**
  - **Sample ID**

### Calculations

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<th>Increment</th>
<th>Acid Added (mL)</th>
<th>pH</th>
<th>Sludge Dry Mass (g)</th>
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### Analytical Reports

- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Type**
- **Instruments**
- **Calculations**
- **Key Results**
- **Description Text**

### Data Entry

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added (mL)</th>
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<th>Measured Sludge Dry Mass (g)</th>
<th>Measured Sludge Wet Mass (g)</th>
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<td>0.36</td>
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### Calculations

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<th>Cumu. Acid Added (mL)</th>
<th>Cumu. Acid Added (mol)</th>
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</tbody>
</table>

### Notes:

- Include in Avg

### Results

- **Fe**, 50.4
- **S**, 4.1
- **Si**, 3.3
- **Al**, 1.9
- **Other**, 40.3
# General Test Information

**Test No.** 1:01  
**Sample Desc:** Red-Black Sludge, Fudge-Like  
**Sample Source:** Directly sampled from bucket, removed Tramp  
**Pre-processing:** Sludge wet mass  
**Sample ID:** SL-2-5  
**Sample Type:** Acid / Unit Mass Sludge (g/g)  

## Initial Test Data

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**Note:** Include in Avg: x

**Values:**  
1. Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.  
2. Create dilute acid solution at desired acid concentration. Record acid concentration.  
3. Calibrate pH meter if necessary.  
4. Perform moisture analysis on at least 3 feed sludge samples.  
5. Add feed sludge to reaction vessel. Record volume and mass.  
6. Add distilled water to achieve desired S:L ratio, record volume of water added.  
7. Set desired temperature and stirrer speed. Thoroughly mix.  
8. Measure and record initial pH of solution.  
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.  
10. Repeat step 9 until endpoint pH is reached (<0.5)  
11. Dispose of any waste according to CHP.

## Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average (pH)</th>
<th>Cumulative Acid Added (mL)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>15.00</td>
<td>15.30</td>
<td>0.13</td>
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</tbody>
</table>

## Graph

![Graph showing pH vs. Acid Added for Sludge SL-2-5](image_url)

**Legend:**  
- **Feed Sludge:** Red-Black Sludge, Fudge-Like  
- **Sludge Dry Mass:** N/A  
- **Red Sludge:** N/A  
- **Si:** 9.9  
- **Al:** 5.3  
- **Fe:** 14.8  
- **Ca:** 1.8  
- **Other:** 68.1
General Test Information

- **Test No.**: 5
- **Acid Type**: 0.01 N HNO₃
- **Acid Concentration**: 0.01 N
- **Initial Volume**: N/A

Moisture Data

- **Sample ID**: SL-2-1
- **Sample Desc.**: Red. Fudge-Like Sludge
- **Directly sampled from bucket, removed Tramp
- **Sample**: SL-2-1

Analytical Report

- **Pre-processing**: N/A
- **Analysis**: S:L ratio
- **Time Step**: 5 minutes

Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Sl. Dry Mass</th>
<th>Unit Mass Sludge (g/g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Component</td>
<td>Added</td>
<td>Measured</td>
<td>Added</td>
</tr>
<tr>
<td>1</td>
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</table>

Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

Legend:

- **Test Result**: pH, Acid / Unit Mass Sludge (g/g), Sludge Dry Mass (g)
- **Key Result**: Add Acid / Unit Mass Sludge (g/g), Sludge Dry Mass (g)
- **Description Text**: N/A
**General Test Information**

**Initial Test Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>1:01</td>
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</tbody>
</table>

**Moisture Data**

<table>
<thead>
<tr>
<th>Test</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g</td>
<td>g</td>
</tr>
<tr>
<td></td>
<td>100.95</td>
<td>61.5</td>
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**Acid Type**

<table>
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<tr>
<th>Acid Type</th>
<th>Test by</th>
<th>Date</th>
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</thead>
<tbody>
<tr>
<td>Nitric</td>
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**Moisture 2**

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<tr>
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<th>Moisture 2: Wet Sample</th>
<th>Moisture 2: Dry sample</th>
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</thead>
<tbody>
<tr>
<td></td>
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<td>g</td>
</tr>
<tr>
<td></td>
<td>100.33</td>
<td>59.24</td>
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</table>

**Date**

<table>
<thead>
<tr>
<th>Date</th>
<th>Acid Concentration</th>
<th>Acid Molar Mass</th>
<th>Sludge Moisture 1</th>
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<tbody>
<tr>
<td>2017.5.30</td>
<td>mol/L</td>
<td>g/mol</td>
<td>--</td>
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<td>3</td>
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**Sludge Moisture**

<table>
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<th>Temperature</th>
<th>Stirrer Speed</th>
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<td>1</td>
<td>°C</td>
<td>RPM</td>
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**Sample ID**

<table>
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<th>Initial Volume</th>
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<td>SL-14-1</td>
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**Pre-processing**

- Sludge: Directly sampled from bucket, removed Tramp.
- Sample ID: SL-14-1

**Notes**

- Include in Avg: x

**Test Notes**

- Add adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- Create dilute acid solution at desired acid concentration. Record acid concentration.
- Calibrate pH meter if necessary.
- Perform moisture analysis on at least 3 feed sludge samples.
- Add feed sludge to reaction vessel. Record volume and mass.
- Add distilled water to achieve desired S:L ratio, record volume of water added.
- Set desired temperature and stirrer speed. Thoroughly mix.
- Measure and record initial pH of solution.
- Add desired volume of acid, wait until reaction is complete, measure and record pH.
- Repeat step 9 until endpoint pH is reached (<0.5).
- Dispose of any waste according to CHP.

**Legend**

- Test Failed: Red
- Resample: Orange
- Completed: Green
- Key Result: Yellow

**Calculations**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>(mL)</td>
<td>(g)</td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
</tr>
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<tr>
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**Diagram**

- SL-14-1: Graph showing pH change with acid addition.
**General Test Information**

- Test No.
- Date
- Sludge Type
- Acid Type
- Acid Concentration
- Acid Molar Mass
- Temperature
- Stirrer Speed
- Water Added
- Sample Desc.
- Pre-processing

**Initial Test Data**

- S:L Mass Ratio
- Moisture
- Test by
- Acid Type
- Date
- Acid Concentration
- Sludge Moisture
- Sludge Volume
- Sample ID
- Stirrer Speed
- Water Added
- Sample Desc.
- Pre-processing

**Moisture Data**

- Test No.
- S:L Mass Ratio
- Moisture
- Test by
- Acid Type
- Date
- Acid Concentration
- Sludge Moisture
- Sludge Volume

**Analytical Reports**

- Rep 1
- Rep 2
- Rep 3
- Rep 4
- Rep 5

**Calculations**

- Increment
- Average
- Cum. Acid
- Acid Added
- Acid Added / Sludge Dry Mass (g/g)

---

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test No.
- Rep
- Date
- Result
- Description Test

---

**Data Entry**

<table>
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<tr>
<th>Increment</th>
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<td>pH</td>
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<td>pH</td>
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**Plot:**

- SL-5-1

**Legend**

- Test No.
- Rep
- Date
- Result
- Description Test
### General Test Information

- **Test No.**: SL-17-1
- **Sample Desc.**: Blue-Green Aqueous Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data

<table>
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<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
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<tr>
<td>1:01</td>
<td>100.77 g</td>
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### Moisture Data

- **Sample ID**: 2017.6.1
- **Temp**: 22°C
- **Sludge Wet Mass**: 100.0 g
- **Sludge Dry Mass**: 8.5 g
- **Water Added**: 100.0 ml
- **Initial Volume**: N/A

### Calculations

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<th>Acid Added</th>
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<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
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<th>Sl. Dev.</th>
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<td></td>
<td>in mL</td>
<td>in mL</td>
<td>in mL</td>
<td>in mL</td>
<td>in mL</td>
<td>in mL</td>
<td>in mL</td>
</tr>
<tr>
<td>1-5</td>
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</tr>
</tbody>
</table>

### Legend

- **Rep 1**: Blue
- **Rep 2**: Green
- **Rep 3**: Red
- **Rep 4**: Black
- **Rep 5**: Grey

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat steps 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Notes:

- Include in Avg
- "x""
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
**General Test Information**

**Initial Test Data**

- **Test No.**
- **Sample ID.**
- **Sample Desc.**
- **Sample Prep.**
- **Pre-processing**
- **Initial Mass**
- **Average Moisture**
- **Initial Weight**

**Moisture Data**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

**Analytical Data**

- **Test by**
- **Acid Type**
- **Nitric**
- **Acid Concentration**
- **mol/L**
- **Sludge Moisture 1**
- **Dry sample**

**Calculations**

<table>
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<tr>
<th>Increment</th>
<th>Acid Added</th>
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<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
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<tr>
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<td></td>
</tr>
</tbody>
</table>

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Equal**
- **Sample Prep.**
- **Calculation**
- **Key Result**
- **Description Text**

**Data Entry**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Lab. Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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### General Test Information

**Test No.**
1

**Date**
2017.6.2

**Sample ID**
SL-6-1

**Sample Desc.**
Red, fudge-like sludge

**Pre-processing**
Directly sampled from bucket, removed tramp

**Sample Processing**
Red, fudge-like sludge

**Test Notes**

---

### Initial Test Data

**Moisture Data**

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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
</tr>
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<td>100.3</td>
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**Acid Type**
Nitric

**Acid Concentration**
mol/L

**Acid Molar Mass**
g/mol

**Temperature**

**Sludge Moisture 1**
--
0.91

**Sample Volume**
ml

**Sample ID**

**Acid Type**
Nitric

**Acid Concentration**
mol/L

**Acid Molar Mass**
g/mol

**Temperature**

**Sludge Moisture 1**
--
0.92

---

### Calculations

<table>
<thead>
<tr>
<th>Increment Number</th>
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<th>Cumulative Acid Added (mol)</th>
<th>Cumulative Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</thead>
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<td>8.10</td>
<td>8.10</td>
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<tr>
<td>3</td>
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### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

### Legend

**Test Result**

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<th>Cumulative Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x

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### Test Notes

- Include in Avg: x
# General Test Information

**Test No.**

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**Date**

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**Sample ID**

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**Sample Desc.**

<table>
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<th>Sample Desc.</th>
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**Moisture Data**

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<th>Moisture 1: Wet Sample</th>
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<th>g</th>
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<th>g</th>
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<tr>
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**Acid Type**

- Nitric

**Acid Concentration**

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<th>mol/L</th>
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**Acid Molar Mass**

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<th>g/mol</th>
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**Sample Volume**

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**Initial Test Data**

**Test Notes**

- Include in Avg
- x

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## Calculations

**Inc. Number**

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---

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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10. Record step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

**Legend**

- Test Equal
- Data Input
- Calculations
- Key Results
- Description Text

---

**Diagram**

Title: Sludge Acid Titration Test - Experimental Data Entry Sheet

---

**Notes**

- Include in Avg
- x
**General Test Information**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
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**Analytical Reports**

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<table>
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<tr>
<th>Date</th>
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<th>Time</th>
<th>Average Moisture</th>
<th>Water Added ml</th>
<th>Initial Volume ml</th>
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**Calculations**

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<th>Cumus. Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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**Test Notes**

- Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- Create dilute acid solution at desired acid concentration. Record acid concentration.
- Calibrate pH meter if necessary.
- Perform moisture analysis on at least 3 feed sludge samples.
- Add feed sludge to reaction vessel. Record volume and mass.
- Add distilled water to achieve desired S:L ratio, record volume of water added.
- Set desired temperature and stirrer speed. Thoroughly mix.
- Measure and record initial pH of solution.
- Perform step 9 until endpoint pH is reached (<0.5, pH 8.5).

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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9. Perform step 9 until endpoint pH is reached (<0.5, pH 8.5).
10. Dispose of any waste according to CHP.

**Legend**

- Test Data
- Initial Test Data
- Moisture Data
- Test by Acid Type
- Date
- Sample ID
- Sample Desc
- Analytical Reports
- Pre-processing
- Test Notes
- Data Entry
- Calculations
- Key Result
- Descriptive Text

**Diagram**

- SL-4-1
- pH vs Acid / Unit Mass Sludge (g / g)

**Table**

- General Test Information
- Analytical Reports
- Data Entry
- Calculations

**Notes**

- Include in Avg
- "a"
**General Test Information**

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**Calculations**

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</table>

**Preparation**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Test Initial**
- **Initial Test Data**
- **Moisture Data**
- **Calculations**
- **Key Result**
- **Descriptive Test**
**General Test Information**

- **Test No.**: SS
- **Sample ID**: SL-4-3
- **Sample Desc.**: Black Sludge, Dirt-Like
- **Sample origin**: Directly sampled from bucket, removed Tramp

**Moisture Data**

- **Test No.**: SS
- **S:L Mass Ratio**: 1:01
- **Moisture 1: Wet Sample g**: 100.52
- **Moisture 1: Dry Sample g**: 51.82
- **Acid Type**: Nitric
- **Acid Concentration** (mol/L): 3
- **Acid Molar Mass (g/mol)**: 63.01
- **Time Step (min)**: 5
- **Stirrer Speed (RPM)**: med
- **Sample Test Date**: 2017.6.7
- **Sample Time**: AM
- **Sample pH**: 7.9

**Analytical Reports**

- **Initial Mass**: Sludge Moisture 1: 100.0 g
- **Initial Volume**: Sludge Volume: 100.0 ml
- **Average Moisture**: 0.49
- **Water Added**: 200.0 ml
- **Sludge Dry Mass**: 51.3 g
- **Sludge Wet Mass**: 100.0 g
- **Sludge Volume**: 100.0 ml

**Data Entry**

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<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
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<th>Sl. Dev.</th>
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**Calculations**

<table>
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<tr>
<th>Increment</th>
<th>Average pH</th>
<th>Cum. Acid Added (mL)</th>
<th>Cum. Acid Added (mol)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
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<td>7.94</td>
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<td>0.00</td>
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</table>

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Input**: Text Input
- **Data Entry**: Numeric Input
- **Calculations**: Calculation
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>5</th>
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</thead>
<tbody>
<tr>
<td>Sample ID</td>
<td>54-3-1</td>
</tr>
<tr>
<td>Date</td>
<td>06/17/17</td>
</tr>
<tr>
<td>Time</td>
<td>AM</td>
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</table>

#### Initial Test Data

<table>
<thead>
<tr>
<th>Moisture Data</th>
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<tbody>
<tr>
<td>Test No.</td>
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</tr>
<tr>
<td>S:L Mass Ratio</td>
<td>1:01</td>
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<tr>
<td>Moisture 1: Wet Sample</td>
<td>100.52</td>
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<td>Temperature</td>
<td>22</td>
</tr>
<tr>
<td>Time</td>
<td>AM</td>
</tr>
<tr>
<td>Stirrer Speed</td>
<td>RPM</td>
</tr>
<tr>
<td>Raw Sludge</td>
<td>N/A</td>
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<tr>
<td>Raw Sludge Moisture</td>
<td>0.48</td>
</tr>
<tr>
<td>Sample Desc.</td>
<td>Black Sludge, Dirt-Like</td>
</tr>
<tr>
<td>Sludge Moisture 1</td>
<td>0.46</td>
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<tr>
<td>Note:</td>
<td>Originally sampled from bucket, removed Tramp</td>
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#### Replicate 1

<table>
<thead>
<tr>
<th>Increment</th>
<th>1</th>
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<tbody>
<tr>
<td>Initial Volume</td>
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<tr>
<td>Sludge Dry Mass</td>
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#### Replicate 2

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</thead>
<tbody>
<tr>
<td>Initial Volume</td>
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<tr>
<td>Sludge Dry Mass</td>
<td>51.3</td>
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#### Replicate 3

<table>
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<tbody>
<tr>
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<tr>
<td>Sludge Dry Mass</td>
<td>51.3</td>
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#### Replicate 4

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<tbody>
<tr>
<td>Initial Volume</td>
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#### Replicate 5

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<tbody>
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<td>Initial Volume</td>
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<tr>
<td>Sludge Dry Mass</td>
<td>51.3</td>
</tr>
</tbody>
</table>

### Test Notes

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat steps 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- Test Date
- Procedure
- Calculations
- Description Text

---

**Procedure:**

1. 1.
2. 2.
3. 3.
4. 4.
5. 5.
6. 6.
7. 7.
8. 8.
9. 9.
10. 10.
11. 11.

**Calculations:**

<table>
<thead>
<tr>
<th>Increment</th>
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<tbody>
<tr>
<td>Average</td>
<td>5.43</td>
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<tr>
<td>Cumu. Acid Added</td>
<td>0.72</td>
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<tr>
<td>Acid Added / Sludge Dry Mass (g/g)</td>
<td>0.23</td>
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**Results:**

<table>
<thead>
<tr>
<th>Increment</th>
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</thead>
<tbody>
<tr>
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<tr>
<td>Cumu. Acid Added</td>
<td>0.72</td>
</tr>
<tr>
<td>Acid Added / Sludge Dry Mass (g/g)</td>
<td>0.23</td>
</tr>
</tbody>
</table>

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**Legend:**

- Test Equal
- Procedure
- Calculations
- Description Text

111
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture Data</th>
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<tbody>
<tr>
<td></td>
<td>--</td>
<td>Wet Sample</td>
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<tr>
<td></td>
<td>--</td>
<td>Dry Sample</td>
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</table>

#### Initial Test Data

<table>
<thead>
<tr>
<th>Test by Acid Type</th>
<th>Nitric</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
<th>100.95</th>
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<tbody>
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<td>Date</td>
<td>2017.6.13</td>
<td>Temperature</td>
<td>°C</td>
<td>22</td>
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<tr>
<td>Sludge Moisture 1</td>
<td>--</td>
<td>Sludge Moisture 1</td>
<td>g</td>
<td>0.66</td>
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<tr>
<td>Sludge Moisture 2</td>
<td>--</td>
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<td>g</td>
<td>0.75</td>
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<tr>
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#### Analytical Reports

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Test Notes</th>
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<td>pH</td>
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<tr>
<td>Endpoint pH</td>
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#### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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<tr>
<td>1</td>
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<td>3</td>
<td>1.00</td>
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#### Pre-processing

- Sludge Wet Mass: 100.0 g
- Sludge Dry Mass: 31.9 g
- Water Added: 100.0 mL
- Initial Volume: 200.0 mL

#### Results

- Average Moisture: 0.68
- Average Sludge Moisture: 0.66

---

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

**Legend**

- **Test**
- **Symbol Select**
- **Symbol Calculations**
- **Key Result**
- **Description Text**

---

**Graphic**

- SL-31-1
- Graph showing Acid / Unit Mass Sludge (g/g) vs. pH

---
**General Test Information**

- Test No.: SS-38-1
- Date: 2017.6.13
- Sample ID: 1
- Sample Desc.: Grey, Sandy Sludge
- Pre-processing: Sample captured from sludge, removed tramp

**Initial Test Data**

- S:L Mass Ratio: 1:01
- Moisture 1: Wet Sample g: 101.67
- Moisture 1: Dry Sample g: 46.53
- Acidity Type: Nitric
- Acid Concentration: 3 mol/L
- Acid Molar Mass: 63.01 g/mol
- Sludge Moisture 1: 0.54
- Sludge Moisture 2: 0.61
- Sludge Moisture 3: 0.59
- Sludge Volume: 100.0 ml

**Moisture Data**

- Moisture 1: Wet Sample g: 101.33
- Moisture 2: Wet Sample g: 101.43
- Moisture 3: Wet Sample g: 101.43
- Moisture 1: Dry Sample g: 40.01
- Moisture 2: Dry Sample g: 39.46
- Moisture 3: Dry Sample g: 39.46

**Calculations**

- Increment: 0.01
- Acid: Measured: 0.00 - 1.00 - 10.00
- Average: Ca, 40.6
- Si, 1.7
- Mn, 0.9
- Fe, 0.7
- Other, 56.1

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio. Record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Name: SL-38-1
- Analysis: pH
- Description Text: Grey, Sandy Sludge
- Sample ID: 1
- Pre-processing: Sample captured from sludge, removed tramp

---

**Analytical Reports**

- Date: 2017.6.13
- Sample ID: 1
- Sample Desc.: Grey, Sandy Sludge
- Pre-processing: Sample captured from sludge, removed tramp
### General Test Information

**Test No.**
- SL-30-RAW

**Sample ID**
- 37

**Sample Desc.**
- Feed Sludge

**Pre-processing**
- Tramp Fe, Cu, Ni, Cr

**Test Notes**
- Include in Avg

### Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
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<tbody>
<tr>
<td>SL-30-RAW</td>
<td>1:01</td>
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**Date**
- 2017.6.13

**Time Step**
- 5 min

**Analytical Reports**
- pH

### Calculations

#### Data Entry

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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</thead>
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<td>0.76</td>
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#### Key Result
- Summary:
- **Calculation:**
  - **Average pH:** 0.10
  - **Cumu. Acid Added (mol):** 0.58
  - **Acid Added (g):** 0.30
  - **Acid Added / Unit Mass Sludge (g/g):** 0.05

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend:
- Test Input
- Replication/Unit
- Calculation
- Key Result
- Description Text

---

### Moisture Data

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moisture</th>
<th>Test by</th>
<th>Acid Type</th>
<th>Moisture 1: Dry Sample</th>
<th>g</th>
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</thead>
<tbody>
<tr>
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<td>Nitric</td>
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**Acid Concentration**
- mol/L

**Acid Molar Mass**
- g/mol

**Temperature**
- °C

**Sludge Moisture 1**
- g

**Sample ID**
- 37

**Sample Desc.**
- Feed Sludge

**Pre-processing**
- Tramp Fe, Cu, Ni, Cr

**Test Notes**
- Include in Avg

---

### List of Elements

- **Fe:** 62.2
- **S:** 1.6
- **Si:** 0.7
- **Al:** 0.6
- **Other:** 34.8

---

### Test Notes

- Include in Avg

---

### Graph

**SL-30-RAW**

- **Acid / Unit Mass Sludge (g/g):**
  - 0.00 to 0.10
  - 0.00 to 0.10
  - 0.00 to 0.10

---

### Data Table

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass (% of total)</th>
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<tr>
<td>S</td>
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<tr>
<td>Si</td>
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<tr>
<td>Al</td>
<td>0.6</td>
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<td>Other</td>
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</table>
General Test Information

Test No.: 1
Date: 06/14/2017
Sample ID: SL-41-1
Sample Desc.: Grey Sludge

Procedure:
1. Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

Legend
- Test Result
- Calculations
- Rep Check
- Description Text

Data Entry

<table>
<thead>
<tr>
<th>Increment</th>
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<tbody>
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Calculations

<table>
<thead>
<tr>
<th>Increment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
</tr>
<tr>
<td>Cumu. Acid Added [mol]</td>
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<tr>
<td>Acid Added [g]</td>
</tr>
<tr>
<td>Acid Added / Sludge Dry Mass [g/g]</td>
</tr>
</tbody>
</table>

Analitical Reports

Moisture Data

- Moisture 1: Wet Sample
- Moisture 2: Dry Sample
- Moisture 3: Wet Sample

Ph-Processing

- pH of sludge
- pH of acid
- pH of reaction

Pre-processing

- Sludge Moisture 1
- Sludge Moisture 2
- Sludge Moisture 3

Test Notes

- Include in Avg
- X

Legend

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text
**General Test Information**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Sample ID</th>
<th>Date</th>
<th>Time</th>
<th>Sludge Moisture 1</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>32-1</td>
<td>2017.6.14</td>
<td>AM</td>
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**Moisture Data**

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<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>ASTM</th>
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<td>1:01</td>
<td>101.58</td>
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**Analytical Reports**

<table>
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<th>Sludge Moisture 1</th>
<th>Acid Type</th>
<th>Nitric</th>
<th>Moisture 1: Dry sample</th>
<th>g</th>
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**Pre-processing**

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<th>Sludge Dry Mass</th>
<th>Sludge Volume</th>
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**Data Entry**

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<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Average Acid / Unit Mass Sludge (g/g)</th>
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</thead>
<tbody>
<tr>
<td></td>
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**Calculations**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Average Acid / Unit Mass Sludge (g/g)</th>
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</thead>
<tbody>
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**Legend**

- Test Equal
- Increment
- Calculated
- Key Result
- Description Text

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
**Test Notes:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**
- Test Failed
- Analysis Complete
- Calculation
- Key Result
- Description Text

**Calculations:**

<table>
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<tr>
<th>Increment Number</th>
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<th>Cumus. Acid Added</th>
<th>Cumus. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added Sludge Dry Mass (g/g)</th>
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<tr>
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<td>(g)</td>
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**Legend:**
- Initial Test Data
- Moisture Data

**Moisture Data:**
- Test No.: 1
- S:L Mass Ratio: 1:01
- Moisture 1: Wet Sample g: 100.16
- Moisture 1: Dry Sample g: 24.81
- Date: 2017.6.14
- Acid Type: Nitric
- Acid Concentration: 3 mol/L
- Temp.: 22
- Stirrer Speed: RPM
- Sample ID: SS-16-1
- Analysis Report
- Sample Desc.: Red Sludge
- Pre-processing: Initially sampled from bucket, removed Tramp

**Test Notes:**

- Initial Volume: ml
- Red Sludge
- N/A
- Directly sampled from bucket, removed Tramp
- SL-16-1
- Sludge Acid Titration Test - Experimental Data Entry Sheet

**General Test Information:**
- Test No.: 1
- Test Date: 2017.6.14
- Sludge Moisture 1: g
- Sludge Moisture 1: Dry sample g
- Test by: Acid Type
- Acid Concentration: mol/L
- Sludge Moisture 1: Sludge Dry Mass g
- Sludge Moisture 1: Sludge Moisture 1: Average
- Temperature: c
- Sludge Moisture 1: Average Moisture
- Time: min
- Stirrer Speed: RPM
- Sample Desc.: Red Sludge
- Pre-processing: Initially sampled from bucket, removed Tramp

**Analytical Reports:**
- Test No.: 1
- Rep 1
- Rep 2
- Rep 3
- Rep 4
- Rep 5
- Increment
- Acid
- Measured
- Measured
- Measured
- Measured
- Average
- St. Dev
- Increment
- Average
- Cumus. Acid
- Cumus. Acid
- Acid
- Added / Sludge Dry Mass (g/g)

**Fe, Si, Mn, Ca, Other:**
- Fe: 55.7
- Si: 2.4
- Mn: 2.3
- Ca: 0.8
- Other: 38.8

**Graph:**
- SL-16-1
- Graph of Acid vs. Unit Mass Sludge (g/g)
- Graph of pH vs. Time

**Procedure:**

1. 1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. 2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. 3. Calibrate pH meter if necessary.
4. 4. Perform moisture analysis on at least 3 feed sludge samples.
5. 5. Add feed sludge to reaction vessel. Record volume and mass.
6. 6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. 7. Set desired temperature and stirrer speed. Thoroughly mix.
8. 8. Measure and record initial pH of solution.
9. 9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. 10. Repeat step 9 until endpoint pH is reached (<0.5).
11. 11. Dispose of any waste according to CHP.
### General Test Information

- **Test No.**: SL-48-1
- **Sample Desc.**: Red Sludge
- **Sample ID**: N/A
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data

- **Test Notes**

### Moisture Data

- **Moisture 1: Wet Sample**
  - g: 101.12
- **Moisture 2: Wet Sample**
  - g: 100.89
- **Moisture 3: Wet Sample**
  - g: 100.41

### Acid Type

- **Nitric**

### Sludge Moisture 1

- **g**: 0.98
- **ml**: 200.0

### Analytical Reports

#### Sludge Wet Mass

- **g**: 100.0

#### Sludge Dry Mass

- **g**: 1.6

#### Sludge Volume

- **ml**: 100.0

### Water Added

- **ml**: 100.0

### Test Notes

1. **Procedure**
   - 1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
   - 2. Create dilute acid solution at desired acid concentration. Record acid concentration.
   - 3. Calibrate pH meter if necessary.
   - 4. Perform moisture analysis on at least 3 feed sludge samples.
   - 5. Add feed sludge to reaction vessel. Record volume and mass.
   - 6. Add distilled water to achieve desired S:L ratio, record volume of water added.
   - 7. Set desired temperature and stirrer speed. Thoroughly mix.
   - 8. Measure and record initial pH of solution.
   - 9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
   - 10. Repeat step 9 until endpoint pH is reached (<0.5).
   - 11. Dispose of any waste according to CHP.

### Calculations

#### Increment

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid / Unit Mass Sludge (g/g)</th>
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#### Increment

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid / Unit Mass Sludge (g/g)</th>
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<tr>
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<tr>
<td>6</td>
<td>4.27</td>
<td>18.31</td>
<td>0.00</td>
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</table>

### Legend

- **Test Initials**: 215
- **Data Entry**: 41
- **Pre-processing**: Red Sludge
- **Sample ID**: SL-48-1
- **Sample Desc.**: Red Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Analysis

- **Fe**, 25.9
- **Al**, 7.0
- **Ca**, 6.4
- **Si**, 5.1
- **Other**, 55.5

### Notes

- Include in Avg: 0
- pH: 0
- Acid / Unit Mass Sludge (g/g): 0

---

**SL-48-1**

![Graph](image)

**SL-48-1**

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### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Test by Acid Type</th>
<th>Moisture 1: Dry sample g</th>
<th>Date</th>
<th>Acid Concentration mol/L</th>
<th>Sample Desc.</th>
<th>Temperature °C</th>
<th>Stirrer Speed RPM / Sludge Moisture 1 g</th>
<th>Pre-processing</th>
<th>Sample ID</th>
<th>Analysis Report</th>
<th>Test Notes</th>
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<tbody>
<tr>
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### Data Entry

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<th>Average (mL)</th>
<th>Cum. Acid Added (mL)</th>
<th>Cum. Acid Added (mmol)</th>
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<th>Avg pH</th>
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<tbody>
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### Calculation

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<th>Increment Number</th>
<th>Average (mL)</th>
<th>Cum. Acid Added (mL)</th>
<th>Cum. Acid Added (mmol)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
<th>Avg pH</th>
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</tbody>
</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Input**: Text Input
- **Numeric Input**: Numeric Input
- **Calculation**: Calculation
- **Key Result**: Key Result
- **Descriptive Text**: Descriptive Text
**General Test Information**

- Test No. 43
- Test No. SS
- Date 2017.6.15
- Sample ID 43
- Sample Desc. Red-Brown Sludge
- Sample Size N/A
- Pre-processing Directly sampled from bucket, removed Tramp

**Test Notes**

- Red-Brown Sludge
- N/A
- Directly sampled from bucket, removed Tramp
- SL-44-5
- Ca, 19.4
- Fe, 9.0
- Si, 7.5
- Al, 5.7
- Other, 58.3

**Initial Test Data**

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<th>Acid Type</th>
<th>Acid Concentration</th>
<th>Acid Molar Mass</th>
<th>Temp.</th>
<th>Stirrer Speed</th>
<th>Initial Volume</th>
<th>pH</th>
<th>St. Dev</th>
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**Moisture Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Dry sample</th>
<th>Moisture 2: Dry sample</th>
<th>Moisture 3: Dry sample</th>
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<tr>
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<td>--</td>
<td>39.11</td>
<td>38.06</td>
<td>38.06</td>
</tr>
</tbody>
</table>

**Analysis Reports**

- Nitric

**Calculations**

- Acid / Unit Mass Sludge (g/g)
- Cumu. Acid Added (mol)
- Acid Added (g)
- Sludge Dry Mass (g)

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
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7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Result
- Data Entry
- Description Text

**Classes**

- Solid
- Acid
- Other

**Data Entry**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Acid Added (mL)</th>
<th>Measured pH 1</th>
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<th>Measured pH 3</th>
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**Graphs**

- **SL-44-5**
  - Graph showing data from the experiment.
### General Test Information
- **Test No.**: SL-51-5
- **Sample Desc.**: Brown Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data
- **Test by**: Acid Type
- **Nitric**
- **Moisture 1**: Wet Sample, g: 42.14
- **Moisture 2**: Wet Sample, g: 47.82
- **Moisture 3**: Wet Sample, g: 42
- **Sludge Moisture 1**: g: 0.60
- **Sludge Moisture 2**: g: 0.62
- **Sludge Moisture 3**: g: 0.61
- **Sample ID**: N/A
- **Concentration (mol/L)**: 3
- **Stirrer Speed (RPM)**: med
- **Temperature (c)**: 22
- **Acid molar mass (g/mol)**: 63.01
- **Water Added (ml)**: 100.0

### Calculations

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### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat steps 8 and 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
### General Test Information
- Test No.
- Sample ID
- Date

### Initial Test Data
- S:L Mass Ratio
- Moisture 1: Wet Sample g
- Moisture 1: Dry Sample g
- Date
- Acid Type
- Acid Concentration mol/L
- pH
- Temperature
- Stirrer Speed RPM
- Mass
- Initial Volume

### Pre-processing
- Sample Desc.
- S:L 57-2
- Sludge Moisture 1
- Sludge Moisture 2
- Sludge Moisture 3
- Sample ID
- Temperature

### Test Notes
- Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Add desired volume of acid, wait until reaction is complete, measure and record pH.
9. Repeat step 8 until endpoint pH is reached (<0.5).
10. Dispose of any waste according to CHP.

### Analytical Reports
- Sludge Wet Mass g
- Sludge Dry Mass g
- Water Added mL
- Average Moisture

### Calculations

#### Data Entry

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<th>Measured pH</th>
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#### Calculations

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### Test Notes

- Ensure adequate PPE and ensure safe laboratory conditions. Consult CHE for questions.
- Create dilute acid solution at desired acid concentration. Record acid concentration.
- Calibrate pH meter if necessary.
- Perform moisture analysis on at least 3 feed sludge samples.
- Add feed sludge to reaction vessel. Record volume and mass.
- Add distilled water to achieve desired S:L ratio, record volume of water added.
- Set desired temperature and stirrer speed. Thoroughly mix.
- Measure and record initial pH of solution.
- Add desired volume of acid, wait until reaction is complete, measure and record pH.
- Repeat step 9 until endpoint pH is reached (<0.5).
- Dispose of any waste according to CHE.

### Data Entry

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### Calculations

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### Legend

- Test Data
- Calculations
- Mössbauer Data
- Key Result
- Description Test

### Graph

- SL-13-1

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### Analytical Reports

- Nitric Acid: 64.91 g
- Sulfuric Acid: 30.8 g
- Sludge Moisture 1: 0.53 g
- Sludge Moisture 1: 0.58 g
- Sludge Moisture 2: 0.49 g
- Sludge Moisture 3: 0.53 g

### Test Notes

- Include in Avg
- x
- 0
- 1
- 2
- 3
- 4
- 5
- 6
- 7
- 8
- 9
- 10

### Sludge Acid Titration Test - Experimental Data Entry Sheet

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<th>Sample Name</th>
<th>Sample Desc.</th>
<th>Pre-processing</th>
<th>Analysis</th>
<th>Time</th>
<th>Stirrer Speed</th>
<th>Sludge Moisture 1</th>
<th>Sludge Moisture 2</th>
<th>Sludge Moisture 3</th>
<th>Water Added</th>
<th>Analytical Reports</th>
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<td>Red-Brown Sludge, gritty</td>
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**Note:**

1. Use appropriate PPE and ensure safe laboratory conditions. Consult CHE for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHE.
**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**

- Fe
- Al
- Si
- S
- Other

**Calculation:**

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<tr>
<th>Increment Number</th>
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<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</tbody>
</table>

**Note:** Include in Avg: x

- x
- x
- x
- x

**Acid / Unit Mass Sludge (g/g):**

- Fe, 32.6
- Si, 7.2
- Al, 6.3
- S, 3.6
- Other, 50.3

**Graph:**

- SL-50-1
- pH vs. Acid / Unit Mass Sludge (g/g)
**General Test Information**

- **Test No.**
- **Sample ID**
- **Sample Desc.**
- **Test by**
- **Date**
- **Acid Type**
- **Acid Concentration**
- **Acid Molar Mass**
- **Sludge Moisture 1**
- **Sludge Moisture 2**
- **Sludge Moisture 3**
- **Sludge Dry Mass**
- **Sludge Volume**
- **Water Added**

**Moisture Data**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry sample**
- **Moisture 2: Wet Sample**
- **Moisture 2: Dry sample**
- **Moisture 3: Wet Sample**
- **Moisture 3: Dry sample**

**Analytical Reports**

- **pH**
- **Sludge Volume**
- **Initial Volume**
- **Sample Desc.**
- **Pre-processing**

**Test Notes**

**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Text Input**
- **Numeric Input**
- **Calculation**

**Red Sludge**

- **N/A**

**Directly sampled from bucket, removed Tramp**

**SL-49-1**

**Ca**, **12.5**  
**Fe**, **10.9**  
**Si**, **8.2**  
**Al**, **4.7**  
**Other**, **63.7**

**Data Entry**

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</table>

**Calculations**

- **Avg pH**
- **Acid / Unit Mass Sludge (g/g)**
- **Cum. Acid Added (mol)**
- **Cum. Acid Added (g)**
- **Acid Added / Sludge Dry Mass (g/g)**

**Legend**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Description Text**

![Diagram](image-url)
**General Test Information**

- **Test No.**
- **Sample ID.**
- **Sample Desc.**
- **Pre-processing**
- **Analytical Reports**

**Initial Test Data**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

**Moisture Data**

- **Sample ID.**
- **Sample Desc.**
- **Acid Type**
- **Acid Concentration**
- **Acid Molar Mass**
- **Temperature**
- **Temperature**
- **Sample Size**

**Initial Volume**

- **Brown-White Aqueous Sludge**
- **Directly sampled from bucket, removed Tramp**

**SL-53-1**

**Analytical Reports**

- **Acid / Unit Mass Sludge (g/g)**
  - **Ca, 25.2**
  - **S, 5.8**
  - **Al, 5.5**
  - **Mg, 4.5**
  - **Other, 59.0**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

**Data Entry**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
<th>Average</th>
<th>Cum. Acid Added (g)</th>
<th>Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
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<td></td>
</tr>
</tbody>
</table>

**Calculations**

- **Increment**
- **Average**
- **Cum. Acid Added (g)**
- **Acid Added (g)**
- **SL-Dry Mass (g / g)**

**Legend**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

**Diagram**

- **SL-53-1**

**Note:**

1. Data adequate PRI and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
### General Test Information
- **Test No.**: SL-46-1
- **Sample Desc.**: Brown Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data
- **Moisture Data**
  - **Test No.**: SL-46-1
  - **Moisture 1**: Wet Sample
    - **g**: 72.06
  - **Moisture 2**: Dry Sample
    - **g**: 15.48

### Analytical Reports
- **Sample ID**: 2017.6.21
- **Acid Type**: Nitric
- **Acid Concentration**: mol/L 3
- **Sludge Moisture 1**: mol/L
  - **g/mL**: 72.06
- **Sludge Moisture 2**: mol/L
  - **g/mL**: 15.48
- **Sample ID**: 2017.6.21

### Calculations
- **Increment**: 0, 0.01, 0.1, 1.0, 10.0
- **Acid / Unit Mass Sludge (g/g)**
  - **SL**: 0.46
  - **1**: 4.70
  - **2**: 4.00
  - **3**: 3.50
  - **4**: 2.70
  - **5**: 85.1

### Test Notes
- **Data Entry**: Rep 1, Rep 2, Rep 3, Rep 4, Rep 5
- **Initial Volume**: ml

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend
- **Text Input**:
- **Numeric Input**: 
- **Calculation**: 
- **Key Result**: 
- **Description Text**

### Sludge Acid Titration Test - Experimental Data Entry Sheet

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<th>Acid Added</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>SL Dry Mass</th>
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</table>

### Sludge Waste Mass
- **Moisture 1**: Wet Sample
- **Moisture 2**: Dry Sample
- **Sludge Volume**: ml
- **Water Added**: ml
- **Initial Volume**: ml

### Water Added
- **ml**: 100.0

### Avg pH

- **Acid / Unit Mass Sludge (g/g)**
- **SL**: 0.46
- **1**: 4.70
- **2**: 4.00
- **3**: 3.50
- **4**: 2.70
- **5**: 85.1

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average pH</th>
<th>Cumulative Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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<tr>
<td>5</td>
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### Analysis

- **Note:** Include in Avg
- **x**: 0
- **0**: 0
- **8.5**: 8.50
- **8.0**: 8.50
- **8.49**: 8.50
- **8.50**: 8.50
- **0.01**: 0.00
- **0.00**: 0.00
- **0.00**: 0.00
- **0.57**: 0.19
- **0.02**: 0.01
- **1.32**: 0.57
- **0.05**: 0.02
- **2.84**: 1.32
- **0.10**: 0.05
- **5.86**: 2.84
- **0.22**: 0.10
- **11.91**: 5.86
- **0.44**: 2.26
- **5.31**: 2.26
- **2.00**: 1.00
- **0.10**: 0.05
- **10.00**: 5.00
- **0.05**: 0.02
- **20.00**: 10.00
- **0.10**: 0.05
- **100.00**: 50.00
- **0.10**: 0.05
- **200.00**: 100.00
- **0.10**: 0.05
- **500.00**: 250.00
- **0.10**: 0.05
- **1000.00**: 500.00
- **0.10**: 0.05

### Diagram

- **SL-46-1**

---

127
## General Test Information

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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
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<th>Moisture 1: Wet Sample</th>
<th>g</th>
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<th>Moisture 1: Dry sample</th>
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### Analytical Reports

- **Date:** 2017.6.21
- **Acid Type:** Nitric
- **Acid Concentration:** 3 mol/L
- **Acid molar mass:** 63.01 g/mol
- **Sample ID:** SL-35-10
- **Sample Desc.:** Black-Grey Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Test Notes

- **Test:** Sludge Acid Titration Test
- **Sample ID:** SL-35-10
- **Sample Desc.:** Black-Grey Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations

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<thead>
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<th>Increment Number</th>
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<th>Acid Added</th>
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<td>(mL)</td>
<td>(g)</td>
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### Legend

- **Test Type:** Sludge Acid Titration Test
- **Sample ID:** SL-35-10
- **Sample Desc.:** Black-Grey Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Sludge Wet Mass

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### Sludge Dry Mass

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### Water Added

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### Average Moisture

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### Data Entry

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</tbody>
</table>

### Note: Include in Avg "x":
- 0.00
- 0.00
- 0.00
- 0.00
- 0.00
- 0.00
- 0.00
- 0.00

### Results

- **Sample ID:** SL-35-10
- **Sample Desc.:** Black-Grey Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Calculations:

- **Average:** 4.64
- **Cumus. Acid Added:** 14.8
- **Acid Added / Unit Mass Sludge (g/g):** 0.04

---

**Legend**

- **Test Type:** Sludge Acid Titration Test
- **Sample ID:** SL-35-10
- **Sample Desc.:** Black-Grey Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

**Graph**

- **Y-axis:** Sludge pH
- **X-axis:** Acid / Unit Mass Sludge (g/g)

---

**Table**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average</th>
<th>Cumus. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>(mL)</td>
<td>(mL)</td>
<td>(g)</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>0.10</td>
<td>0.00</td>
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**Diagram**

- **Y-axis:** Sludge pH
- **X-axis:** Acid / Unit Mass Sludge (g/g)
General Test Information

Test No. 1
Test No. 2
Date 2017.6.21
Sample ID I-3
Sample Desc. Brown Sludge
Pre-processing Directly sampled from bucket, removed Tramp

Moisture Data

<table>
<thead>
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<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
<th>Moisture 1: Dry sample</th>
<th>g</th>
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</thead>
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<td>1:01</td>
<td>74.44</td>
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| Test Notes |

| Test Notes |

Data Entry

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<tr>
<th>Increment</th>
<th>Acid Added</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Average</th>
<th>Sl. Dev</th>
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| Notes |

| Notes |

Calculations

<table>
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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added (mL)</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tr>
</tbody>
</table>

| Notes |

| Notes |

Legend

| Legend |

| Legend |

Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.
### General Test Information
- Test No.: 0
- Test Date: N/A
- Site: N/A
- Sample ID: N/A
- Sample Desc.: N/A
- Pre-processing: N/A
- Water Added: N/A
- Initial Volume: N/A
- Sample ID: N/A
- Sample Desc.: N/A
- Water Added: N/A

### Initial Test Data
- S:L Mass Ratio: N/A
- Date: N/A
- Acid Type: N/A
- Acid Concentration: N/A
- Acid Molar Mass: N/A
- Temperature: N/A
- Stirrer Speed: N/A

### Moisture Data
- Test No.: 0
- S:L Mass Ratio: 1:01
- Moisture 1: Wet Sample g: 103.83
- Moisture 1: Dry sample g: 11.73
- Date: N/A
- Acid Type: N/A
- Acid Concentration: N/A
- Acid Molar Mass: N/A
- Temperature: N/A
- Stirrer Speed: N/A

### Analytical Reports
- Test No.: 0
- S:L Mass Ratio: 0.89
- Temperature: 22
- Stirrer Speed: N/A

### Test Notes
- Procedure:
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

### Calculations
- Increment Number: 0
- Average:
- Cumu. Acid Added (g): N/A
- Acid Added (g):
- Acid Added / Sludge Dry Mass (g/g):

### Data Entry

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<tr>
<td>Sl. Dev</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>

### Data Entry
- Increment: 0
- Average:
- Cumu. Acid Added (g): N/A
- Acid Added (g):
- Acid Added / Sludge Dry Mass (g/g):

### Results
- Rep 1:
- Rep 2:
- Rep 3:
- Rep 4:
- Rep 5:

### Key Result
- Rep 1:
- Rep 2:
- Rep 3:
- Rep 4:
- Rep 5:

### Legend
- Key Result: N/A

### Diagram
- SL-15-1

130
**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**
- Text Input
- Numeric Input
- Calculation

---

### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Test by</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
<th>Temperature</th>
<th>Stirrer Speed</th>
<th>Test Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1:01</td>
<td></td>
<td>66.5</td>
<td>Nitric</td>
<td>1</td>
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### Initial Test Data

<table>
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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Dry sample</th>
<th>Test by</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
<th>Temperature</th>
<th>Stirrer Speed</th>
<th>Test Notes</th>
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<td></td>
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### Moisture Data

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<th>Acid molar mass</th>
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### Analytical Reports

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<th>Date</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
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<th>Stirrer Speed</th>
<th>Test Notes</th>
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<td>3</td>
<td>63.01</td>
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### Pre-processing

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<th>S:L Mass Ratio</th>
<th>Sludge Moist 3</th>
<th>Date</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
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<th>Stirrer Speed</th>
<th>Test Notes</th>
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### Water Added

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<th>Date</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
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<td>63.01</td>
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</tr>
</tbody>
</table>

### Test Notes

- Include in Avg

---

### Data Entry

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
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<td>1</td>
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<tr>
<td>2</td>
<td>0.01</td>
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<tr>
<td>3</td>
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### Calculations

<table>
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<th>Increment</th>
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</thead>
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<tr>
<td>5</td>
<td>10.00</td>
</tr>
</tbody>
</table>

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**Legend:**
- Test Equal
- Water Added
- Calculations
- Key Result

---

**Graph:**

![Graph of pH vs. Acid Added](image-url)
### Sludge Acid Titration Test - Experimental Data Entry Sheet

#### General Test Information
- **Test No.**: 5
- **Acid Type**: Nitric
- **Test Date**: 2017.6.21
- **Sample ID**: SL-58-5
- **Sample Description**: Grey, Fudge-like Sludge, directly sampled from bucket, removed Tramp

#### Data Entry

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<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>Sl. Dev.</th>
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#### Calculations

<table>
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<tr>
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<th>Average pH</th>
<th>Cum. Acid (g)</th>
<th>Cum. Acid (mol)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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#### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

#### PowerPoint Diagram

- **Legend**
  - Gray Sludge
  - Si, 24.5
  - Al, 8.8
  - Fe, 4.9
  - Ca, 2.3
  - Other, 59.5

---

**Note:**
- Include in Avg "x" when applicable.

---

**Figures:**
- Final pH vs. Acid Added
- Pictorial Representation of Sludge Composition

---

**Recap:**
- Ensure PPE and safe lab conditions.
- Prepare dilute acid solution.
- Calibrate pH meter if necessary.
- Analyze feed sludge moisture.
- Adjust S:L ratio with distilled water.
- Set reaction conditions.
- Measure initial pH.
- Add acid in measured volumes.
- Measure final pH.
- Record endpoint pH.
- Dispose of waste.

---

**Additional Information:**
- Consult CHP for questions.
- Record all measurements accurately.

---

**Key Result:**
- Final pH vs. Acid Added
- Sludge Composition Analysis
- Moisture Data Summary

---

**Description:**
- Sludge Moisture 1: Wet Sample
- Sludge Moisture 1: Dry Sample
- Sludge Moisture 2: Wet Sample
- Sludge Moisture 2: Dry Sample
- Average Moisture
### General Test Information
- **Test No.**: 3
- **S:L Mass Ratio**: 1:01
- **Initial Test Data**
  - **Date**: 2017.6.22
  - **Acid Type**: Nitric
  - **Acid Concentration**: 20 mol/L
  - **Stirrer Speed**: 10 RPM
  - **Sample Desc.**: Brown Aqueous Sludge
  - **Pre-processing**: Directly sampled from bucket, removed Tramp

### Analytical Reports
- **Brown Aqueous Sludge**: Sludge Moisture 1: Wet Sample, g: 103.62; Dry sample, g: 14.15
- **Brown Aqueous Sludge**: Sludge Moisture 1: Wet Sample, g: 100.28; Dry sample, g: 10.83
- **Brown Aqueous Sludge**: Sludge Moisture 1: Wet Sample, g: 97.95; Dry sample, g: 8.78

### Calculations

#### Raw Text
- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

#### Table: Acid / Unit Mass Sludge (g/g)

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Average</th>
<th>Cumu. Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
<th>Sludge Moisture 1 (g)</th>
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</thead>
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#### Graph: SL-33-1

---

**Legend**
- **Test Data**
  - **Sample ID**: SL-33-1
  - **Sample Desc.**: Brown Aqueous Sludge
  - **Pre-processing**: Directly sampled from bucket, removed Tramp
  - **Sample ID**: SL-33-1
  - **Test by**: Acid Type
  - **Acid Concentration**: 20 mol/L
  - **Stirrer Speed**: 10 RPM
  - **Sample Desc.**: Brown Aqueous Sludge
  - **Pre-processing**: Directly sampled from bucket, removed Tramp

---

**Data Entry**

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<th>Acid Added (g)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
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**Calculations**

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<th>Increment</th>
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<th>Sludge Moisture 1 (g)</th>
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</tbody>
</table>

---

**Legend**
- **Test Notes**
  - **Date**: 2017.6.22
  - **Acid Type**: Nitric
  - **Acid Concentration**: 20 mol/L
  - **Stirrer Speed**: 10 RPM
  - **Sample Desc.**: Brown Aqueous Sludge
  - **Pre-processing**: Directly sampled from bucket, removed Tramp

---

**Note:**
Include in Avg: 0, 0, 7.18, 7.29, 7.29, 7.25, 0.06, 0, 7.25, 0, 0.00, 0.00, 0.00

---

**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample**
  - **Moisture 1: Dry Sample**

**Test by Acid Type**

- **Nitric Acid Concentration**
- **Nitric Acid Molar Mass**
- **Sample ID**
- **Date**
- **Stirrer Speed**
- **Moisture 2: Wet Sample**
- **Moisture 2: Dry Sample**

**Pre-processing**

- **Sample ID**
- **Sample Desc.**
- **Sample Description**
- **Stirrer Speed**
- **SL-56-1**

**Analysis**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

**Initial Test Data**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

**Analytical Reports**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**

**Calculations**

- **Increment**
- **Acid Added**
- **Measured pH**
- **Measured pH**
- **Uncorrected Sample pH**
- **Corrected Sample pH**
- **Uncorrected sludge pH**
- **Corrected sludge pH**

**Data Entry**

- **Test Notes**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Fluid**
- **Sample Weight**
- **Conversion**
- **Key Result**
- **Description Text**

**SL-56-1**

**Acid / Unit Mass Sludge (g/g)**

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<tr>
<th>Increment</th>
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<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g / g)</th>
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**Calculations**

- **Increment**
- **Average**
- **Cum. Acid Added**
- **Acid Added**
- **Acid Added / Sludge Dry Mass (g / g)**

**Note:**

- Include in Avg "x"
### General Test Information
- **Test No.**: 5
- **Test Type**: Acid Titration
- **Date**: 2017.6.22
- **Sample Desc.**: Black, Earthy Sludge, Directly sampled from bucket, removed Tramp
- **Sample ID**: SS

### Initial Test Data
- **S:L Mass Ratio**: 1:01

### Moisture Data
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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry sample g</th>
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</tbody>
</table>

### Date
- **Acid Type**: Nitric
- **Acid Concentration**: mol/L 3
- **Sludge Moisture 1**: -- 0.26
- **Time**: min 5
- **Acid molar mass**: g/mol 63.01
- **Sludge Moisture 2**: -- 0.22
- **Temperature**: °C 22
- **Sample Desc.**: Black, Earthy Sludge
- **Acid Added**: g 200.0

### Pre-processing
- **Sample ID**: SL-43-5
- **Black, Earthy Sludge**: Directly sampled from bucket, removed Tramp

### Data Entry

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### Calculations

<table>
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<tr>
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<th>Cum. Acid Added (mol)</th>
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</table>

### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend
- **Data Input**: Black
- **Calculations**: Earthy
- **Key Result**: Sludge
- **Description Text**: Black, Earthy Sludge

![Sludge Acid Titration Test - Experimental Data Entry Sheet](image-url)
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration mol/L</th>
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<tbody>
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<td>68.31</td>
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<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Desc</th>
<th>Initial Test Data</th>
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<tbody>
<tr>
<td>SL-51-1</td>
<td>Red Aqueous Sludge</td>
<td>Directly sampled from bucket, removed Tramp</td>
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### Analytical Reports

<table>
<thead>
<tr>
<th>Pre-processing</th>
<th>Sludge Moisture 1</th>
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### Data Entry

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</table>

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Sludge Dry Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number</td>
<td>pH</td>
<td>(mol)</td>
<td>(g)</td>
<td>(g/g)</td>
<td></td>
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<tr>
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<td>0.01</td>
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</tbody>
</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 as endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- Test Equip
- Materials
- Calculations
- Key Result
- Description Text
### General Test Information

**Test No.**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
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<th>1:01</th>
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**Moisture Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>1:01</th>
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</thead>
<tbody>
<tr>
<td><strong>Wet Sample</strong></td>
<td><strong>g</strong></td>
<td><strong>63.19</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Dry Sample</strong></td>
<td><strong>g</strong></td>
<td><strong>12.41</strong></td>
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</table>

**Date**

| Date | 2017.6.22 |

**Acid Type**

<table>
<thead>
<tr>
<th>Acid Type</th>
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<tbody>
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</table>

**Acid Concentration**

<table>
<thead>
<tr>
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**Acid Molar Mass**

<table>
<thead>
<tr>
<th>Acid Molar Mass</th>
<th>g/mol</th>
<th>63.01</th>
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**Moisture 1: Wet Sample**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>1:01</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Wet Sample</strong></td>
<td><strong>g</strong></td>
<td><strong>68.63</strong></td>
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</tr>
<tr>
<td><strong>Dry Sample</strong></td>
<td><strong>g</strong></td>
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**Sample ID**

<table>
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**Sample Desc.**

<table>
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**Pre-processing**

| Pre-processing | Directly sampled from bucket, removed Tramp |

**Result Notes**

- Includes in Avg (x)

### Data Entry

<table>
<thead>
<tr>
<th>Data Entry</th>
<th>Increment Number</th>
<th>Acid Added</th>
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<th>Measured pH</th>
<th>Measured Sl. Dry Mass</th>
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### Calculations

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<th>Cumus. Acid Added g</th>
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<td>0.10</td>
</tr>
</tbody>
</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test No.**
- **Sample ID**
- **Sample Desc.**
- **Pre-processing**
- **Result Notes**

---

**Calculation Table:**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average Sl. Dry Mass</th>
<th>Cumus. Acid Added</th>
<th>Cumus. Acid Added g</th>
<th>Acid Added</th>
<th>Acid Added Sludge Dry Mass g/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>9.23</td>
<td>9.23</td>
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</tbody>
</table>

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**Diagram:**

- **SL-30-1**
- **Acid / Unit Mass Sludge (g/g)**
- **pH**
- **Sludge Dry Mass**
- **Sludge Moisture 1**

---

**Note:**
Include in Avg (x)
## General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Date</th>
<th>Moitey Data</th>
<th>Sample ID</th>
<th>Sample Desc.</th>
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</table>
|          | 6/22/17| Moisture 1: Wet Sample | 37-1 | White, Sand-like Sludge
|          |        | Moisture 2: Wet Sample | 37-2 | White, Sand-like Sludge
|          |        | Moisture 3: Wet Sample | 37-3 | White, Sand-like Sludge

## Analytical Reports

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<td>1200 RPM</td>
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<td>12.41</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

### Data Entry

#### Pre-processing

- Sludge Moisture 1: Weight sampled from bucket; removed Tramp.
- Sample ID: SL-37-1

#### Calculations

1. **Incident**
   - Number
   - Acid Added (mol)

2. **Rep 1**
   - Measured pH
   - Sludge Moisture 1
   - Ca
   - Mg
   - Si
   - Other

3. **Rep 2**
   - Measured pH
   - Sludge Moisture 2
   - Ca
   - Mg
   - Si
   - Other

4. **Rep 3**
   - Measured pH
   - Sludge Moisture 3
   - Ca
   - Mg
   - Si
   - Other

5. **Rep 4**
   - Measured pH
   - Sludge Moisture 4
   - Ca
   - Mg
   - Si
   - Other

6. **Rep 5**
   - Measured pH
   - Sludge Moisture 5
   - Ca
   - Mg
   - Si
   - Other

#### Legend

- **Test No.**
- **Date**
- **Moisture Data**
- **Sample Desc.**
- **PPI**
- **Acid Type**
- **Acidity**
- **Average**
- **Cumulative Acid**
- **Acidity Added**
- **Sludge Dry Mass**
- **Sludge Volume**
- **Temperature**
- **Stirrer Speed**
- **Time**
- **Average pH**
- **Cumulative Acid**
- **Acidity Added**

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat steps 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations

- **Incident**
  - Number
  - Acid Added (mol)
- **Rep 1**
  - Measured pH
- **Rep 2**
  - Measured pH
  - Sludge Moisture 1
  - Ca
  - Mg
  - Si
  - Other
- **Rep 3**
  - Measured pH
  - Sludge Moisture 2
  - Ca
  - Mg
  - Si
  - Other
- **Rep 4**
  - Measured pH
  - Sludge Moisture 3
  - Ca
  - Mg
  - Si
  - Other
- **Rep 5**
  - Measured pH
  - Sludge Moisture 4
  - Ca
  - Mg
  - Si
  - Other

### Notes

- Include in Avg
- "x"
### General Test Information

<table>
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<tbody>
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<td></td>
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</tbody>
</table>

| Date     | 06/07/2023   |

| Sample ID | 304          |

| Sample Description | Wet storage/other |

### Initial Test Data

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<table>
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### Test Notes

- Analytical Reports
- Pre-processing

### Analytical Reports

- Nitric Acid
- Acids

### Data Entry

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<tr>
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### Calculations

<table>
<thead>
<tr>
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<th>Increment</th>
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<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- Test Input
- Test Notes
- Description Test

### Calculations

1. For the above procedures, calculate the average values for each test.
2. Determine the cumulative acid added for each test.
3. Calculate the acid added per unit mass of sludge (g/g).

### General Test Information

<table>
<thead>
<tr>
<th>Initial Test Data</th>
<th>Moisture Data</th>
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<td>Moisture 1: Dry sample</td>
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### Analysis Reports

- Nitric Acid
- Acids

### Data Entry

<table>
<thead>
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<th>Data Entry</th>
<th>Rep 1</th>
<th>Rep 2</th>
<th>Rep 3</th>
<th>Rep 4</th>
<th>Rep 5</th>
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### Calculations

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<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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<td></td>
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<td></td>
</tr>
</tbody>
</table>

### Procedure:

1. For the above procedures, calculate the average values for each test.
2. Determine the cumulative acid added for each test.
3. Calculate the acid added per unit mass of sludge (g/g).

### Legend

- Test Input
- Test Notes
- Description Test

### Calculations

1. For the above procedures, calculate the average values for each test.
2. Determine the cumulative acid added for each test.
3. Calculate the acid added per unit mass of sludge (g/g).

### General Test Information

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### Analysis Reports

- Nitric Acid
- Acids

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### Procedure:

1. For the above procedures, calculate the average values for each test.
2. Determine the cumulative acid added for each test.
3. Calculate the acid added per unit mass of sludge (g/g).

### Legend

- Test Input
- Test Notes
- Description Test

### Calculations

1. For the above procedures, calculate the average values for each test.
2. Determine the cumulative acid added for each test.
3. Calculate the acid added per unit mass of sludge (g/g).
### General Test Information

- **Test No.**
- **S:L Mass Ratio**
- **1:01**
- **Moisture Data**
  - **1: Wet Sample g**
  - **104.09**
- **Test by**
  - **Acid Type**
  - **Nitric**
- **Moisture 1: Dry Sample g**
  - **65.34**
- **Date**
  - **2017.6.29**
- **Sample Description**
  - **Black Sludge**
- **Sample ID**
  - **SL-118-2**
- **Processing**
  - **Black Sludge obtained from bucket, removed Tramp**
- **Sample Notes**

### Analytical Reports

- **Initial Test Data**
  - **Moisture Data**
  - **Test No.**
    - **S:L Mass Ratio**
    - **1:01**
    - **Moisture 1: Wet Sample g**
      - **104.09**
    - **Test by**
      - **Acid Type**
        - **Nitric**
    - **Moisture 1: Dry Sample g**
      - **65.34**
    - **Sample ID**
      - **SL-118-2**
    - **Processing**
      - **Black Sludge obtained from bucket, removed Tramp**

### Calculations

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### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Entry**
- **Data Input**
- **Calculation**
- **Key Result**
- **Description Text**
### General Test Information

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### Pre-processing

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### Test Notes

1. Always wear appropriate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Perform moisture analysis on at least 3 feed sludge samples.
4. Add feed sludge to reaction vessel. Record volume and mass.
5. Add distilled water to achieve desired S:L ratio. Record volume of water added.
7. Measure and record initial pH of solution.
8. Add desired volume of acid, wait until reaction is complete, measure and record pH.
9. Repeat step 8 until endpoint pH is reached (<0.5).
10. Dispose of any waste according to CHP.

### Calculations

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### Legend

- **Test Input**
- **Calculation**
- **Key Result**
- **Description Text**

---

**Procedure:**

1. Always wear appropriate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Perform moisture analysis on at least 3 feed sludge samples.
4. Add feed sludge to reaction vessel. Record volume and mass.
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7. Measure and record initial pH of solution.
8. Add desired volume of acid, wait until reaction is complete, measure and record pH.
9. Repeat step 8 until endpoint pH is reached (<0.5).
10. Dispose of any waste according to CHP.

**Legend**

- **Test Input**
- **Calculation**
- **Key Result**
- **Description Text**

---

**Data Entry**

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**Diagram:**

- **Graph:**
  - **X-axis:** Acid / Unit Mass Sludge (g/g)
  - **Y-axis:** pH
  - **Legend:**
    - Fe, 48.3
    - Ca, 3.0
    - Si, 2.2
    - Other, 45.1

---

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### General Test Information

- **Test No.**
- **Date.** 2017.7.03
- **Sample ID.** SL-102-20
- **Sample Desc.** Orange-Black Sludge

### Analytical Reports

- **Pre-processing Notes:** initially sampled from bucket, removed Tramp

### Test Notes

- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).
  11. Dispose of any waste according to CHP.

### Calculations

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### Legend

- **Test Type:**
- **Sample Origin:**
- **Key Result:**
- **Description Text:**
### General Test Information
- **Test No.**: 36
- **Sample ID**: SL-106-1
- **Sample Desc.**: Brown Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data
- **Test by**: Acidity
- **Acid Type**: Nitric

### Analytical Reports
- **Date**: 2017.7.03

### Initial Volume
- **Brown-Sludge**: N/A

### Test Notes
- **Sample ID**: SL-106-1
  - **Fe**, 17.3
  - **Si**, 13.9
  - **Al**, 5.8
  - **S**, 2.4
  - **Other**, 60.7

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### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend
- **Test Type**: Pre-processing
- **Test Note**: Directly sampled from bucket, removed Tramp
Sludge Acid Titration Test - Experimental Data Entry Sheet

**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend:**
- **Key Result:**
  - **Water Added:** mL
  - **Initial Volume:** mL
  - **Sample Desc.:**
  - **Sample ID:**
  - **Sample Type:**
  - **Sample Date:**
  - **Pre-processing:**
  - **Test Notes:**
  - **Test by:**

**Data Entry**

**Calculations**

**Note:** Include in Avg "x"
### Data Entry

#### Incubation Number
- **Rep 1**: [Data]
- **Rep 2**: [Data]
- **Rep 3**: [Data]
- **Rep 4**: [Data]
- **Rep 5**: [Data]

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<th>Measured pH</th>
<th>Measured pH</th>
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<th>Average pH</th>
<th>Sl. Dry Mass (g)</th>
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#### Calculations

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#### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHF for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHF.

#### Legend
- **Feed Inlet**
- **Sample Inlet**
- **Pre-processing**
- **Descriptive Test**

### Sludge Acid Titration Test - Experimental Data Entry Sheet

#### General Test Information
- **Test No.**
- **Sample ID**
- **Sample Desc.**
- **Date**
- **Acid Type**
- **Acid Concentration**
- **Acid molar mass**
- **Temperature**
- **Stirrer Speed**
- **Water Added**
- **Initial Volume**
- **Average Moisture**

#### Calculations

### Graph

**SL-115-1**

**Graph Data**

- **Acid / Unit Mass Sludge (g/g)**
- **Avg pH**
- **0.00**
- **0.01**
- **1.00**
- **10.00**

---

**Note:**
- Include in Avg = "x"
- "x" indicates a value not included in the average.
### General Test Information
- **Test No.:** 316
- **Test Date:** 07/07/2017
- **Sample ID:** 316-1
- **Sample Description:** NP

### Analytical Report
- **Sample processing:** Sludge wet mass, samples taken from buckets, removed Tramp

### Initial Test Data
- **S:L Mass Ratio:** -- 1:01
- **Moisture 1: Wet Sample:** g

### Moisture Data
- **Acid Type:** Nitric
- **Acid Concentration:** mol/L
- **Acid Molar Mass:** g/mol
- **Moisture 1: Dry Sample:** g
- **Moisture 2: Wet Sample:** g
- **Moisture 2: Dry Sample:** g
- **Moisture 3: Wet Sample:** g
- **Moisture 3: Dry Sample:** g

### Sample Notes
- “X” test, water only.

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend
- **Test Result:**
- **Calculation:**
- **Descriptive Text:**

### Calculations
- **Acid Added (mL):**
- **Cumu. Acid (mL):**
- **Acid Added / Sludge Dry Mass (g/g):**

### Graphs
- **Graph 1:**
  - **Y-axis:** pH
  - **X-axis:** Acid / Unit Mass Sludge (g/g)

---

**Note:** Include in Avg "X".
**General Test Information**

### Initial Test Data

| Test No. | S:L Mass Ratio | Moisture 1: Wet Sample | g |
|----------|----------------|------------------------|
|          |                |                        | 45.06 |

### Test by Acid Type

- **Nitric**:
  - Moisture 1: Dry sample | g | 4.24 |

### Date

- **2017-7-15**

### Sludge Moisture 1

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<tr>
<th>Test Notes</th>
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<tbody>
<tr>
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<tr>
<td>Moisture 1: Dry sample</td>
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</tbody>
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### Sample ID

- **SL-104-1**

### Sample Desc.

- Black-Brown Aqueous

### Pre-processing

- Directly sampled from bucket, removed Tramp

### Analytical Reports

- **Black-Brown Aqueous**
  - Moisture 1: Wet sample | g | 45.06 |
  - Moisture 1: Dry sample | g | 4.24 |

### Analytical Reports

- **Black-Brown Aqueous**
  - Moisture 1: Wet sample | g | 45.06 |
  - Moisture 1: Dry sample | g | 4.24 |

### Initial Volume

- **0.00 mL**

### Data Entry

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<th>Measured pH</th>
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<th>Sl. Dev.</th>
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### Calculations

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<th>Increment</th>
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<th>Cum. Acid Added (mol)</th>
<th>Acid Added (mol)</th>
<th>Sludge Dry Mass (g)</th>
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</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Type**
- **Sample ID**
- **Calculations**
- **Key Result**
- **Description Test**
### General Test Information

- **Test No.**: 1
- **Sample ID**: 1-20-5
- **Sample Desc.**: Wet Sludge (Clay-Like)
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data

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<th>Test Notes</th>
<th>Characteristics</th>
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<td>Acid Type</td>
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<td>Acid Concentration</td>
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<td>Acid Molar mass</td>
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### Moisture Data

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<table>
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<table>
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### Test by Acid Type

- **Acid Type**: Nitric

### Analytical Reports

- **Sample ID**: 1-20-5
- **Sample Desc.**: Wet Sludge (Clay-Like)
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Data Entry

#### Rep 1
- **Sample ID**: SL-120-5
- **Ca**: 19.8
- **Fe**: 17.9
- **S**: 5.3
- **Si**: 3.9
- **Other**: 53.1

### Calculations

#### Data Entry

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<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Measured (g/g)</th>
<th>Average (g/g)</th>
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#### Calculations

- **Sl. Dev (g/g)**
- **Acid Added (g/L)**
- **Sludge Dry Mass (g)**
- **Sludge Volume (ml)**
- **Water Added (ml)**

### Procedure:

1. Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- **Test Fluid**
- **pH Readings**
- **Calculation**
- **Key Result**
- **Description Text**

### Sludge Acid Titration Test - Experimental Data Entry Sheet

- **Initial Volume**
- **Red-Brown Sludge (Clay-Like)**
- **Sample ID**: SL-120-5
- **Ca**: 19.8
- **Fe**: 17.9
- **S**: 5.3
- **Si**: 3.9
- **Other**: 53.1

---

**Note:** Include in Avg
Sludge Acid Titration Test - Experimental Data Entry Sheet

### General Test Information
- **Sample ID**: 122-2
- **Sample Desc.**: Red Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Initial Test Data
- **Test No.**
  - **S:L Mass Ratio**: 1:01
- **Moisture Data**
  - **Moisture 1**: Wet Sample
    - **g**: 35.24
  - **Moisture 2**: Dry Sample
    - **g**: 16.69
  - **Sludge Moisture 1**: **g**: 0.53
  - **Sludge Moisture 2**: **g**: 0.51

### Moisture Data
- **Test Date**: 2017.7.05 AM
- **Acid Type**: Nitric
- **Acid Concentration**: 1 M
- **Acid molar mass**: 63.01 g/mol
- **Temperature**: 22°C
- **Stirrer Speed**: 150 RPM
- **Initial Volume**: 100.0 mL
- **Water Added**: 100.0 mL

### Analytical Reports
- **Average Moisture**: 0.52

### Test Notes

### Calculations

#### Data Entry

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</tbody>
</table>

#### Key Result
- **Ca**, 37.8 ppm
- **Fe**, 8.7 ppm
- **Si**, 1.7 ppm
- **S**, 1.2 ppm
- **Other**, 50.6 ppm

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
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10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend
- **Test Date**
- **Sample ID**
- **Sludge Dry Mass**
- **Sludge Moisture 1**
- **Sludge Moisture 2**
- **Moisture 1**
- **Moisture 2**
- **Acid molar mass**
- **Initial Volume**
- **Water Added**
- **Average Moisture**
- **Ca**, 37.8 ppm
- **Fe**, 8.7 ppm
- **Si**, 1.7 ppm
- **S**, 1.2 ppm
- **Other**, 50.6 ppm

### Graph
- **Acid / Unit Mass Sludge (g/g)** vs. **pH**
### Sludge Acid Titration Test - Experimental Data Entry Sheet

#### General Test Information
- **Test No.:** 73
- **Date:** 2017.7.6
- **Sample ID:** SL-117-1
- **Pre-processing:** Slightly contaminated from bucket, removed Tramp
- **Sample Desc.:** Brown-dirt like

#### Analytical Results
- **Test by:** Acid Type
  - Nitric
- **Acid Concentration:** mol/L
  - 3
- **Acid Molar Mass:** g/mol
  - 63.01
- **Sludge Moisture 1:**
  - S:L Mass Ratio: --
  - Moisture 1: Wet Sample: g --
  - Moisture 1: Dry Sample: g --
- **Sludge Moisture 1:**
  - Acid Concentration: mol/L
  - Acid Molar Mass: g/mol
  - Sludge Moisture 1: --
  - Sludge Moisture 1: Wet Sample: g --
  - Sludge Moisture 1: Dry Sample: g --
- **Sludge Moisture 2:**
  - Acid Concentration: mol/L
  - Acid Molar Mass: g/mol
  - Sludge Moisture 2: --
  - Sludge Moisture 2: Wet Sample: g --
  - Sludge Moisture 2: Dry Sample: g --
- **Sludge Moisture 3:**
  - Acid Concentration: mol/L
  - Acid Molar Mass: g/mol
  - Sludge Moisture 3: --
  - Sludge Moisture 3: Wet Sample: g --
  - Sludge Moisture 3: Dry Sample: g --

#### Test Notes
- **Brown-Dirt Like**
  - Remained Tramp
- **SL-117-1**

### Data Entry

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**Notes:** Include in Avg 

### Calculations

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### Procedure:

1. **Safety:** Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. **Dilute Acid Solution:** Create dilute acid solution at desired acid concentration. Record acid concentration.
3. **Calibrate pH Meter:** Calibrate pH meter if necessary.
4. **Perform Moisture Analysis:** Perform moisture analysis on at least 3 feed sludge samples.
5. **Add Feed Sludge:** Add feed sludge to reaction vessel. Record volume and mass.
6. **Add Distilled Water:** Add distilled water to achieve desired S:L ratio, record volume of water added.
7. **Set Desired Temperature and Stirrer Speed:** Set desired temperature and stirrer speed. Thoroughly mix.
8. **Measure Initial pH:** Measure and record initial pH of solution.
9. **Add Desired Volume of Acid:** Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. **Repeat Step 9:** Repeat step 9 until endpoint pH is reached (<0.5).
11. **Dispose of Waste:** Dispose of any waste according to CHP.

### Legend

- **Test Fluid:** Sludge Acid Titration Test
- **S-L Mass Ratio:** Sludge:Liquid Mass Ratio
- **P-H pH:** Sludge:Liquid pH Ratio
- **Calculation:** Sludge:Liquid Calculation
- **Calculation Result:** Sludge:Liquid Result
- **Final Result:** Sludge:Liquid Final Result
- **Description Text:** Sludge:Liquid Description Text

### Graph

**Graph Title:** Sludge Acid Titration Test - Graph

**Graph Description:**

- **X-axis:** Acid / Unit Mass Sludge (g/g)
- **Y-axis:** Sl pH

**Data Points:**

1. (10.80, 0.00)
2. (10.60, 0.14)
3. (10.15, 0.28)
4. (10.15, 0.53)
5. (10.15, 0.81)
6. (10.15, 1.05)
7. (10.15, 1.53)
8. (10.15, 2.08)

**Graph Note:**

- **Note:** Include in Avg
**General Test Information**

**Test No.**

**S:L Mass Ratio**

**Moisture 1: Wet Sample g**

**Test No.**

**Acid Type**

**Nitric**

**Moisture 1: Dry Sample g**

**Sample ID**

**SS-122-1**

**Sample Desc.**

**Acid Type**

**H2SO4**

**Sample Note**

**Acid Molar Mass g/mol**

63.01

**Analytical Report**

**Test Notes**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Input
- Data Input
- Calculations
- Test Output

**Data Entry**

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**Calculations**

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**Note:** Include in Avg

*x* = N/A
General Test Information

Test No. 5
Sample ID 5
Sample Desc. Brown Aquoues Sludge
Pre-processing Directly sampled from bucket, removed Tramp

Analytical Reports

Moisture Data
Test No. S:L Mass Ratio -- 1:01
Moisture 1: Wet Sample g 43.52
Moisture 2: Wet Sample g 46.48
Moisture 3: Wet Sample g 39.88
Moisture 1: Dry sample g 10.74
Moisture 2: Dry sample g 14.66
Moisture 3: Dry sample g 7.41

Test Notes

Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Legend
- Raw Data
- Calculated
- Key Result
- Description Text

Calculations:

Increment Number Average Measured Measured Measured Measured Average Sl. Dev. Sludge Acid Titration Test - Experimental Data Entry Sheet

Data Entry

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Avg pH
Acid / Unit Mass Sludge (g/g)
SL-114-1
Si, 23.7
Al, 7.9
Fe, 5.4
S, 1.5
Other, 61.5

Note:
Include in Avg

SiO₂
Al₂O₃
Fe₂O₃
H₂O

Moisture Data

Moisture 1: Wet sample g 43.52
Moisture 1: Dry sample g 10.74
Moisture 2: Wet sample g 46.48
Moisture 2: Dry sample g 14.66
Moisture 3: Wet sample g 39.88
Moisture 3: Dry sample g 7.41

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Graph:

SL-114-1

152
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repate step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.
### General Test Information

- **Test No.:** Test 1
- **Date:** 2017.7.07
- **Sample Desc.:** Brown-White Sludge, directly sampled from bucket, removed Tramp

### Moisture Data

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### Pre-processing Notes
- Initially sampled from bucket, removed Tramp

### Analytical Reports

- **Sample ID:** SL-119-1
- **Sample Type:** Sludge

### Test Notes

- **Test Equipment:**
  - **Start:**
  - **End:**
- **Calculation:**
  - **Key Result:**
    - **Descriptive Text:**

#### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- Test Equipment:
  - **Start:**
  - **End:**
- **Description Text:**

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### Data Entry

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### Calculations

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### Graph

- **SL-119-1**

---

### Note:

Include in Avg "x" 0 0 12.48 12.49 12.45 12.47 0.02 0
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2 0 12.45 0 0.00 0.19 0.01
3 0 12.42 0 0.00 0.01 0.02
4 0 12.34 0 0.00 0.57 0.03
5 0 10.90 0 0.00 1.32 0.04
6 0 10.48 0 0.00 5.86 0.16
7 0 10.48 0 0.00 11.91 0.33
8 0 10.48 0 0.00 24.01 0.67
**General Test Information**

- Test No. 1
- S:L Mass Ratio -- 1:01
- Moisture 1: Wet Sample g 64
- Moisture 1: Dry sample g 20.61
- Date 2017-7-21
- Test by Acid Type Nitric
- Sludge Moisture 1 g 0.68
- Temperature °C 22
- Stirrer Speed RPM 600
- Sample ID SL-108-1
- Sample Desc. Wet Sludge

---

**Moisture Data**

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<th>Sludge Moisture 3</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Nitric</td>
<td></td>
<td></td>
<td>0.68 g</td>
<td>0.12 g</td>
<td>0.25 g</td>
</tr>
</tbody>
</table>

**Analytical Reports**

- Analysis of constituent elements
  - Fe, 61.5 g
  - Si, 1.6 g
  - Al, 0.5 g
  - Ca, 0.4 g
  - Other, 36.0 g

---

**Test Notes**

**Procedure:**

1. Wear adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

**Legend:**

- Test Equal
- Data Input
- Calculations
- Key Result
- Description Text
### General Test Information

- **Test No.**: 1
- **Sample ID**: SL-128-1
- **Sample Desc.**: Brown Sludge

### Initial Data

<table>
<thead>
<tr>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:01</td>
<td>76.11</td>
<td>43.58</td>
</tr>
</tbody>
</table>

### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Acid Type</th>
<th>Acid Concentration mol/L</th>
<th>Sludge Moisture 1 g</th>
<th>Sludge Moisture 2 g</th>
<th>Sludge Moisture 3 g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Nitric</td>
<td></td>
<td>0.43</td>
<td>0.48</td>
<td>0.51</td>
</tr>
</tbody>
</table>

### Analytical Reports

- **Date**: 2017.7.07
- **Acid Concentration**: 3 mol/L

### Pre-processing

- **Sample ID**: SL-128-1
- **Sample Desc.**: Brown Sludge
- **Moisture**: 0.43 g

### Test Notes

- **Sample ID**: SL-128-1
- **Sample Desc.**: Brown Sludge
- **Moisture**: 0.43 g

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average pH</th>
<th>Cumu. Acid Added mL</th>
<th>Acid Added g</th>
<th>Acid Added Sludge Dry Mass g/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.98</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>6.95</td>
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<td>0.19</td>
<td>0.01</td>
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<tr>
<td>3</td>
<td>6.92</td>
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<td>0.01</td>
</tr>
<tr>
<td>5</td>
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<td>3.45</td>
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<td>6.87</td>
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<td>0.01</td>
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</tbody>
</table>

### Legend

- **Test ID**: SL-128-1
- **Sample ID**: SL-128-1
- **Sample Desc.**: Brown Sludge
- **Moisture**: 0.43 g

---

### Graph

- **X-axis**: Acid / Unit Mass Sludge (g/g)
- **Y-axis**: Avg pH
- **Legend**: Other, Fe, Si, Al, S, Other, Fe, Si, Al, S

---

156
**General Test Information**

- **Test No.**
- **Sample ID**
- **Sample Desc.**
- **Pre-processing**

**Initial Test Data**

- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample g**
  - **Date**
  - **Acid Type**
  - **Acid Concentration mol/L**
  - **Temperature**
  - **Binary Speed RPM**

**Moisture Data**

- **Test by**
- **Acid Molar Mass g/mol**

**Analytical Reports**

- **Sample ID**
- **Time**
- **Stirrer Speed RPM**

**Test Notes**

---

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

---

**Legend**

- **Test Equal**
- **Reagents/Reagent**
- **Calculations**
- **Key Result**
- **Description Test**

---

**Calculations:**

- **Increment Number**
- **Average pH**
- **Cumu. Acid Added (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

---

**Graph:**

- **SL-107-1**

---

**Data Entry:**

- **Increment Number**
- **Acid Added (g)**
- **pH**
- **pH**
- **pH**
- **pH**

---

**Note:**

- Include in Avg
- pH

---

**Table:**

- **Increment Number**
- **Acid Added (g)**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Average pH**
- **St. Dev**

---

**Legend:**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

---

**Feign Test Information**

- **Initial Test Data**
- **Moisture Data**

---

**Data Entry:**

- **Increment Number**
- **Acid Added (g)**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Measured pH**
- **Average pH**
- **St. Dev**

---

**Calculations:**

- **Increment Number**
- **Average pH**
- **Cumu. Acid Added (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

---

**Legend:**

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

---

**Graph:**

- **SL-107-1**

---

**Note:**

- Include in Avg
- pH

---
### General Test Information

**Test No.:** 31  
**Sample ID:** 31-01  
**Sample Desc.:** N/A  
**Pre-processing:** Directly sampled from bucket, removed Tramp

### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>31-01</td>
<td>1:01</td>
<td>33.02 g</td>
<td>1.25 g</td>
</tr>
</tbody>
</table>

### Analytical Reports

- **Acid Type:** Nitric
- **Acid Concentration:** 3 mol/L
- **Sludge Moisture 1:** 0.96%
- **Temperature:** 22°C
- **Stirrer Speed:** 630 RPM
- **Date:** 2017.7.10 AM
- **Sample ID:** SL-110-1

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average pH</th>
<th>Average</th>
<th>Cumulative Acid Added (mol)</th>
<th>Cumulative Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.63</td>
<td>6.06</td>
<td>0.00</td>
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<td>0.00</td>
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<tr>
<td>2</td>
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<td>0.00</td>
</tr>
<tr>
<td>3</td>
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<td>0.01</td>
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<td>3.67</td>
<td>0.94</td>
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</tr>
<tr>
<td>15</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test:** 31
- **Results:** N/A
- **Calculations:** N/A
- **Description:** N/A

### Sludge Acid Titration Test - Graph

![Graph showing average pH against acid added per unit mass of sludge](image-url)
### General Test Information

- **Test No.** 82
- **Date** 2017.7.10
- **Sludge Sample Code** SL-95-1

### Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
</tr>
</thead>
<tbody>
<tr>
<td>82</td>
<td>1:01</td>
<td>42.67</td>
<td>8.08</td>
</tr>
</tbody>
</table>

### Analytical Reports

- **Sample Desc.** Sludge Wet Mass g 100.0
- **Sample Desc.** Sludge Moisture 1 g 0.81
- **Sample Desc.** Sludge Dry Mass g 16.8
- **Sample Desc.** Sludge Volume ml 100.0

### Test Notes

- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).
  11. Dispose of any waste according to CHP.

### Calculations

#### Increment

<table>
<thead>
<tr>
<th>Number</th>
<th>Acid Added (mL)</th>
<th>Measured pH</th>
<th>Increment</th>
<th>Average pH</th>
<th>Sl. Dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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</tr>
</tbody>
</table>

### Legend

- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**

### Data Entry

- **Data Table**

### Graph

- **SL-95-1**

- **Graph Title:**

- **Graph Description:**

---

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### General Test Information
- **Test No.**
- **Material:** Red Dirt Sludge
- **Sample ID:** SL-140-2
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>SL-140-2</td>
<td>1:01</td>
<td>36.92 g</td>
<td>29.86 g</td>
</tr>
</tbody>
</table>

### Date
- **Date:** 2017.7.10
- **AM:**

### Analytical Reports
- **Acid Type:** Nitric
- **Acid Concentration:** 3 mol/L
- **Acid Molar Mass:** 63.01 g/mol

### Sludge Moisture
- **Moisture 1:**
  - **Wet Sample:** 36.92 g
  - **Dry Sample:** 29.86 g

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge</th>
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### Procedure
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend
- **Key Graph:**
- **Description Text:**
**General Test Information**

- **Test No.**: 39
- **Date**: 2017-7-11
- **Sample ID**: SL-129-1
- **Sample Desc.**: Red Sludge

**Initial Test Data**

- **S:L Mass Ratio**: 1:01
- **Moisture 1: Wet Sample**: 66.62 g
- **Moisture 1: Dry Sample**: 24.52 g
- **Acid Type**: Nitric
- **Nitric Acid Concentration**: 50% aq
- **Nitric Acid molar mass**: 63.01 g/mol
- **Acid Concentration**: 3 mol/L
- **Acid molar mass**: 63.01 g/mol
- **Temperature**: 22°C
- **Stirrer Speed**: RPM
- **Initial Volume**: ml

**Pre-processing**

- **Red Sludge**: Directly sampled from bucket, removed Tramp

**Data Entry**

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<tr>
<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Measured pH</th>
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</table>

**Calculations**

- **Average pH**: 7.62
- **Cumu. Acid Added (mL)**: 5.00
- **Acid Added / Unit Mass Sludge (g/g)**: 0.10

**Legend**

- **Fe, 25.3**: Fe
- **Ca, 25.1**: Ca
- **Si, 1.1**: Si
- **S, 0.9**: S
- **Other, 47.6**: Other

**Procedure**

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11. Dispose of any waste according to CHP.

**Diagram**

- **SL-129-1**

- **Graph**: pH vs. Acid / Unit Mass Sludge (g/g)

---

**Note:** Include in Avg "x"
### General Test Information
- Test No: 1
- Date: 2017-7-11
- Sample ID: 0101

### Initial Test Data
- S:L Mass Ratio: 1:01
- Moisture Data:
  - Test 1: Wet Sample, g: 38.29
  - Test 2: Dry Sample, g: 0.57

### Analytical Reports
- Acid Type: Nitric
- Sample Desc: Red Aqueous Sludge
- Pre-processing: Directly sampled from bucket, removed Tramp
- Red Aqueous Sludge:
  - Sample ID: SL-111-1
  - Analysis: Fe, Ca, Si, S, Other
  - Analysis Details: 49.3% Fe, 3.1% Ca, 2.0% S, 1.0% Si, 44.7% Other

### Test Notes
- Data Entry:
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<thead>
<tr>
<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Measured pH</th>
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<th>Measured pH</th>
<th>Measured pH</th>
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### Data Entry
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### Calculations
- Calculations:
<table>
<thead>
<tr>
<th>Increment</th>
<th>Average pH</th>
<th>Cum. Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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### Legend
- Test Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text

### Diagram
- Diagram of the reaction process.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
<th>Moisture 1: Dry Sample (g)</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration (mol/L)</th>
<th>Temp</th>
<th>Stirrer Speed (RPM)</th>
<th>Water Added (mL)</th>
<th>Sludge Moisture 1 (g)</th>
<th>Sludge Dry Mass (g)</th>
<th>Average Sludge Moisture (g)</th>
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</table>

**Procedure:**
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**Legend:**
- **Test Data:**
  - Initial pH
  - Cumulative Acid
  - Acid Added
  - Sludge Dry Mass (g/g)
  - Sludge Wet Mass (g)
  - Sludge Volume (mL)
  - Water Added (mL)
  - Average Moisture (g)
  - Other Components (g)

- **Calculations:**
  - Increment Number
  - Average
  - Cumulative Acid (mol)
  - Acid Added (mL)
  - Acid Added / Unit Mass Sludge (g/g)

**Data Entry:**

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<th>Increment</th>
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**Calculations:**

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**Graph:**

**Legend:**

- **Key Result:**
  - Description Text
### General Test Information
- **Test No.**: 1
- **Sample Desc.**: Brown-Red Sludge
- **Sample ID**: N/A
- **Pre-processing**: Directly sampled from bucket, removed Tramp
- **Sample Size**: Brown-Red Sludge

### Initial Test Data
- **Moisture 1**: Wet Sample
  - **g**: 33.6

### Analytical Reports
- **Date**: 2017.7.11
- **Stirrer Speed**: RPM

### Test Notes
- **Temperature**: 22°C
- **Stirrer Speed**: RPM

### Calculations

#### Raw Text

**Procedure:**
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### Data Entry

#### Table

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### Legend
- **Fe**, **Ca**, **Al**, **Si**, **Other**

### Notes
- Include in Avg: "x"
**General Test Information**

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<th>Test No.</th>
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**Initial Test Data**

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**Moisture Data**

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**Analytical Reports**

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**Pre-processing**

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**Calculations**

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<th>Average</th>
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- Test Vector
- Validation
- Calculation
- Key Result
- Description Text

**Data Entry**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>2</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
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<tr>
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<td>1.00</td>
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<td>4</td>
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<td>11.10</td>
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**Calculations:**

<table>
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<td>11.10</td>
</tr>
</tbody>
</table>

**Legend**

- Test Vector
- Validation
- Calculation
- Key Result
- Description Text

**Data Entry**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
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</tr>
</thead>
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<td>1</td>
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<td>11.10</td>
</tr>
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**Calculations:**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>2</td>
<td>0.10</td>
<td>0.10</td>
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<td>1.00</td>
<td>1.10</td>
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<tr>
<td>4</td>
<td>10.00</td>
<td>11.10</td>
<td>11.10</td>
</tr>
</tbody>
</table>

**Legend**

- Test Vector
- Validation
- Calculation
- Key Result
- Description Text
## General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Date</th>
<th>Site</th>
<th>Sample ID</th>
<th>Sample Desc.</th>
<th>Pre-processing</th>
<th>Analytical Reports</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2017-7-12</td>
<td>SL-126-1</td>
<td>Orange Sludge</td>
<td>Directly sampled from bucket, removed Tramp</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.67</td>
<td>0.59</td>
<td>0.39</td>
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</tbody>
</table>

### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Sludge Moisture 1</th>
<th>Sludge Moisture 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.61</td>
<td>0.59</td>
</tr>
</tbody>
</table>

### Test Notes

- 1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- 2. Create dilute acid solution at desired acid concentration. Record acid concentration.
- 3. Calibrate pH meter if necessary.
- 4. Perform moisture analysis on at least 3 feed sludge samples.
- 5. Add feed sludge to reaction vessel. Record volume and mass.
- 6. Add distilled water to achieve desired S:L ratio, record volume of water added.
- 7. Set desired temperature and stirrer speed. Thoroughly mix.
- 8. Measure and record initial pH of solution.
- 9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
- 10. Repeat step 9 until endpoint pH is reached (<0.5)
- 11. Dispose of any waste according to CHP.

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added</th>
<th>Sludge Dry Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.00</td>
<td>3.00</td>
<td>100.0</td>
</tr>
<tr>
<td>2</td>
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<td>3</td>
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</tr>
<tr>
<td>5</td>
<td>3.00</td>
<td>15.00</td>
<td>100.0</td>
</tr>
</tbody>
</table>

### Legend

- Test Sludge
- Water Added
- Initial Volume
- Final Volume
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

Legend:
- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text
### General Test Information

**Test No.**

1. 1:01

**Sample ID**

SL-109-1

**Sample Type**

Red-Brown Sludge

**Pre-processing**

- Directly sampled from bucket, removed Tramp
- SL-109-1

**Procedure**

1. **Data Entry**:
   - **Calculation**: Increment
   - **Descriptive Text**: Included in Avg

2. **Legend**:
   - **Text Input**: Fe, Si, Al, Ca, Other
   - **Numeric Input**: 35.9, 8.8, 2.8, 1.9, 50.5

### Initial Test Data

**Test No.**

1. 1:01

**Acid Type**

- Nitric

**Acid Concentration**: 3 mol/L

**Acid Molar Mass**: 63.01 g/mol

**Time Step**: 5 min

**Stirrer Speed**: RPM

**Sample Desc.**

Red-Brown Sludge

**Average Moisture**: 0.99

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>0.00</td>
<td>0.00</td>
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<td>3</td>
<td>0.00</td>
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<tr>
<td>10</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

**Legend**

- **Text Input**: Fe, Si, Al, Ca, Other
- **Numeric Input**: 35.9, 8.8, 2.8, 1.9, 50.5

### Analytical Reports

#### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:01</td>
<td>36.22</td>
<td>0.43</td>
</tr>
</tbody>
</table>

The diagram shows a trend line labeled SL-109-1 with points indicating changes in pH and acid added. The pH range is from 0 to 14, and the acid added range is from 0.0 to 14.0. The diagram illustrates the relationship between acid added and pH over time.
**General Test Information**

- **Date:** 2017.10.12
- **Sample ID:** SL-62-2
- **Sample Desc.:** White Sludge
- **Pre-processing:** Directly sampled from bucket, removed Tramp

**Note:** 
- 1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- 2. Create dilute acid solution at desired acid concentration. Record acid concentration.
- 3. Calibrate pH meter if necessary.
- 4. Perform moisture analysis on at least 3 feed sludge samples.
- 5. Add feed sludge to reaction vessel. Record volume and mass.
- 6. Add distilled water to achieve desired S:L ratio. Record volume of water added.
- 7. Set desired temperature and stirrer speed. Thoroughly mix.
- 8. Measure and record initial pH of solution.
- 9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
- 10. Repeat step 9 until endpoint pH is reached (<0.5).
- 11. Dispose of any waste according to CHP.

**Legend**
- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumus. Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g / g)</th>
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<td></td>
</tr>
</tbody>
</table>

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
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**Data Entry**

<table>
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<tr>
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<th>Rep 3</th>
<th>Rep 4</th>
<th>Rep 5</th>
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<td>12.90</td>
<td>12.89</td>
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</tbody>
</table>

**Initial Test Data**

- **Test No.:** 3
- **S:L Mass Ratio:** 1:01
- **Moisture 1: Wet Sample g:** 116.85
- **Moisture 1: Dry Sample g:** 52.31
- **Acid Type:** Nitric
- **Acid Concentration:** 3 mol/L
- **Acid molar mass:** 63.01 g/mol
- **Bomber Speed:** RPM
- **Date:** 2017.10.12

**Moisture Data**

- **Moisture 1: Wet Sample g:** 116.85
- **Moisture 1: Dry Sample g:** 52.31
- **Moisture 2: Wet Sample g:** 116.85
- **Moisture 2: Dry Sample g:** 52.31
- **Moisture 3: Wet Sample g:** 116.85
- **Moisture 3: Dry Sample g:** 52.31

**Water Added**

- **ml:** 200.0

**Average Moisture**

- **g:** 0.58
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>Moisture 1: Wet Sample g</th>
</tr>
</thead>
<tbody>
<tr>
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### Initial Test Data

<table>
<thead>
<tr>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration mol/L</th>
<th>pH</th>
<th>Acid molar mass g/mol</th>
<th>Temperature °C</th>
<th>Stirrer Speed RPM</th>
<th>Initial Volume ml</th>
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<tbody>
<tr>
<td>2017.10.12</td>
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<td>3</td>
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</table>

### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>Moisture 1: Dry sample g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tr>
</tbody>
</table>

### Test Notes

- Include in Avg "x"
- Notes:
  - Data adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  - Create dilute acid solution at desired acid concentration. Record acid concentration.
  - Calibrate pH meter if necessary.
  - Perform moisture analysis on at least 3 feed sludge samples.
  - Add feed sludge to reaction vessel. Record volume and mass.
  - Add distilled water to achieve desired S:L ratio, record volume of water added.
  - Set desired temperature and stirrer speed. Thoroughly mix.
  - Measure and record initial pH of solution.
  - Add desired volume of acid, wait until reaction is complete, measure and record pH.
  - Repeat step 9 until endpoint pH is reached (<0.5)
  - Dispose of any waste according to CHP.

### Data Entry

<table>
<thead>
<tr>
<th>Increment Number</th>
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<th>Average</th>
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<td>2.75</td>
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<tr>
<td></td>
<td>3</td>
<td>2.75</td>
<td>2.75</td>
<td>2.75</td>
</tr>
</tbody>
</table>

### Calculations

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average</th>
<th>Cumulative Acid Added (mL)</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.75</td>
<td>2.75</td>
<td>2.75</td>
</tr>
</tbody>
</table>

### Legend

- Red Orange Sludge
- N/A
- Directly sampled from bucket, removed Tramp
- SL-72-1
- Fe, 38.9
- Si, 6.6
- S, 4.6
- Al, 2.8
- Other, 47.1
- SL-72-1

### Diagram

[Sludge Acid Titration Test - Experimental Data Entry Sheet]

---

170
### General Test Information

- **Test No.**: [1:01]
- **S:L Mass Ratio**: --
- **Moisture 1: Wet Sample**: g 69.75
- **Moisture 1: Dry Sample**: g 39.56
- **Date**: 2017.10.12
- **Sample ID**: SL-80-2
- **Sample Desc.**: Grey Sludge, directly sampled from bucket, removed Tramp

### Initial Test Data

- **Acid Type**: Nitric
- **Acid Concentration**: mol/L 3
- **Acid Molar Mass**: g/mol 63.01
- **Sludge Moisture 1**: g 0.43
- **Time**: min 5
- **Acid Added**: mL 200.0

### Moisture Data

- **Sample ID**: SL-80-2
- **Sample Desc.**: Grey Sludge
- **Pre-processing**: Nutrients removed from feed sample
- **Water Added**: mL 100.0
- **Initial Volume**: mL 100.0
- **Average Moisture**: -- 0.44

### Analitical Reports

- **Calculation**: Average Moisture 0.42

### Test Notes

- **Procedure**:
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stir speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).
  11. Dispose of any waste according to CHP.

### Data Entry

<table>
<thead>
<tr>
<th>Sludge Acid Titration Test - Experimental Data Entry Sheet</th>
<th>Calculations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Increment</td>
<td>Average</td>
</tr>
<tr>
<td>Number</td>
<td>(mL)</td>
</tr>
<tr>
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<tr>
<td>9</td>
<td>0.00</td>
</tr>
<tr>
<td>10</td>
<td>0.00</td>
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</tbody>
</table>

### Legend

- **Test Liquid**: [Grey Sludge]
- **Sample ID**: SL-80-2
- **Description**: Test Sludge
**General Test Information**

- **Test No.**: SL-82-1
- **Sample Code**: N/A
- **Sample Decr.**: Directly sampled from bucket, removed Tramp

---

**Initial Test Data**

- **S:L Mass Ratio**: 1:01
- **Moisture 1: Wet Sample**: 51.59 g
- **Acid Type**: Nitric
- **Moisture 1: Dry Sample**: 1.2 g
- **Date**: 2017.10.12 AM
- **Acid Concentration**: 3 mol/L
- **Sample ID**: 3
- **Acid Molar Mass**: 63.01 g/mol
- **Temperature**: 32
- **Stirrer Speed**: RPM
- **Time**: 5
- **Sample ID**: 3

---

**Moisture Data**

- **Sludge Moisture 1**: 0.98
- **Sludge Moisture 2**: 0.98
- **Sludge Moisture 3**: 0.98
- **Water Added**: 200.0
- **Stirrer Speed**: med

---

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added (ml)</th>
<th>Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
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</thead>
<tbody>
<tr>
<td>Rep 1</td>
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<td>Rep 5</td>
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---

**Legend**

- **Test Entry**: Data Input
- **Data Output**: Calculations
- **Key Result**: Description Test

---

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

---

**Legend**

- **Test Entry**: Data Input
- **Data Output**: Calculations
- **Key Result**: Description Test

---

**Graph**

- **SL-82-1**
  - **pH vs. Acid / Unit Mass Sludge (g/g)**
  - **Sludge Volume**: 100.0 ml
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio --</th>
<th>Moisture 1: Wet Sample g</th>
<th>Test by Acidity</th>
<th>Sludge Moisture 1 --</th>
<th>Moisture 1: Dry sample g</th>
<th>Date</th>
<th>Temperature °C</th>
<th>Sludge Moisture 1 --</th>
<th>Moisture 2: Wet Sample g</th>
<th>Test by Acidity</th>
<th>Sludge Moisture 1 --</th>
<th>Moisture 2: Dry sample g</th>
<th>Date</th>
<th>Temperature °C</th>
<th>Sludge Moisture 1 --</th>
<th>Moisture 3: Wet Sample g</th>
<th>Test by Acidity</th>
<th>Sludge Moisture 1 --</th>
<th>Moisture 3: Dry sample g</th>
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<tbody>
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Test Notes

Data Entry

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<th>Data Yield</th>
<th>Step 1</th>
<th>Step 2</th>
<th>Step 3</th>
<th>Step 4</th>
<th>Step 5</th>
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<tbody>
<tr>
<td></td>
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</tbody>
</table>

Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

Legend

<table>
<thead>
<tr>
<th>Test Yield</th>
<th>Data Yield</th>
<th>Calculation</th>
<th>Key Result</th>
<th>Description Test</th>
</tr>
</thead>
</table>

97

SL-81-2
**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Red** = incinerator
- **Blue** = incinerator ash
- **White** = sludge

---

**Note:**

Include in Avg: 0 0 12.35 12.38 12.36 0.02 0 12.36 0 0.00 0.00 0.00 0.57 0.19 0.00 0.00 0.00 1.32 0.57 0.00 0.00 0.00 2.84 0.12 0.00 0.00 0.00 5.86 0.24 0.00 0.00 0.00 11.91 0.24 0.00 0.00 0.00


**General Test Information**

- **Test No.**
- **Initial Test Data**
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample g**
  - **Date**
  - **Acid Type**
  - **Acid Concentration mol/L**
  - **Acid molar mass g/mol**
  - **Sludge Moisture 1**
  - **Temperature**
  - **Stirrer Speed RPM**
  - **Moisture 2: Wet Sample g**
  - **Sample ID**
  - **Sample Desc.**
  - **Moisture 2: Dry sample g**
  - **Sludge Moisture 1**
  - **Time**
  - **Acid molar mass g/mol**
  - **Moisture 3: Wet Sample g**
  - **Moisture 3: Dry sample g**
  - **Sample ID**
  - **Temperature**
  - **Sample Desc.**
  - **Pre-processing**

**Analytical Reports**

- **Moisture Data**
  - **Moisture 1: Wet Sample g**
  - **Moisture 1: Dry sample g**
  - **Sludge Moisture 1**
  - **Stirrer Speed RPM**
  - **Moisture 2: Wet Sample g**
  - **Moisture 2: Dry sample g**
  - **Sludge Moisture 1**
  - **Sludge Dry Mass g**
  - **Time Step min**
  - **Sample ID**

**Pre-processing**

- **Moisture Data**
  - **Sludge Wet Mass g**
  - **Sludge Volume ml**
  - **Sample ID**
  - **Water Added ml**

**Test Notes**

- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

**Legend**

- **Test Equal**
- **Normalized**
- **Calculation**
- **Key Result**
- **Description Text**

<table>
<thead>
<tr>
<th>Data Entry</th>
<th>Rep 1</th>
<th>Rep 2</th>
<th>Rep 3</th>
<th>Rep 4</th>
<th>Rep 5</th>
<th>Average</th>
<th>Sl. Dev</th>
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<tbody>
<tr>
<td>Increment</td>
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**Calculations**

<table>
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<tr>
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<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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</tbody>
</table>

**Legend**

- **Test Equal**
- **Normalized**
- **Calculation**
- **Key Result**
- **Description Text**

**Data Entry**

- **Step 1**: Add desired volume of acid, wait until reaction is complete, measure and record pH.
- **Step 2**: Repeat step 9 until endpoint pH is reached (<0.5).
- **Step 3**: Dispose of any waste according to CHP.
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration mol/L</th>
<th>Sludge Moisture 1: g</th>
<th>Sludge Moisture 2: g</th>
<th>Sludge Moisture 3: g</th>
<th>Acidity Concentration mol/L</th>
<th>Acid Molar Mass g/mol</th>
<th>Temperature ºC</th>
<th>Stirrer Speed RPM</th>
<th>Water Volume ml</th>
<th>Sludge Volume ml</th>
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</thead>
<tbody>
<tr>
<td>01</td>
<td>1:01</td>
<td>96.66</td>
<td>21.17</td>
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### Calculations

<table>
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<th>Acid Added / Unit Mass Sludge (g/g)</th>
<th>pH</th>
<th>Sl. Dev</th>
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</table>

### Legend

- **Test Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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11. Dispose of any waste according to CHP.

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Data Entry</th>
<th>Rep 1</th>
<th>Rep 2</th>
<th>Rep 3</th>
<th>Rep 4</th>
<th>Rep 5</th>
<th>Average</th>
<th>Cumulative Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Increment</td>
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### Moisture Data

<table>
<thead>
<tr>
<th>Moisture 1: Wet Sample g</th>
<th>Moisture 1: Dry Sample g</th>
<th>Moisture 2: Wet Sample g</th>
<th>Moisture 2: Dry Sample g</th>
<th>Moisture 3: Wet Sample g</th>
<th>Moisture 3: Dry Sample g</th>
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</thead>
<tbody>
<tr>
<td>96.66</td>
<td>21.17</td>
<td>0.78</td>
<td>0.84</td>
<td>0.81</td>
<td>0.81</td>
</tr>
</tbody>
</table>

### Sludge Analysis Reports

- **Pre-processing**
  - Sludge Moisture 1: N/A
  - White Sludge: 100.0
  - Directly sampled from bucket, removed Tramp
- **Final Volume**
  - White Sludge: N/A

### Water Added

<table>
<thead>
<tr>
<th>Added</th>
<th>Cumulative Value (g)</th>
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<tbody>
<tr>
<td>White Sludge</td>
<td>100.0</td>
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</table>

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
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</tbody>
</table>

### Diagram

**SL-73-1**

- **Avg pH**
- **Acid / Unit Mass Sludge (g/g)
# Sludge Acid Titration Test - Experimental Data Entry Sheet

### General Test Information
- **Test No.**
- **Sample ID.**
- **Sample Desc.**
- **Pre-processing:**
  - Sludge moisture 1
- **Initial Volume:**
  - Brown Sludge
  - SL-80-1

### Initial Test Data
- **Test by:**
- **Acid Type:**
- **Acid Concentration:**
- **Temperature:**
- **Chemical:**
- **Initial Sludge Volume:**
- **Initial Sludge Mass:**
- **Water Added:**

### Moisture Data
- **Moisture 1:**
  - Wet Sample:
  - Dry Sample:

### Analytical Reports
- **Stirrer speed:**
- **Sludge moisture 1:**
- **Sludge moisture 2:**
- **Sludge moisture 3:**

### Calculations
- **Acid added:**
- **Acid added / Unit Mass Sludge:**

### Key Result
- **Other:**
  - Fe
  - Al
  - Ca
  - Si

### Data Entry

<table>
<thead>
<tr>
<th>Data Entry</th>
<th>Test 1</th>
<th>Test 2</th>
<th>Test 3</th>
<th>Test 4</th>
<th>Test 5</th>
<th>Average</th>
<th>Sl. Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Include in Avg</td>
<td>&quot;x&quot;</td>
<td>a</td>
<td>b</td>
<td>c</td>
<td>d</td>
<td>e</td>
<td>f</td>
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</tbody>
</table>

### Note:
- Include in Avg: "x" a b c d e f g h i j k l m n o p q r s t u v w x y z aa bb cc dd ee ff gg hh ii jj kk ll mm nn oo pp qq rr ss tt uu vv ww xx yy zz

### Legend:
- **Test Input:**
- **Calculation:**
- **Key Result:**
- **Description Text:

---

**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Graph:
- **Graph Title:** Sludge Acid Titration Test - Experimental Data Entry Sheet
- **Axes:**
  - Acid / Unit Mass Sludge (g / g)
  - pH
- **Data Points:**
  - SL-80-1
  - Other
  - Fe
  - Al
  - Ca
  - Si

---

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### General Test Information
- **Test No.**: 102
- **Date**: 2017.10.30
- **Time**: AM

### Initial Test Data
- **Sample Desc.**: White Sludge
- **Sludge Moisture 1**: 0.62
- **Sludge Moisture 2**: 0.66
- **Sludge Moisture 3**: 0.68

### Test Notes
- **Procedure**:
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).
  11. Dispose of any waste according to CHP.

### Data Entry

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<th>Increment</th>
<th>Acid Added (mL)</th>
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<th>Measured pH 3</th>
<th>Measured pH 4</th>
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<th>Sl. Dev</th>
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### Legend
- **Test Exit**: Final
- **Replication**
- **Calculations**
- **Key Result**
- **Description Text**
### General Test Information
- **Test No.**
- **Sample ID.**
- **Sample Desc.**
- **Sample Type.**
- **Acid Type.**
- **Acid Concentration.**
- **Acid Molar Mass.**
- **Initial Volume.**
- **Acid Added / Unit Mass Sludge (g/g).**

### Initial Test Data
- **Test by Acid Type.**
- **Acid Concentration.**
- **Acid Molar Mass.**
- **Initial Volume.**
- **Acid Added / Unit Mass Sludge (g/g).**

### Moisture Data
- **Test No.**
- **S:L Mass Ratio.**
- **Moisture 1: Wet Sample.**
- **Moisture 1: Dry Sample.**

### Analytical Reports
- **Analytical Report.**
- **Date.**
- **Temperature.**
- **Time.**
- **Stirrer Speed.**

### Pre-processing
- **Pre-processing Notes.**
- **Moisture 1: Wet Sample.**
- **Moisture 1: Dry Sample.**

### Test Notes
- **Test Notes.**
- **Initial Volume.**
- **Initial Volume.**
- **Initial Volume.**
- **Initial Volume.**

### Calculations
- **Calculations.**
- **Increment.**
- **Acid Added.**
- **Cumu. Acid Added.**
- **Acid Added / Unit Mass Sludge (g/g).**

### Data Entry
- **Data Entry.**
- **Increment Number.**
- **Acid Added.**
- **Measured.**
- **Measured.**
- **Measured.**
- **Measured.**
- **Measured.**
- **Average.**
- **St. Dev.**

### Legend
- **Legend.**
- **Key Result.**
- **Description Text.**

### Graph
- **Graph.**
- **Acid / Unit Mass Sludge (g/g).**
- **Avg pH.**
- **SL-87-5.**
- ** отметить.**
- **реакция окончена.**
- **Следующий этап.**
- **тест завершен.**

---

1. Conduct adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

**Note:**
- Include in Avg
- "x"
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
<th>Moisture 1: Dry Sample (g)</th>
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### Test Notes

- Sample ID: SL-77-2
- Sample Desc.: Brown Grey Sludge
- Pre-processing: Directly sampled from bucket, removed Tramp
- Brown Sludge

### Analytical Reports

- Nitric Acid
- Concentration: 3 mol/L
- Molar mass: 63.01 g/mol
- Sludge Moisture 1: 0.84%
- Sludge Moisture 2: 0.80%
- Sludge Moisture 3: 0.73%

### Test Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added (mL)</th>
<th>Cumu. Acid Added (mL)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
<th>Avg pH</th>
<th>Cumu. Acid Added / Sludge Dry Mass (g/g)</th>
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### Legend

- Test Result: Calculated
- Key Result: Description Text

### Graph

- SL-77-2

---

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### General Test Information

- Test No.: 105
- Date: 2017.11.2
- Sample ID: SS

### Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
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### Moisture Data

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### Analytical Reports

- Analysis Type: Nitric Acid
- Acid Concentration: 3 mol/L
- Acid Molar Mass: 63.01 g/mol
- Sludge Moisture 1: 0.93

### Pre-processing

- Sample ID: SL-97-5
- Sample Description: Directly sampled from bucket, removed Tramp
- Initial Volume: N/A

### Test Notes

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Key Result

- Avg. pH: 0.00
- Acid / Unit Mass Sludge (g/g): SL-97-5
- Fe: 14.5
- Al: 8.3
- Ca: 5.4
- S: 4.3
- Other: 67.4

---

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

---

### Legend

- Test Fail
- Data Entry
- Calculations
- Key Result
- Description Text

---

### Calculations

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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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### Diagram

- Title: SL-97-5
- X-axis: Acid / Unit Mass Sludge (g/g)
- Y-axis: Avg. pH
- Data points: Fe, Al, Ca, S, Other

---

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### General Test Information

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### Analytical Reports

**Sample ID**: 02-22-2222

**Sample Description**: N/A

**Pre-processing**: Grey sludge, directly sampled from bucket, removed tramp

### Initial Test Data

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### Test Notes

- **Stirrer Speed (RPM)**: med
- **Sample ID**: SL-63-2
- **Test by**: nitric
- **Sample Desc.**: Grey sludge

### Calculations

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**Note**: Include in Avg

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</tbody>
</table>

### Key Result

**Avg pH**

**Acid / Unit Mass Sludge (g/g)**

**Calcium, Aluminum, Iron, Other**

**Notes**: Include in Avg

#### Legend

- **Grey Sludge**
- **Directly sampled from bucket, removed tramp**

#### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

#### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Moisture Data</th>
<th>Moisture 1: Wet sample g</th>
<th>--</th>
<th>Moisture 1: Dry sample g</th>
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<tbody>
<tr>
<td></td>
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**General Test Information**

**Initial Test Data**

**Moisture Data**

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<th>Test No.</th>
<th>S:L Mass Ratio</th>
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<th>Moisture 1: Wet Sample g</th>
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<th>Moisture 1: Dry sample g</th>
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### Calculations

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<th>Cumu. Acid (mol)</th>
<th>Acid Added (g)</th>
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### Graph

**SL-63-2**

- **Acid / Unit Mass Sludge (g/g)**
- **Calcium, Aluminum, Iron, Other**

#### Notes:

Include in Avg

---

183
### General Test Information

- **Test No.**:
- **S:L Mass Ratio**:
- **Moisture 1: Wet Sample**: 87.88 g
- **Acid Type**: Nitric
- **Moisture 1: Dry sample**: 78.75 g
- **Sample ID**: SL-138-1
- **Sample Desc.**: Chalky White Sludge
- **Pre-processing**: Chalky sample collected from bucket, removed Tramp

### Analytical Reports

- **Moisture 2: Wet Sample**: 59.9 g
- **Sample ID**: SL-138-1
- **Sample Desc.**: Directly sampled from bucket, removed Tramp

### Test Notes

- **Sample ID**: SL-138-1
- **Sample Desc.**: Directly sampled from bucket, removed Tramp

### Calculations

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### Test Notes

- **Test Notes**:

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Type**: Initial Test Data
- **Sample ID**: SL-138-1
- **Sample Desc.**: Chalky White Sludge
- **Pre-processing**: Chalky sample collected from bucket, removed Tramp

---

**Note:**

Include in Avg "x"
## General Test Information

<table>
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<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
<th>Test by Acid Type</th>
<th>Moisture 1: Dry Sample (g)</th>
<th>Date</th>
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</table>

## Test Notes

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
### General Test Information

- **Test No.**: SL-64-2
- **Sample Desc.**: Red Orange Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

### Analytical Reports

- **Sample ID**: SL-64-2
- **Date**: 2017.11.03
- **Time Step**: 5 min

### Data Entry

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### Calculations

1. **General Procedure**:
   1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
   2. Create dilute acid solution at desired acid concentration. Record acid concentration.
   3. Calibrate pH meter if necessary.
   4. Perform moisture analysis on at least 3 feed sludge samples.
   5. Add feed sludge to reaction vessel. Record volume and mass.
   6. Add distilled water to achieve desired S:L ratio, record volume of water added.
   7. Set desired temperature and stirrer speed. Thoroughly mix.
   8. Measure and record initial pH of solution.
   9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
   10. Repeat step 9 until endpoint pH is reached (<0.5)
   11. Dispose of any waste according to CHP.

### Legend

- **Test Initial**: Red Orange Sludge
- **Pre-processing**: Directly sampled from bucket, removed Tramp

---

**Diagram**: SL-64-2

Legend:
- Test Initial: Red Orange Sludge
- Pre-processing: Directly sampled from bucket, removed Tramp

---

**Note**: Include in Avg x x x x x x
### General Test Information
- **Activity:** General Test Information
- **Initial Test Data:**
  - **Moisture Data:**
    - **Test No.:** Moisture 1: Wet Sample
    - **S:L Mass Ratio:** 1:01
    - **Moisture 1:** 93.84 g
    - **Acid Type:** Nitric
    - **Moisture 1:** Dry Sample
    - **g:** 34.66
- **Sample ID:** SL-95-2
- **Sample Desc:** Pre-processing
- **Test Notes:**

### Initial Test Data

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### Calculations

- **pH:**
  - **Acid / Unit Mass Sludge (g/g):**
  - **SL-95-2:**
    - **Si:** 18.5
    - **Al:** 11.2
    - **Fe:** 2.9
    - **Mg:** 2.4
    - **Other:** 65.0

### Test Notes
- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

### Legend
- **Text Input:**
- **Key Results:**
- **Description Text**

### Diagram

- **SL-95-2**
  - **Graph:**
    - **X-axis:** Acid / Unit Mass Sludge (g/g)
    - **Y-axis:** pH
    - **Data Points:**
      - **pH:** 4.12
      - **pH:** 6.78

---

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**Budge Acid Titration Test - Experimental Data Entry Sheet**

### General Test Information
- **Test No.** 111
- **Sample ID.** SS
- **Sample Desc.** Brown Dirt-like Sludge
- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

### Initial Test Data
- **Sample Desc:** Brown Dirt-like Sludge
- **Pre-processing:** Sample was comprised from bucket, removed Tramp

### Moisture Data
- **Moisture 1:**
  - **Wet Sample:** g 78.78
  - **Dry Sample:** g 38.15

### Analytical Reports
- **Sample Desc:** Brown Dirt-like Sludge
- **Pre-processing:** Sample was comprised from bucket, removed Tramp

### Calculations

#### Data Entry

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#### Key Results
- **SL-94-5**

### Legend
- Test Input
- Numeric Input
- Calculation
- Key Result
- Description Text

---

**Note:** Include in Avg "x"
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1:** Wet Sample (g)
- **Acid Type**
- **Acid Concentration** (mol/L)
- **Acid Molar Mass** (g/mol)
- **Temperature** (°C)
- **Stirrer Speed** (RPM)
- **Sample ID**
- **Test by**
- **Moisture 1:** Dry sample (g)
- **Date**
- **Sample Desc.**
- **Moisture 2:** Wet Sample (g)
- **Moisture 2:** Dry sample (g)
- **Acid Added / Unit Mass Sludge (g/g)**
- **Oxalic Acid (mol/L)**
- **Acid Molar Mass** (g/mol)
- **Sample ID**
- **Test by**
- **Moisture 3:** Wet Sample (g)
- **Moisture 3:** Dry sample (g)
- **Sample ID**
- **Test by**

**Calculations**

- **Increment Number**
- **Acid Added (g)**
- **Measured Acid (g)**
- **Avg. pH**
- **Cumu. Acid Added (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

**Legend**

- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Type**
- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**

**Sludge Acid Titration Test - Experimental Data Entry Sheet**

**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1:** Wet Sample (g)
- **Acid Type**
- **Acid Concentration** (mol/L)
- **Acid Molar Mass** (g/mol)
- **Temperature** (°C)
- **Stirrer Speed** (RPM)
- **Sample ID**
- **Test by**
- **Moisture 1:** Dry sample (g)
- **Date**
- **Sample Desc.**
- **Moisture 2:** Wet Sample (g)
- **Moisture 2:** Dry sample (g)
- **Acid Added / Unit Mass Sludge (g/g)**
- **Oxalic Acid (mol/L)**
- **Acid Molar Mass** (g/mol)
- **Sample ID**
- **Test by**
- **Moisture 3:** Wet Sample (g)
- **Moisture 3:** Dry sample (g)
- **Sample ID**
- **Test by**

**Calculations**

- **Increment Number**
- **Acid Added (g)**
- **Measured Acid (g)**
- **Avg. pH**
- **Cumu. Acid Added (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

**Legend**

- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Type**
- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**

**Sludge Acid Titration Test - Experimental Data Entry Sheet**

**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1:** Wet Sample (g)
- **Acid Type**
- **Acid Concentration** (mol/L)
- **Acid Molar Mass** (g/mol)
- **Temperature** (°C)
- **Stirrer Speed** (RPM)
- **Sample ID**
- **Test by**
- **Moisture 1:** Dry sample (g)
- **Date**
- **Sample Desc.**
- **Moisture 2:** Wet Sample (g)
- **Moisture 2:** Dry sample (g)
- **Acid Added / Unit Mass Sludge (g/g)**
- **Oxalic Acid (mol/L)**
- **Acid Molar Mass** (g/mol)
- **Sample ID**
- **Test by**
- **Moisture 3:** Wet Sample (g)
- **Moisture 3:** Dry sample (g)
- **Sample ID**
- **Test by**

**Calculations**

- **Increment Number**
- **Acid Added (g)**
- **Measured Acid (g)**
- **Avg. pH**
- **Cumu. Acid Added (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

**Legend**

- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- **Test Type**
- **Data Entry**
- **Calculations**
- **Key Result**
- **Description Text**
General Test Information

- Test No.: 1
- Sample ID: SL-32-1
- Date: 2017.11.9
- Sample Desc.: Brown Aqueous sludge
- Sample Type: Directly sampled from bucket, removed Tramp
- Pre-processing: Easily sampled from bucket, removed Tramp

Initial Test Data

- Moisture 1: Wet Sample g: 81.12
- Moisture 2: Wet Sample g: 88.67
- Moisture 3: Wet Sample g: 92.68
- Sludge Moisture 1: 0.86
- Sludge Moisture 2: 0.81
- Sludge Moisture 3: 0.77

Analytical Reports

- Acid Type: Nitric
- Acid Concentration: 3 mol/L
- Acid molar mass: 63.01 g/mol
- Time Step: 5 min
- Stirrer Speed: RPM
- Water Added: 100 ml
- Initial Volume = 100 ml

Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Legend

- Test Ident: SS
- Calculations: SL
- Key Result: NL
- Description Text

SL-32-1

Acid / Unit Mass Sludge (g/g)

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average Acid Added (ml)</th>
<th>Cum. Acid Added (mol)</th>
<th>Cum. Acid Added Sludge Dry Mass (g / g)</th>
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<tr>
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<table>
<thead>
<tr>
<th>Data Entry</th>
<th>Increment</th>
<th>Rep 1</th>
<th>Rep 2</th>
<th>Rep 3</th>
<th>Rep 4</th>
<th>Rep 5</th>
<th>Average</th>
<th>SL Dry Mass (g)</th>
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Notes

- Include in Avg: x

Calculation

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<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Cum. Acid Added Sludge Dry Mass (g / g)</th>
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Fe, 13.3
Mn, 8.6
Al, 7.3
Mg, 5.1
Other, 65.6

Note:

Include in Avg: x

Rep 1

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<tr>
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</table>

Abbreviations:

- Acid: ml
- Unit Mass Sludge: g/g
- Average: ml
- Cum. Acid: ml
- Cum. Acid Added Sludge Dry Mass (g / g): g/ml

Graph

- X-axis: Acid / Unit Mass Sludge (g/g)
- Y-axis: Avg pH

- SL-32-1

- All variables are within the limits:
  - Fe: 13.3
  - Mn: 8.6
  - Al: 7.3
  - Mg: 5.1
  - Other: 65.6
### General Test Information

- **Initial Test Data**
  - **Test No.**
  - **S:L Mass Ratio** -- 1:01
  - **Moisture 1: Wet Sample**
    - **g** 106.51
  - **Test by Acid Type**
    - **Nitric**
  - **Moisture 1: Dry sample**
    - **g** 65.87
  - **Date**
  - **Acid Concentration**
    - **mol/L** 3
  - **Sludge Moisture 1**
    - **%** 0.38
  - **Temperature**
  - **Speed RPM**
  - **Mixing Time**
  - **Stirrer Speed**
    - **RPM** med
  - **Sample Desc.**
    - **Red Coarse Dirt Sludge**
  - **Pre-processing**
    - **Wet sample was sampled from bucket, removed Tramp**
  - **Test Notes**
    - **Sample ID**
    - **Temperature**
      - **c** 22
    - **Sample ID**
      - **SL-132-1**
    - **Sample Desc.**
      - **Red Coarse Dirt Sludge**
    - **Pre-processing**
      - **Wet sample was sampled from bucket, removed Tramp**
    - **Test Notes**
      - **Sample ID**
      - **Temperature**
        - **c** 22

### Data Entry

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<th>Increment Number</th>
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<th>Acid Added / Sludge Dry Mass (g/g)</th>
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</table>

### Calculations

- **Initial Test Data**
  - **Sludge Moisture 1**
    - **%** 0.38
  - **Sludge Dry Mass**
    - **g** 61.7
  - **Water Added**
    - **ml** 100.0
  - **Sludge Wet Mass**
    - **g** 100.0
  - **Sludge Volume**
    - **ml** 100.0

### Analytical Reports

- **Average Moisture**
  - **%** 0.38

### Test Notes

- **Note:**
  - Include in Avg

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**
# General Test Information

## Initial Test Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
<th>Test by</th>
<th>Acid Type</th>
<th>Moisture 1: Dry Sample (g)</th>
<th>Date</th>
<th>Acid Concentration (mol/L)</th>
<th>Acid Molar Mass (g/mol)</th>
<th>Sludge Moisture 1 (g)</th>
<th>Temperature (°C)</th>
<th>Stirrer Speed (RPM)</th>
<th>Note</th>
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<tbody>
<tr>
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</table>

## Moisture Data

- **Sample ID**: 115-SS
- **Sample Desc.**: Red Orange, Lots of Leaves
- **Pre-processing**: Directly sampled from bucket, removed Tramp
- **Initial Volume**: N/A
- **SL-73-P**
- **Fe, Al, S, Si, Other**: 46.6, 5.4, 3.7, 2.0, 42.4

## Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

## Calculations

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average</th>
<th>Cumu. Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>10</td>
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</tbody>
</table>

**Legend**

- **Test Result**: Calculations
- **Key Result**: Description Text

![Graph](image-url)
### General Test Information

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Date</th>
<th>Sample ID</th>
<th>Sample Desc.</th>
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<tbody>
<tr>
<td>1</td>
<td>2017.11.9</td>
<td>SI-78-1</td>
<td>Red Fudge Like Sludge</td>
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</tbody>
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### Moisture Data

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:01</td>
<td>103.4</td>
<td>36.4</td>
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</tbody>
</table>

### Initial Volume

- Red Fudge Like Sludge
- Directly sampled from bucket, removed Tramp
- Sample ID: SI-78-1

### Analytical Reports

- pH
  - Before Acid: 7.09
  - After Acid: 7.11
- Acid Type: Nitric
- Acid Concentration: 3 mol/L
- Acid Molar Mass: 63.01 g/mol

### Calculations

- Increment: 0.00 0.01 0.10 1.00 10.00
- Acid Added / Unit Mass Sludge (g/g)
- SL-78-1
- Fe, 58.4
- Mg, 2.4
- Si, 2.1
- Ca, 1.1
- Other, 36.0

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- Test Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text
### General Test Information

- **Test No.**: 117
- **Sample Desc.**: Brown/Black Sludge
- **Sample ID**: SL-69-5
- **Sample Date**: 2017.11.13
- **Acid**: SS

### Initial Test Data

- **Moisture 1**: Wet Sample, g: 104.11
- **Moisture 2**: Wet Sample, g: 146.59
- **Moisture 3**: Wet Sample, g: 95.19

### Test Notes

- **Procedure**:
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

### Analytical Reports

- **Water Added**: ml: 100.0
- **Atomic Composition**:
  - Si: 14.4
  - Al: 11.5
  - Ca: 6.0
  - Fe: 4.5
  - Other: 63.6

### Calculations

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<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added</th>
<th>Cumulative Acid Added</th>
<th>Acid Added per Unit Mass Sludge (g/g)</th>
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### Legend

- **Test Date**
- **Sample ID**
- **Sample Desc.**
- **Sample Date**

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### Diagam:

- **SL-69-5**

---

194
## General Test Information

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<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample g</th>
<th>Test by Acid Type</th>
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<th>Moisture 1: Dry sample g</th>
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### Initial Test Data

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<th>Acid Molar Mass (g/mol)</th>
<th>Sludge Moisture 1</th>
<th>Temperature °C</th>
<th>Stirrer Speed RPM</th>
<th>Water Added mL</th>
<th>Initial Sludge Mass (g)</th>
<th>Initial Volume ml</th>
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### Test Notes

- Data Entry
- Calculations
- Key Result
- Description Text

### Calculations

<table>
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<th>Increment Number</th>
<th>Average</th>
<th>Cumulative Acid Added (mol)</th>
<th>Acid Added (g)</th>
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### Results

- **Moisture Data**
  - Moisture 1: Wet sample
  - Moisture 2: Wet sample
  - Moisture 3: Wet sample
  - Moisture 1: Dry sample
  - Moisture 2: Dry sample
  - Moisture 3: Dry sample

- **Rep 1**
  - Average pH
  - Acid / Unit Mass Sludge (g/g)

- **Rep 2**
  - Average pH
  - Acid / Unit Mass Sludge (g/g)

- **Rep 3**
  - Average pH
  - Acid / Unit Mass Sludge (g/g)

- **Rep 4**
  - Average pH
  - Acid / Unit Mass Sludge (g/g)

- **Rep 5**
  - Average pH
  - Acid / Unit Mass Sludge (g/g)

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- Test Type
- Calculations
- Key Result
- Description Text

### Notes

- Include in Avg
- Note: 0.00
### General Test Information
- **Initial Test Data**
  - Test No.
  - Sample ID
- **Moisture Data**
  - Test by Acid Type
  - **Nitric**
    - **Moisture 1:** Wet Sample
      - g 64.02
    - **Moisture 2:** Dry sample
      - g 14.64
  - **Table:** Sludge Moisture 1
    - Sludge Moisture 1: Wet Sample
      - g 68.87
    - Sludge Moisture 1: Dry Sample
      - g 11.25

### Analytical Reports
- **Pre-processing:**
  - Sample was collected from bucket, removed Tramp
  - Initial Volume
  - Red Sludge
  - N/A
  - Directly sampled from bucket, removed Tramp
- **Sample Desc.**
  - SL-68-1
- **SL-68-1**
  - **Si** 19.1
  - **Fe** 9.5
  - **Al** 4.9
  - **S** 2.2
  - Other 64.2

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Data Entry
<table>
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<tr>
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<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
</tr>
</thead>
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<td></td>
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<td>(mL)</td>
<td>(mol)</td>
<td>(g)</td>
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</table>

### Calculations

### Legend
- **Test Equal**
- **Recovery Equal**
- **Conclusion**
- **Key Result**
- **Description Test**

---

### Graph

![Graph](image-url)
### General Test Information

<table>
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<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Wet Sample</th>
<th>Dry Sample</th>
<th>Temperature</th>
<th>Sludge Moisture 1</th>
<th>Dry sample</th>
<th>Average Moisture</th>
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### Analytical Reports

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<th>Acid Concentration</th>
<th>Acid molar mass</th>
<th>Sludge Moisture 1</th>
<th>Dry sample</th>
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### Test Notes

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 if endpoint pH is not reached (<0.5).
11. Dispose of any waste according to CHP.

### Calculations

<table>
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<th>Inc.</th>
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<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Sludge Dry Mass</th>
<th>Sludge Wet Mass</th>
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### Legend

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text

### Data Entry

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</tbody>
</table>

### Procedure:

1. Set adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 if endpoint pH is not reached (<0.5).
11. Dispose of any waste according to CHP.

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Sludge</th>
<th>SL-90-1</th>
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<tbody>
<tr>
<td>Al,</td>
<td>11.1</td>
</tr>
<tr>
<td>Si,</td>
<td>9.3</td>
</tr>
<tr>
<td>Fe,</td>
<td>5.0</td>
</tr>
<tr>
<td>Mn,</td>
<td>3.9</td>
</tr>
<tr>
<td>Other,</td>
<td>70.7</td>
</tr>
</tbody>
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197
**General Test Information**

- **Test No.**: 1
- **Date**: 2017-11-11
- **Sample ID**: SL-65-1

**Analytical Report**

- **Sample Desc.**: Brown Sludge
- **Pre-processing**: Sludge sampled from bucket, removed Tramp

**Procedure**:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Test Fluid**
- **Analysis Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

### Data Entry

<table>
<thead>
<tr>
<th>Increment</th>
<th>Acid Added</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Average</th>
<th>Sl. Dev</th>
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<td>[ml]</td>
<td>[ml]</td>
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</table>

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g / g)</th>
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<tr>
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<td>[ml]</td>
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<td>[g]</td>
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</tbody>
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**Legend**

- **Include in Avg**: x
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**
- **pH**

**Note**: Include in Avg "x"
### General Test Information

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### Moisture Data

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</thead>
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<table>
<thead>
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<th>Moisture 1: Dry sample</th>
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### Test Notes

- Include in Avg

### Data Entry

<table>
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<th>Increment Number</th>
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<td>Al, 1.7</td>
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<td>Si, 1.3</td>
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<td>Other, 38.9</td>
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### Calculations

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average Sl. Dry Mass / Add. Acid (g/g)</th>
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</thead>
<tbody>
<tr>
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<td>S, 5.0</td>
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<td></td>
<td>Si, 1.3</td>
</tr>
<tr>
<td></td>
<td>Other, 38.9</td>
</tr>
</tbody>
</table>

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
### General Test Information

- **Test No.:** 123
- **Sample ID:** SL-71-1
- **Pre-processing:** Directly sampled from bucket, removed Tramp

### Test Notes

- **Red Black Sludge**
- **N/A**
- **Directly sampled from bucket, removed Tramp**

### Analytical Data

#### Moisture Data

- **Test No.:** 123
- **S:L Mass Ratio:** 1:01
- **Moisture 1:** Wet Sample
  - **g:** 146.5
- **Moisture 2:** Wet Sample
  - **g:** 150.51
- **Sludge Moisture 1**
  - **g:** 100.0
  - **%:** 0.76
- **Sludge Moisture 2**
  - **g:** 23.8
  - **%:** 0.77

### Test Notes

- **Sample Desc.:** Red Black Sludge
- **Sample Description:** Directly sampled from bucket, removed Tramp

### Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
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11. Dispose of any waste according to CHP.

### Data Entry

<table>
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<tr>
<th>Increment</th>
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<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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### Calculations

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<th>Increment</th>
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<th>Cumulative Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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### Legend

- **Note:** Include in Avg

### Graph

**SL-71-1**

- **Graph Title:** Acid / Unit Mass Sludge (g/g)
- **X-axis:** Acid / Unit Mass Sludge (g/g)
- **Y-axis:** Avg pH

### Other Information

- **Fe, Si, S, Al, Other:**
  - **Fe:** 47.6
  - **Si:** 4.0
  - **S:** 2.0
  - **Al:** 1.5
  - **Other:** 44.9

---

**Data Entry**

- **Increment:** 1, 2, 3, 4, 5
- **Acid Added:** 1.5, 2.5, 3.5, 4.5, 5.5
- **Measured:** 1.50, 2.50, 3.50, 4.50, 5.50
- **Average:** 1.50, 2.50, 3.50, 4.50, 5.50
- **Cumulative Acid Added:** 1.50, 2.00, 3.00, 4.00, 5.00
- **Acid Added:** 1.00, 2.00, 3.00, 4.00, 5.00
- **Acid Added / Sludge Dry Mass (g/g):** 1.00, 0.80, 1.50, 2.00, 2.50

---

**Note:** Include in Avg
# General Test Information

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Acid Type**
- **Acid Concentration**
- **Acid Molar Mass**
- **Temperature**
- **Sludge Moisture 1**
- **Sample Desc.**
- **Pre-processing**

## Initial Test Data

- **Test by**
- **Acid Type**
- **Acid Concentration**
- **Acid Molar Mass**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**
- **Date**
- **Sample ID**

## Moisture Data

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**
- **Sample Desc.**
- **Pre-processing**

## Initial Volume

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<th>Measured pH</th>
<th>Measured pH</th>
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</tr>
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</table>

## Calculations

- **Increment**
- **Average**
- **Cumu. Acid (g)**
- **Acid Added / Unit Mass Sludge (g/g)**

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

---

**Data Entry**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
<th>Date</th>
<th>Sample ID</th>
<th>Sample Desc.</th>
<th>Pre-processing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1:01</td>
<td>98.95</td>
<td>6.77</td>
<td>2017.11.16</td>
<td>124</td>
<td>Red Aqueous Sludge</td>
<td>Fully sampled from bucket, removed Tramp</td>
</tr>
</tbody>
</table>

---

**Graph**

- **x-axis:** Acid / Unit Mass Sludge (g/g)
- **y-axis:** Avg pH

---

**Table:**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Added Acid / Unit Mass Sludge (g/g)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>SL Dry Mass</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>1.25</td>
<td>1.29</td>
<td>0.78</td>
<td>0.78</td>
<td>0.79</td>
<td>0.79</td>
<td>0.79</td>
<td>0.79</td>
</tr>
<tr>
<td>2</td>
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<td>1.25</td>
<td>1.29</td>
<td>0.78</td>
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<tr>
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<td>1.25</td>
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</tr>
</tbody>
</table>

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**Note:** Include in Avg "x"
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample** (g)
  - **Moisture 1: Dry Sample** (g)

**Analytical Reports**

- **Acid Type**
- **Acid Concentration** (mol/L)
- **Acid Molar Mass** (g/mol)

**Date**

- **Acid Concentration**
- **Temperature**
- **Sludge Moisture 1**

**Results**

- **Sample ID**
- **Sample Desc.**
- **Pre-processing**
- **Moisture 2:**
  - **Wet Sample** (g)
  - **Dry Sample** (g)

**Calculations**

- **Test Notes**
- **Data Entry**
  - **Increment Number**
  - **Acid Added** (g)
  - **Measured pH**
  - **Sl. Dry Mass** (g)

**Legend**

- **Test Fluid**
- **Water Added**
- **Stirrer Speed**
- **pH**

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Test Fluid**
- **Water Added**
- **Stirrer Speed**
- **pH**
**General Test Information**

- **Test No.** 1
- **Sample ID.** 312-2
- **Sample Desc.** White Chalky Sludge
- **Pre-processing**:
  - Directly sampled from bucket, removed Tramp
  - SL-138-2

**Analytical Reports**

- **Acid Type**: Nitric
- **Stirrer Speed**: RPM
- **Date**: 11.16.2017
- **Acid Concentration**: mol/L
- **Temperature**: °C
- **Sludge Moisture 1**: g
- **Sample ID**: N/A
- **Stirrer Speed**: RPM
- **Time Step**: min
- **Sample ID**: AM
- **Analysis**: White Chalky Sludge
- **Nitric Acid Concentration**: mol/L
- **Moisture 2**: g
- **Time**: min
- **Acid Molar Mass**: g/mol
- **Moisture 3**: g
- **Mass**: g
- **Moisture 4**: g
- **Sample Diameter**: cm
- **Moisture 5**: g
- **Sample Date**: 11.16.2017
- **Moisture 1**: g
- **Sample Grade**: g
- **Moisture 6**: g
- **Sample Description**: White Chalky Sludge
- **Moisture 7**: g
- **Sample ID**: AM
- **Moisture 8**: g

**Test Notes**

**Data Entry**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Acid Added</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>Sl. Dev.</th>
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<tbody>
<tr>
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**Calculations**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Average pH</th>
<th>Cum. Acid Added (mol)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>10.59</td>
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</tbody>
</table>

**Legend**

- **Test Start**: Date
- **Test End**: Date
- **Calculation**: Calculation
- **Data Entry**: Data Entry
- **Key Result**: Result
- **Description Test**: Test Notes

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Table:**

<table>
<thead>
<tr>
<th>Increment Number</th>
<th>Acid Added (g)</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
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</thead>
<tbody>
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</tr>
</tbody>
</table>

**Note:**

- Include in Avg "x"
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
  - **Temperature**
  - **Time**
  - **Stirrer Speed**
  - **Pre-processing**
  - **Moisture 2: Wet Sample**
  - **Date**
  - **Acid Concentration**
  - **Sludge Moisture 1**
  - **Temperature**
  - **Time**
  - **Stirrer Speed**
  - **Pre-processing**
  - **Moisture 3: Wet Sample**
  - **Sample ID**
  - **Sample Description**
  - **Analytical Reports**
  - **Test Notes**

**Calculation**

- **Data Entry**
  - **Increment**
  - **Acid Added**
  - **Measured**
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  - **Meas
**Moisture Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample (g)</th>
<th>Moisture 1: Dry Sample (g)</th>
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<tbody>
<tr>
<td>1</td>
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<td>10.73</td>
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</tbody>
</table>

**Sample Details**

- Sludge: Brown Aquous Sludge
- Sample Desc: Directly sampled from bucket, removed Tramp
- Date: 2017.11.16
- Time: AM
- Temperature: 30.0°

**Sample ID:** SL-94-1

**Pre-processing:**
- Sludge: Moisture 1
- Water Added: 100.0 ml
- Initial Volume: 128 ml

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added (mol)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</tr>
</tbody>
</table>

**Legend**

- pH
- Sludge
- Increment
- Average
- Cum. Acid Added

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Graph**

- Graph showing pH over time with data points.
- Legend: Sl-94-1
- Additional notes on graph.
**General Test Information**

**Initial Test Data**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>S:L Mass Ratio</th>
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</tr>
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<td>Moisture 1: Wet Sample</td>
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</tr>
<tr>
<td>Test by Acid Type</td>
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<tr>
<td>Nitric</td>
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<td>Moisture 1: Dry sample</td>
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**Analytical Reports**

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**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 8 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

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**Note:**

- Include in Avg "x"
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*Pre-processing:
- sludge sampled from bucket, removed trash

*Notes:
- include in avg: x

### Initial Test Data

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### Moisture Data

- Sludge Moisture 1: Wet Sample g 111.55 |
- Sludge Moisture 1: Dry Sample g 45.34 |
- Sludge Moisture 2: Wet Sample g 88.23 |
- Sludge Moisture 2: Dry Sample g 33.61 |
- Sludge Moisture 3: Wet Sample g 101.34 |
- Sludge Moisture 3: Dry Sample g 40.28 |

### Analytical Reports

- Average Moisture g 0.60

### Calculations

#### Acid / Unit Mass Sludge (g/g)

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**Legend**

- Data Entry:
  - First Input
  - Calculations
  - Description Text

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- Data Entry:
  - First Input
  - Calculations
  - Description Text

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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- Data Entry:
  - First Input
  - Calculations
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9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

**Legend**

- Data Entry:
  - First Input
  - Calculations
  - Description Text
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| Sludge Moisture 1 | -- |

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### Test Notes

- Ensure adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- Create dilute acid solution at desired acid concentration. Record acid concentration.
- Calibrate pH meter if necessary.
- Perform moisture analysis on at least 3 feed sludge samples.
- Add feed sludge to reaction vessel. Record volume and mass.
- Add distilled water to achieve desired S:L ratio, record volume of water added.
- Set desired temperature and stirrer speed. Thoroughly mix.
- Measure and record initial pH of solution.
- Add desired volume of acid, wait until reaction is complete, measure and record pH.
- Use curve step 9 acid endpoint pH is reached (pH 5).
- Dispose of any waste according to CHP.

### Calculations

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### Legend

- **Test Type**: Acid Titration Test
- **Sample ID**: SL-72-P
- **Sample Desc.**: Directly sampled from bucket, removed Tramp
- **Acid Type**: Nitric
- **Date**: 2017-11-20
- **Temperature**: 22°C

### Sludge Acid Titration Test - Experimental Data Entry Sheet

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### Data Entry

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### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Results

- **Initial Volume**: ml
- **Sample ID**: SL-72-P
- **Sample Desc.**: Directly sampled from bucket, removed Tramp
- **Acid Type**: Nitric
- **Date**: 2017-11-20
- **Temperature**: 22°C

### Calculations

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<tr>
<th>Increment</th>
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<th>Acid Added / Sludge Dry Mass (g / g)</th>
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<tr>
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<td>0.00</td>
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</tbody>
</table>

### Legend

- **Test Type**: Acid Titration Test
- **Sample ID**: SL-72-P
- **Sample Desc.**: Directly sampled from bucket, removed Tramp
- **Acid Type**: Nitric
- **Date**: 2017-11-20
- **Temperature**: 22°C

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Step</th>
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<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Measured</th>
<th>Average</th>
<th>SL Dry Mass</th>
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</tbody>
</table>

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Test Type**: Acid Titration Test
- **Sample ID**: SL-72-P
- **Sample Desc.**: Directly sampled from bucket, removed Tramp
- **Acid Type**: Nitric
- **Date**: 2017-11-20
- **Temperature**: 22°C

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Step</th>
<th>Increment</th>
<th>Acid Added</th>
<th>Measured</th>
<th>Measured</th>
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<th>SL Dry Mass</th>
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General Test Information

<table>
<thead>
<tr>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
<th>Temperature</th>
<th>Sludge Moisture 1: Dry Sample g</th>
<th>Sludge Moisture 1: Wet Sample g</th>
<th>Sludge Moisture 1: Average Moisture g</th>
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<tbody>
<tr>
<td>30-07-17 17:11</td>
<td>Nitric</td>
<td>35.0</td>
<td>63.01</td>
<td>37.52</td>
<td>94.35</td>
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Analytical Reports

<table>
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<tbody>
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<td>30-07-17 17:11</td>
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Pre-processing

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<th>Sample ID</th>
<th>Date</th>
<th>Acid Type</th>
<th>Acid Concentration</th>
<th>Acid molar mass</th>
<th>Temperature</th>
<th>Sludge Moisture 1: Dry Sample g</th>
<th>Sludge Moisture 1: Wet Sample g</th>
<th>Sludge Moisture 1: Average Moisture g</th>
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</thead>
<tbody>
<tr>
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<td>37.52</td>
<td>94.35</td>
<td>0.60</td>
<td>0.65</td>
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</table>

Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Legend:

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text

Graph:

- SL-66-4
- pH
- Acid / Unit Mass Sludge (g/g)
- Avg pH
- Sludge Moisture 1
- Moisture 1: Wet Sample
- Moisture 1: Dry Sample
- Moisture 2: Wet Sample
- Moisture 2: Dry Sample
- Moisture 3: Wet Sample
- Moisture 3: Dry Sample
- Average Moisture
- Other

209
**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**
- **Test Type:**
- **Note:**
- **Calculation:**
- **Key Result:**
- **Description Test**

**SL-70-P**

**Analytical Report**
- **Sample Desc.:** Directly sampled from bucket, removed Tramp
- **Red Sludge:** N/A
- **Sample ID:** SL-70-P
- **Test by:** txt
- **Date:** 2017.11.20
- **Acid Type:** Nitric

**Initial Test Data**
- **Test No.:** Rep 1
  - **S:L Mass Ratio:** 1:01
  - **Moisture 1: Wet Sample:** 97.97 g
  - **Moisture 1: Dry sample:** 28.45 g
- **Sample Desc.:** Red Sludge
- **Acid Concentration:** 3 mol/L
- **Sample ID:** SL-70-P
- **Moisture 2: Wet Sample:** 107.14 g
  - **Sludge Moisture 1:** 0.71%
  - **Temperature:** 22 c
- **Sludge Moisture 1:** 0.70%
- **Acid molar mass:** 63.01 g/mol
- **Time:** 5 min
- **Acid Concentration:** 3 mol/L

**Pre-processing**
- **Stirrer Speed:** med
- **Sample ID:** SL-70-P
- **Sludge Moisture 1:** 0.70%
- **Temperature:** 22 c
  - **Sludge Moisture 1:** 0.70%
- **Sample ID:** SL-70-P
- **Stirrer Speed:** med
  - **Sludge Moisture 1:** 0.70%
- **Temperature:** 22 c

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumu. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sl. Dry Mass (g/g)</th>
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<tbody>
<tr>
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<th>Sl. Dry Mass (g)</th>
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**Note:** Include in Avg

<table>
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**Legend**
- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**

**General Test Information**
- **Test No.:** Rep 1
  - **Date:** 2017.11.20
  - **Acid Type:** Nitric
- **Sample Desc.:** Red Sludge
Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

Legend
- Text Input
- Numeric Input
- Calculation
- Key Result
- Description Text
**General Test Information**

<table>
<thead>
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<th>Test No.</th>
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**Test Notes**

- General Test Information
- Initial Test Data
- Moisture Data

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<th>Test No.</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Moisture 1: Dry Sample</th>
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**Acid Type**

- Nitric

**Sample Desc.**

- Brown Sludge

---

**Data Entry**

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<th>Acids Added (mL)</th>
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<th>Measured (pH)</th>
<th>Measured (pH)</th>
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**Calculations**

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<tr>
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<th>Cumulative Acid (mL)</th>
<th>Acid Added (g)</th>
<th>Acid Added / Sl. Dev. Mass (g/g)</th>
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</tbody>
</table>

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**Legend**

- Test Std
- Calculated
- Conclusion
- Key Result
- Description Text
**General Test Information**

- **Test No.**
- **S:L Mass Ratio**
- **Moisture Data**
  - **Moisture 1:** Wet Sample (g) 75.48
  - **Moisture 2:** Wet Sample (g) 70.89
  - **Moisture 3:** Wet Sample (g) 75.27
  - **Moisture 1:** Dry Sample (g) 49.65
  - **Moisture 2:** Dry Sample (g) 45.51
  - **Moisture 3:** Dry Sample (g) 48.41

**Analytical Reports**

- **Date**
- **Acid Type**
- **Acid Concentration**
- **Acid Molar Mass (g/mol)**
- **Sludge Moisture 1**
  - **Sludge Moisture 1:** Sludge Moisture (g) 100.0
  - **Sludge Moisture 1:** Sludge Volume (ml) 100.0
  - **Sludge Moisture 1:** Sludge Dry Mass (g) 64.8
  - **Sludge Moisture 1:** Water Added (ml) 200.0

<table>
<thead>
<tr>
<th>Test Notes</th>
<th>Pre-processing Notes</th>
<th>Analysis Notes</th>
<th>Test Notes</th>
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**Test Notes**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 acid endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend**

- **Test Input**
- **Calculation**
- **Key Result**
- **Description Test**

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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</table>

**Graph**

- **SL-65-5**
- **Other | Sl 5**
- **

---

**Procedure**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 acid endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.
General Test Information

- Test No.
- S:L Mass Ratio
- 1:01
- Moisture 1: Wet Sample
- g
- 111.09
- Test by
- Acid Type
- Nitric
- Moisture 1: Dry Sample
- g
- 9.85
- Date
- 2017.11.28
- Temperature
- 22
- Sludge Moisture 1
- g
- 0.91
- Time
- 3
- Acid Concentration
- mol/L
- 63.01
- Sludge Moisture 1
- g
- 0.91
- Sample ID
- 02-32-1
- Stirrer Speed
- RPM
- 900
- Sludge Moisture 1
- g
- 0.91
- Sample Desc.
- Grey Aqueous Sludge
- Directly sampled from bucket, removed Tramp
- SL-62-1

Moisture Data

- Test
- Initial Test Data
- Moisture Data
- Test No.
- S:L Mass Ratio
- 1:01
- Moisture 1: Wet Sample
- g
- 111.09
- Test by
- Acid Type
- Nitric
- Moisture 1: Dry Sample
- g
- 9.85
- Date
- 2017.11.28
- Temperature
- 22
- Sludge Moisture 1
- g
- 0.91
- Time
- 3
- Acid Concentration
- mol/L
- 63.01
- Sludge Moisture 1
- g
- 0.91
- Sample ID
- 02-32-1
- Stirrer Speed
- RPM
- 900
- Sludge Moisture 1
- g
- 0.91
- Sample Desc.
- Grey Aqueous Sludge
- Directly sampled from bucket, removed Tramp
- SL-62-1

Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

Legend:

- Test: Final
- Present/Planned: Calculation
- Key Result: Description Text

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Calculations:

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SL-62-1

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**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text

---

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**Legend:**

- Text Input
- Numeric Input
- Calculation
- Key Result
- Descriptive Text
SLudge Acid Titration Test - Experimental Data Entry Sheet

Initial Test Data

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Procedure:

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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Legend

- Text Input
  - Numeric Input
  - Calculation
  - Key Result
  - Descriptive Text

Data Entry

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Data Notes:

- Include in Avg "x"  x  x  x  x

Calculations

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<th>Cumulative Acid Added (g)</th>
<th>Sludge Dry Mass (g)</th>
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Graph: SL-76-1

Legend

- Acid / Unit Mass Sludge (g/g)

Acid added to Sludge Dry Mass (g/g)

Other (R2).7

Ca, Mg, S, Si, Other

Note:

Include in Avg "x."
### General Test Information

<table>
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<tr>
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<th>S:L Mass Ratio</th>
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<th>Test by Acid Type</th>
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### Initial Test Data

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<th>Sludge Moisture 1</th>
<th>Time Step (min)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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### Moisture Data

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<th>Acid Molar Mass (g/mol)</th>
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<th>Time Step (min)</th>
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### Test Notes

- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).

### Legend
- **Key Input:**
  - Text Input
  - Numeric Input
  - Calculation

### Data Entry

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<th>Increment Number</th>
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<th>Measured pH</th>
<th>Measured pH</th>
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### Calculations

<table>
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<tr>
<th>Increment Number</th>
<th>Average pH</th>
<th>Cum. Acid Added (mL)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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### Sludge Acid Titration Test - Experimental Data Entry Sheet

- **Date:** 2017.11.28
- **AM:**
  - Sludge Moisture 1: Wet Sample
  - Sludge Moisture 1: Dry Sample
  - Sludge Moisture 2: Wet Sample
  - Sludge Moisture 2: Dry Sample
  - Sludge Moisture 3: Wet Sample
  - Sludge Moisture 3: Dry Sample

### Sludge Volume

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Description</th>
<th>Date</th>
<th>Acid Concentration (mol/L)</th>
<th>Acid Molar Mass (g/mol)</th>
<th>Sludge Moisture 1</th>
<th>Time Step (min)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Black Brown Sludge</td>
<td>2017.11.28</td>
<td>3</td>
<td>63.01</td>
<td>0.88</td>
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</tr>
<tr>
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<td>2018.01.01</td>
<td>3</td>
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<th>Sludge Moisture 1</th>
<th>Time Step (min)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</thead>
<tbody>
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<td>2017.11.28</td>
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<td>63.01</td>
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<td>3</td>
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### Water Added

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<td>0.89</td>
<td>5</td>
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</tbody>
</table>

### Pre-processing

- Sludge Moisture 1: Black Brown Sludge, directly sampled from bucket, removed Tramp
- Sludge Moisture 2: Black Brown Sludge, directly sampled from bucket, removed Tramp
- Sludge Moisture 3: Black Brown Sludge, directly sampled from bucket, removed Tramp

---

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- **Key Result:**
  - Text Input
  - Numeric Input
  - Calculation

### Descriptive Text

**Note:**
Inclue in Avg...
### General Test Information
- **Test No.**
- **S:L Mass Ratio**
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio**
  - **Moisture 1: Wet Sample**
  - **Moisture 1: Dry Sample**
- **Analytical Reports**
  - **Sample Desc.**
  - **Moisture 1**
  - **Sludge Moisture 1**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
  - **Temperature**
  - **Stirrer Speed**
  - **Sample ID**
  - **Moisture 2**
  - **Sludge Moisture 2**
  - **Sample Date**
  - **Moisture 3**
  - **Sludge Moisture 3**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
- **Additional Data**
  - **Time**
  - **Temperature**
  - **Stirrer Speed**

### Data Entry

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### Test Notes
- **Data Entry**
- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**
- **Average**
- **Sl. Dev**

### Calculations

<table>
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### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
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10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Evaluation
- **General Test Information**
- **Initial Test Data**
- **Moisture Data**
  - **Moisture 1**
  - **Sludge Moisture 1**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
  - **Temperature**
  - **Stirrer Speed**
  - **Sample ID**
  - **Moisture 2**
  - **Sludge Moisture 2**
  - **Sample Date**
  - **Moisture 3**
  - **Sludge Moisture 3**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
- **Additional Data**
  - **Time**
  - **Temperature**
  - **Sludge Moisture 1**
  - **Sludge Moisture 2**
  - **Sludge Moisture 3**
  - **Acid Type**
  - **Acid Concentration**
  - **Acid Molar Mass**
- **Data Entry**
  - **Rep 1**
  - **Rep 2**
  - **Rep 3**
  - **Rep 4**
  - **Rep 5**
  - **Average**
  - **Sl. Dev**
- **Calculations**
  - **Increment**
  - **Average**
  - **Cumu. Acid Added**
  - **Acid Added**
  - **Acid Added / Sludge Dry Mass (g / g)**

### Legend
- **Test Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Description Text**

---

**Note:**
- Include in Avg
- Add in Avg
- "x"
**General Test Information**

**Initial Test Data**

- **Test No.**
  - 1:01
- **Sample ID**
  - 316:2:1

**Moisture Data**

- **Test No.**
  - 1:01
- **Moisture 1: Wet Sample**
  - g: 110.17
- **Sample Desc.**
  - Black Brown Sludge

**Analytical Reports**

- **Acid Type**
  - Nitric
- **Acid Concentration**
  - mol/L: 3
- **Acid Molar Mass**
  - g/mol: 63.01

**Date**

- 2017.11.28

**Temperature**

- 22°C

**Sludge Moisture 1**

- **--**
  - 0.97

**Stirrer Speed**

- RPM: 600

**Test Notes**

- **Sample ID**
  - SL-84-2
- **Sample Desc.**
  - Directly sampled from bucket, removed Tramp
- **Pre-processing**
  - Black Brown Sludge

---

**Data Entry**

<table>
<thead>
<tr>
<th>Step 1</th>
<th>Step 2</th>
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<th>St. Dev</th>
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<th>Average</th>
<th>St. Dev</th>
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</table>

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added</th>
<th>Sludge Dry Mass (g/g)</th>
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<tr>
<td></td>
<td></td>
<td>(mol)</td>
<td>(mol)</td>
<td>(g)</td>
<td>(g)</td>
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</table>

---

**Legend**

- **Test Fluid**
- **Moisture Data**
- **Calculations**
- **Key Result**
- **Description Test**

---

**Procedure:**

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio. Record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

---

**SL-84-2**

**Note:**

Include in Avg.
### General Test Information

**Test No.**
2017.11.28

**Sample Desc.**
White Sludge

**Pre-processing**
Directly sampled from bucket, removed Tramp

**White Sludge**
SL-70-1

### Analytical Reports

**Initial Volume**

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### Data Entry

#### Calculations

**Acid / Unit Mass Sludge (g/g)**

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<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
</tr>
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<tbody>
<tr>
<td>Rep 1</td>
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<td>Rep 5</td>
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#### Procedure:

1. **Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.**
2. **Create dilute acid solution at desired acid concentration. Record acid concentration.**
3. **Calibrate pH meter if necessary.**
4. **Perform moisture analysis on at least 3 feed sludge samples.**
5. **Add feed sludge to reaction vessel. Record volume and mass.**
6. **Add distilled water to achieve desired S:L ratio, record volume of water added.**
7. **Set desired temperature and stirrer speed. Thoroughly mix.**
8. **Measure and record initial pH of solution.**
9. **Add desired volume of acid, wait until reaction is complete, measure and record pH.**
10. **Repeat step 9 until endpoint pH is reached (<0.5).**
11. **Dispose of any waste according to CHP.**

### Legend

- **Test:**
- **Replicate:**
- **Calculation:**
- **Key Result:**

### Sludge Acid Titration Test - Experimental Data Entry Sheet

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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<tbody>
<tr>
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### Calculations

**Acid / Unit Mass Sludge (g/g)**

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<th>Acid Added / Sludge Dry Mass (g/g)</th>
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<td>Rep 5</td>
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</table>
**Procedure:**
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

**Legend:**
- Test Failed
- Result Discounted
- Calculation
- Key Result
- Description Test

---

**Data Entry**

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**Calculations**

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**Analytical Reports**

**SS**: Black Sludge
- N/A
- Directly sampled from bucket, removed Tramp

**SL-91-2**

**Moisture Data**

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**Moisture 1: Dry sample**

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**Date**

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**Acid Concentration**

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**Sludge Moisture 1**

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**Sludge Moisture 2**

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**Sludge Moisture 3**

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**Analysis Reports**

**Pre-processing**

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**Calculations**

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**Note:**

Include in Avg "x"
**General Test Information**

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**Analytical Reports**

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**Data Entry**

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**Calculations**

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**Legend**

- **Test 1**: Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
- **Test 2**: Create dilute acid solution at desired acid concentration. Record acid concentration.
- **Test 3**: Calibrate pH meter if necessary.
- **Test 4**: Perform moisture analysis on at least 3 feed sludge samples.
- **Test 5**: Add feed sludge to reaction vessel. Record volume and mass.
- **Test 6**: Add distilled water to achieve desired S:L ratio, record volume of water added.
- **Test 7**: Set desired temperature and stirrer speed. Thoroughly mix.
- **Test 8**: Measure and record initial pH of solution.
- **Test 9**: Add desired volume of acid, wait until reaction is complete, measure and record pH.
- **Test 10**: Repeat step 9 until endpoint pH is reached (<0.5).
- **Test 11**: Dispose of any waste according to CHP.

**Legend**

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- **Test 2**: Create dilute acid solution at desired acid concentration. Record acid concentration.
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**Legend**

- **Test 1**: Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
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- **Test 10**: Repeat step 9 until endpoint pH is reached (<0.5).
- **Test 11**: Dispose of any waste according to CHP.
### General Test Information
- **Test No.**
- **S:L Mass Ratio** --
- **Moisture Data**
  - **Test No.**
  - **S:L Mass Ratio** --
  - **Moisture 1: Wet Sample** g
  - **Test by**
  - **Acid Type**
  - **Moisture 1: Dry sample** g
  - **Date**
  - **Acid Concentration** mol/L
  - **Moisture 2: Wet Sample** g
  - **Sample ID**
  - **Sludge Moisture 1** --
  - **Sample Desc.**
  - **Time**
  - **Acid molar mass** g/mol
  - **Moisture 2: Dry sample** g
  - **Sludge Volume** ml
  - **Sludge Moisture 1** --
  - **Sample ID**
  - **Temperature** c
  - **Sludge Wet Mass** g
  - **Sample Desc.**
  - **Stirrer Speed** RPM
  - **Analytical Reports**
  - **Sludge Moisture 1** --
  - **Note:**
  - **Pre-processing**
  - **Water Added** ml
  - **Test Notes**

### Analytical Reports
- **Rep 1**
- **Increment**
- **Acid**
- **Number**
- **pH**
- **Number**
- **pH**
- **Number**
- **pH**
- **Average**
- **Acid**
- **Acid Added**
- **Acid**
- **Acid Added**
- **Acid**
- **Acid Added**

### Data Entry
- **Step 1**
- **Rep 1**
- **Rep 2**
- **Rep 3**
- **Rep 4**
- **Rep 5**
- **Average**
- **SL Dry Mass** g
- **SL Wet Mass** g
- **SL-75-P**

### Calculations
- **Increment**
- **Average**
- **Cum. Acid**
- **Acid**
- **Cum. Acid**
- **Acid**
- **Cum. Acid**
- **Acid**

### Procedure:
1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
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11. Dispose of any waste according to CHP.

### Legend
- Test Equip
- Sample Prep
- Calculations
- Key Results
- Description Text
**General Test Information**

- **Test No.**
- **Sample ID:**
- **Sample Desc.:**
- **Date:** 11/17/17
- **Time:**
- **Stirrer Speed:**
- **pH:**
- **pH Added:**
- **Initial Volume:**
- **Procedure:**
  1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
  5. Add feed sludge to reaction vessel. Record volume and mass.
  6. Add distilled water to achieve desired S:L ratio, record volume of water added.
  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5).
  11. Dispose of any waste according to CHP.

**Calculations**

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid</th>
<th>Average</th>
<th>Sl. Dry Mass</th>
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**Legend**

- **Test Result:**
- **Calculations:**
- **Test Notes:**

**Data Entry**

- **Calculation**
- **Key Result**
- **Description Text**

**Initial Test Data**

- **S:L Mass Ratio:** 1:01
- **Moisture 1:** Wet Sample
  - **g:** 113.34
  - **pH:**
- **Moisture 2:** Wet Sample
  - **g:** 139.39
  - **pH:**
- **Sludge Moisture 1:**
  - **g:** 100.0
  - **pH:**
- **Sludge Moisture 2:**
  - **g:** 5.6
  - **pH:**
- **Sludge Moisture 3:**
  - **g:** 5.6
  - **pH:**

**Moisture Data**

- **Moisture 1:** Dry Sample
  - **g:** 6.25
  - **pH:**
- **Moisture 2:** Dry Sample
  - **g:** 7.71
  - **pH:**
- **Sludge Moisture 1:**
  - **g:** 0.94
  - **pH:**
- **Sludge Moisture 2:**
  - **g:** 0.94
  - **pH:**
- **Sludge Moisture 3:**
  - **g:** 0.94
  - **pH:**

**Analytical Reports**

- **Nitric Acid**
  - **Acid Type:**
  - **Acid Concentration:** 3 mol/L
  - **Acid molar mass:** 63.01 g/mol
  - **Sludge Moisture 1:**
    - **g:** 100.0
    - **pH:**
  - **Sludge Moisture 2:**
    - **g:** 5.6
    - **pH:**
  - **Sludge Moisture 3:**
    - **g:** 5.6
    - **pH:**

**Analytical Reports**

- **Acid Type:**
  - **Acid Concentration:**
  - **Acid molar mass:**

**Note:**

Include in Avg "x"
## General Test Information

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<thead>
<tr>
<th>Test No.</th>
<th>Date</th>
<th>S:L Mass Ratio</th>
<th>--</th>
<th>Moisture 1: Wet Sample</th>
<th>g</th>
<th>Moisture 1: Dry Sample</th>
<th>g</th>
<th>Temperature °C</th>
<th>--</th>
<th>Sludge Moisture 1</th>
<th>--</th>
<th>Acid Type</th>
<th>--</th>
<th>Acid Concentration</th>
<th>mol/L</th>
<th>Acid Molar Mass g/mol</th>
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## Test Notes

- Nitric Acid
- Moisture 1: Sludge Moisture 1
- Moisture 2: Moisture 2
- Moisture 3: Moisture 3
- Sludge Moisture 1: Sludge Moisture 1
- Sludge Moisture 2: Sludge Moisture 2
- Sludge Moisture 3: Sludge Moisture 3
- Sample ID: SL-98-1
- Sample Desc: Grey Sludge
- Pre-processing: Directly sampled from bucket, removed Tramp

## Analytical Reports

- Wet Mass: 100.0 g
- Dry Mass: 16.6 g
- Volume: 100.0 ml
- Water Added: 100.0 ml

## Data Entry

### Calculations

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<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added</th>
<th>Acid Added</th>
<th>Acid Added / Sludge Dry Mass (g/g)</th>
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<td></td>
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<td>(mol)</td>
<td>(g)</td>
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</tbody>
</table>

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5).
11. Dispose of any waste according to CHP.

### Legend

- Test Result
- Calculated
- Description Test

---

### Graph

![Graph](image-url)
## Sludge Acid Titration Test - Experimental Data Entry Sheet

### General Test Information

| Test No. | Date | Time | Sample ID | Sample Desc. | Stirrer Speed | Sludge Moisture 1 | Sludge Moisture 2 | Sludge Moisture 3 | Temperature | Acid Concentration | Acid Acid Added | pH | pH | pH | pH | pH | pH | pH | pH | pH | pH |
|----------|------|------|-----------|--------------|--------------|------------------|------------------|------------------|--------------|-------------------|----------------|----|----|----|----|----|----|----|----|----|----|----|
|          |      |      |           |              |              |                  |                  |                  |              |                   |               |    |    |    |    |    |    |    |    |    |    |

### Initial Test Data

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### Data Entry

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<th>Acid Added</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Measured pH</th>
<th>Average pH</th>
<th>Sl. Dev</th>
<th>Avg pH</th>
<th>Acid / Unit Mass Sludge (g/g)</th>
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### Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cum. Acid Added (ml)</th>
<th>Acid Added (ml)</th>
<th>Sludge Dry Mass (mL)</th>
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<tbody>
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</table>

### Procedure

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
4. Perform moisture analysis on at least 3 feed sludge samples.
5. Add feed sludge to reaction vessel. Record volume and mass.
6. Add distilled water to achieve desired S:L ratio, record volume of water added.
7. Set desired temperature and stirrer speed. Thoroughly mix.
8. Measure and record initial pH of solution.
9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

### Legend

- **Text Input**
- **Numeric Input**
- **Calculation**
- **Key Result**
- **Descriptive Text**

---

## Sludge Acid Titration Test - Data Sheet

### General Test Information

- **Test No.**
- **Date**
- **Time**
- **Sample ID**
- **Sample Desc.**
- **Stirrer Speed**
- **Sludge Moisture 1**
- **Sludge Moisture 2**
- **Sludge Moisture 3**
- **Temperature**
- **Acid Concentration**

### Initial Test Data

- **Test No.**
- **S:L Mass Ratio**
- **Moisture 1: Wet Sample**
- **Moisture 1: Dry Sample**
- **Moisture 2: Wet Sample**
- **Moisture 2: Dry Sample**
- **Moisture 3: Wet Sample**
- **Moisture 3: Dry Sample**
- **Sludge Dry Mass**
- **Sludge Wet Mass**
- **Sludge Volume**
- **Water Added**

### Data Entry

- **Increment**
- **Acid Added**
- **Measured pH**
- **Average pH**
- **Sl. Dev**
- **Cum. Acid Added (ml)**
- **Acid Added (ml)**
- **Sludge Dry Mass (mL)**

### Calculations

- **Increment**
- **Average**
- **Cum. Acid Added (ml)**
- **Acid Added (ml)**
- **Sludge Dry Mass (mL)**

---

### Graph

- **SL-87-1**
- **pH**
- **Acid / Unit Mass Sludge (g/g)**
- **Other Sludge**
- **Shale**
- **Al 1.47**
- **Ca 0.00**
- **Si 0.00**
- **Fe 0.00**
- **Other 0.00**

---

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## General Test Information

- **Test No.**: SL-81-1
- **Date**: 11/30/2017
- **Sample No.**: 1
- **Sample Desc.**: Red/Brown Aquoues Sludge – Directly sampled from bucket, removed Tramp
- **Pre-processing**: Sludge Wet Mass: 100.0 g
- **Analytical Reports**: Water Added: 100.0 ml

## Initial Test Data

<table>
<thead>
<tr>
<th>Test Notes</th>
<th>S:L Mass Ratio</th>
<th>Moisture 1: Wet Sample</th>
<th>Acidity / Unit Mass Sludge (g/g)</th>
<th>Test by Acid Type</th>
<th>Date</th>
<th>Acid Concentration mol/L</th>
<th>Temperature °F</th>
<th>Sludge Moisture 1</th>
<th>Acid Molar Mass g/mol</th>
<th>Sludge Moisture 2: Wet Sample</th>
<th>Sludge Moisture 2: Dry Sample</th>
<th>Sludge Moisture 3: Wet Sample</th>
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</table>

## Test Notes

1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
2. Create dilute acid solution at desired acid concentration. Record acid concentration.
3. Calibrate pH meter if necessary.
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10. Repeat step 9 until endpoint pH is reached (<0.5)
11. Dispose of any waste according to CHP.

## Calculations

<table>
<thead>
<tr>
<th>Increment</th>
<th>Average</th>
<th>Cumulative Acid Added (ml)</th>
<th>Acid Added / Unit Mass Sludge (g/g)</th>
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</tbody>
</table>

## Data Entry

- **Test Notes**: Include in Avg •x
- **Procedure**: 1. Don adequate PPE and ensure safe laboratory conditions. Consult CHP for questions.
  2. Create dilute acid solution at desired acid concentration. Record acid concentration.
  3. Calibrate pH meter if necessary.
  4. Perform moisture analysis on at least 3 feed sludge samples.
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  7. Set desired temperature and stirrer speed. Thoroughly mix.
  8. Measure and record initial pH of solution.
  9. Add desired volume of acid, wait until reaction is complete, measure and record pH.
  10. Repeat step 9 until endpoint pH is reached (<0.5)
  11. Dispose of any waste according to CHP.

## Legend

- **Test No.**: SL-81-1
- **Key Result**: Description Text
- **Note**: Data Entry